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Determining The Limitations of Class F Fly Ash Compared To Class G Cement In Hydrocarbon Wells Application

A THESIS SUBMITTED BY

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TO THE

Petroleum and Energy Engineering Department

SUPERVISED BY

Dr. Sherif Fakher

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in partial fulfilment of the requirements for the Degree of Master of Science in Petroleum and Energy Engineering

Declaration of Authorship

I, Youssef Ayman Sayed Helmy, declare that this thesis titled, "Determining The Limitations of Class F Fly Ash Compared To Class G Cement In Hydrocarbon Wells Application" and the work presented in it are my own. I confirm that:

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- I have acknowledged all main sources of help.
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Signed:

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A Thesis Submitted by

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To the

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Abstract

Cement is a material commonly used in multiple applications in different industries. Among the industries that uses cement is the oil and gas industry. API (American Petroleum Institute) Class G cement that is the most incorporated form of cement used in the cementation of oil and gas wells to provide several forms of protection and stabilization for the casings. However, Class G cement is contested in terms of efficiency, costs, and environmental awareness against Class F fly ash geopolymers by researchers. This research aims to explore the potential limitations that fly ash geopolymer might have for its use in the oil and gas industry for cementing wells. A background study was conducted on both materials with functional batches formulated from workable ratios and concentrations, materials, and procedures according to the findings and methodologies of other researchers. The working sample batches were then experimented on to test their performances in water retention, durability, structural integrity, thickening time, pumpability, compressive strength, and reactions at different temperatures, pressures, and chemical conditions. The results showed that the geopolymer generally retained more water, was more durable and showed increased strength with temperature, water, and curing time than Class G cement. However, Class F fly ash limits geopolymers from industrial usage due to its inconsistent aluminosilicate compositions, its thickening time, and lack of information on its reactions with various drilling fluids. Class G retains higher compressive strength and requires less complexity in adjusting it with additives. The geopolymer may be limited for use to surface and intermediate casings and even used in two stage cementing using a diverter, but only at depths away from the production zone and substances such as HCl.

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Chapter 1: Introduction

Among the fields that require the use of cement is the petroleum industry. Cement is crucial in supporting the well and maintaining its integrity against many hazardous materials and occurrences in the formation when conducting post-drilling operations [1]. It is also crucial in zonal isolation, protecting and centralizing the casing, and preventing potential uncontrolled flows of hydrocarbons to the surface that could result in a blowout [2].

In 2010, an ultra-deepwater offshore drilling rig, Deepwater Horizon, stationed in the Macondo Prospect of the Gulf of Mexico suffered a massive loss of control of hydrocarbon flow which resulted in an explosive blowout [2, 3]. The damages of the disaster included 11 deaths and 17 injuries from a total of 126 crew members on the rig, as well as the largest marine oil spill in history. This resulted in the devastation of the surrounding marine life and environment in the Gulf of Mexico [4]. Upon the completion of investigations, the US Department of Justice ruled that among the parties responsible for the blowout was the cementing contractor for the rig, Halliburton, for gross negligence [5]. The National Aeronautics and Space Administration (NASA) published a case study report on the Macondo blowout, in which its Academy of Program/Project and Engineering Leadership (APPEL) noted that Halliburton's cement, meant for the well, did not pass its safety and stability tests in their own laboratories on multiple trials, but was approved for the cement job. The cement failure resulted in a process called "channelling" in which the hydrocarbons flowed through the pores of the cement and permeated to the surface. What followed was a "kick" in which hydrocarbons flowed in an uncontrolled manner into the wellbore due to a loss in pressure. After some time of the pressure continuing to drop, and the continuous flow of hydrocarbons to the surface through the cement's pores, the blowout preventor (which had also been used with negligence) could not stop the explosion from occurring.

It is worth noting that conventional cement exhibits many challenges at high-temperature and high-pressure (HTHP) environments [6, 7]. Aside from that, the cement industry's expenditures continue to grow, and this warrants a high demand in raw materials, consumables, resources, and energy to power the machines and factories for manufacturing. This leads to a major contribution in producing solid wastes into the environment and the emission of greenhouse gasses into the earth's atmosphere [8].

With the great and growing environmental and financial concerns that Class G cement poses, it is paramount to investigate a cement alternative that could become less impacting on the environment, less demanding of consumables and is highly efficient at much lower costs. One of the new alternative materials proposed is geopolymer cement [2].

This research focuses on determining the mechanical and operational limitations of geopolymer cement compared to Class G cement that have prevented geopolymer from being used in oil and gas wells. The process carried out in the research involves gathering and studying past findings on the theoretical concept behind Class F fly ash-based geopolymers and its uses. The background study was then followed by formulating geopolymer slurry designs of different concentrations of fly ash, water, and a single alkaline activator. Lastly, the formulations were prepared for mixing using laboratory utilities to mix the ingredient materials at the formulations' concentrations with specific mixing procedures. The resulting slurries were poured into a tray to set and harden in batches to later examine. The most efficient and most well-set formulation samples were used as references for later testing to determine their performances chemically, mechanically, and thermally compared to Class G cement samples.

Chapter 2: Literature Review

2.1. What Is Cement?

Figure 1 – Cement [9]

Cement is a pozzolanic material with binding properties used to bind material together. In the oil and gas industry, the dry cement, as shown above in Figure 1, is mixed into a slurry with water and other additives (such as a thickener, accelerator, retarder, caustic soda…etc) and is given time to set downhole around the wellbore, forming a cement barrier. This barrier protects the casing, maintains wellbore integrity, and protects the well overall from invasive fluids and hydrocarbons that could potentially permeate and channel up the well, causing an uncontrolled flow, or worse, a blowout [10].

With the world continuously developing every day, the demand for cement continues to grow. Market Data Centre published a research paper on ready-mix cement that found it to have a market value of $788.2 billion in 2021, and projects that the market value will reach a potential $1.4 trillion by 2030 [11].

Cement production is often viewed by economists to be a pillar of a country's economic strength and development. This is due to the great amount of purchasing power required, as well as a great number of consumables such as raw material, resources, energy to power machines and factories used for manufacturing [12]. While this view is exhibited by many countries around the world who maintain a strong economy, this also poses a great challenge from an environmental perspective. The massive consumption of energy and material has results in the cement industry being among the greatest emitters of greenhouse gasses and solid waste around the world [13].

2.2. Cement In The Petroleum Industry

Cement is a vital material needed in the oil and gas industry. Among the main functions of cement in the well, it is meant to: [14]

- Protect the well integrity throughout its lifetime.
- Protect the well casings from surrounding pressures.
- Isolate the well from unwanted invasive and corrosive substances.
- Prevent the channeling of formation fluids to the surface.
- Centralize the casing in the borehole.

As demonstrated in Figure 2, the well is drilled, then the casing is placed in the resulting borehole. The cement slurry is then pumped downhole and circulated back up to the top of the casing, filling the borehole space between the formation and the casing, and set in place as a barrier. This process continues with every required casing in the well until it is complete and reaches the target zone. The dried cement barrier then assumes the previously mentioned functions, even after perforation is executed in the target zone [15].

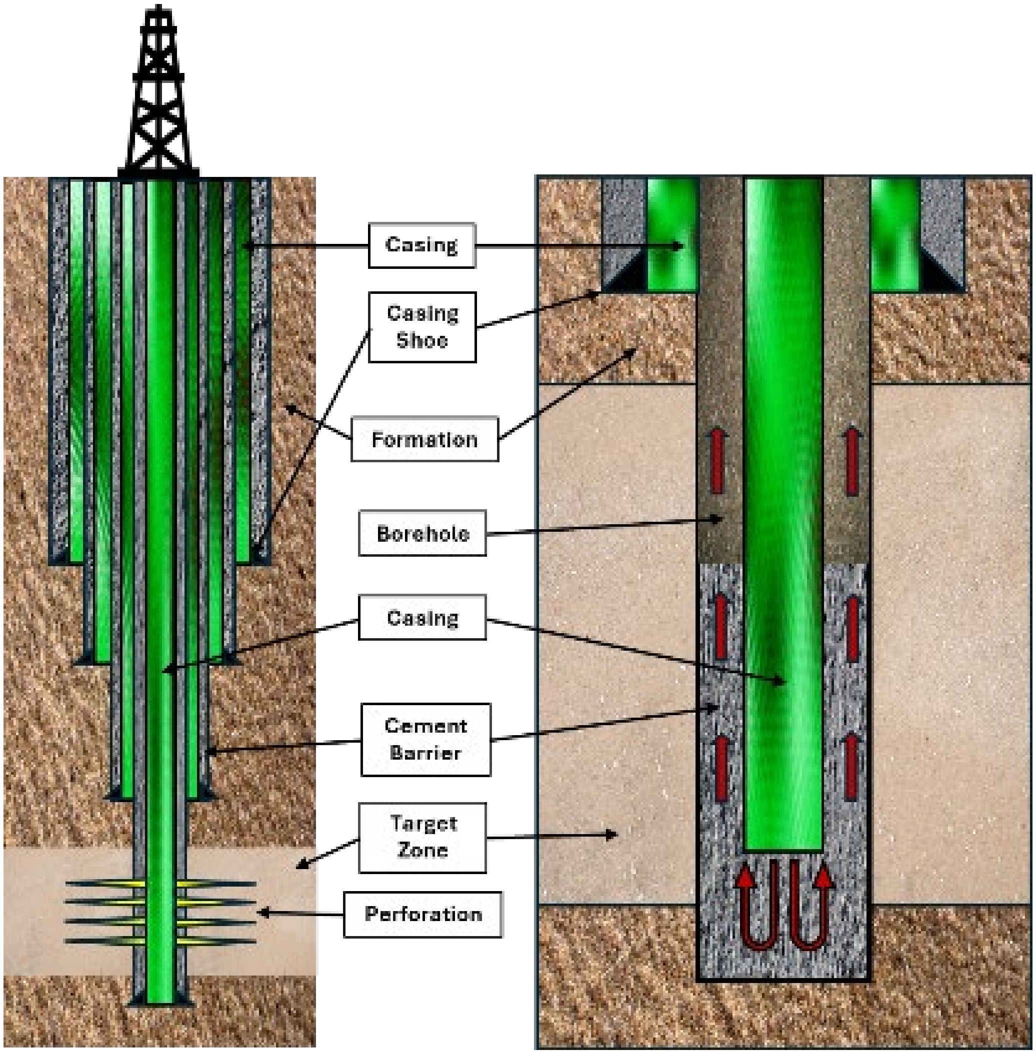


Figure 2 – Well Cementing

2.3. Cement Types and Properties

Table 1 shows that cement has different types and forms according to standards and designs established by the American Petroleum Institute (API). Each cement class has different properties and uses depending on downhole environments [14]. In Table 1, a list of cement types can be found along with their respective properties and designated depths for which they are designed to be used in.

Table 1 – API Classes of Cement

API Class	Depth (ft)	Special Properties	Other
A	Surface – 6,000	• None	Similar to ASTM C 150, Type I
B	Surface – 6,000	• Moderate to high sulphate resistance	Similar to ASTM C 150, Types II
C	Surface – 6,000	• High early strength • Moderate to high sulphate resistance	Similar to ASTM C 150, Types III
D	6,000 – 10,000	• Moderately high pressure and temperature • Moderate to high sulphate resistance	-
E	10,000 – 14,000	• High pressure and temperature • Moderate to high sulphate resistance	-
F	10,000 – 16,000	• Extremely high pressure and temperature • Moderate to high sulphate resistance	-
G	Surface – 8,000	• Can be used with accelerators and retarders for other specs • Moderate to high sulphate resistance • No addition other than calcium sulphate or water	
H	Surface – 8,000	• Can be used with accelerators and retarders for other specifications. • Moderate to high sulphate resistance • No addition other than calcium sulphate or water	
J	12,000 – 16,000	• Extremely high pressure and temperature • Can be used with accelerators and retarders for other specifications. • Moderate to high sulphate resistance • No addition other than calcium sulphate or water	

When it comes to the selection of the most suitable cement type for the cementation of a well, certain properties in the cement may be important, depending on the conditions downhole [14]. These properties include:

- **Slurry Density** – Should be equivalent to mud density to minimize kicks, blowouts or lost circulations.

- **Thickening Time** – Determines the length of time the slurry can be pumped. The time necessary for a slurry consistency to reach 100 Bearden units of consistency (Bcs) within a simulated downhole pressure and temperature condition.
- **Cement Strength** – Strength against static stress due to the weight exerted by the pipe and surrounding environment, and dynamic stress due to drilling.
- **Filtration** – Water loss from the cement is proportionate with slurry density.
- **Permeability** – Should be as low as possible.
    - Bentonite cement has higher permeability.
    - Latex cement has lower permeability.
- **Perforating Qualities** – Cement generally fractures easily upon perforation.
    - Additives such as bentonite, pozzolan and latex increase the ductility and integrity of the cement.
- **Corrosion Resistance** – Cement should be able to resist deterioration against acidic and corrosive fluids.
- **Bond Requirements** – The bond between the cement with rock and metal increases with temperature and time as the cement sets [14].

2.4. Cement Failures

Although cement has been used extensively for years, it is still prone to failure. Class G cement has been found to face challenges in completely stopping contaminants from flowing within its pores. Cement failure modes downhole can be any one or more of the following [16]:

- **Poor Cement Quality:** If the quality and design of the cement is insufficient to the required depth and environment, the cement sheath's integrity may be impacted in one or more ways, as shown in Figure 3:
    a) *Radial Fractures*: Cracks resulting from surrounding formation stresses.
    b) *Plastic Deformation*: Deformation due to surrounding formation stresses.

c) *Casing-Cement Debonding*: Spaces between the casing and the dry cement, creating an inner micro-annulus.

d) *Cement-formation Debonding*: Spaces between the formation and the dry cement, creating an outer micro-annulus.

e) *Incomplete Cementing*: An isolated space within the cement sheath

f) *Channeling*: Formation fluid flows to the surface by way of inner cracks.

- **Temperature and Pressure Change:** Certain enhanced oil recovery and stimulation operations can impact the temperature and pressure conditions within the well, and thus impact the cement barrier.
- **Cement Shrinkage:** Shrinking in volume of cement while hardening due to the surrounding radial stresses, which can lead to cracks.
- **Chemical Degradation:** A reaction resulting in the corrosion of the cement's matrix due to its interaction with formation acidic substances, which can create vugs or cavities in the cement sheath.
- **Perforation:** The explosives in the perforation gun form cracks in the cement [16].

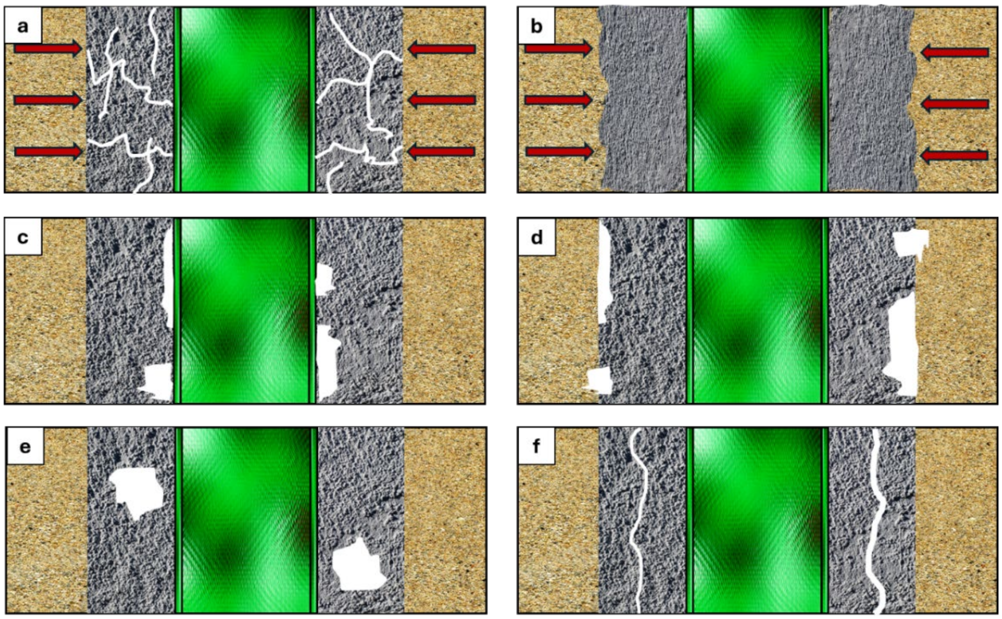


Figure 3 – Cement Failure Modes: a) Radial Fracture, b) Plastic Deformation, c) Cement-Casing Debonding, d) Cement-Formation Debonding, e) Incomplete Cementing, f) Channeling

2.5. Geopolymer Cement

Geopolymers are alternative inorganic binders made by activating material comprising of aluminosilicates, like fly ash from coal-fired power plants, shown in Figure 4, with low-cost alkali activators. The concept of geopolymers has become popular in the cement industry over time since this material recycles waste and reincorporates it in the same function as cement [12].

Figure 4 – Fly Ash Geopolymer Cubes [18]

Geopolymers can comprise of different materials based on their environmental requirement and the need that they will serve. According to Zain, et al. [19], these materials are used to make different sorts of geopolymers:

- Fly Ash
- Palm Oil Fuel Ash (POFA)
- Kaolin
- Metakaolin
- Dolomite

Each of these different materials consists of a chemical composition with components that are vital to the geopolymerization process of the slurry and the formation of an alternative solid compared to conventional cement. Table 2 shows the chemical composition of different geopolymers made from the above-mentioned 5 materials using x-ray fluorescence (XRF) [19].

As shown, the most prevalent compounds of each of these geopolymers are silicates (SiO_2) and alumina (Al_2O_3). Natural minerals such as kaolinites and clays tend to have higher concentrations of these compounds, whereas fly ash, silica fume, slag, metakaolin and other material could be used as supporting materials to produce a geopolymer.

Table 2 – Chemical Composition of Geopolymer Materials Using XRF

Chemical Composition	Fly Ash	POFA	Kaolin	Metakaolin	Dolomite
SiO_2	52.11	51.18	52.00	55.90	15.37
Al_2O_3	23.59	4.61	35.00	37.20	1.69
Fe_2O_3	7.39	3.42	1.00	1.70	0.51
TiO_2	0.88		0.90	2.40	0.015
CaO	2.61	6.93	<0.05	0.11	23.00
MgO	0.78	4.02	0.70	0.24	17.20
K_2O	0.80	5.52	2.00	0.18	0.195
Na_2O	0.42	0.06	0.05	0.27	0.013
SO_3	0.49			0.02	
P_2O_5	1.31			0.17	0.019
Loss In Ignition		21.6		0.80	

Table 3 shows that fly ash possesses a high composition of alumina and silica, exhibits no impacting loss in ignition, and does not require natural resources that provide essential need to human life to use a waste material productively [19].

Table 3 – Geopolymer Types and Their Sources [19]

Geopolymer Types	Highest Chemical Composition	Sources of Raw Materials
Fly Ash	SiO_2 & Al_2O_3	Waste product from coal mining
Metakaolin	SiO_2 & Al_2O_3	Natural resources
Kaolin	SiO_2 & Al_2O_3	Natural resources
POFA	SiO_2	Waste product from palm oil industry
Dolomite	SiO_2, CaO & SiO_3	Natural resources

Fly ash, shown in Figure 5, is essentially the residue of coal combustion from facilities in which coal is used for energy, such as coal-fired power plants. This residue pollutes the environment and is in great amounts. This material is referred to as an aluminosilicate, meaning that its composition is abundant in alumina and silica [19]. It is also very cheap to purchase, generally being 18% cheaper than Class G cement [20].

Figure 5 – Class F Fly Ash [21]

When fly ash is mixed with an alkaline solution, the reaction forms an aluminosilicate gel within the slurry that binds and dries the ash and fluids together, respectively, into a solid geopolymer. These gels make the geopolymer highly dense and resistant to several corrosive substances such as sulphates, carbon dioxide, and chloride. The alkaline solution is conventionally a fluid comprised of alkaline activators mixed with water that trigger the binding in the aluminosilicate; these activators are usually sodium silicate (Na_2SiO_3, Figure 6(a)) and sodium hydroxide (NaOH, Figure 6(b)).

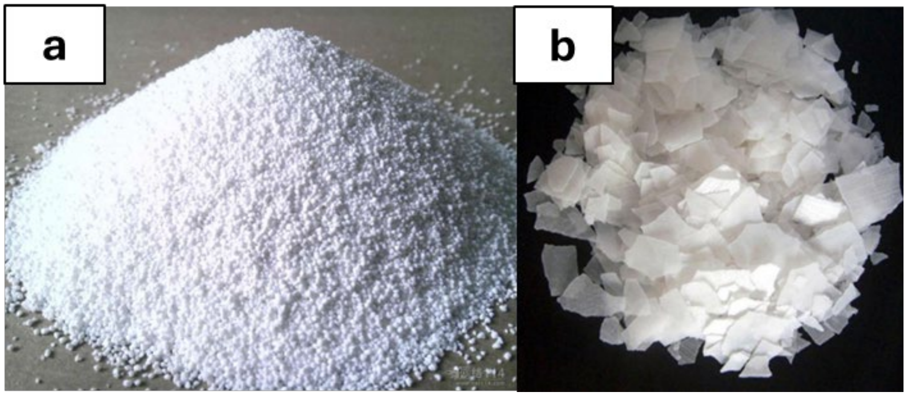


Figure 6 – Alkaline Activators: (a) Sodium Silicate [22], (b) Sodium Hydroxide [23]

Class F fly ash is a pozzolanic material used for mixing slurries with light weight. This material has a specific gravity of 2.32, whereas Class G cement is 3.15. As the curing time for the geopolymer passes, its compressive strength increases. The material's permeability could decrease depending on how the geopolymer slurry's design is formulated, and depending on the type of binder it is exposed to. This decrease may aid in reducing the risk of corrosion from hazardous substances downhole like sulphate. Fly ash's inconsistency in its alumina-silica ratio could result in different degrees of reactions and inconsistencies when exposed to certain additives and heat. It can even react differently depending on its Calcium Oxide (CaO) composition, which can range anywhere between 2% and 30% of the weight of the total fly ash used in that geopolymer. Additionally, while Class F is its own form of fly ash (Aside from Class C), there also exists a "True" Class F, with 10% or lower CaO. This means that Class F fly ash reactions can vary with different batches [24].

Table 4 exhibits the findings by authors who tested approaches to designing a functioning geopolymer. The table includes the testing methods each author used, the alkaline activating materials they incorporated, and the conclusive remarks and recommendations made based on the observations noted and the results determined from the tests.

Table 4 – Test Methods Used By Authors

Author	Methods	Activator(s)	Conclusion
Ahdaya, et al. [6]	• Rheology tests • Density tests • Compressive strength test • Fluid loss test	• Sodium silicate • Sodium hydroxide	• Workable activator ratios make geopolymer a suitable alternative to cement.
Yu et. al. [25]	• Unconfined compressive strength test	• ASM	• ASM can be used without NaOH. • Geopolymer can be used for soil stabilization.
Haruna et al. [26]	• Hardened density test • Compressive strength test • Flexural strength test • Water absorption test • Efflorescence test • Microstructure test	• Sodium silicate	• ASM increases compressive strength. • Geopolymer is a suitable alternative to cement.
Rasuli et al. [27]	• Compressive strength test • Flexural strength test • Drying shrinkage test • Setting time	• Sodium metasilicate • Slag-based alkali activated material (AAM) • NaOH	• Sodium metasilicate is a superior activator. • AAM can increase compressive strength.
Dong, et al. [28]	• 18 one-part geopolymers • Activator comparison tests	• Sodium metasilicate pentahydrates • Liquid activators	• Sodium metasilicate gives a high compressive strength.

Given these answers, the geopolymer samples with different concentrations still must be tested under specific pressures, temperatures, environments, strength tests, resistance to deformation and absorbing water, and reactions to certain substances. This is to determine its abilities to be used in field operations and industrial applications.

Chapter 3: Research Question, Hypothesis and Contribution

3.1. Research Question

What factors limit using Class F geopolymer cement as an alternative to using Class G cement in the cementation process of drilling operations of oil and gas wells?

3.2. Hypothesis

The formulation of a functioning geopolymer using a single alkaline activator and Class F fly ash at high pressure and high temperature conditions faces limitations compared to API Class G cement that prevent it from being utilized as its alternative in the cementing of oil and gas wells in the petroleum industry.

3.3. Planned Contribution

3.3.1. Intellectual Rationale

This geopolymer developed in this research uses Class F fly ash and one low-cost alkali activator (sodium metasilicate, Na_2SiO_3). By relying on a single alkaline activator, this will eliminate the dangerous handling of further chemicals, as well as test the capabilities of the concentrated liquid form of sodium metasilicate when reacting to an aluminosilicate.

3.3.2. Relevance Of This Research

The results and findings of this research could be used to help further future research about safer, more durable, cheaper, less resourceful, environmentally friendly, and waste recyclable alternatives for cement. Though it has shown success at a laboratory level, it still requires further improvement to sustain usage at an industrial level and to prevent hazards caused by high pressure and high temperature well environments including corrosive fluids. This research is for the purpose of testing potential material that could improve cementation and oil and gas operations while protecting the well, the environment and human life near powerplants and/or on oil and gas rigs.

3.3.3. Theoretical and Practical Impact

On a theoretical level, the geopolymer's capabilities and properties provide a cost-efficient sustainable alternative for a vital artificial material for the petroleum industry and the world. The geopolymer could serve in providing solutions for preserving human health, overall operational safety, and the protection of the environment. In practice, the findings of this research will highlight the strengths and weaknesses of Class F fly ash according to the tests conducted, and how that may impact its usage in oil and gas well cementing operations.

3.3.4. Contribution Of This Research

This research can provide insight into the potential drawbacks and limits of geopolymers. This is done by examining the behaviors of Class F fly ash, sodium metasilicate and water in reaction to one another under different concentrations, as well as the physical, chemical, thermal, and rheological capabilities of geopolymer.

3.4. Specific Research Aims

To properly give context and provide a more informative outlook on this research, there are four specific research aims that are needed to be investigated:

1. To determine the required ratios of the weights of the fly ash and alkaline needed to mix the geopolymer slurry.
2. To produce different formulations of geopolymer batches using the ratios found and narrow down the most optimal formulations for testing.
3. To determine the optimal speed and mixing time needed for the components to be mixed into a cement slurry to avoid the effect of thickening while mixing or high temperature from the blender's blade.
4. To test the narrowed down batches for property performance under specific pressure, thermal, chemical, and mechanical conditions.
5. To compare the geopolymer's results with that of Class G cement and determine the potential limits of geopolymer at an industrial level.

Chapter 4: Experimentation

4.1. Scope of Experimentation

The process of the experimentation phase of the research is initiated by preparing a geopolymer sample and a cement sample, then proceeding with the finalized samples toward experimentation. The scope of work and process for preparing the geopolymer sample covers three tasks, as shown and explained in Figure 7:

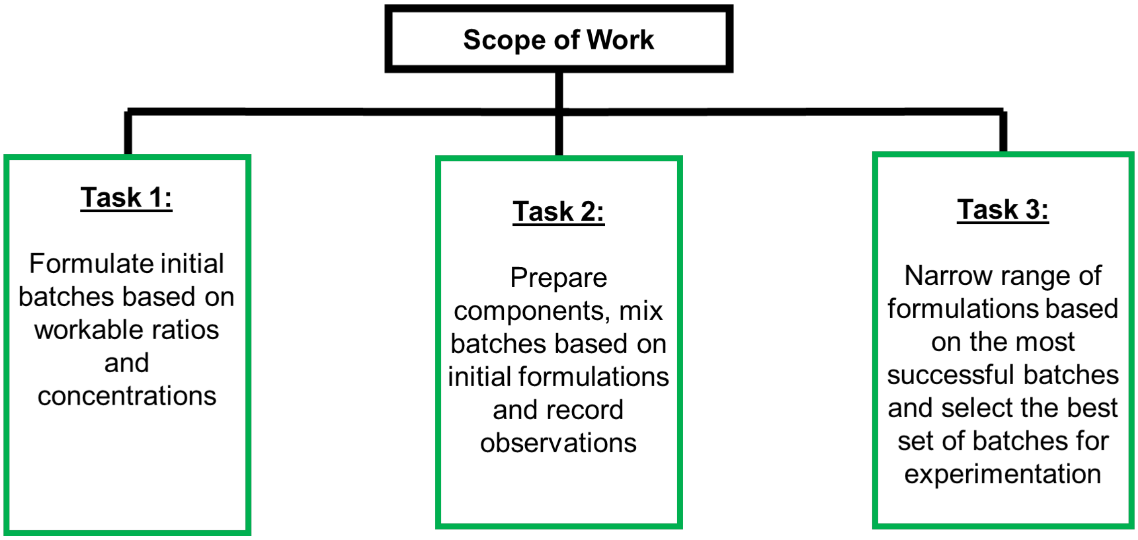


Figure 7 – Scope of Work

Task 1 commences with initial formulations for mixing the geopolymer samples. These formulations are varying batch recipes for the geopolymer determined using the workable weight ratios of the fly ash and Na_2SiO_3 and the water concentrations used by the previously cited authors in the literature review. Task 2 proceeds with preparing the components needed to mix the geopolymer, setting the parameters and time of mixing the slurry, pouring the slurry and allowing it to set, followed by recording initial observations. Finally, Task 3 entails selecting the most successful batch recipe of the geopolymer to proceed to experimentation and comparison with the Class G cement.

4.2. Procedure

4.2.1. Geopolymer Formulation Calculations

For the purposes of this approach to the research, the following material ingredients were used to mix the geopolymer slurry, based on the research conducted by authors referenced in the literature review: [24]

4.2.1.1. Geopolymer Ingredients:

- ***Class F Fly Ash (SiO_2 + Al_2O_3 + Fe_2O_3 + CaO)***: The fly ash material incorporated in this geopolymer is comprised of silica, alumina, hematite and a low content of lime.
- ***Sodium Metasilicate (Na_2SiO_3)***: The alkaline activator of this geopolymer is the solid and concentrated variant of sodium silicate. The use of this variant is to activate the silicates of the fly ash and increase the bonding of the aluminosilicate gels.
- ***Room Temperature Tap Water (H_2O)***: The water is used as part of the alkaline solution that will be applied to the fly ash to form a slurry and form aluminosilicate gels.

In formulating the geopolymer slurries to be narrowed down, and to investigate the impact of each ingredient on the geopolymer's performance, the following data in Table 5 was listed based on the workable ratios and concentrations established by the researchers in the literature review: [1, 6, 7, 8, 12, 13, 19, 25, 26, 27, 28]

4.2.1.2. *Known Workable Ratios and Concentrations:*

Table 5 - Initial Geopolymer Formulations

Fly Ash Weight (g)	*100*						
Fly Ash-Binder Ratio	15	20	25	30	35		
Water Concentrations (%)	10	15	20	25	30	35	40

The water concentrations listed are used to determine the weight of the water required for the alkaline solution, with which it will be mixed with the binder, sodium metasilicate. The fly-ash binder ratios listed are used to determine the weight of the sodium metasilicate required based on the weight of the fly ash. These ranges will result in 35 formulations.

4.2.1.3. *Solving For Remaining Weights And Concentrations:*

To provide further view of potentially affecting factors in the reaction of the geopolymer while setting, a series of equations were formulated to determine any missing values:

$$\boldsymbol{Binder\ (g) = \frac{Fly\ Ash\ (g)}{Fly\ Ash{:}\ Binder\ Ratio}}$$

Eq. (1)

Eq. (1) is used to determine the weight of the binder of the sample by dividing the single weight of fly ash used for all batches in the experimental phase by the chosen fly ash-binder ratio, as expressed by Table 6.

Table 6 - Sodium Metasilicate Weight For Each Fly Ash-Binder Ratio

Fly Ash Weight (g)	*100*				
Fly Ash-Binder Ratio	15	20	25	30	35
Binder Weight (g)	6.67	5	4	3.33	2.86

The five weights of the sodium metasilicate determined in Table 6 are each mixed with the seven water concentrations mentioned in Table 5. The concentrations are based on the weight of the total weight of the slurry.

$$\boldsymbol{Total\ (g)} = \frac{\boldsymbol{Fly\ Ash\ (g) + Binder\ (g)}}{\boldsymbol{1 - Water}\ (\%)}$$

Eq. (2)

Eq. (2) determines the total weight of the geopolymer cube sample by first adding the weight of the binder, determined in Eq. (1), with the weight of the fly ash, then dividing it by the concentration of solids in the sample. The concentration of solids is determined by subtracting the concentration of water chosen for that sample.

$$\boldsymbol{Water\ (g) = Total\ (g) - Binder\ (g) - Fly\ Ash\ (g)}$$

Eq. (3)

Having determined the total weight of the sample in Eq. (2), the weight of the water in the sample is determined in Eq. (3) by subtracting the determined sodium metasilicate weight and the established weight of the fly ash.

$$\boldsymbol{Binder}\ (\%) = \frac{\boldsymbol{Binder\ (g)}}{\boldsymbol{Total\ (g)}}$$

Eq. (4)

$$\boldsymbol{Fly\ Ash}\ (\%) = \frac{\boldsymbol{Fly\ Ash}\ (\boldsymbol{g})}{\boldsymbol{Total}\ (\boldsymbol{g})}$$

Eq. (5)

Lastly, the concentrations of the sodium metasilicate and the fly ash in each sample are determined using Eq. (4) and Eq. (5), respectively. To determine the sodium metasilicate concentration, simply divide the determined binder weight by the determined total weight of its sample. As for the fly ash, the same concept applies by dividing the established fly ash weight by the total weight of the sample.

4.2.1.4. Formulation Batches:

Based on the previously mentioned procedure, formulations were calculated and listed in a table of component weights, concentrations and ratios (can be found in Appendix Table A1). Initial observations from mixing the slurries into cubes showed that the samples with the smallest fly ash-binder ratio settled best, so a decrease in the ratio is warranted. The concentrations of water also revealed that the cubes settled best with 20% water without deteriorating. In an effort to reach greater results, a second round of formulations was conducted. A different approach was taken in which the water concentration was fixed at 20%, while different concentrations of sodium metasilicate were tested for each sample.

Table 7 – Final Geopolymer Batch Formulations

TEST (#)	COMPOSITION (%)			WEIGHT (g)				Ratio	
Serial Number	Binder	Water	Fly Ash	Binder	Water	Fly Ash	Total	FA/B	B/W
B10W20	10%	20%	70%	14.29	28.57	100	142.86	7.00	0.50
B12W20	12%	20%	68%	17.65	29.41	100	147.06	5.67	0.60
B15W20	15%	20%	65%	23.08	30.77	100	153.85	4.33	0.75
B17W20	17%	20%	63%	26.98	31.75	100	158.73	3.71	0.85
B20W20	20%	20%	60%	33.33	33.33	100	166.67	3.00	1.00

The concentrations in Table 7 were selected to increase the binder content and decrease the fly ash-binder ratio for optimum settling. The mixing time remained the same for each mix, but the speed remained at 2000 RPM to not allow the heat induced by the blender's blade to affect the settling of the cubes. The samples were given serial names based on the binder concentration tested for each one, followed by the fixed water content of 20% in each sample. Each batch also produced 12 samples to provide more results.

Table 8 – Class G Cement Formulation

TEST (#)	**COMPOSITION (g)**	**WEIGHT (g)**		
Serial Number	**Water**	**API Class G Cement**	**Water**	**Total**
API Class G Cement	44%	100	44	144

The Class G cement samples were mixed in a similar approach by mixing the Class G cement with room temperature tap water in the API standard ratios in Table 8. The objective of this approach was to formulate a baseline reference without any additives to test its basic capabilities when tested against the geopolymers.

4.2.1.5. *Mixing Process:*

1. Gather the fly ash, water, and sodium metasilicate
2. Prepare each component at a weight based on the formulations determined using the workable ratios and concentrations and Eq. (1) to Eq. (5)
3. Pour the water and sodium metasilicate into the mixer (Fann Constant Speed Mixer Model 686CS)

4. Mix water with the sodium metasilicate for 1 minute at 2000 RPM to produce the alkaline activating solution
5. Pour the fly ash into the solution while mixing to produce the geopolymer slurry and let it mix for 5 minutes at 2000 RPM
6. Once the time is up, stop the mixer and pour the slurry into cubes and allow them to set, as shown in Figure 8

Figure 8 – 12 Samples of B15W20 As A Slurry (Left), Then Cured (Right)

4.2.2. Baseline Cement Recipe

To prepare a cement sample for which a comparative analysis would be conducted, samples of a single cement recipe are mixed in accordance with API standards (API 10A:2011) and procedures [29]. The samples consist of 100g of API Class G cement and 44g of room temperature tap water. This would serve to be the lab equivalent recipe of the industry standard amounts for this experiment. The final dried samples can be shown in Figure 9.

Figure 9 – Cement Samples

4.2.3. Experiments

The experiments required for this research, which will be further explained below, can be summarized in brief by the flow chart in Figure 10. There will be a set of experiments conducted on the optimum batches of fly ash geopolymer, in which each test will be conducted with different constraints to determine the limits and capabilities of these samples.

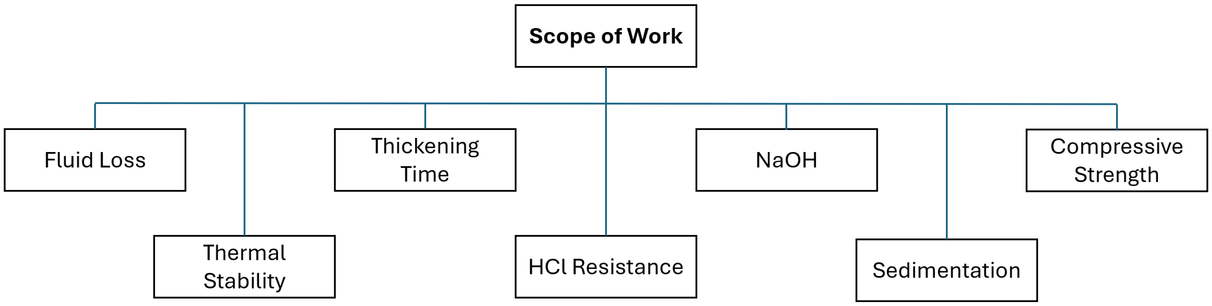


Figure 10 – Scope of Work

Following the completion of the above-mentioned experiments on the geopolymer samples, the same experiments will be conducted on the cement samples, from which a comparative analysis could be produced. All materials and procedures are in accordance with API Specification 10A [29].

4.2.3.1. Experiment Setup and Procedures:

Fluid Loss Test

Description

The Fluid Loss Test is an experiment in which the sample slurry is poured into a vessel and is placed under specific temperature and pressure conditions to determine a cement sample's ability to retain water under said conditions, meant to resemble that of downhole high-temperature and high-pressure conditions. The ability of the cement to retain water under these conditions is measured by draining the sample's potential water volume and measuring that volume. The sample's ability to control the amount of fluid lost reduces the potential for changes in slurry properties and the appearance of cracks, which are among the biggest problems associated with fluid loss in oil and gas wells [1, 6].

Test Setup (As shown in Figure 11)

- Fann HPHT Filter Press Model 175CT
- Graduated Cylinder

Procedure

1. Place filter membrane into the experiment vessel
2. Pour sample batch onto the filter membrane inside the vessel
3. Seal the vessel with six bolts
4. Use the pressure and temperature regulators to set the pressure to 1000 psi and the back pressure to 500 psi, and the temperature of the vessel for each test to 20^{o}C, 60^{o}C and 100^{o}C
5. Slightly open the bottom nozzle of the vessel to allow any water volume within to drop into the graduated cylinder
6. Collect the water volume and analyze the results

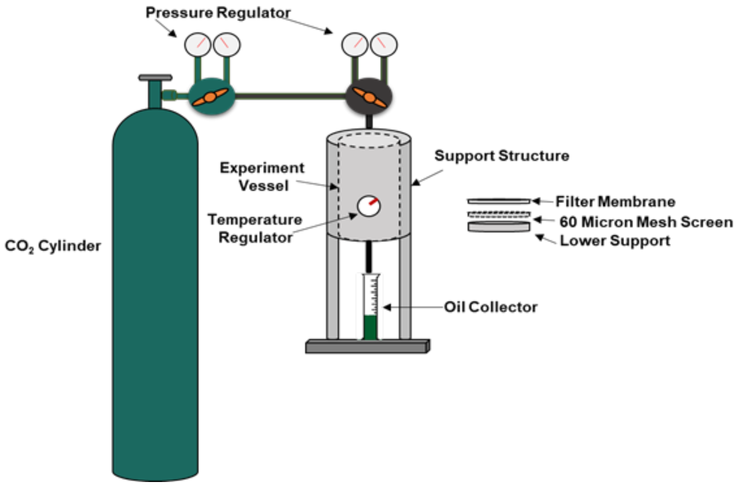


Figure 11 – Fluid Loss Test

Thermal Stability Test

Description

This is an experiment in which the sample slurry is left to set in several sample cubes, and then measured for their dry weight. The cubes are then placed in a water bath with water at a high temperature and are measured at different intervals of time for potential changes in weight. Changes in weight indicate whether the cement samples experience stability or deterioration under high temperature or high pressure (HPHT) conditions.

Test Setup

- Memmert Water Bath WTB
- Tap water

Procedure

1. Pour the sample batches into the tray of cubes and wait for them to set
2. Weigh the hardened cubes
3. Set the water bath temperature to 95^{o}C

4. Place the cubes into the water bath (As shown in Figure 12)
5. Set the bath to the required temperature
6. Weigh the sample cubes each day for possible water retention

Figure 12 – Water Bath

Thickening Time Test

Description

The Thickening Time Test is an experiment in which the cement slurry is poured into a chamber that is placed in a consistometer, and that chamber is then subjected to a given temperature and pressure condition to resemble that of the annulus in an oil or gas well. The test times how long it takes for the given cement slurry to thicken under said conditions, how stable the cement is after the test is completed under those consistent conditions, as well as other rheological properties during that time.

Test Setup

- Fann M290 Consistometer (As shown in Figure 13)

Procedure

1. Pour the sample batch slurry into the experiment vessel and seal it tightly
2. Set the pressure conditions to 1000 psi, and the temperatures to 20°C, 60°C and 100°C for each test
3. Start the test using the internal software on the consistometer's computer
4. Monitor the sample until the consistency reaches 100 Bc, then record the time

Figure 13 – Consistometer

Sedimentation Test

Description

The Sedimentation Test is an experiment in which the cement slurry is poured into a tube to then set in a cylindrical shape, or a “core”. This cement cylinder is then divided into three segments of equal height, or “core plugs”, then placed into a volume of water, and finally measured for the weight and density of each cement plug. The objective of the test is to determine if the core maintains its desired properties throughout its plugs, as an indicator of stability in the cement’s integrity in the well [6].

Test Setup

- Cardboard Paper
- Filament Tape
- Electric Saw

Procedure

1. Pour sample batches into cylindrical tubes and wait to set
2. Cut the tubes into 3 equal cylinder segments (As shown in Figure 13)
3. Measure the weight of each cylinder segment
4. Pour a volume of water into a beaker
5. Place each cylinder segment into the water beaker and measure the displacement volume
6. Determine the density of the cylinder segment for each binder concentration and section

Figure 14 – Cylinder Segments

NaOH and HCl Tests

Description

This test placed cement slurry cubes in different concentrations of acids for intervals of time. The main purpose is to test the samples' reactions when exposed to certain chemicals and compounds. The samples are weighed before being placed, then after to determine weight or shape change. This is to reflect the cement in the annulus when exposed to the same conditions.

Test Setup

- NaOH, Lab Grade
- HCl 28% Solution, Lab Grade
- Plastic Containers
- Weight Scale

Procedure

1. Place the required amount of NaOH with water into the container
2. Weigh the geopolymer cube samples
3. Place the samples in their designated NaOH concentrations (As shown in Figure 14)
4. Take the samples out and weigh them again, monitor any changes
5. Repeat the process with HCl

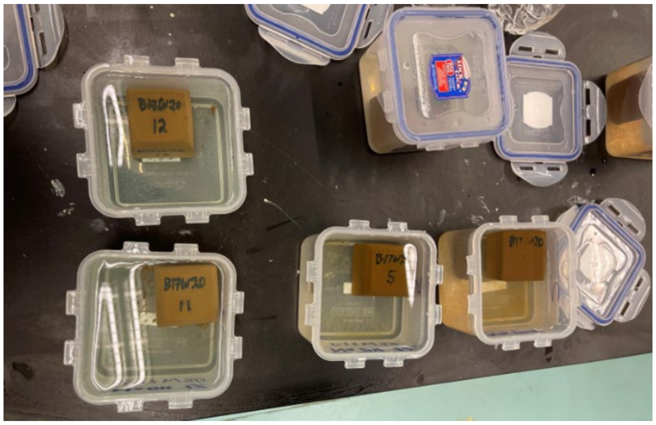


Figure 15 – NaOH and HCl tests

Compressive Strength Test

Description

The Compressive Strength Test applies force in MPa to the sample at a consistent rate until it reaches mechanical failure and fractures. This is meant to reflect the mechanical conditions that the cement will experience [1]. The test uses the machine in Figure 16.

Test Setup

- Electro-Hydraulic Servo Control Compression Testing Machine Press (Cangzhou Lovely Technology Co.)

Procedure

1. Measure the geopolymer cube's surface area and place it under the press
2. Apply load gradually using the computer until specimen failure
3. Divide failure load by cross-sectional area of specimen to get the compressive strength in psi

Figure 16 – Compressive Strength Test [30]

Chapter 5: Results

In Figures 17 and 18, the samples were settled, taken out, and examined. B10W20 did not show enough strength upon initially settling and was thus eliminated from the testing process. Over time, samples B20W10 and later B17W10 began showing cracks and signs of deterioration, which then resulted in their elimination as well, resulting in samples B12W20 and B15W20 being the optimum two to be tested on for their properties and compared to cement.

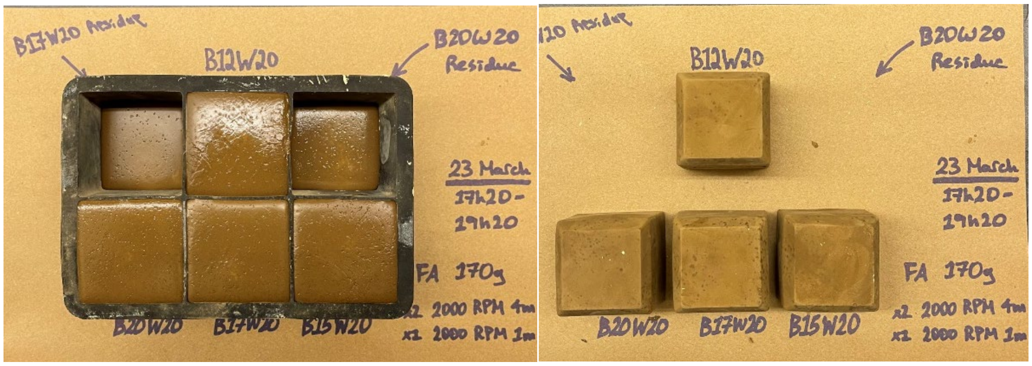


Figure 17 – Narrowed Samples Wet (Left), Dry (Right)

Figure 18 – Deteriorated Samples (B17W20 and B20W20)

In Figure 19, the samples were settled and taken out. The cement samples were found to be hard, well set and did not show any signs of serious deterioration. Upon comparing the two different materials, it was much easier to crush the geopolymer samples by hand than it was to do so with the cement samples. The cement showed little to no signs of deterioration or degradation.

Figure 19 – Cement Samples After Settling

5.1. Fluid Loss Test

The fluid loss test was conducted at 1000 psi with a back pressure of 500 psi at different temperatures of 20, 60 and 100 degrees Celsius for the geopolymer samples and the baseline cement sample. Figures 20 and 21 show how the fluid slurry for each test batch came out of the fluid loss tester after each condition. Figure 22 expresses the fluid lost in milliliters from each sample at each temperature.

At 20 degrees Celsius, the geopolymer samples had a relatively high level of fluid lost, between 28 and 29 mL, and the samples came out of the fluid loss tester in a smooth core plug shape. In comparison, the cement sample had a lower fluid loss of 24 mL, with a relatively smooth core plug; however, it had edges that were chipped off that the top.

At 60 degrees Celsius, the geopolymer samples had less fluid loss than before, ranging between 12 and 15 mL, and the samples were taken out of the tester intact but showing signs of slight decalcification. The cement sample, however, produced 28 mL of fluid lost, with the core plug coming out almost completely crumbled due to the high pressure, high temperature conditions, as shown in Figure 21.

At 100 degrees, B15W20 showed a much less fluid loss of 16.5 mL than B12W20, which showed a fluid loss of 31 mL. Both samples emerged from the tester with even greater signs of deterioration due to the high temperature and high pressure. B17W20 showed the least amount of fluid loss with only 7 mL but was also highly deteriorated. Due to the heavy pressure and temperature, the 100-degree test sample came out destroyed and the fluid within the sample evaporated upon commencing the test.

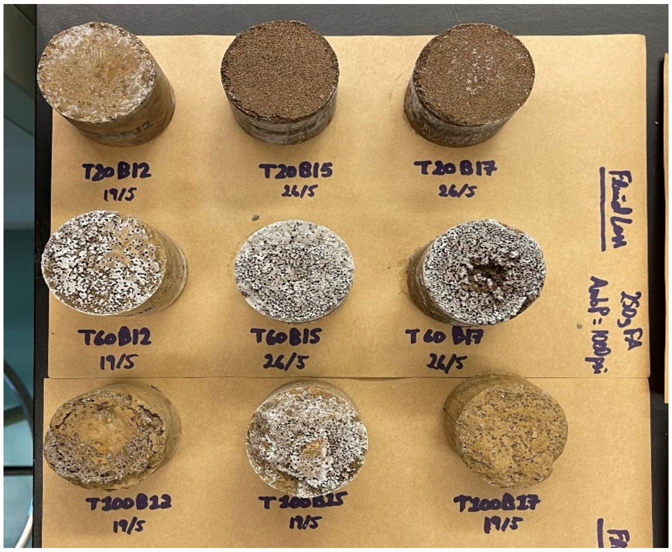
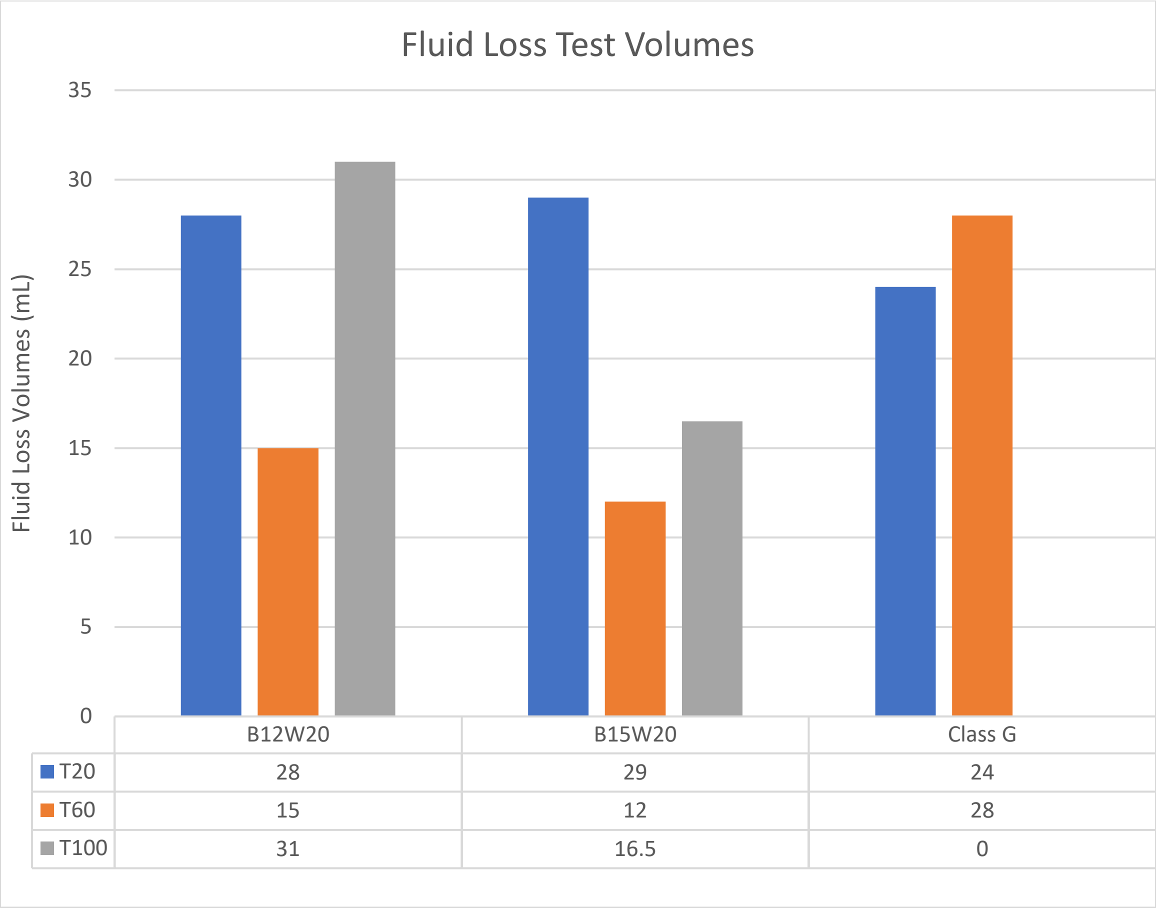


Figure 20 – Geopolymer Fluid Loss Test Residue

Figure 21 – Cement Fluid Loss Test Residue T20 (Left) and T60 (Right)



	B12W20	B15W20	Class G
■ T20	28	29	24
■ T60	15	12	28
■ T100	31	16.5	0

Figure 22 – Fluid Loss Test Volumes

As Suppiah, et al. [1] as well as Ahdaya et al. [6] find in their report, a possible reason for the reduction of volume for fluid loss in geopolymers more than cement, as shown in Figure 22, could be due to the reaction of silicates with aluminum and water to form aluminosilicate gels. Furthermore, this indicates These gels optimize the geopolymer's retention ability and thus do not require the incorporation of fluid loss additives as Class G cement requires.

This experiment showed that Lake, et. al. [24] were correct in the Petroleum Engineering Handbook in indicating that different batches of Class F fly ash were inconsistent in their reactions with different fluids and temperatures. Upon applying the fly ash from two different bags of the same order from the same supplier, the fluid loss resulting from the fly ash became inconsistent with the increase of temperature, as shown by the 100-degree test.

5.2. Thermal Stability

The thermal stability test was conducted by gathering samples of the Class F fly ash geopolymer (B12W20 and B15W20) as well as the Class G cement. For each test, the samples are placed in a hot water bath at 95^{o}C for the geopolymers, and 20^{o}C, 40^{o}C, and 60^{o}C for the cement. They are weighed after intervals of time to determine any water absorption or weight gain compared to their dry weight. The intervals were 1 day, 4 days, and 1 week since their placement in the water bath. All samples' shapes remained intact and did not face deterioration; however, the weight results showed different patterns.

Based on the results exhibited in Figure 23, it is shown that the geopolymer samples generally gained weight in the hot water over the course of the week. The B12W20 samples showed a 10% to 13% growth in weight (11% on average), whereas the B15W20 samples with the higher sodium metasilicate composition showed a growth of 2% to 8%. As shown in Figure 24, the trend shows that by the end of the curing period, with the increased sodium metasilicate of 15%, the geopolymer sample absorbs less water.

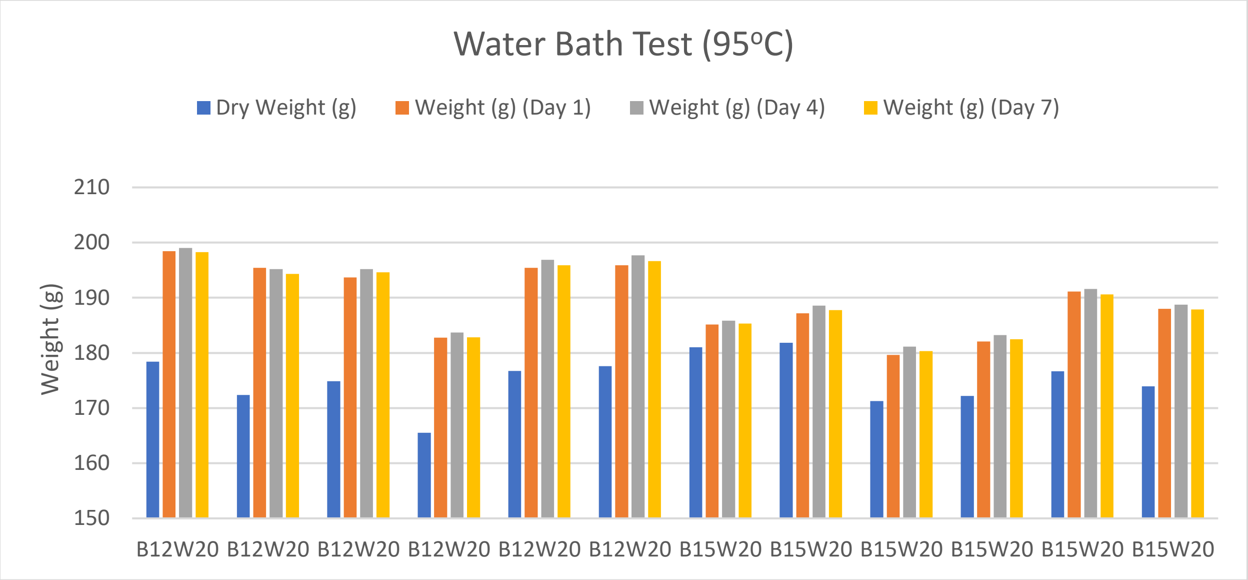


Figure 23 – Thermal Stability Weights (Geopolymers)

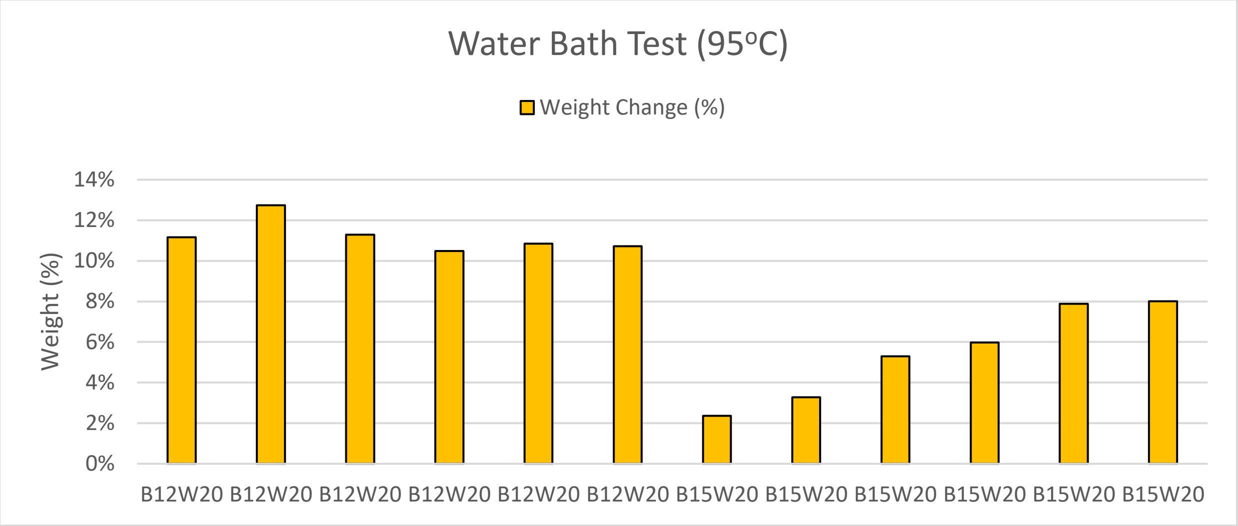


Figure 24 – Thermal Stability Weight Change (Geopolymers)

In Figure 25 and Figure 26, it is apparent that the cement samples gained weight by 3.5% to 3.6% under ambient temperatures (20◦C). Upon increasing the temperature of the water to 40◦C, as shown in Figure 27 and Figure 28, the weight change was also gained by 4.3% to 4.7%. Finally, upon testing at 60◦C, as shown in Figure 29 and Figure 30, the weight gain only increased by 2%. The samples emerged from the water bath without cracks or deterioration and showed that with temperature, they can prevent unwanted fluids from permeating and channeling through the cement without additives being involved.

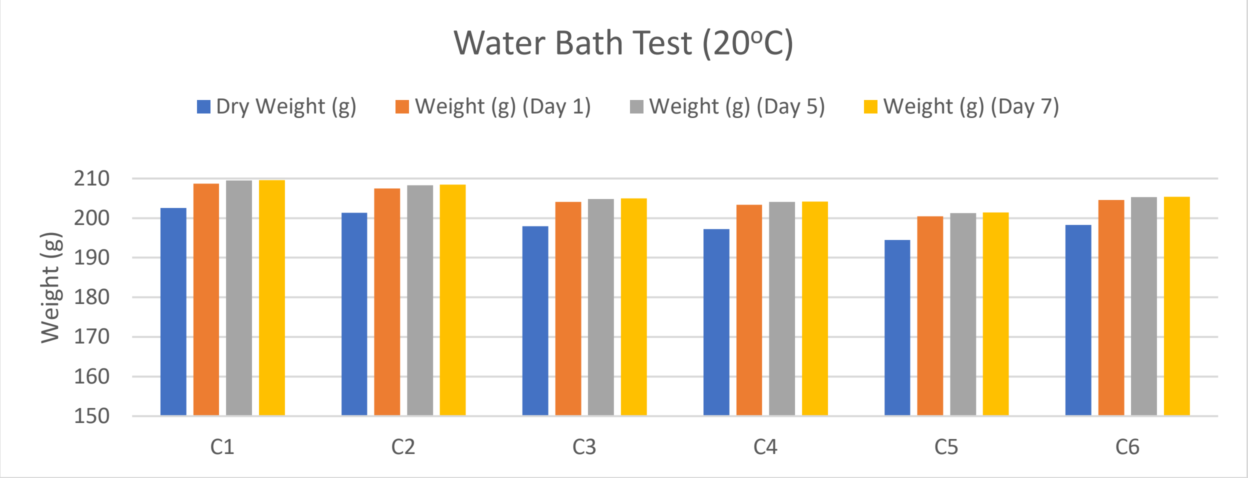


Figure 25 – Thermal Stability Weights (20 C)

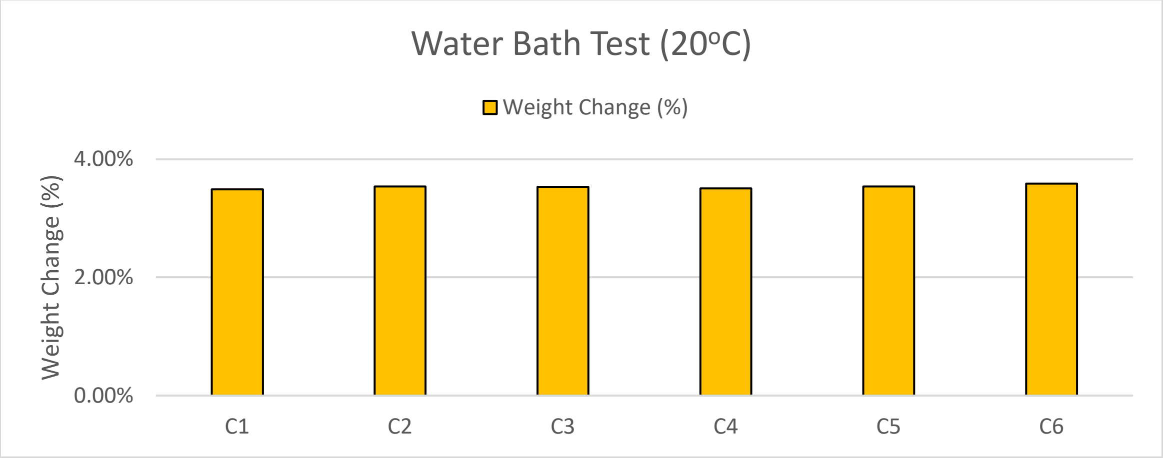


Figure 26 – Thermal Stability Weight Change (20 C)

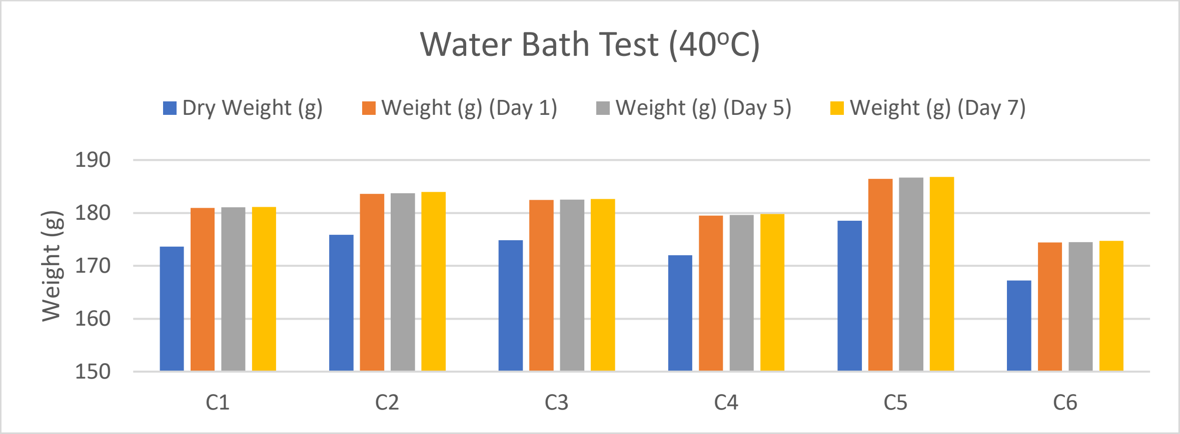


Figure 27 – Thermal Stability Weights (40 C)

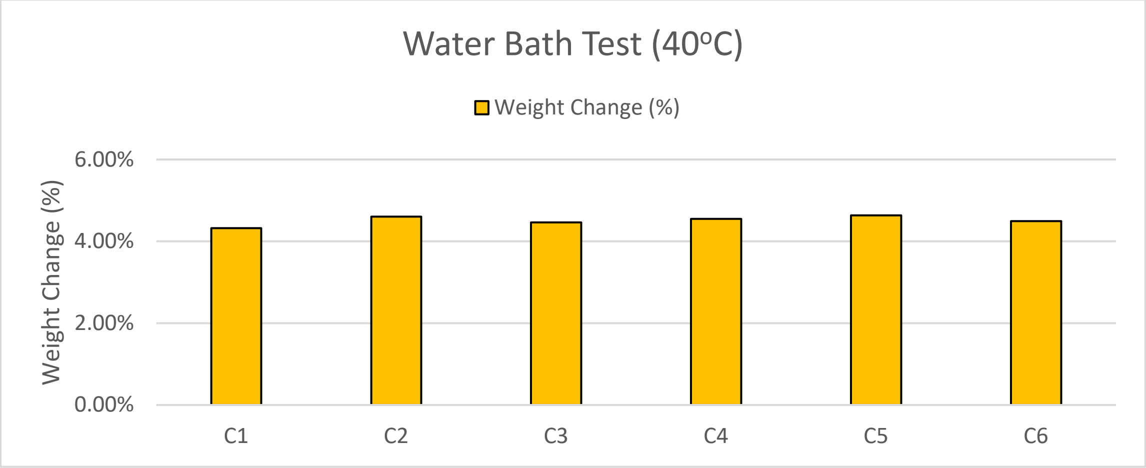


Figure 28 – Thermal Stability Weight Change (40 C)

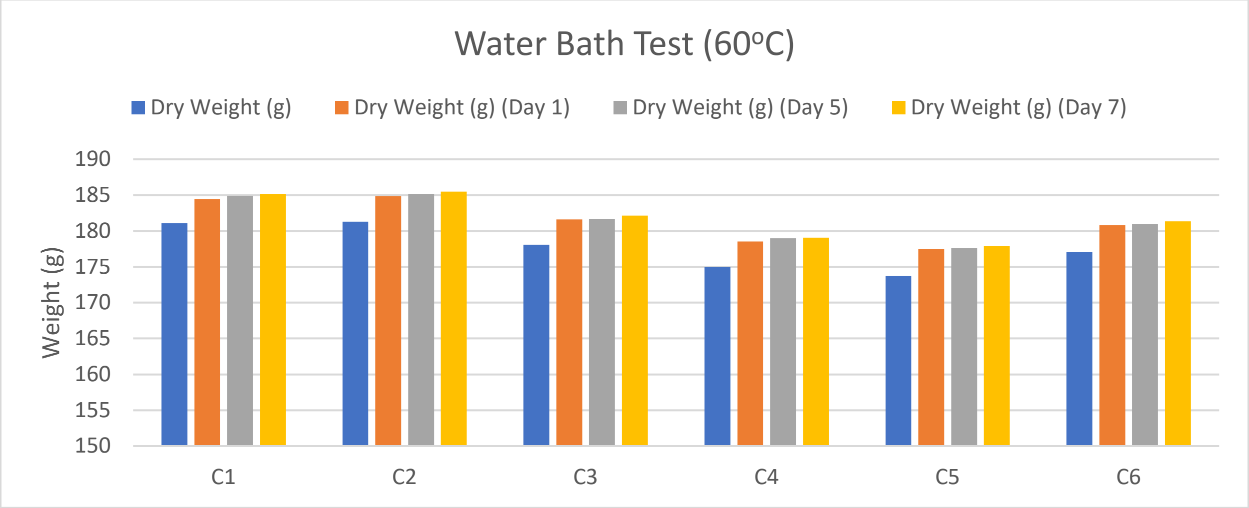


Figure 29 – Thermal Stability Weights (60 C)

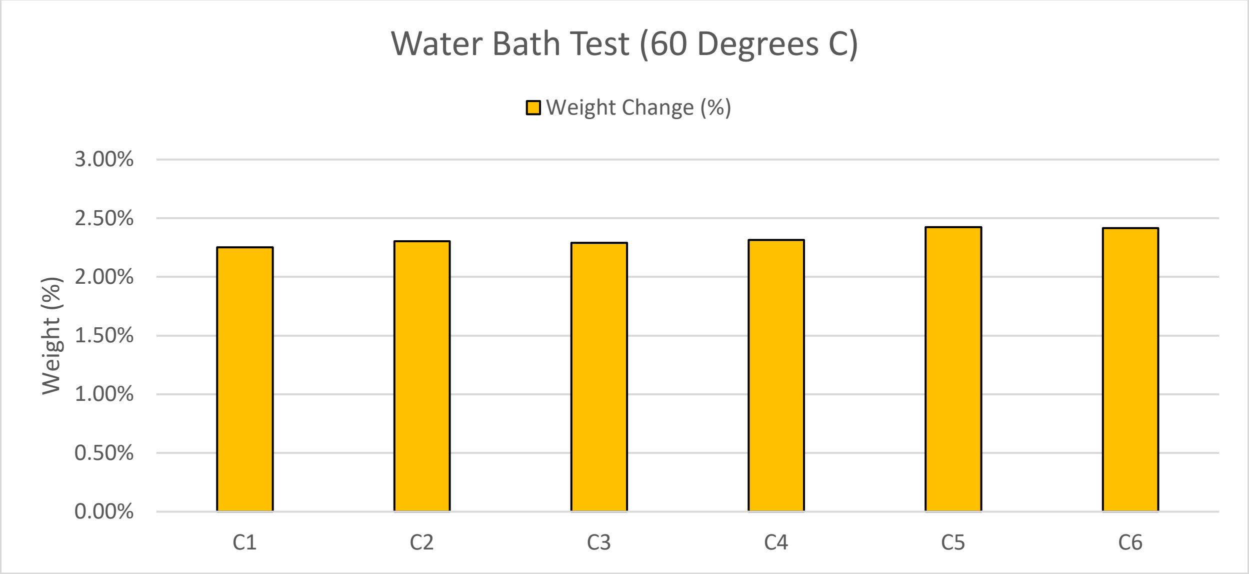
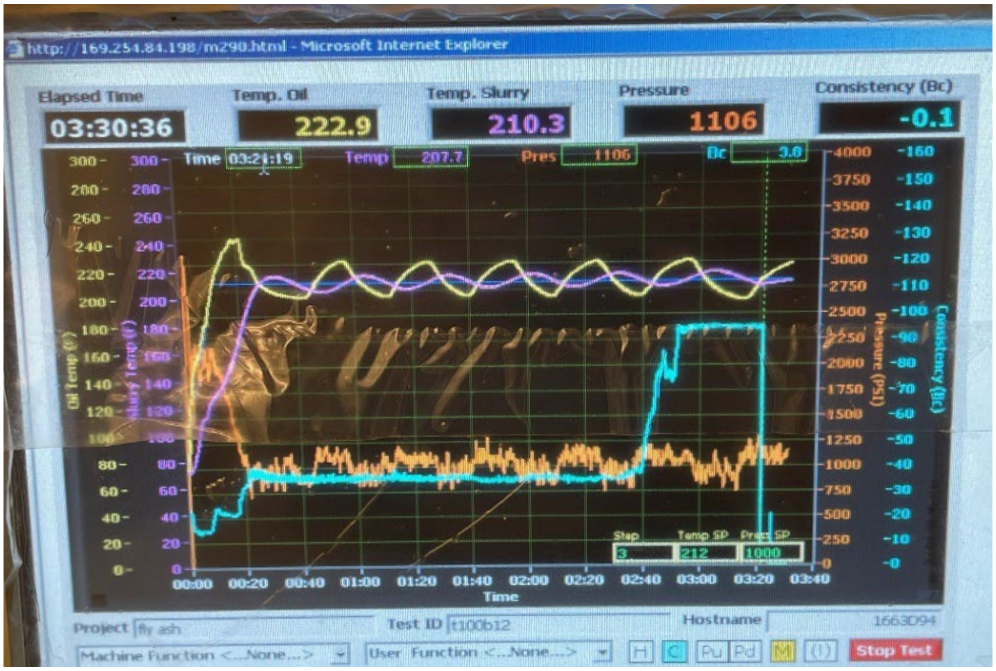


Figure 30 – Thermal Stability Weight Change (60 C)

5.3. Thickening Time Test

The thickening time test was conducted by pouring the B12W20 slurry into the consistometer’s chamber, and setting an ambient pressure of 1000 psi and three sets of temperatures of 20-, 60-, and 100-degrees C. Upon testing the B12W20 sample at 20 and 60 degrees C, the sample remained at 17 Bc (Bearden Consistency Unit) for over 8 hours, meaning that the temperature may be too low for it to set and thicken in time. At 100 degrees C, the sample reached a consistency of 100 Bc in just over 3 hours and 30 minutes (as shown in Figure 31), and Figure 32 shows the sample came out hardened around the rod within the chamber.



http://169.254.84.198/m290.html - Microsoft Internet Explorer

Elapsed Time 03:30:36
Temp. Oil 222.9
Temp. Slurry 210.3
Pressure 1106
Consistency (Bc) -0.1
Time 03:21:19
Temp 207.7
Pres 1106
Bc 3.0
Step 3
Temp SP 212
Pres SP 1000
Project fly ash
Test ID t100b12
Hostname 1663D94
Machine Function <...None...>
User Function <...None...>

Figure 31 – Geopolymer Consistometer Readings

Figure 32 – Thickened Geopolymer

The thickening time test for the cement was conducted by pouring the cement slurry into the consistometer's chamber and setting the same ambient pressure and temperature conditions as that of the test conducted for the geopolymer sample (1000 psi with the previously mentioned temperatures). At 20 degrees, the sample reached a consistency of 30 Bc in 4 hours (as shown in Figure 33), and Figure 34 shows the sample came out of the chamber as a slurry.

http://169.254.84.198/m290.html - Microsoft Internet Explorer

Elapsed Time 04:02:58
Temp. Oil 77.9
Temp. Slurry 78.5
Pressure 996
Consistency (Bc) 30.0

Time 00:00:00
Temp 75.9
Pres -469
Bc 30.6

Step 1
Temp SP 39
Press SP 0

Figure 33 – Cement Consistometer Readings (20◦C)

Figure 34 – Cement Sample After Thickening Time Test (20◦C)

The 60 degree test reached a consistency of 92.8 Bc in 6 hours and 33 minutes (as shown in Figure 35), and Figure 36 shows the sample came out only well shaped as the consistometer’s chamber; however, the sample did not harden in the chamber, as the geopolymer did.

http: 169.254.84.198/m290.html - Microsoft Internet Explorer

Elapsed Time: 06:33:20
Temp. Oil: 137.3
Temp. Slurry: 140.3
Pressure: 923
Consistency (Bc): 92.9

Time 00:00:00
Temp 76.2
Pres -469
Bc 29.4

Step 2
Temp SP 140
Pres SP 1000

Figure 35 – Cement Consistometer Readings (60◦C)

Figure 36 – Cement Sample After Thickening Time Test (60◦C)

The 100 degree cement test sample reached a consistency of 100 Bc in just under 1 hour and 49 minutes (as shown in Figure 37), and Figure 38 shows the sample came out hardened around the rod within the chamber, with some debris.

http://169.254.84.198/m290.html - Microsoft Internet Explorer

Elapsed Time 01:48:46
Temp. Oil 196.6
Temp. Slurry 222.5
Pressure 1069
Consistency (Bc) 9.2

Time 00:00:20
Temp 82.7
Pres -159
Bc 0.0

Step 3
Temp SP 212
Pres SP 1000

Project cementq
Test ID cement youssef 18july23
Hostname 1663D94

Figure 37 – Cement Consistometer Readings (100◦C)

Figure 38 – Cement Sample After Thickening Time Test (100◦C)

The thickened geopolymer sample is shown to have had an overall stronger bond in the chamber and around the rod when extracted. Regarding time, certain additives such as accelerators or retarders can be used to serve the setting and thickening needs required of the cement. However, it should be noted that the geopolymer includes Class F fly ash, which is an extender, and sodium metasilicate, which is also a chemical extender and an accelerator.

5.4. Sedimentation Test

The sedimentation test was conducted by having the batches of B12W20 and B15W20 geopolymer and the cement batch mixed and poured into cylindrical tubes, left to set and harden, then cut into three equal segments. The segments for each batch are top, middle, and bottom. Each segment was weighed, then placed in 300 milliliters of water to determine each sample's segment's weight (as seen in Figure 39) in order to determine each segment's density (as seen in Figure 40). This is needed to observe how the density of each segment of the geopolymer will react with respect to depth and setting.

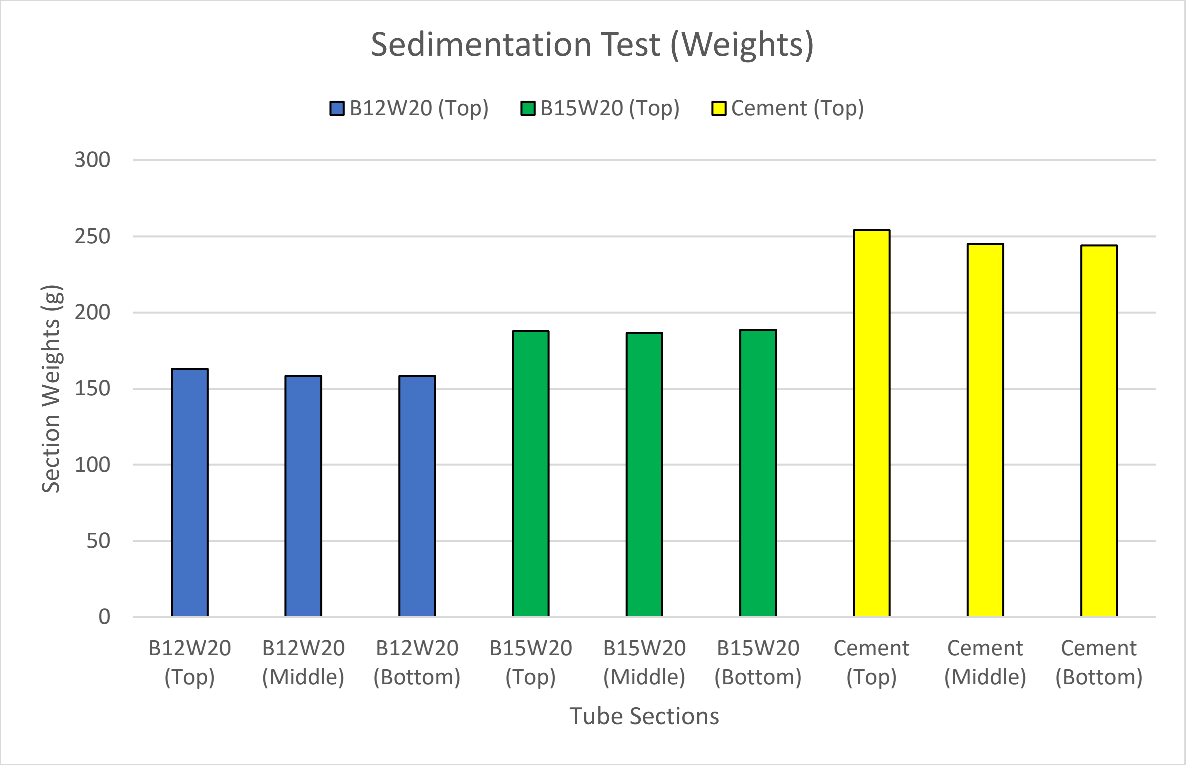


Figure 39 – Sedimentation Test Weights and Volumes

Figure 40 shows that the density in the two geopolymer samples (B12W20 and B15W20) don't exhibit as much consistency as the cement, which shows a presence of sedimentation.

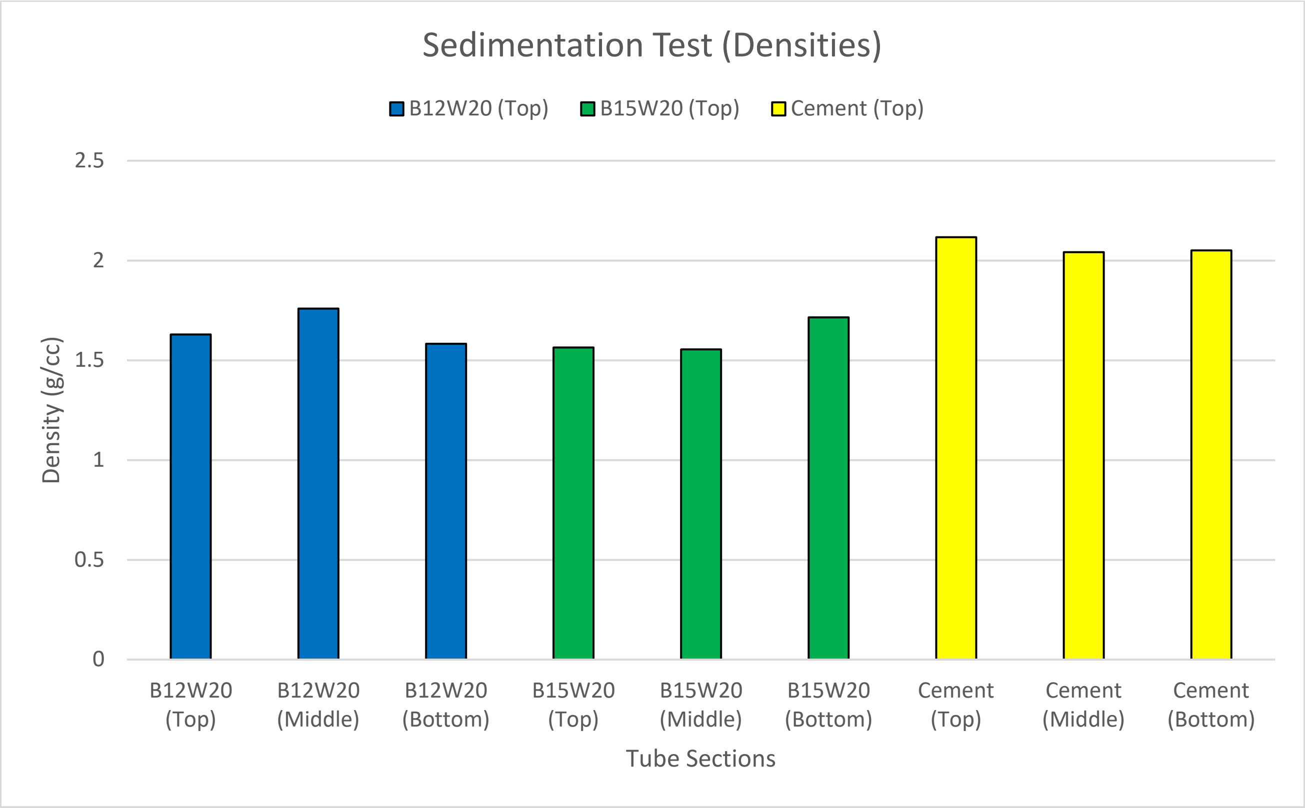


Figure 40 – Sedimentation Test Densities

5.5. NaOH Immersion Test

2 samples of each geopolymer batch were placed in 1%, 10% and 20% solutions of NaOH mixed with water after being dry-weighed, in an effort to determine if there is any weight gain or deterioration in the samples and compare the effects on the geopolymers with that of the cement. All samples were left to set for 3 days and showed no signs of deterioration.

It is shown in Figures 41 to 46, that small concentrations of NaOH will generally result in a slight weight loss due to the NaOH binding effect, whereas an increase in the concentration will eventually lead to weight gain. This is potentially due to the excess of hydroxide in the geopolymers, as sodium metasilicate, an already existing ingredient and binder in these geopolymer samples, contains sodium hydroxide, which acts as a binder [31].

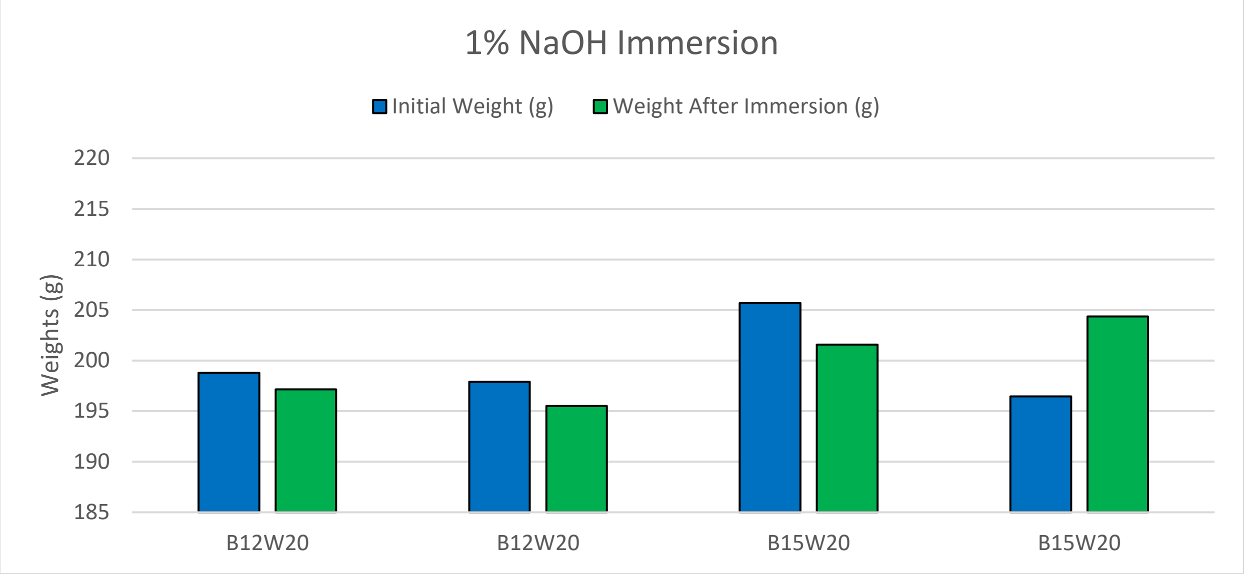


Figure 41 – Geopolymer NaOH Test (1%)

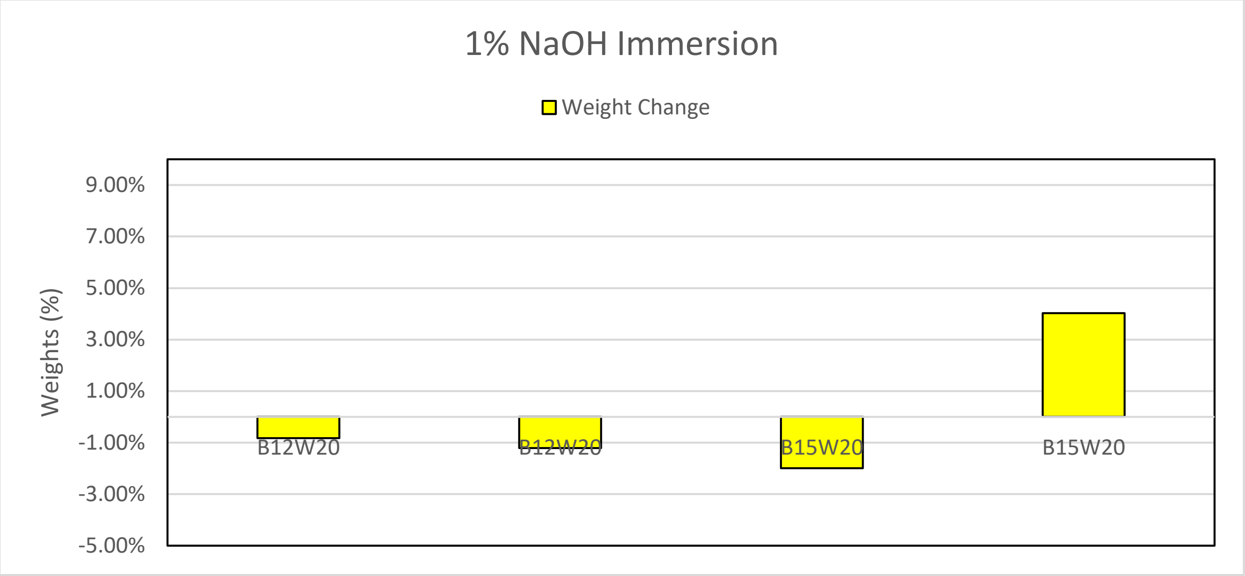


Figure 42 – Geopolymer NaOH Weight Change (1%)

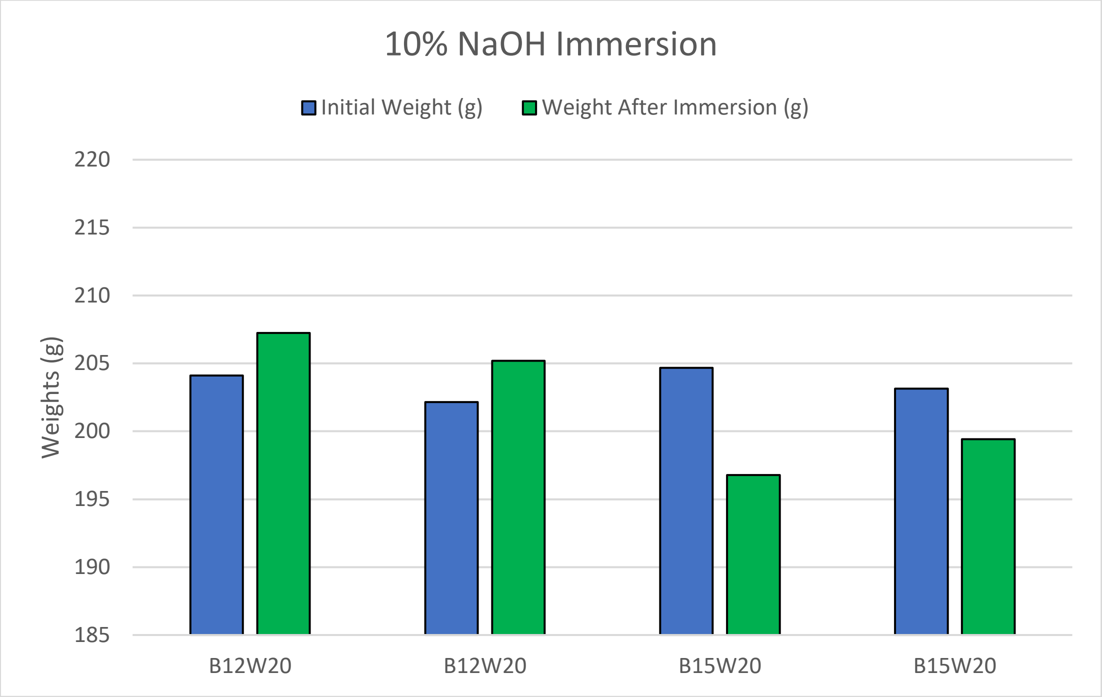


Figure 43 – Geopolymer NaOH Test (10%)

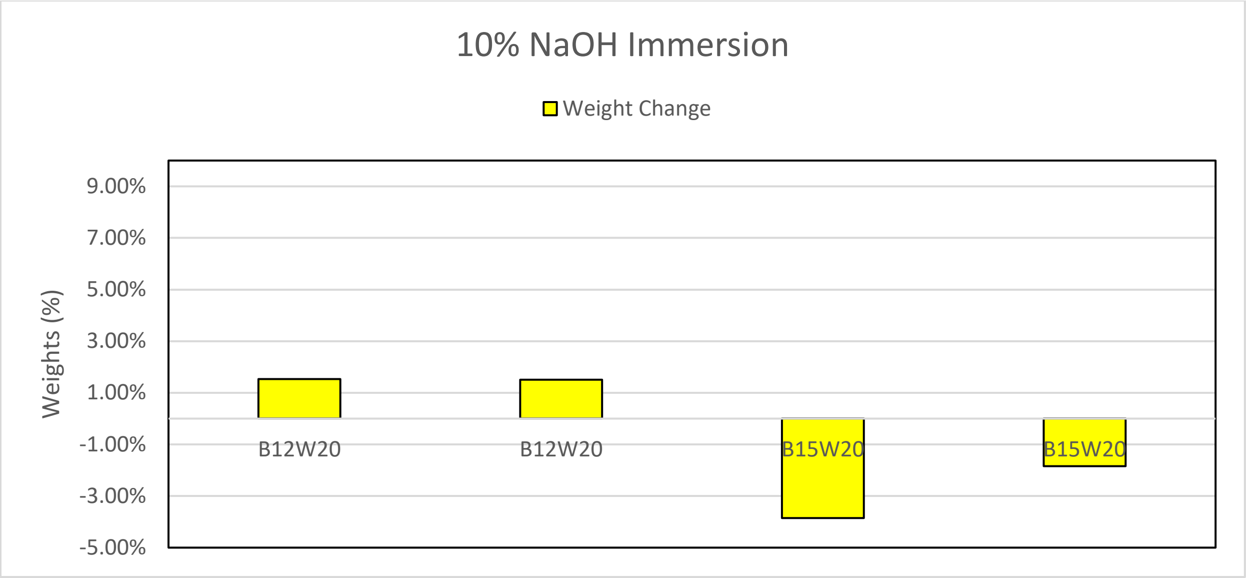


Figure 44 - Geopolymer NaOH Weight Change (10%)

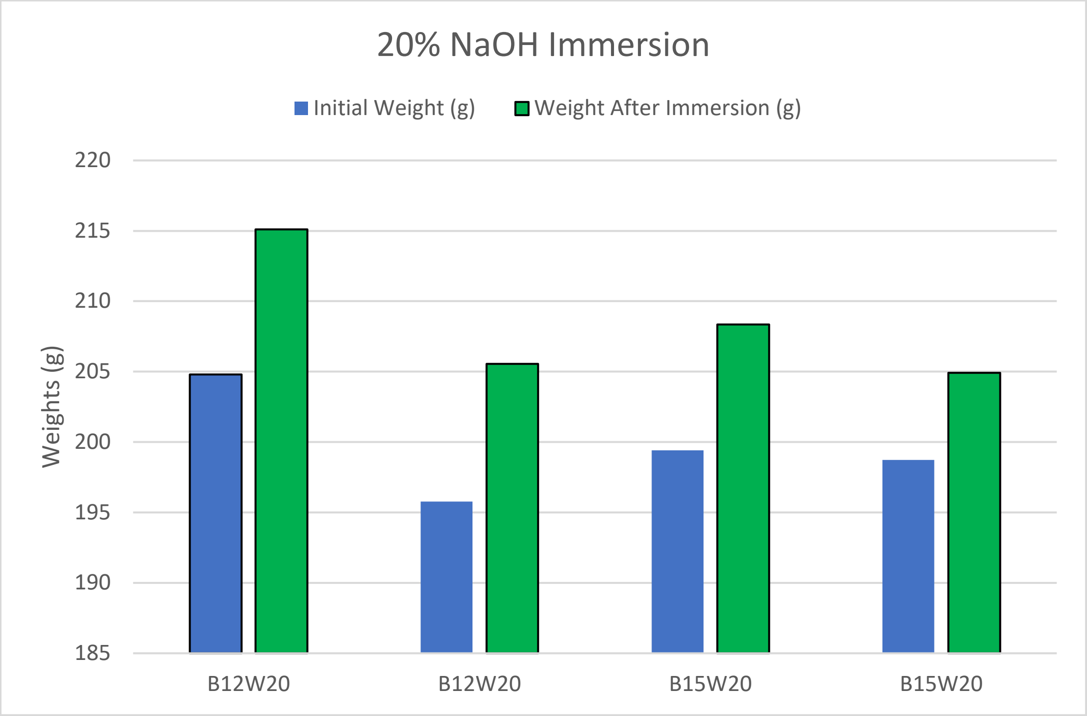


Figure 45 – Geopolymer NaOH Test (20%)

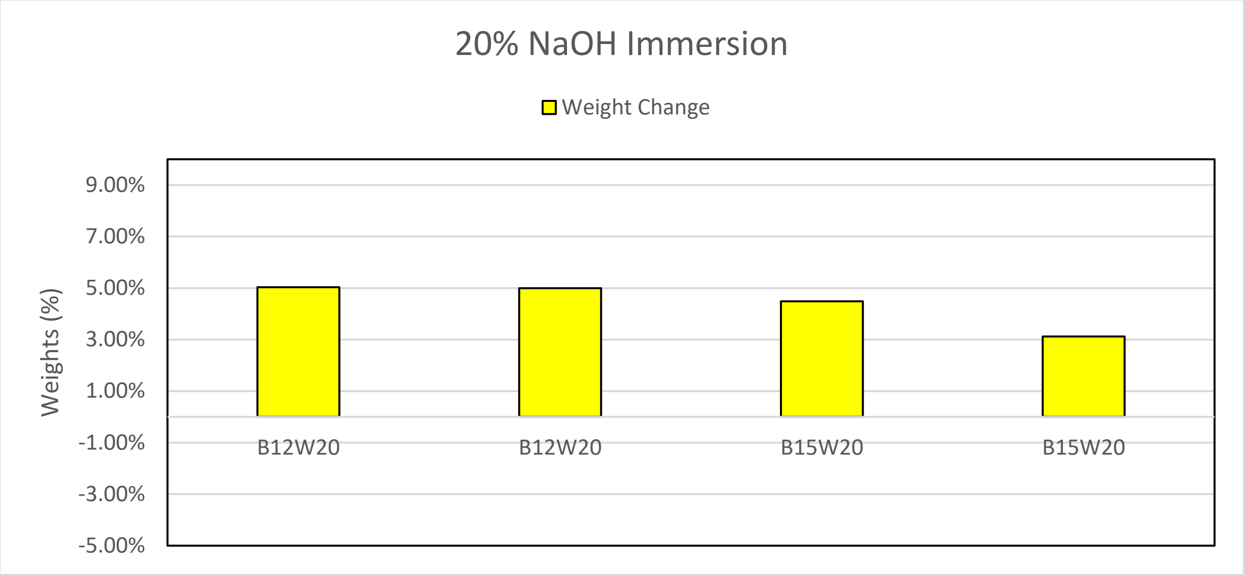


Figure 46 - Geopolymer NaOH Weight Change (20%)

The cement samples were placed in the NaOH solution as the geopolymer samples. The samples exhibited a 6% weight gain with 5% concentration (as shown in Figure 47), and a 7% weight gain with the 15% concentration (as in Figure 48). This leads to the consideration that increasing NaOH to the cement samples will gain weight (as demonstrated in Figure 49).

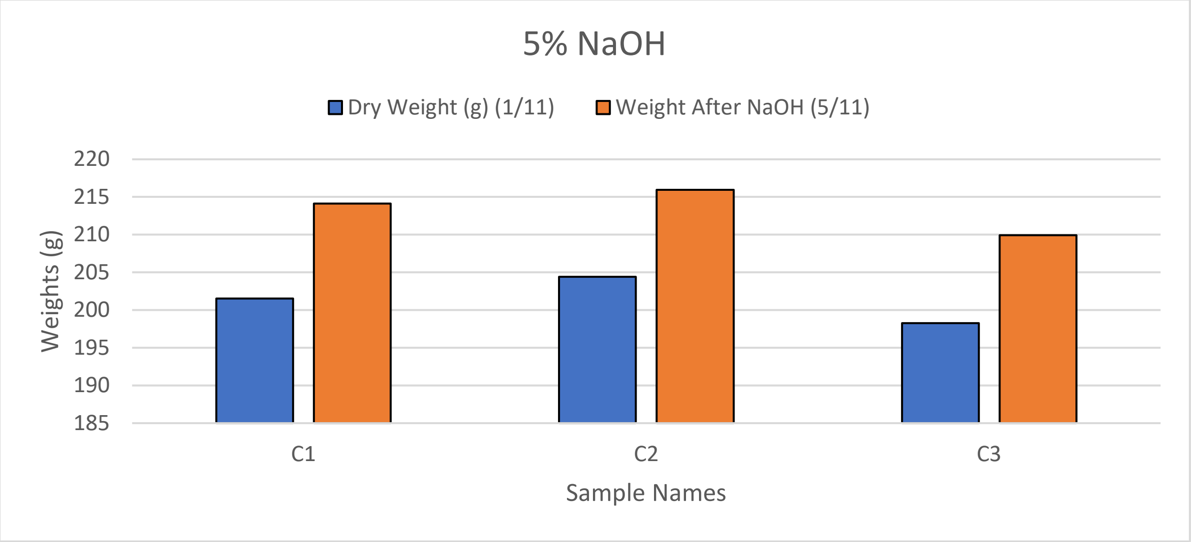


Figure 47 – Cement NaOH Test (5%)

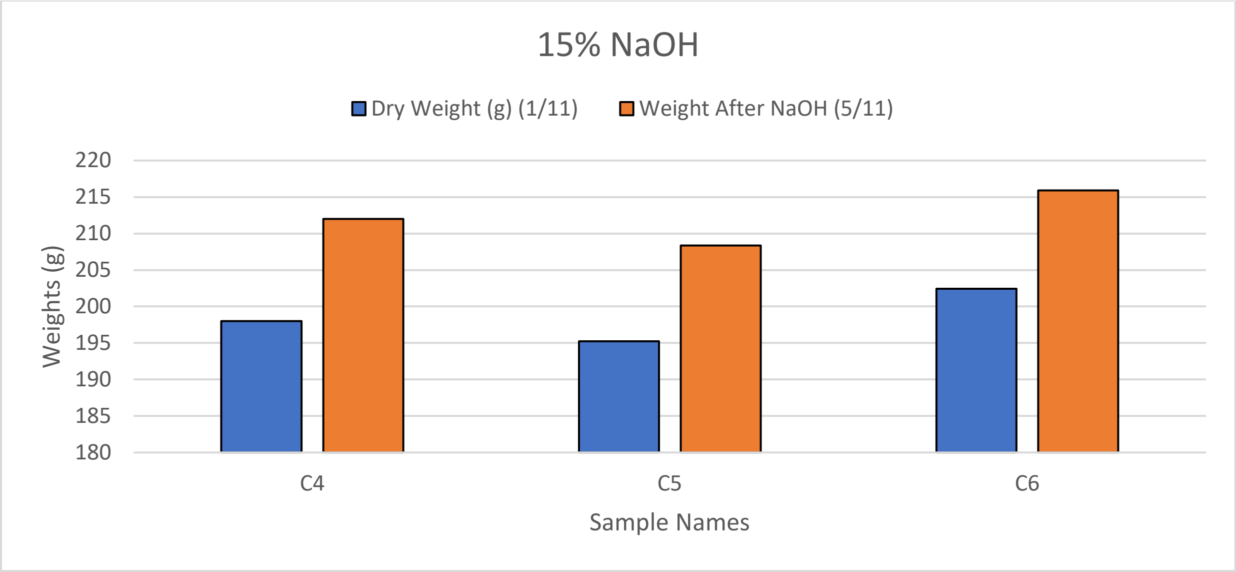


Figure 48 – Cement NaOH Test (15%)

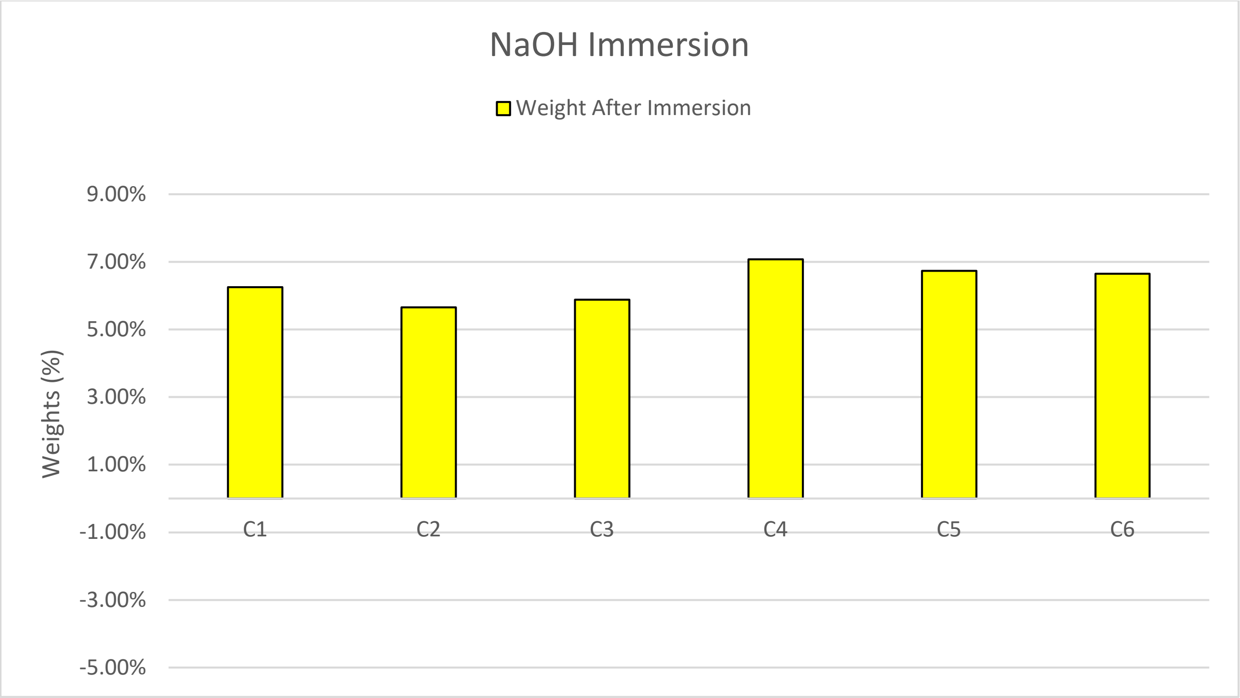


Figure 49 - Cement NaOH Immersion Weight Change

5.6. HCl Test

3 samples of Class G cement were placed in a solution with water and 2 different concentrations of hydrochloric acid (HCl). The test is conducted to examine the potentially aggressive effects of HCl on the samples. One test contained hydrochloric acid at 5% of the solution's weight (as shown in Figure 50), and the other contained 15% (as shown in Figure 51). As shown in Figures 50 and 52, the Class G cement samples in 5% HCl solution generally gained weight, while only one sample slightly lost some weight due to being corroded.

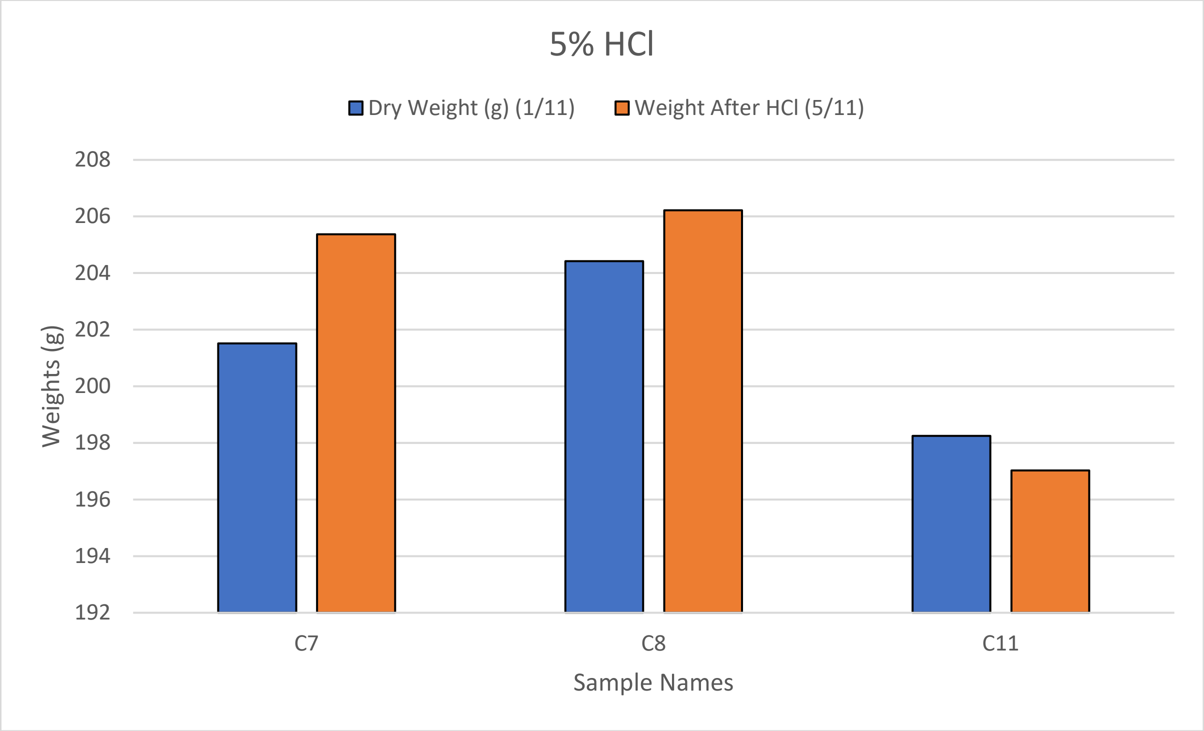


Figure 50 – Cement HCl Test (5%)

The samples in 15% HCl solution also generally lost weight due to corrosion, but one sample remained intact and gained weight (as shown in Figures 51 and 52). This leads to the consideration that Class G cement is limited to a certain low composition of HCl resistance.

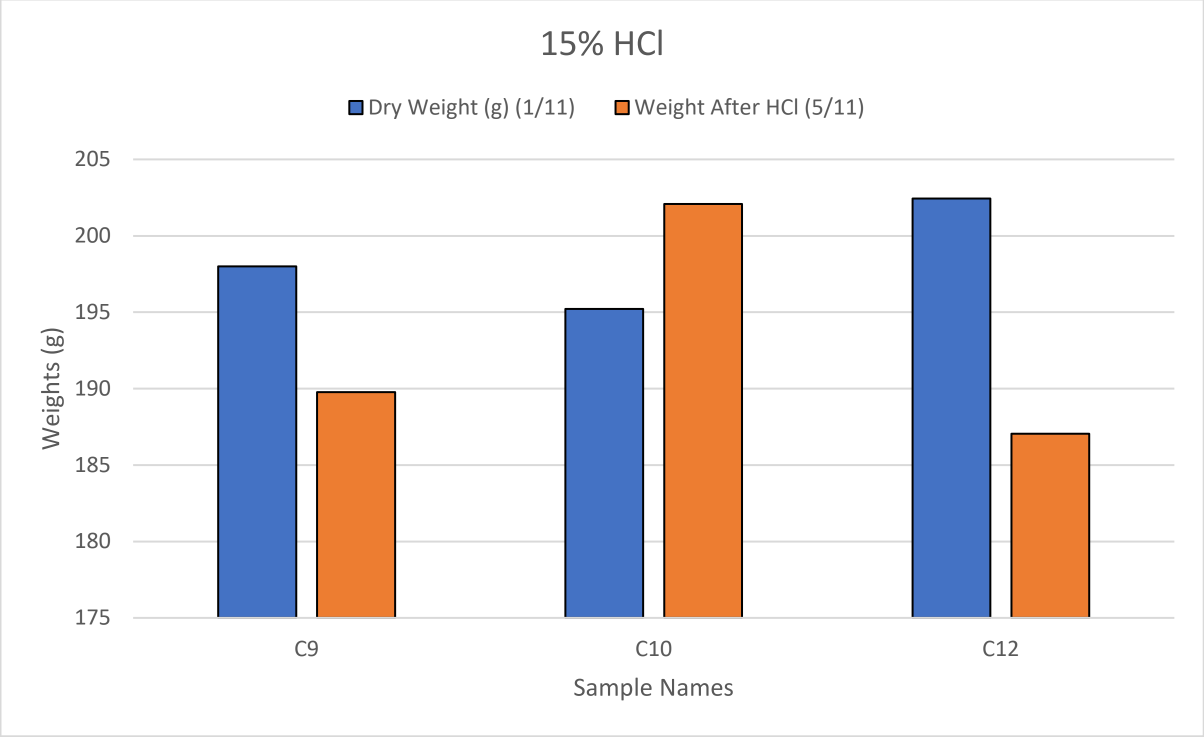


Figure 51 – Cement HCl Test (20%)

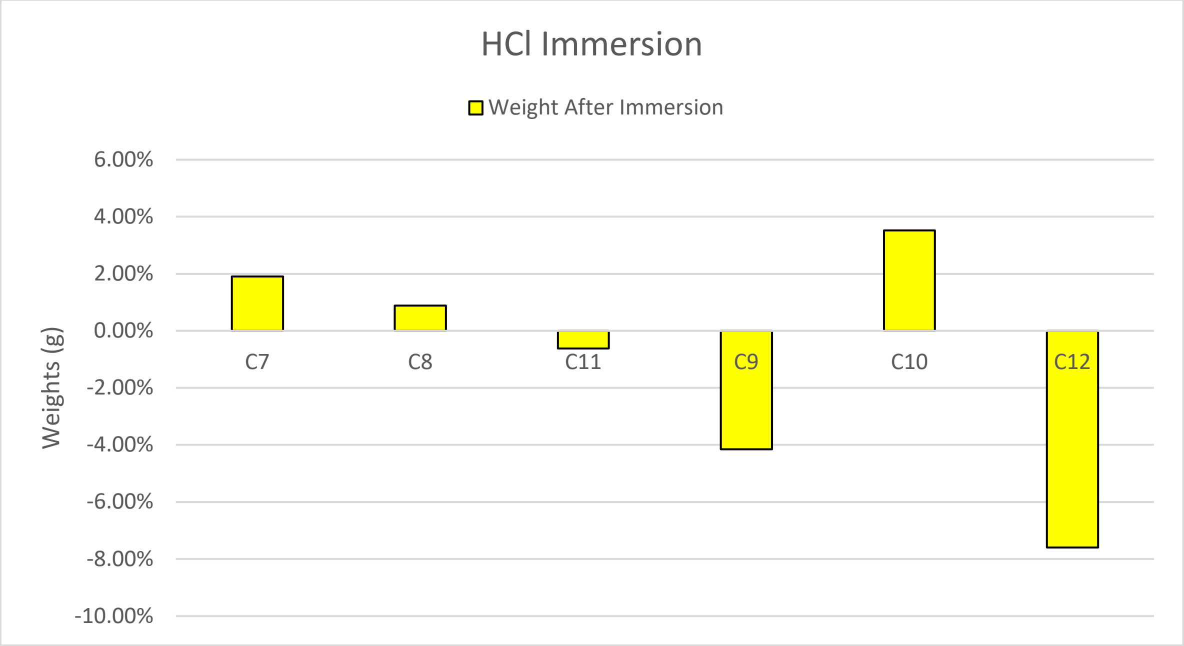


Figure 52 - Cement HCl Weight Change

5.7. Compressive Strength Test

Using a universal testing machine, the surface areas of samples used in previous experiments were logged in, and force was gradually applied on the samples over time until they failed. After the forces were obtained, they were divided by the surface area to determine the force per unit area, then converted to pressure in pounds per square inch (psi), to replicate the effect of downhole pressures and stresses surrounding the well.

Figure 53 shows the compressive strength of the geopolymer samples subjected to 95 degree C for 7 days at different concentrations of Na_2SiO_3. The samples that had 15% content had a compressive strength between 1200 psi to 1400 psi at point of failure. The 12% Na_2SiO_3 content samples had a compressive strength between 600 psi to 1000 psi at point of failure.

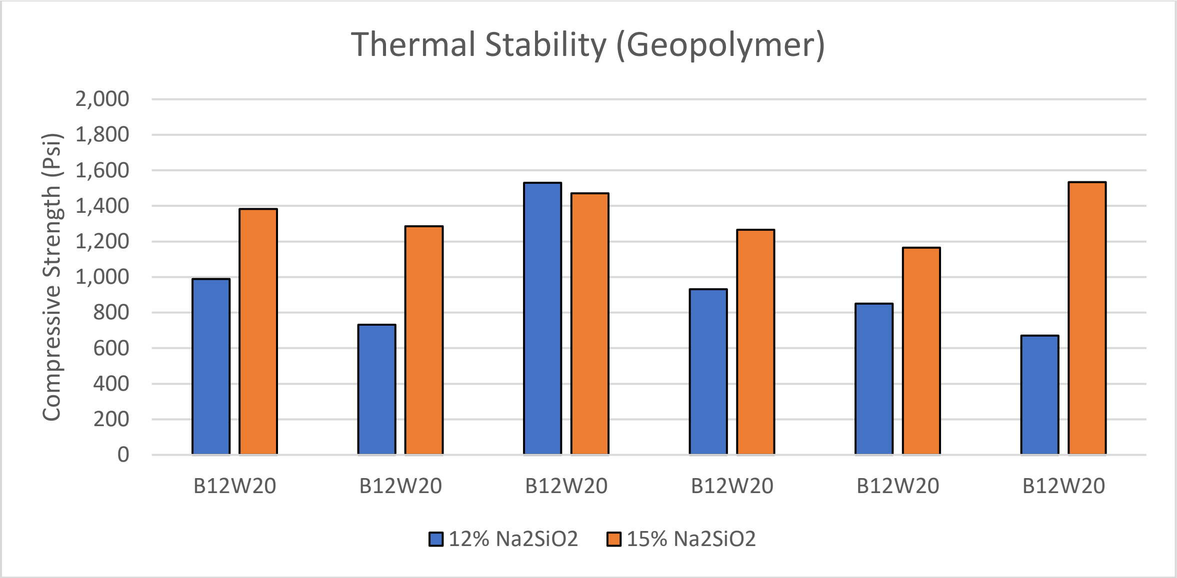


Figure 53 – Compressive Strength of Thermal Stability Geopolymer Samples

Figure 54 shows the compressive strength of the cement samples subjected to different temperatures for 7 days. The samples at 60 degrees C showed the most consistency compared to the samples at lower temperatures, with a point of failure between 1600 psi and 2400 psi. It can be considered that the less absorbent cement samples are more durable than geopolymers at lower temperatures and could potentially require more strength to fail at higher temperatures.

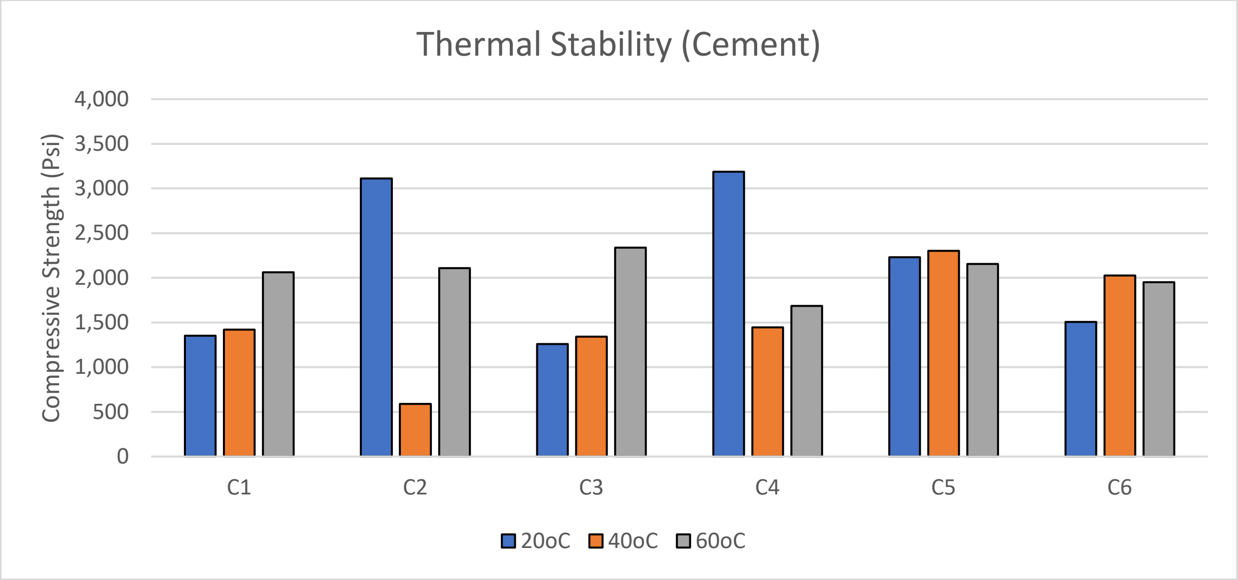


Figure 54 – Compressive Strength of Thermal Stability Cement Samples

Figure 55 shows the compressive strength of geopolymer samples of different concentrations of Na_2SiO_3 subject to 1% NaOH solution for 7 days. The samples with 12% Na_2SiO_3 failed at 652 psi and 839 psi of compressive strength, whereas the 15% Na_2SiO_3 samples failed at 742 psi and 778 psi. The 15% Na_2SiO_3 samples show the most stable range of failure.

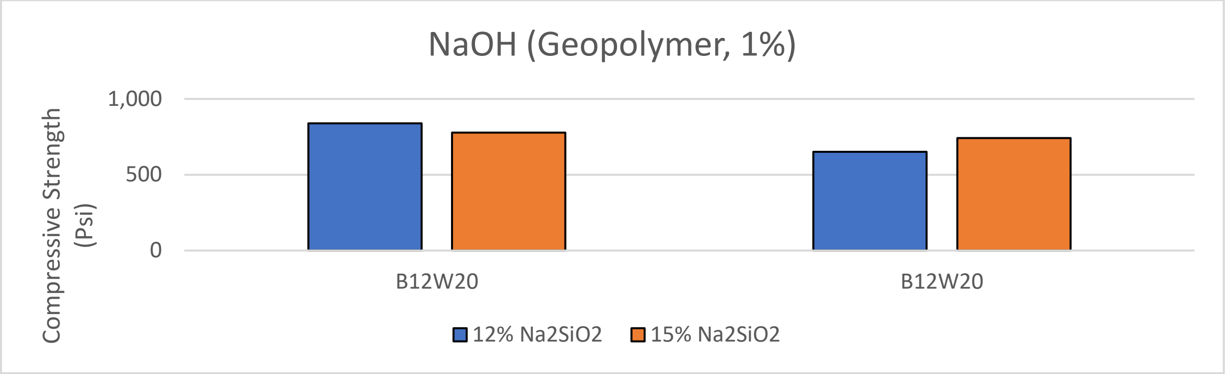


Figure 55 – Compressive Strength of 1% NaOH Geopolymer Samples

Figure 56 shows the compressive strength of geopolymer samples of different concentrations of Na_2SiO_3 subject to 10% NaOH solution for 7 days. The samples with 12% Na_2SiO_3 failed at 1380 psi and 1400 psi, whereas the 15% samples failed at 2402 psi and 3591 psi. As the NaOH increases, the sample with higher silica reaches a higher point of failure. The 12% Na_2SiO_3 samples show consistency in terms of its range for the point of failure.

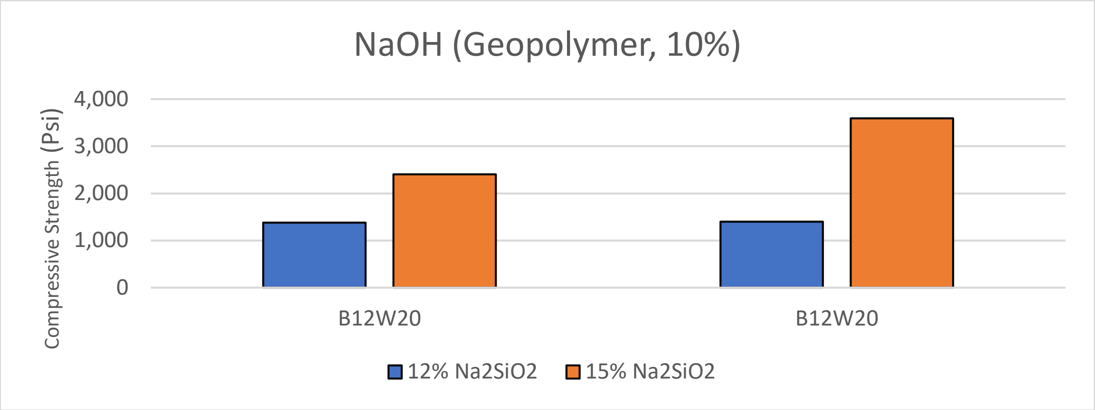


Figure 56 – Compressive Strength of 10% NaOH Geopolymer Samples

Figure 57 shows the compressive strength of geopolymer samples of different concentrations of Na_2SiO_3 subject to 20% NaOH solution for 7 days. Samples with 12% Na_2SiO_3 failed at 2714 psi and 3043 psi, whereas the 15% Na_2SiO_3 samples failed at 1765 psi and 2258 psi. The 12% Na_2SiO_3 high points of failure could be due to the activator being abundant in silica.

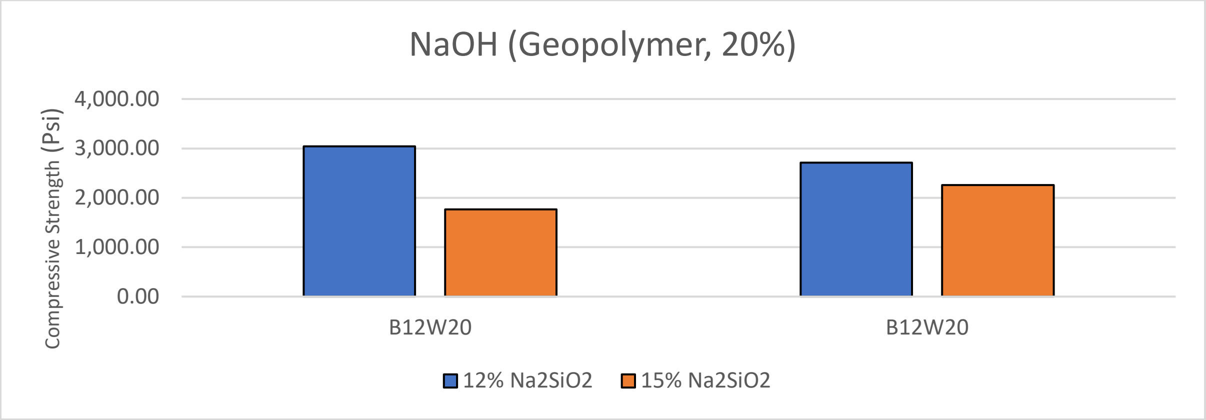


Figure 57 – Compressive Strength of 20% NaOH Geopolymer Samples

Figure 58 shows the compressive strength of cement samples subject to different concentrations of NaOH solution for 7 days. Cement in 5% NaOH solution failed between 2100 psi and 2541 psi, whereas samples in 15% NaOH solution failed between 2520 psi and 2566 psi, with an outlier point of failure at 1208 psi. The NaOH content slightly strengthens the cement.

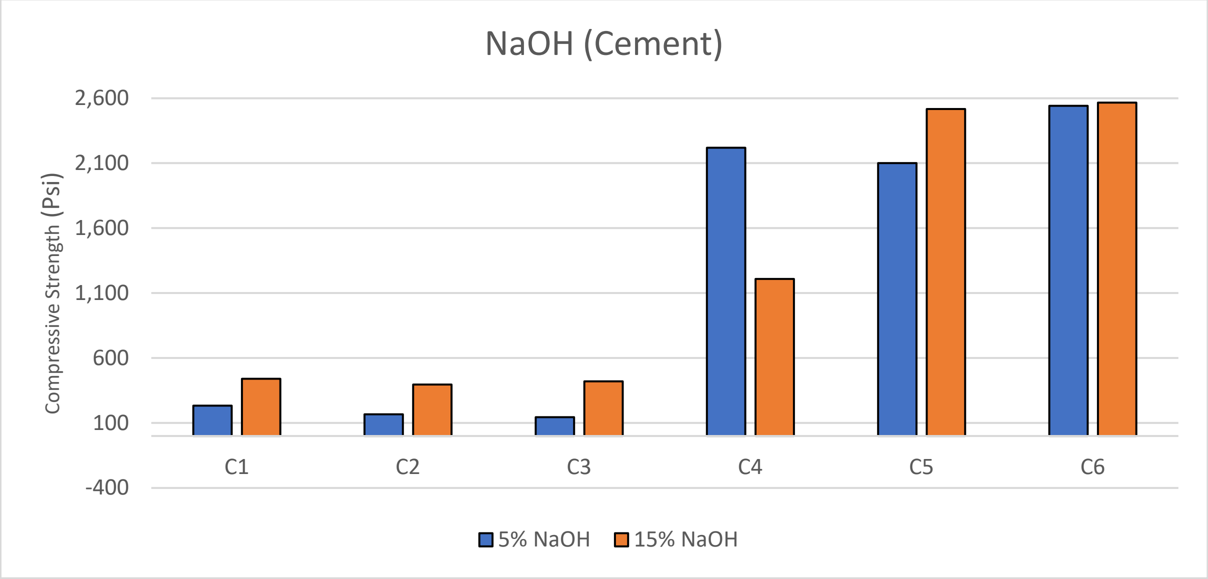


Figure 58 – Compressive Strength of NaOH Cement Samples

Figure 59 shows the compressive strength of cement and geopolymer samples subject to 5% HCl solution for 7 days. The geopolymer samples showed points of failure ranging from 160 psi to 180 psi, whereas the cement samples showed points of failure from 2132 psi to 2250 psi.

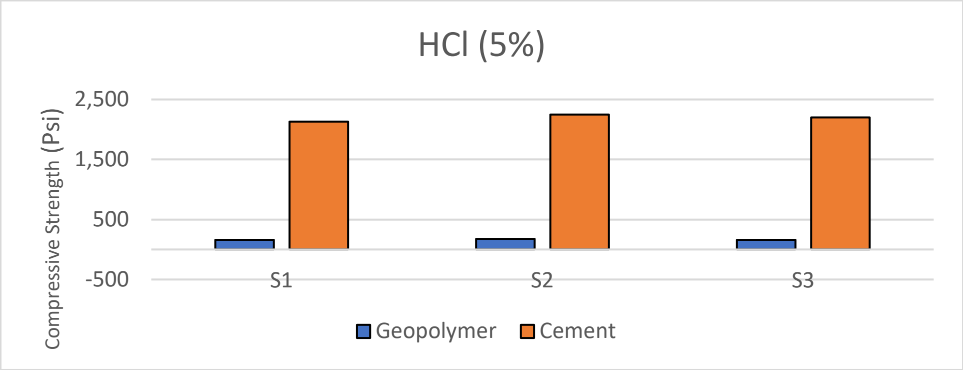


Figure 59 – Compressive Strength of 5% HCl Samples

Figure 60 shows the compressive strength of cement and geopolymer samples subject to 15% HCl solution for 7 days. The geopolymer samples failed between 200 psi and 255 psi of compressive strength, whereas the cement samples failed between 1240 psi and 1290 psi, with an outlier of 2066 psi. The compressive strength for increased for geopolymers and decreased for cement. The cement will still require more strength to fail than the strengthened geopolymer.

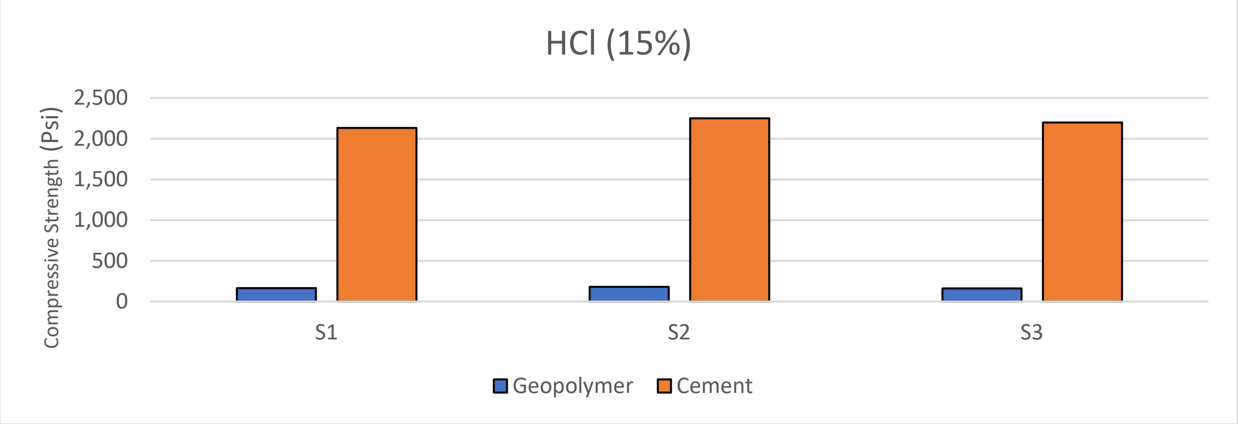


Figure 60 - Compressive Strength of 15% HCl Samples

Figure 61 shows the compressive strength of cement and geopolymer samples subject to black crude oil saturation for 7 days. The geopolymer failed between 460 psi and 572 psi, and the cement failed between 2223 psi and 2414 psi. This shows that cement is more durable than geopolymer when saturated in oil in downhole environments.

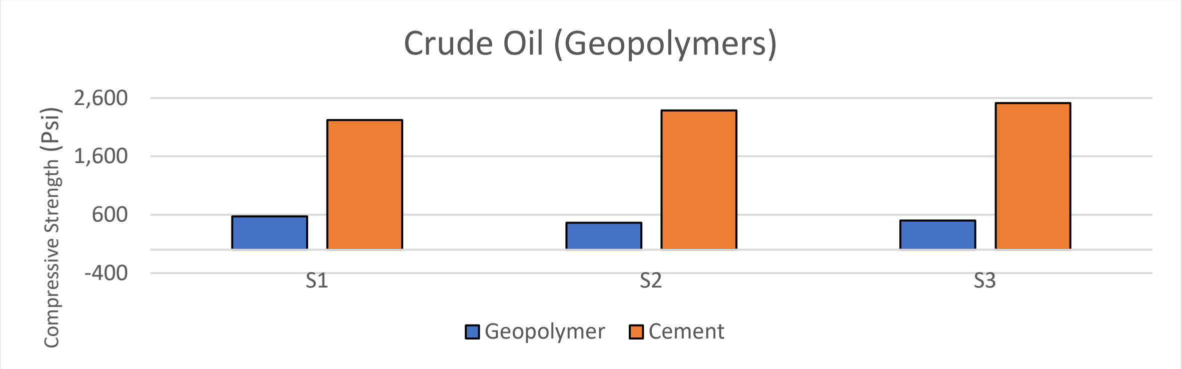


Figure 61 – Compressive Strength of Crude Oil Samples

Chapter 6: Summary and Conclusion

6.1. Future Tests

While plenty of data was gathered and obtained to assist in determining an acceptable sample of fly ash geopolymer to be an alternative to Class G cement, further tests are needed to further verify and confirm this:

- Different conditions for tests that have already been conducted.
- Using Class F fly ash as a supplementary aggregate to the cement could be examined.
- Slight additives can be used to modify or adjust the slurry's behavior.

6.2. Conclusion

The research and the testing process showed that using a single binder of sodium metasilicate for a geopolymer cement has its advantages, yet it requires modifications to properly function:

- Using Class G cement with minor adjustments would continue to provide success in cementing oil and gas wells and would not include the complexity of checking the base material's chemical content, while focusing on designing the slurry for that particular job, and conducting safer operations for personnel on the rig, and the well.
- Class F fly ash faces a challenge with differing alumina and silica content and its potentially differing reactions at high temperature and high-pressure conditions, as well as downhole fluids and substances.
- The use of this geopolymer would require intense design and an incorporation of excessive chemicals and additives to adjust the material's integrity and the fly ash's consistency.

- This geopolymer’s use may be limited to only the surface and intermediate casings, and zones close to, but not in, the production zone. In such a case, two stage cementing may be incorporated along with API cement using a diverter.

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Appendix

Table A1 will provide the full list of the initial 35 geopolymer formulations with each ingredient’s weight, concentration and ratios:

Table A1 - Initial Geopolymer Batch Formulations

SERIAL NUMBER	FA/BINDER RATIO	COMPOSITION (%)			WEIGHT (g)			
		Fly Ash	Binder	Water	Fly Ash	Binder	Water	Total
1	15	84%	6%	10%	100	6.67	11.85	118.519
2	15	80%	5%	15%	100	6.67	18.82	125.49
3	15	75%	5%	20%	100	6.67	26.67	133.333
4	15	70%	5%	25%	100	6.67	35.56	142.222
5	15	66%	4%	30%	100	6.67	45.71	152.381
6	15	61%	4%	35%	100	6.67	57.44	164.103
7	15	56%	4%	40%	100	6.67	71.11	177.778
8	20	86%	4%	10%	100	5.00	11.67	116.667
9	20	81%	4%	15%	100	5.00	18.53	123.529
10	20	76%	4%	20%	100	5.00	26.25	131.25
11	20	71%	4%	25%	100	5.00	35.00	140
12	20	67%	3%	30%	100	5.00	45.00	150
13	20	62%	3%	35%	100	5.00	56.54	161.538
14	20	57%	3%	40%	100	5.00	70.00	175
15	25	87%	3%	10%	100	4.00	11.56	115.556
16	25	82%	3%	15%	100	4.00	18.35	122.353
17	25	77%	3%	20%	100	4.00	26.00	130
18	25	72%	3%	25%	100	4.00	34.67	138.667
19	25	67%	3%	30%	100	4.00	44.57	148.571
20	25	63%	3%	35%	100	4.00	56.00	160
21	25	58%	2%	40%	100	4.00	69.33	173.333
22	30	87%	3%	10%	100	3.33	11.48	114.815
23	30	82%	3%	15%	100	3.33	18.24	121.569
24	30	77%	3%	20%	100	3.33	25.83	129.167
25	30	73%	2%	25%	100	3.33	34.44	137.778
26	30	68%	2%	30%	100	3.33	44.29	147.619
27	30	63%	2%	35%	100	3.33	55.64	158.974

28	30	58%	2%	40%	100	3.33	68.89	172.222	
29	35	88%	3%	10%	100	2.86	11.43	114.286	
30	35	83%	2%	15%	100	2.86	18.15	121.008	
31	35	78%	2%	20%	100	2.86	25.71	128.571	
32	35	73%	2%	25%	100	2.86	34.29	137.143	
33	35	68%	2%	30%	100	2.86	44.08	146.939	
34	35	63%	2%	35%	100	2.86	55.38	158.242	
35	35	58%	2%	40%	100	2.86	68.57	171.429	