

University of Southern Queensland
Faculty of Engineering and Surveying

The Effect of Efflorescence on the Mechanical Properties of Geopolymers

A dissertation submitted by

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Abstract

The purpose of this research project was to find out and compare the effect of efflorescence on different composition of Alkali Activated Fly Ash based Geopolymer Paste (AAFAGP). Since geopolymers are gaining worldwide recognition as a greener alternative to the more popular Ordinary Portland Cement a better understanding of the behaviour of AAFAGP is needed. As far as the author is aware there is no literature available on the effects of efflorescence on the mechanical properties of AAFAGP, this is the part of the literature which this research intends to fill.

Various mix designs or composition of AAFAGP was used in this research in order to obtain the compressive strengths of the composition. Each mix design or composition is exposed to three different environments:

- Immersed in water
- Dry exposure
- Partially Immersed in water

The partially immersed simulated the efflorescence effect. These were left for 28 days before being crushed to compare the compressive strength of the each mix design being exposed to different environments.

The results formulated from this report show the efflorescence compressive strength was tested after 28 days and compared with the dry sample compressive strength. Along with this the compressive modulus was also obtained. From the investigation done it was found that efflorescence lowered the compressive strengths of various mix designs these included:

- Mix design 1: Gladstone 1
- Mix design 2: Gladstone 2
- Mix design 3: Callide 1
- Mix design 4: Callide 2
- Mix design 5: MM1-MgO

However for the rest of the various mix designs there was no conclusive evidence that showed efflorescence had a clear effect, this was because for each mix design had 5 specimens and at times the compressive strength of the efflorescence was greater than that of the dry specimen. Modulus of elasticity is shown to have a direct relationship with compressive strength.

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1.0 Introduction

1.1 Problem Statement

Alkali- activated geo-polymer provides a greener alternative to the use of Ordinary Portland Cement which emits harmful carbon dioxide gas into the atmosphere during production. This greener solution provides a material that can provide the same strength but also suffers the same problem of efflorescence as does its predecessor. As stated by ŠKVÁRA, et al., (2009) sodium is bonded in the aluminosilicate polymer structure as $\text{Na}(\text{H}_2\text{O})_n^+$ and not as Na^+ . The Na bond in the aluminosilicate polymer structure is weak and because of this weak structure aluminosilicate polymer material forms efflorescence in a humid environment.

1.2 Background

Geopolymer is relatively a new material which was developed after World War 2. It is seen as an alternative to OPC. It is widely known that geopolymer is more environmentally friendly since its production does not release carbon dioxide (CO_2) into the air. This new material was only extensively studied during the early 1970s by Joseph Davidovits. This resulted in a material that robust and is applicable to almost any conditions. Because of this robustness geopolymers are used in fire- and heat-resistant coatings and adhesives, medicinal applications, high-temperature ceramics, new binders for fire-resistant fibre composites, toxic and radioactive waste encapsulation and new cements for concrete

The application of geopolymers in civil engineering structures is of great interest because of the properties it possesses. The illustration below summarises (Davidovits, 1994) the applications of geopolymers.

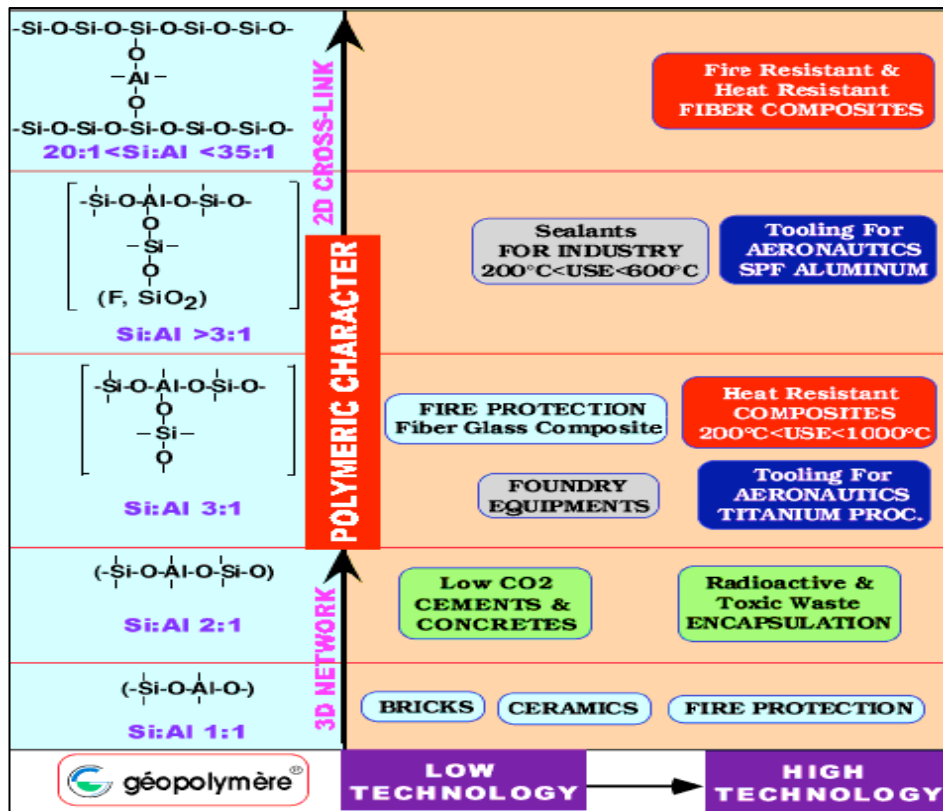


Figure 1: Different Applications of Geopolymers Source: (Davidovits, 1994)

1.3 Aims and Objectives

In this research project the efflorescence effect on the mechanical properties will be investigated on various samples of fly ash based geopolymer and at different exposure conditions.

- Compression Modulus
- Compressive Strength

1.4 Scope

The scope of this project thesis encompasses the research of the mechanical properties (compressive strength) of geopolymer paste under efflorescence. Various mix designs will be tested this under this condition (efflorescence) and will be compared to two other condition scenarios, in dry condition and fully immersed in water. Tests were conducted after 28 days for each mix design under the three conditions.

The production or manufacture of Ordinary Portland cement (OPC) is an energy intensive process which releases a large amount of carbon dioxide into the atmosphere. Fly ash based geopolymer provide an alternative to this harmful production process as it

uses fly ash, which is a by-product of the coal driven power stations. Past researchers have indicated that this new “cement” has superior qualities to that of OPC.

Efflorescence has been seen to be a big issue in OPC from an aesthetic point view. This has also been seen to be a problem with geopolymers as The Na bond in the aluminosilicate polymer structure is weak and because of this weak structure aluminosilicate polymer material forms efflorescence in a humid environment (ŠKVÁRA, et al., 2009).

By comparing the results from the various tests done to the different mix design at different conditions a better understanding of the effect of efflorescence should be obtained.

1.5 Limitations

This major limitation faced in this research project was time. At the initial stage of the project the flexural strength was included as one of the objectives. However this was being left out due to time constraints. Other academic commitments, since this research project was done simultaneously with my final year units to complete my BENG (Civil) it contributed to the limitation in time.

1.6 Risk Assessment and Safety

Safety is a major issue while conducting experiments with corrosive solutions such as alkali activators. Before the initial or trial experiments were conducted a risk assessment of the potential hazards was done. Also a safety induction process on how to safely operate machines was conducted. All University of Southern Queensland safety protocols were followed before the commencement of any activity in the laboratory. The risk assessment is summarised in the following page. These are divided into the separate steps being taken:

Element or Sub Element/Process step	The Risk: What can happen and what will be the result	Existing Controls
Alkaline Solution Preparation	High pH solution]may cause chemical burn on skin if it spills out of the container or may spill on the concrete floor, not dangerous to the environment but may cause slipping	(1) wear long sleeve overalls to protect arm and skin,safety boots to protect feet, wear glasses to protect eyes, wear chemical resistant gloves to protect hands(2) All the PPE are prepared by the centre, and gloves and glasses are put into P2, in file cabinet third drawer (3) If alkaline spills on skin use dry cloth to clean and use running water to wash; the dry cloth and water are at P2, beside and front door (opposite to P3) (4) Alkaline solution are all stored in the chemical cabinets before and after using; once it spills on the floor(rare), use dry cloth and the chemical leakage bucket to clean it.
Mixing alkaline solution with solution with fly ash	(1) Electric shock when turn on the mixing (2) The fine powder may fly of the mixing bowl and people may breath in ; (3) fingers can touch the turning paddle	(1) wear safety boots (2) wear mask during weighting and mixing (3) Cover the mixing bowl with a paper to stop the ash particles flying out and to protect fingers.
Casting and curing of geopolymers in oven	(1) The pH slurry may spill on hand and burn skin during casting. (2) Electric shock when turning on the oven(P2) (3) high temperature (40° - 80°C) in the oven may hurt hand	(1) wear safety boots (2) wear gloves to protect hand skin (3) open the door of oven for a while after it cools down, then remove the samples
Strength testing of hardened samples	(1) Electric shock when turn on the universal testingmachine (in P9) and hydraulic testing machine (in P11) (2) The cracked chips may fly out and hurt eyes.	(1) wear safety boots (2) wear safety glasses (3) get professional testing training from technical staff

Table 1: Risk Assessment table

1.7 Dissertation Outline

The structure of the dissertation is being summarised below from this section forward:

- Chapter 2 : Literature Review
In this section the relevant literature of the research are being discussed. These include; description of geopolymer, constituents of geopolymer, alkali activators, curing, mechanical properties and application.
- Chapter 3: Methodology
In this section the process of preparing, casting and testing of the geopolymer paste is described.
- Chapter 4: Results and Discussion
In this section the results from the various tests are being presented and discussed. The effect of efflorescence is being discussed in this section when compared to other conditions.
- Chapter 5: Conclusion and Future Work
This part of the dissertation discusses the conclusions made from the results presented and provides recommendations for which work.

2.0 Literature Review

2.1 What is geopolymer?

The French scientist Joseph Davidovits first proposed the term “geopolymer” in 1978 (Davidovits, 1994). Davidovits developed an amorphous to semi crystalline three dimensional silico – aluminate which he gave the term geo-polymer i.e. mineral polymers resulting from geo-chemistry or geo-synthesis. The developed of geopolymer by Davidovits came after catastrophic fires in France between 1970 – 1973 which involved common plastics hence began his research on non-flammable and non-combustible plastic material.

Geo-polymer cement is created by the mixing aluminosilicate – reactive material with strong alkali solutions such as sodium hydroxide, potassium hydroxide and sodium silicate or potassium silicate. Somna, et al., (2011) .This is called alkali activated geopolymer cements. The alkali solution plays an important role in the process in that it activates the aluminosilicate raw materials in the geopolymerization reactions and to charge balance the growing aluminosilicate gel framework. This charge balance is the requirement for electrical stability in silicate and other ionic compounds means that the sum of positive and negative charges on the ions must balance. This activation creates a sufficiently high pH which activates the aluminosilicate raw materials. The process of geopolymerisation is usually divided into 3 main stages:

1. **Dissolution of raw materials in alkali solution.**

In the dissolution of aluminosilicate in strong alkali solution the aluminosilicate reactive materials dissolve and form free and tetrahedral units

2. **Reorientation of the dissolved species.**

Water is gradually removed and the tetrahedral clusters are joined to yield polymeric precursors through the sharing of all oxygen atoms between two tetrahedral units, resulting in the formation of amorphous geopolymers.

3. **Polycondensation of networked structures.**

Polycondensation process is actually the setting reaction which leads onto the formation of amorphous to semi-crystalline aluminosilicate as mentioned

earlier. The schematic diagram of the formation of the geopolymer material is shown by the two reactions (Xu and Van Deventer 2000):

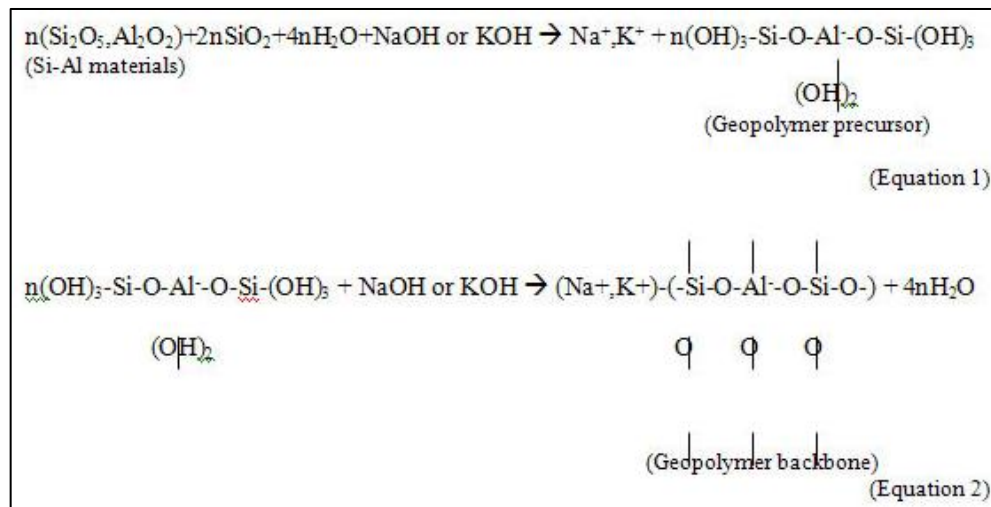


Figure 2: Schematic diagram of the formation of the geopolymer

2.2 Main Issues Concerning Concrete

Concrete is the most widely used material used in construction, with the rate of development in developing countries the demand of this material is growing at an exponential rate. OPC is still the most common binder ingredient in the production of concrete with a current rate of 1m^3 per person per year. The worlds yearly cement production of 1.6 billion tons accounts for about 7% of the global loading of carbon dioxide into the atmosphere (Mehta, 2001). OPC however has a high embodied energy, with carbon dioxide equivalent ($\text{CO}_2\text{-e}$). This is the measure used to compare the emissions from various greenhouse gases based upon their global warming potential, ranging from 0.66 to 0.88kg of CO_2 emitted for every kilogram manufactured. (Peng J, 2013) (Li C, 2011) (Huntzinger & Eatmon, 2009).

According to The Intergovernmental panel on Climate Change's (IPCC) there are four scenarios to project future cement demand. (Humphreys & Mahasanen, 2002). These 4 scenarios are:

1. A1). The income gap between developed and developing countries closes: there is rapid economic growth and low population growth; new and more efficient technologies are rapidly introduced; and the world has high per-capita

energy use.

2. B1). The income gap between developed and developing countries decreases but does not close; material intensities decline; the world experiences low population growth and a shift toward a service and information economy; and the world has a low per-capita energy use.
3. A2). The income gap between developed and developing countries does not come close to closing; level of economic growth varies among countries and the world experiences high population growth.
4. B2). The income gap between developed and developing countries does not close; the world experiences intermediate levels of economic growth and moderate population growth.

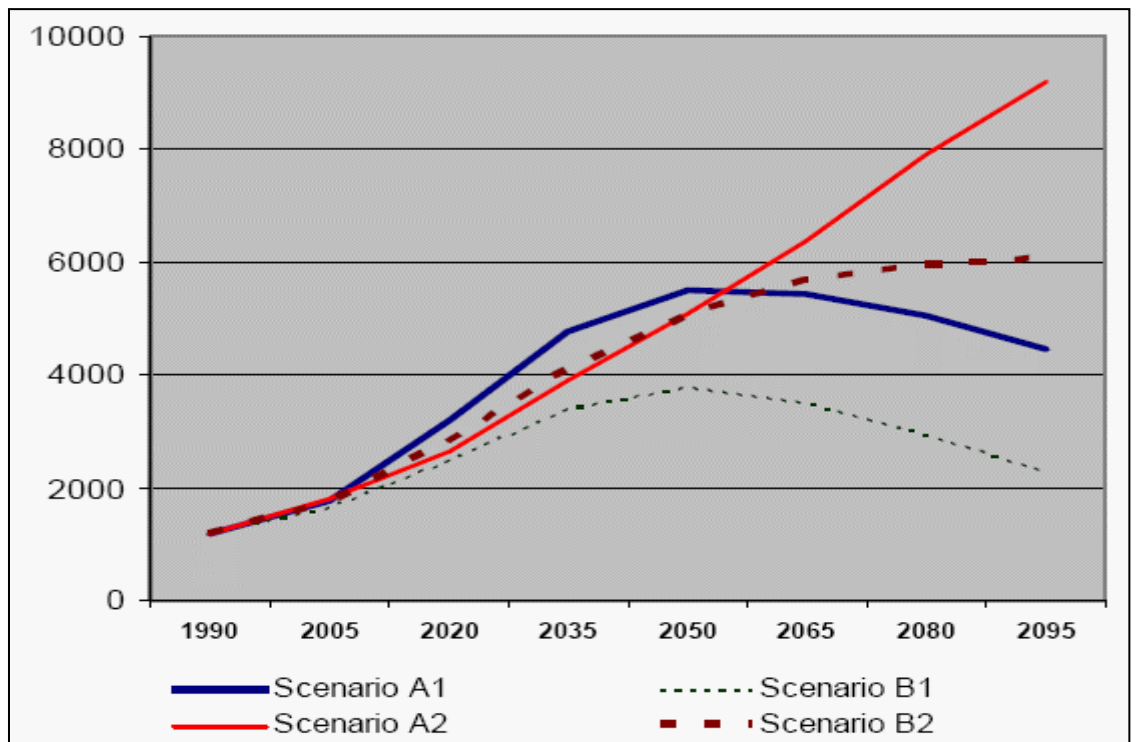


Figure 3: Cement Demand Projection

2.3 Constituents of Geopolymer

2.3.1 Source Materials

The main source materials for the manufacture of geopolymer are materials that contain mostly Silicon (Si) and Aluminium (Al) in the amorphous state which theoretically can be alkali activated. To add to this some mineral and industrial by-product material has been investigated into possibly being used for the manufacture of geopolymer. The following prime materials for geopolymer manufacture have been investigated (Torgal & Jalali, n.d.):

1. Kaolinite clays (Davidovits & Sawyer, 1994; Rahier, et al., 1996; Rahier, et al., 1997; (Barbosaa,etal.,2000)
2. Metakaolin (Davidovits, 1999; Barbosaa, et al., 2000)
3. Fly ashes (Palomoa, et al., 1999; Fernández-Jiménez & Palomo, 2005)
4. Blast furnace slag (Purdon, 1940; Wang, 1995; Fernández-Jiménez, 1999; Adolf, 2007)
5. Mixtures of fly ashes and slag (Puertas, 2003)
6. Mixture of fly ashes and metakaolin (Swanepoel, 2002)
7. Mixture of red mud and metakaolin (Cheng, 2003)
8. Mixture of slag and red mud (Pan, 2002)
9. Mixture of fly ashes and non – calcined materials (kaolin and stilbite) (Xu, 2002)

2.3.1.1 Metakaolin

Metakaolin or calcined kaolin is according to the xxxxx is refined clay that is fired (calcined) under carefully controlled conditions to produce an amorphous aluminosilicate that is reactive in concrete. As with other pozzolans fly ash and silica fume this material reacts with calcium hydroxide by-products during cement hydration. Metakaolin is obtained by the calcination of kaolinite. This material is widely used as pozzolanic material in mortar and concrete. According to (Siddique & Klaus, 2009) this material (metakaolin) exhibited considerable influence in enhancing the mechanical and durability properties of mortar and concrete. The use of by-products like fly ash, granulated blast furnace slag, silica fume, rice husk ash, and metakaolin in cement and concrete has gained significant importance because of the requirements of environmental protection and sustainable construction in the future. The conclusion that (Siddique & Klaus, 2009) made is that the metakaolin:

1. The use metakaolin enhances the early age mechanical properties as well as long-term strength properties of cement paste/mortar/concrete.
2. The water penetration by capillary action is reduced by partially replacing cement with metakaolin.
3. Metakaolin reduces the permeability resulting in the transportation of water and diffusion of harmful ions which lead to the deterioration of the matrix; this is because the addition of the metakaolin modifies the pore structure of the cement, mortar and concrete.
4. The addition of metakaolin as a replacement to cement improves the sulphate attack resistance of the concrete. As the replacement level of the metakaolin increases so does the resistance level to sulphate attack, 10% -15% replacement of cement with metakaolin showed the best durability to sulphate attack. Metakaolin has a high rate of dissolution in the reactant solution and this is why it is preferred by the niche product developers (Gourley, 2003) and also is very expensive.

2.3.1.2 Blast Furnace Slag

Blast furnace slag is a by-product in the production of molten iron product which can be cast into iron products. Production of iron involves charging iron ore, iron scrap, and fluxes (limestone and/or dolomite) with coke, the coke is combusted which reduces iron ore to a molten iron product. It is this product that is used to manufacture iron products. The blast furnace slag is the non-metallic by product in the process and this material is primarily consists of silicates, aluminosilicate, and calcium-alumina-silicates. (Administration, 2012)

In 1940 (Purdon, 1940) reported on the rapid hardening binder by the slag activation. In the 1950s the United States army used NaCl and NaOH to activate slag to produce binder for use in Military applications (Malone, et al., 1986) . Also during the 1950s (Gluchovsky, 1959) subsequently developed alkali activated systems, this contained calcium silicate hydrate and aluminosilicate phases. Later literature showed that products of fly ash/slag pastes activated NaOH (10M) and Sodium silicate and found that the compressive strength of specimen with 50% fly ash/50% slag and cured at 25°C was more than 50 MPa at the age of 28 days (Fernández-Jiménez, 1999)

2.3.1.3 Mixtures of fly ashes and slag

The cements obtained from a mixture of ground blast furnace slag and aluminosilicate fly ash have not been studied consistently. (Puertas & Fernández-Jiménez, 2003) The study carried out by Puertas, et al., 2000 on the strength behaviour and hydration products of fly ash/slag pastes activated with NaOH. It was observed that the mixture 50% fly ash/50% slag and activated with 10 M NaOH solution and cured at 25 °C develops a compressive strength of 50Mpa. The ratio of 50% fly ash/50% slag is the most relevant factor on the strength development. Hydrated calcium silicate, like CSH gel, with high amount of tetraco-ordinated Al in its structure is the main product from the reaction.

The investigation conducted by Smith & Osborne, (1977) investigated cements made from the combination of 60% finely ground blast furnace slag and 40% fly ash activated by 7% sodium hydroxide. From their research they found that early strength properties were good but there was little gain in strength beyond 28 days though improved strength might be obtained by varying the proportions of slag and fly ash or by increasing the fineness of the slag. It was postulated by Puertas, et al., (2000) that the main reaction product in these pastes is a hydrated calcium silicate, like CSH gel, with high amounts of tetracoordinated Al in its structure, as well as Na ions in the interlayer spaces. There was no hydrated alkaline alumino-silicates with three-dimensional structure characteristics of the alkaline activation of fly ashes were formed.

2.3.1.4 Mixture of slag and red mud

In the research done by Pan, et al., (2002) for Alkali slag red mud cement it was found out that hydration products of ASRC cement are mostly C-S-H gel with very low Ca/Si ratio of 0.8–1.2, very small particle size of hundreds of nanometers and very irregular shape in morphology. According to this study neither crystallized calcium hydroxide nor ettringite, which are normally found in hardened Portland cement paste system, nor zeolite-like phase were found in hardened ASRC cement paste system. (Pan, et al., 2002) concluding that the hydration products of ASRC cement remain similar, regardless of its kind of activators, simple or composite, and curing condition, ambient temperature curing or steam curing.

Pan, et al., (2003) recorded the Properties and microstructure of the hardened alkali-activated red mud–slag cementitious material and found that cement (ASRC) has high

early and ultimate strength and a good strength development, without any trend of strength rebounding with curing ages. Also that ASRC cement has good resistance against various chemical attacks and freeze and thaw cycles.

Also in the study done by Pan, et al., (2003) found that:

- Hardened ASRC cement paste has lower total porosity, less portion of larger pore and more portion of smaller pore, as compared with those of hardened Portland cement paste.
- The hardened ASRC cement paste has more compacted and continued more integrated microstructure than hardened Portland cement paste.
- The denser microstructure and the better pore structure are the physical reasons contributing to the good strength performance and resistance against chemical attacks.

2.2 Mixture Proportions

The majority of the works carried out on geopolymer material currently refer to the properties of the geopolymer paste and this is measured using small size samples. For this research project P.V.C pipes of approximately 2.5cm in diameter and 2.8cm in height are used. The study carried out by Palomo, et al., (1999) on the geopolymerisation of ASTM class F fly ash used four different alkali activator solutions which had a solution- to – fly ash ratio by mass of 0.25 to 0.30. The size of the specimens used was 10x10x60 mm. in this study Palomo, et al., (1999) obtained the top compressive strength of 60MPa after 24 hrs curing at 65°C using a combination of sodium hydroxide and sodium silicate alkali activator solution. According to Xu & Van Deventer, (2000) the optimum proportion of alkaline solution to alumina silicate powder by mass should be around 0.33. This is to allow for geopolymeric reactions to occur. As reported by Xu & Van Deventer, (2000) a thick gel formed as soon as alumino-silicate powder was added to the alkaline solutions. Specimen size used by these two researchers was 20mm x20mm x20 mm and this achieved a maximum compressive strength of 19 MPa after 72 hrs curing at 35°C with stilbite as source material. In comparison to Xu & Van Deventer, (2000) van Jaarsveld et al (1998) used 0.39 as mass ratio of the solution to the powder and achieved a maximum compressive strength of 75MPa using fly ash and builders' waste as the source material. This design was a combination of 57% fly ash was mixed with 15% kaolin or calcined kaolin. This was

then activated by a mixture of 3.5% sodium silicate, 20% water and 4% sodium or potassium hydroxide.

Barbosa, et al., 2000 found that the optimum composition occurred when the ratio of $\text{Na}_2\text{O}/\text{SiO}_2$ was 0.25, the ratio of $\text{H}_2\text{O}/\text{Na}_2\text{O}$ was 10.0, and the ratio of $\text{SiO}_2/\text{Al}_2\text{O}_3$ was 3.3. This was after preparing and testing seven mixture compositions of geopolymers paste in the following range of molar oxide ratios: $0.2 < \text{Na}_2\text{O}/\text{SiO}_2 < 0.48$; $3.3 < \text{SiO}_2/\text{Al}_2\text{O}_3 < 4.5$ and $10 < \text{H}_2\text{O}/\text{Na}_2\text{O} < 25$. It was noticed by Barbosa, et al., 2000 that mixture with high water content, i.e. $\text{H}_2\text{O}/\text{Na}_2\text{O} = 25$, developed very low compressive strengths, and thus underlying the importance of water content in polymerisation.

2.3 Fly ash

Fly ash is a by-product from the combustion of pulverized coal in furnaces of thermal power plant; it largely consists of inorganic content. It is collected by electrostatic precipitators when it solidifies while being suspended in the exhaust gases. This by-product from the coal power station is currently being used as a component in geopolymers, where the reactivity of the fly ash glasses is used to generate a binder that is comparable to hydrated OPC in appearance and properties but with possibly reduced CO_2 emissions. The particles of fly ash are usually spherical in shape and maybe finer or coarser than cement. According to the Concrete Institute of Australia Fly ash used in geopolymer production can be divided into two categories:

- Class F: when the sum of SiO_2 , Al_2O_3 and Fe_2 is not less than 70%
- Class C: when the sum of SiO_2 , Al_2O_3 and Fe_2 is not less than 50%

The characteristics of fly ash depend mainly upon its collection process used. The current systems in use are mechanical collectors, fabric filters, wet scrubbers and electrostatic precipitators. From these systems mechanical collectors and electrostatic precipitators are most widely used. The efficiency of collection depends on the particle size for each type of collector system. (Shao, 2002)

The table below shows the Typical Composition of Fly Ash and Type A Portland Cement %

Compound	Fly Ash			Cement Type A
	NSW	QLD	SA	
SiO ₂	58 - 63	53 - 66	54	22
Al ₂ O ₃	24 - 29	24 - 29	30	4.5
Fe ₂ O ₃	3 - 5	1 - 9	4.5	3.5
CaO	0.5 - 2.5	0.2 - 5	55	64
MgO	0.5 - 1.5	0.2 - 1.5	1.8	1.4
Na ₂ O	0.1 - 1.0	0.1 - 0.4	3	0.2
K ₂ O	0.9 - 2.0	0.2 - 1.4	0.9	0.5
SO ₃	0.2 - 0.4	0 - 0.4	0.3	2.4
Loss of ignition	1.0 - 3.0	1.0 - 3.0	0.3	1.1

Table 2: Composition of fly ash Source: Concrete Institute of Australia sited at 4:20pm 7/9/13

The majority of fly ash produced by Australian power plants is of low calcium (Class F) which contains calcium content of less than 5% by mass and is light to mid grey in colour. In order for this type of fly ash to be used as binder in geopolymer concrete it requires to have a silicon and aluminium content of about 80%, with a Si-Al ratio of 2 – 1 (Rangan, 2008).

The use of fly ash to replace the conventional OPC has advantages and also disadvantages. The research done highlighted by (Shao, 2002) showed that fly ash provided better workability, higher long term strength and improved durability of the concrete. The disadvantages found were that fly ash based concrete accompanied increased setting time and decreased early strength. Different techniques to overcome come these setbacks is proposed by (Shao, 2002) which includes grinding, elevated temperature curing and the use of chemical activators in order to overcome these disadvantages.

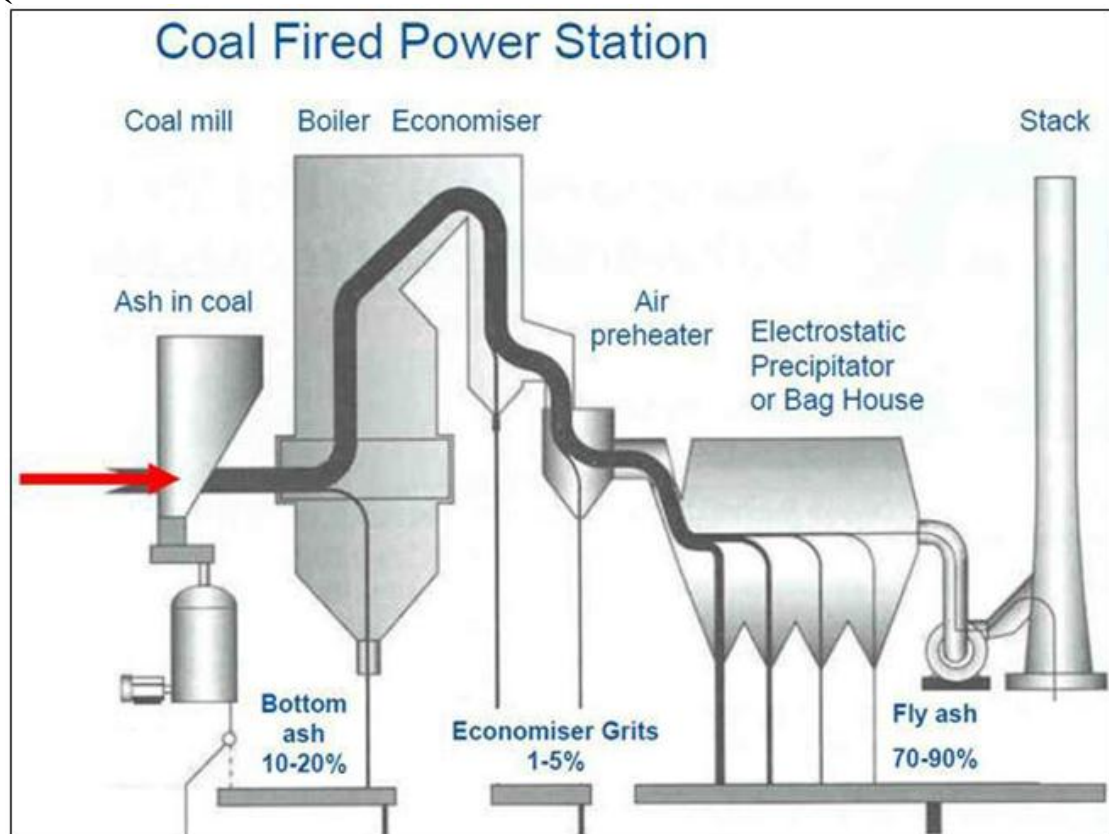


Figure 4: Fly ash from coal power plant.

Source: *Fly Ash Australia 2010*

2.4 Efflorescence

Efflorescence is the formation of salt deposits on or near the surface of concrete causing a change in colour or discolouration. According to the concrete.net.au efflorescence is generally harmless and is considered to be a skin trouble and not deep disease. (Anon., 2009)

Efflorescence in Portland cement is caused when water in the concrete mixes to produce alkaline products. Efflorescence mainly occurs on vertical surfaces or inclined surfaces. The maximum thickness of this water membrane is limited by capillarity or surface roughness or attitude, which means that the water film is <1mm. According to (Dowa, 2003) the formation of efflorescence is controlled by processes that are if it is assumed that water has already moistened the surface of the structure the cement substances begin to dissolve at the water – solid interface. These dissolving species are divided into two types:

- Those with essentially unlimited solubility, relative to the potential of the source to supply and saturate the film
- Those with limited solubility that is they may react with the formation of a sparingly soluble salt.

The examples of such elements in the first group are sodium and potassium and the example of the second group is calcium, aluminium and silicon. The 3rd and 4th processes are coupled, will describe relevant solubilities. In these two processes the chemical matters supplied from the cement dissolves and is free to diffuse throughout the thin solution layer. The 5th process assumes that on account of the thinness of the water layer, the two interfaces are at a distance of ranging from a few microns up to a few hundred microns. With this distance between the two layers diffusion occurs principally across the thinnest dimension. The solvency of water for CO₂ is affected only once it is chemically modified by the dissolution of the cement components. This results in a new dissolution equilibrium being established. Initially CO₂ dissolves as molecular CO₂ but this depends on the pH and composition of the solution. The speciations of the CO₂ will adjust to the new equilibrium CO₂(aqueous), H₂CO₃ and CO₃⁻² species. At last the dissolved CO₃ reacts with the Ca species from the cement thus precipitating CaCO₃ once its solubility limit is attained. This reaction is shown in the 6th process including the solubility of CaCO₃

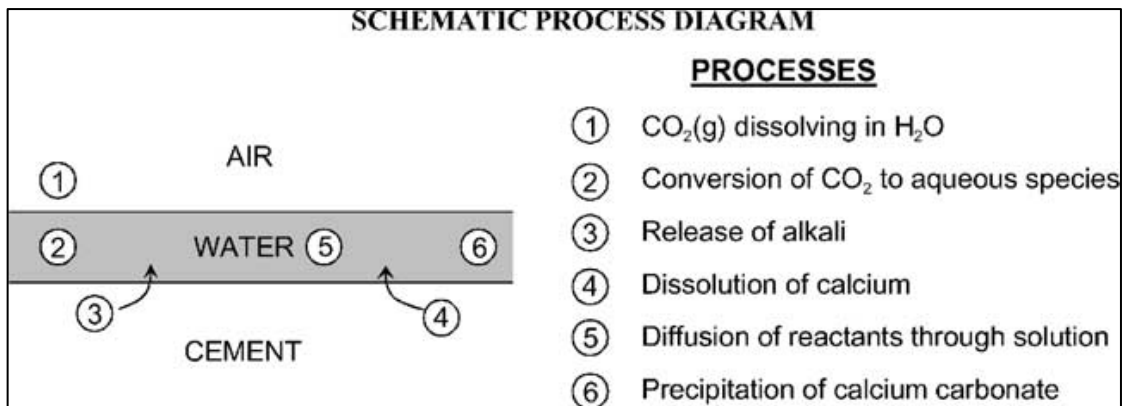


Figure 5: Schematic diagram for the formation of efflorescence. (Source: (Dow & F.P, 2003))

As with OPC efflorescence also occurs in geopolymers according to (Ebrahim, et al., 2011) the tendency towards efflorescence is because of some materials having a very open microstructure which have a lower extent of reaction. This is partly due to the high alkali concentration in the pore solution. Also the weak binding of Na in the geopolymer structure can contribute to this problem.

2.4.1 Control of Efflorescence in Geopolymers

Studies have been conducted into the possibility of controlling or eliminating the efflorescence effect on geopolymers. Attempts have been made to reduce efflorescence by adding potassium hydroxide instead of sodium hydroxide in the activator (Szklorzova & Bilek, 2008). This is done because potassium is more strongly bound to the aminosilicate gel framework, and also potassium carbonate is less visually evident than their sodium counterparts. In 2011 Ebrahim, et al., conducted a study into controls for efflorescence in geopolymer. It was found that hydrothermal curing at elevated temperatures showed a positive effect on the reduction of efflorescence. Adding to this calcium aluminate cements showed the greatest effect in the reduction of efflorescence. This is attributed to the dissolution of calcium aluminate in alkaline media releasing high amounts of alumina into the aluminosilicate geopolymer.

It is known that alkali-activated binders are prone to the formation of efflorescence's. According to Pacheco-Torgal, et al., 2012 this advantage can be greatly minimised when using hydrothermal curing treatments or calcium aluminate admixtures. It should be noted that this type of curing has limited applications for on situ concrete placement operations and the use of a calcium based admixture raises issues about its acid resistance. Moreover, alkali-activated binders that contain calcium based admixture have a greater global warming impact than alkali-activated Si–Al mixtures.

2.4.2 Efflorescence Control in OPC Concrete

According to Dow & Glasser, 2003 the best practical advice to mitigate calcium carbonate efflorescence is to:

- Use low-alkali cements, if this is not available then sodium is preferred to potassium. It is best that alkali contents are compared on a mole basis rather than weight percentage which can be misleading.
- Impermeable mortars should be used so that the availability of alkali is reduced and there is a decrease of alkali and decrease the effective thickness per unit time of the alkali-depleted layer.
- The exterior faces should be washed after the final set of the cement in order to dilute and flush away alkalis. Acidic washers should be avoided as this will only enhance the permeability of the cement surface and may subsequently worsen the problem.

2.5 Alkali Activators

Sodium silicate and potassium hydroxide are the most widely used alkaline solution in the production of fly ash based geopolymer. These two materials are usually mixed with sodium hydroxide to produce the alkaline solution, with the molarity (M) of alkaline solution is from 7 to 10 M. (Mohd Mustafa Al Bakri, 2011). Alkaline solution is prepared a day before being mixed with the fly ash. After the alkaline solution is prepared (1 day after) it is then mixed together with the other material (fine aggregate and coarse material) to make the concrete after which curing takes place. Chindaprasirt et al. (2007) established that to produce higher strength geopolymer, the optimum range of the sodium silicate to sodium hydroxide should be a ratio ranging between 0.67 to 1.00.

2.6 Alkali Concentration

In the research conducted by Palomo, et al., 1999 it was concluded that the type of activator plays an important role in the polymerisation process. Alkaline activator that contains soluble silicate, either sodium or potassium silicate had a high rate of reaction when compared to the use of only alkaline hydroxides

It was also noted by (Chindaprasirta, 2007) that the concentration of sodium hydroxide (NaOH) between 10 and 20M had minimal effect on the strength of concrete. According to (Kiatsuda Somna, 2011) an increase in the concentration of NaOH from 4.5M to 14.0M increases the strength of ground fly ash (GFA) geopolymer pastes. Microstructure studies done on NaOH showed that concentrations of 12M to 14M created new crystalline products of sodium silicate (Somna, et al., 2011). The compressive strength at 28 days of 20 to 23 MPa were obtained with the concentrations of 9.5M to 14.0M. It was also found that increasing the NaOH concentration beyond this point resulted in the decrease in strength of the paste due to the early precipitation of aluminosilicate products.

It was confirmed by Xu & Van Deventer, 2000 that the addition of sodium silicate solution to sodium hydroxide alkaline activator improved the reaction between the fly ash (source material) and the alkaline solution. Xu & Van Deventer, 2000 concluded the NaOH solution caused a higher dissolution of minerals than the KOH solution.

2.6 Curing

Curing is the process of controlling the rate and extent of moisture loss from concrete during hydration. The curing process maybe done after the concrete is placed in its final position. This provides time for the hydration of cement to occur. In order to for concrete to obtain its potential strength and durability curing may take days or even weeks since it takes time for cement to hydrate. (Anon., 2006).

2.6.1 Curing At Ambient Temperature

Geopolymer concrete is widely known for achieving high compressive strength when cured at high temperature. Hence it is very expensive to cast geopolymer on site. This provides a need to create a geopolymer concrete that can be cured at ambient temperature at the same time having a relatively high compressive strength. Research into this subject has yielded a number of methods. (Temuujin, et al., 2009) concluded that the mechanical activation of fly ash in a vibration mill lead to an 80% increase in the compressive strength of the geopolymer paste when compared with geopolymer made from raw fly ash, when cured at ambient temperature. According to this finding the reduction of particle size and change in the particle shape contributed to the increase in the compressive strength since this change allowed a greater dissolution rate of the fly ash. NaOH – activated ground fly ash geopolymer cured at ambient temperature gave a higher strength geopolymer paste (Kiatsuda Somna, 2011). Concentration of NaOH of 4.5 – 16.5 M was used with ground fly ash of a median size of 10.5 μ m.

2.6.2 Curing At Elevated Temperature

In (Mohd Mustafa Al Bakri, 2011) it highlights that the setting time of geopolymer depend on many factors like composition of alkaline liquid to fly ash by mass. It is also noted that the curing temperature is the most important factor in the in the geopolymer process. According to (Chanh et al.2008). When the curing is taking place the geopolymer concrete experiences a polymerization process. With the increasing temperature, polymerization become more rapid and the concrete can gain 70% of its strength within 3-4 hrs of curing (Kong and Sanjayan, 2008). Geopolymer cured at ambient temperature show similar strengths to traditional OPC concrete (Kiatsuda Somna, 2011). In order to increase the strength of geopolymer cured at ambient

temperature there should be an increase in NaOH concentration from 4.5 to 14.0 M increased the strength of GFA geopolymer pastes. (Van Jaarsveld, 2002) came to the conclusion that curing and water content along with the calcining condition of kaolin clay affected the properties of geopolymers. It was also stated that the curing at too high temperature caused cracking and a negative effect on the properties of the material and so they finally recommended the use of mild curing to improve the physical properties of the material.

2.7 Mechanical Properties of Geopolymers

2.7.1 Creep

Creep is defined as the tendency of a solid material to move slowly or deform under the influence of sustained load. According to (Zhu Pan, 2011) Transient Thermal creep' (TTc), plays a major role in providing fire resistance in Ordinary Portland Cement (OPC) concrete. In its investigation of this effect on Class F fly ash with sodium silicate and sodium hydroxide as activators. It was found that at temperature below 250°C, geopolymer showed significantly higher level of creep (TTc) when compared to the OPC paste. From the range of 250 °C - 550 °C, the geopolymer did not show any pronounced increase in transient creep. However the OPC paste developed significant transient creep. (Zhu Pan, 2011)

2.7.2 Workability

Workability is the ease of which concrete can be poured or discharged onto a mould or formwork. Geopolymer concrete also has this attribute. In concrete the workability is affected by factors such as water content, cement content, aggregate particle shape and size (Anon., 2006). In (M. Fareed Ahmed, 2011) study of the workability of Low-Calcium Fly ash-based self-compacting concrete it was concluded that just like OPC the addition of extra water improved the workability of the concrete mix, however the inclusion of water beyond a certain limit resulted in the bleeding and segregation of fresh concrete and decreased the compressive strength of the concrete. Also the compressive strength of self-compacting geopolymer concrete was significantly decreased as the amount of additional water exceeded 12% by mass of fly ash. (M. Fareed Ahmed, 2011) Workability for High – Calcium Fly Ash based concrete depended on the concentration of NaOH and the amount sodium silicate. With the increase in the concentration of the NOH and the amount of sodium

silicate this decreases the workability of the mixes. The increase in NaOH concentration increases the viscosity of the solution and thus reduces the flow of the mortar. The sodium silicate solution is itself a solution of very high viscosity and hence an increase in the amount also reduces the flow of the mortar. For a suitable flow, extra water is needed. (Chindaprasirta, 2007). The addition of high range water reducing admixture, up to approximately 2% of fly ash by mass, improved workability of the fresh geopolymer concrete (D Hardjito, 2004).

2.7.3 Compressive Strength

Compressive strength is one of the most important properties of concrete OPC and geopolymer based concrete. It is the measure of its ability to resist loads which tend to crush it. (Anon., 2006) . Compressive strength in Alkali Activated fly ash geopolymer is affected by a number of factors. A study conducted by (D Hardjito, 2004) on the factors which influence the compressive strength of Fly Ash based geopolymer concrete of which the binder which is the geopolymer paste is class F fly ash which is activated by a combination of sodium silicate solution and sodium hydroxide solution. Class F fly ash was used since it was rich in silicon and aluminium. From this (D Hardjito, 2004) concluded that the compressive strength of Fly Ash based geopolymer concrete was affected by the following factors:

1. Higher concentration of sodium hydroxide solution corresponds to the increase in the compressive strength of the geopolymer concrete.
2. The higher the ratio of silicate-to-sodium hydroxide liquid ratio by mass results in the higher compressive strength of geopolymer concrete.
3. As the curing temperature in the range of 30°C to 90°C increases so does the compressive strength of the geopolymer concrete.
4. The longer the curing time the higher the compressive strength. This is from a range of 6hrs to 96hrs

2.7.4 Durability

Durability is a very important aspect of a material having the ability to withstand harsh and severe environmental conditions, natural or those due to human activity. This durability includes properties such as freeze – thaw resistance, sulphate resistance. Ghosh & Nasser, 1994 showed that high strength concrete containing lignite fly ash and silica fume had a generally good freeze-thaw resistance. The inclusion of a higher fly ash content contributed to the low freeze-thaw durability. This was based on microstructure study showing that ettringite formed pores after freeze thaw cycle.

2.8 Leaching

Leaching is described as the process of extracting minerals from a solid by dissolving them in a liquid, either in nature or through an industrial process. Various studies have been done on the leaching effect of fly ash based geopolymers. Maria Izquierdo, (2009) sought to determine the role that curing in uncovered conditions may play on the environmental performance of geopolymer-like structures. Their findings concluded that leachates were highly alkaline, with pH values practically unchanged whatever the curing conditions used. Also found is that differences in leaching cannot be attributed to pH variations. This study brought light the impact of open air cured geopolymers with those cured in protected environment on the environmental performance of the final product in comparison of leachable concentrations. Maria Izquierdo, (2009) and L. Ly, (2007) confirmed that the most leachable species in water solutions derived from correctly made geopolymers exposed to deionised water are alkalis. Findings from this study also concluded that the leaching of all elements generally increased with time; however with less durable sample it was difficult to explain the kinetics since it had a tendency to disintegrate on exposure to water.

3.0 Methodology

The method used in preparing the geopolymer material follows the general geopolymer preparation techniques and testing. This section is divided into three major sections:

- Materials
- Sample Mix Preparation
- Sample Testing

3.1 Materials

All the materials and equipment used in the preparation of the samples was provided by the Centre of Excellence Engineered Fibre Composites (CEEFC) at the University of Southern Queensland.

3.1.1 Fly Ash

The fly ash used in the preparation of geopolymer binder was obtained from three power stations in the state of Queensland. The fly ash used is of Class F grade this is when the sum of SiO₂, Al₂O₃ and Fe₂ is not less than 70%. Each of the three fly ash batch was checked for large particles before being used.



Millmerran



Callide



Gladstone

Figure 6: Various types of fly ash used

3.1.3 Sodium Silicate

The sodium silicate (class D) supplied by the CEEFC was obtained from P Q Australia Ltd. This class D sodium silicate has the following chemical composition:

- Weight ratio $\text{SiO}_2/\text{Na}_2\text{O}$ – 3.22
- NaO_2 % - 14.7%
- SiO_2 % - 29.4%
- Density (g/cm^3) – 1.38
- pH – 12.8
- Viscosity - 400

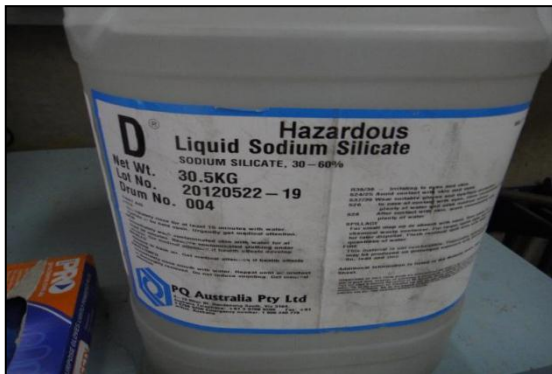


Figure 7: Sodium Silicate solution

3.1.4 Sodium Hydroxide

The sodium hydroxide is an alkali activator that is being used for the activation of the various fly ashes and for the different geopolymer mix designs. This alkali activator was provided by the CEEFC.



Figure 8: Sodium Hydroxide

3.1.5 Magnesium Oxide



Figure 9: Magnesium Oxide

3.1.6 Glass Fibre



Figure 10: Glass Fibre

3.1.7 Potassium Aluminium Sulphate



Figure 11: Potassium Aluminium Sulphate

3.1.7 Fine Sand



Figure 12: Fine Sand

3.1 Preparation of Alkaline Solution

Alkaline solution is prepared by mixing the sodium hydroxide together with the sodium silicate solution.

- Sodium Silicate, $M_s = 1.5$ ($\text{SiO}_2/\text{Na}_2\text{O} = 1.5$)

Since the original solution from the manufacturer (P Q Australia Ltd) had an $M_s = 2$ $\text{SiO}_2/\text{Na}_2\text{O} = 2$ the following steps were taken to obtain an $M_s = 1.5$

1. The original sodium silicate solution contains:

$$\text{Na}_2\text{O} = 14.7 \text{ g} = 0.2371 \text{ mol}$$

$$\text{SiO}_2 = 29.4 \text{ g} = 0.49 \text{ mol}$$

2. To obtain a ratio of 1.5, additional Na_2O needs to be added to the solution.

Calculating the additional Na_2O to be added it was found that 5.55g of Na_2O needs to be added to obtain the ratio of 1.5. This is done by adding NaOH which contains Na_2O . 1g of NaOH contains approximately 0.775g of Na_2O

So $5.55\text{g}/0.775\text{g} = 7.16\text{g}$ of NaOH. This is the amount of NaOH that is to be added to the sodium silicate solution, so that the $\text{SiO}_2/\text{Na}_2\text{O} \approx 1.5$.

$$\text{The total solid} = 14.7\text{g} + 29.4\text{g} + 5.55\text{g} = 49.5\text{g}$$

$$\text{The total solution} = 100\text{g} + 7.16\text{g} = 107.16\text{g}$$

3. To make a solid content of 35% additional water (H_2O) needs to be added

Therefore x is equal to 34.7g of H_2O

The solution was mixed and left for 24 hrs before being used.

3.2 Sample Mix Preparation

In this research various different mix composition of geopolymer paste was used. This is tabulated below:

Number	Mix	Fly Ash		Alkali Activator		Water	Additive				Added Water (g)
		Source	Mass (g)	Type	Mass (g)		Type	Mass	Type	Mass	
1	GS1	Gladstone	500	Sodium Silicate	180		na	na	na	na	na
2	CL1	Callide	600	Sodium Silicate	180	36	na	na	na	na	na
3	CL2	Callide	600	Sodium Silicate	216	24	na	na	na	na	na
4	GS2	Gladstone	600	Sodium Hydroxide	158		na	na	na	na	na
5	MM1	Millmerran	600	Sodium Silicate	162	74	na	na	na	na	na
6	MM2	Millmerran	600	Sodium Hydroxide	158	60	na	na	na	na	na
7	MM3	Millmerran	600	Sodium Silicate	116.064	64	na	na	na	na	na
8	MO-0	Millmerran	600	Sodium Silicate	162	74	Glass Fibre	3	MgO	0	42
9	MO-2	Millmerran	600	Sodium Silicate	162	74	Glass Fibre	3	MgO	12	46
10	MO-4	Millmerran	600	Sodium Silicate	162	74	Glass Fibre	3	MgO	24	50
11	MO-6	Millmerran	600	Sodium Silicate	162	74	Glass Fibre	3	MgO	36	55
12	MM1-MgO-0	Millmerran	600	Sodium Silicate	162	74	MgO	0	na	na	42
13	MM1-MgO-2	Millmerran	600	Sodium Silicate	162	74	MgO	12	na	na	46
14	MM1-MgO-4	Millmerran	600	Sodium Silicate	162	74	MgO	24	na	na	50
15	MM1-MgO-6	Millmerran	600	Sodium Silicate	162	74	MgO	36	na	na	55
16	MM1-0	Millmerran	600	Sodium Silicate	162	74	Kal(SO ₄) ₂	0	na	na	40
17	MM1-2	Millmerran	600	Sodium Silicate	162	74	Kal(SO ₄) ₃	12	na	na	44
18	MM1-4	Millmerran	600	Sodium Silicate	162	74	Kal(SO ₄) ₄	24	na	na	46
19	MM1-6	Millmerran	600	Sodium Silicate	162	74	Kal(SO ₄) ₅	36	na	na	48
20	MMS-1	Millmerran	600	Sodium Silicate	162	74	Fine Sand	0	na	na	0
21	MMS-2	Millmerran	600	Sodium Silicate	162	74	Fine Sand	60	na	na	-4
22	MMS-3	Millmerran	600	Sodium Silicate	162	74	Fine Sand	120	na	na	-11
23	MMS-4	Millmerran	600	Sodium Silicate	162	74	Fine Sand	240	na	na	0

3.2.1 Preparation of Geopolymer Paste

All mixing was done in P2 at the CEEFC with accordance to the safety standards of USQ and under the guidance of Phd mentor Zhua Zang. It was ensured that all safety procedures and protocol was followed before the commencement of mixing. Goggles, gloves and a dust mask were worn throughout the duration of the mixing. All of the different mix composition followed the same procedure. The procedure of the mixing is listed below:

1. The container that was used to hold the fly ash while being weighed was first cleaned off impurities and weighed. The electronic scale was then zeroed so that only the mass of the fly ash was measured when added to the electronic scale.



Figure 13: Empty container weighed

2. The required mass of fly ash is obtained from the container and weighed in a separate container on the electronic scale. Since fly ash is very fine in nature care was taken when transferring the fly ash between containers. After weighing the container holding the fly ash was set aside.



Figure 14: Fly ash weighed

- The water was next to being weighed. A volumetric beaker was first weighed on the scale and zeroed so that the only the required volume of water could be measured by the scale.

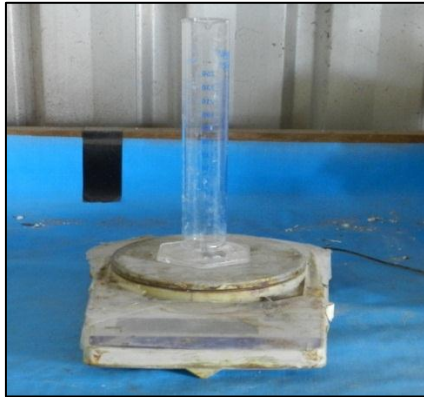


Figure 15: Volumetric beaker empty weighed

- Another container was obtained for the measurement of the alkali activator which was either sodium silicate or sodium hydroxide. The container was first weighed and then zeroed. Alkali activator was then added according to the mix composition.

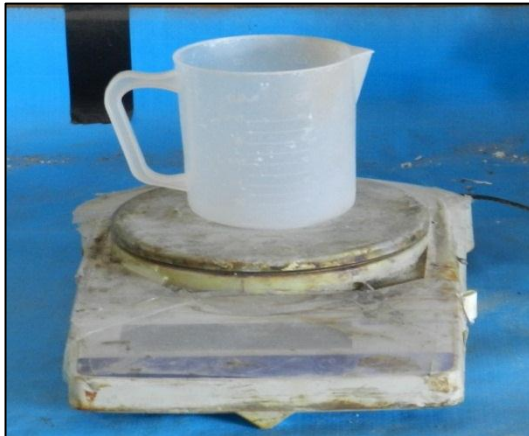


Figure 16: Alkali Activator container being weighed

- Additional additives followed the same procedure for the measurement of its mass.



Figure 17: Additional additive being weighed

- Mixer bowl was removed and filled with fly ash; it was then re-attached to the mixer which was then activated at low speed.



Figure 18: Electronic mixer

- The alkali activator was then mixed with the water and then gradually poured into the mixture being stirred until all was finished.



Figure 19: Alkali Activator being added to fly ash

- The speed of the mixer was increased and allowed to mix for about 3-4 minutes until it could be seen that the materials had formed a paste with uniform colour and texture.



Figure 20: Electronic mixer mixing at high speed

9. Additional water was added if the workability was seen to be low. This was then noted.

3.2.2 Casting and Curing

Casting for all the mix composition followed the same procedure and used the same moulds. Curing was done at temperature of 80°C. All moulds were cleaned off any impurities before casting. The procedure followed for the casting is listed below:

1. Moulds are made of PVC 25mm dia pipe cut into small lengths of 40mm. The bottom side was taped to contain the paste.



Figure 21: PVC moulds

2. The mixing bowl was detached from the mixer. A spatula was used to fill the moulds. Approximately 25 moulds were filled from each mix composition.



Figure 22: Moulds ready to be cast

3. After filling each mould it was then compacted in order to displace any air bubbles. After which the top was struck off to ensure a flat top surface. It was then laid out ready to be heat cured.



Figure 23: Compacting of geopolymer paste

4. The samples were then sealed with plastic and placed into the oven at 80°C for 24 hrs.



Figure 24: Container covered with plastic for curing

3.2.3 De-Moulding

After 48hrs of being in the oven the samples have been sufficiently hardened. The following steps describe the removal of the samples from the oven and the different environments they are exposed to:

1. Sufficiently hardened the samples are removed from the oven are demoulded. This required removing the tap from the bottom side of the specimen. The specimen was carefully removed from the mould so that no part of the specimen was left stuck onto the mould.



Figure 25: Specimen de-moulded

2. It is ensured that the samples are correctly labelled according to their mix composition to avoid any confusion during and after the experiment phase. After the specimens were labelled it then stored into glass jars where the casting date, mix design and chemical additive was carefully labelled and glued onto the side of the glass jar.

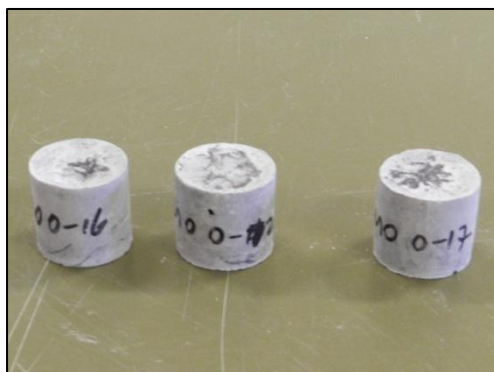


Figure 26: Specimens labelled and stored

3. Five samples from each mix composition are randomly chosen and are placed into three different environments:
 - a. Container 1, water just up to the base of the sample.
 - b. Container 2, dry container with just sample (control).
 - c. Container 3, sample submerged in water.

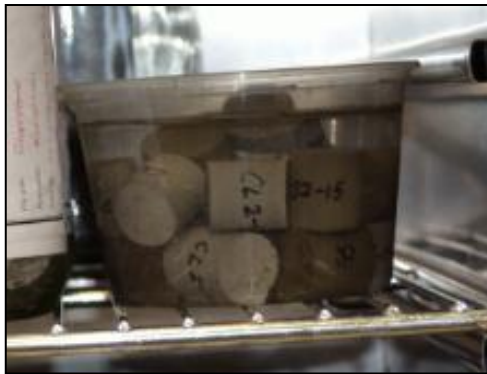


Figure 27: Specimens exposed to different condition

4. A sample from each mix composition was placed into a closed container containing 50ml of distilled so that leaching could be tested.

3.3 Sample Testing

This section describes the various tests conducted on the samples after 28 days of being exposed to the three different environment conditions. The tests include:

- i. Compressive Test
- ii. Leaching
- iii. pH level.

Compressive testing was conducted at P9 while the leaching and the pH testing were conducted at the lab at Z1 under the supervision of Mrs Freidriker Eberhard and Mr Raed. A risk assessment and safety induction was conducted before any experiment was to proceed.

5.3.1 Compressive Test

This test was conducted in the P9 laboratory at the CEEFC. This test was done under supervision. All crushing was done in P9 at the CEEFC with accordance to the safety standards of USQ and under the guidance of Phd mentor Zhua Zang. It was ensured that all safety procedures and protocol was followed before the commencement of crushing. Goggles, gloves and a dust mask were worn throughout the duration of the compressive test. The following procedure was followed during the testing of each sample.

1. The samples are divided into their different exposure environment and different mix composition. Note that the picture below illustrates the three exposure environment; rom left to right; submerged in water, in contact with water (efflorescence simulation) and dry condition.



Figure 28: Specimens layed out after 28 days

2. Before the crushing it was ensured that the surface of the sample was reasonably flat. This was done by measuring with a micro-metre. If the sample was not flat then it was rubbed onto sandpaper to even out the surface.



Figure 29: Specimens prepared for crushing

3. The compression machine was switched on and the cross-hair was set according to the desired limits.

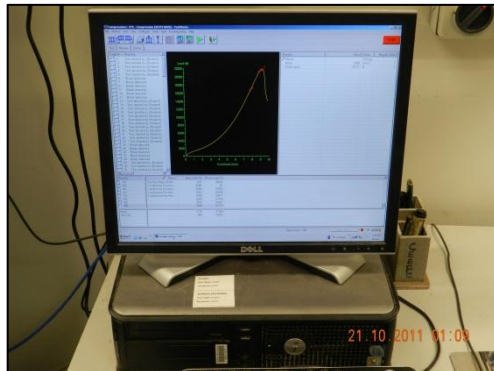


Figure 30: Computer recording data

4. Sample is placed onto between the crushing planes. The top plane was then lowered so that it was just about to be in contact with sample, this was manually.



Figure 31: Lowering compression plates

- The machine was switched on to automatic mode where by the machine slowly transfer load onto the sample. While this done the computer connected to the crushing machines plots the graph as the load gradually increased until the sample fails. This is recorded by the computer and also by hand.

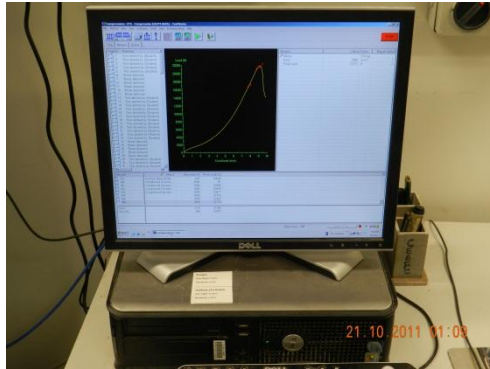


Figure 32: Data recording

- A crushed sample from each exposure environment and mix composition was placed in a container, clearly labelled for further analysis.

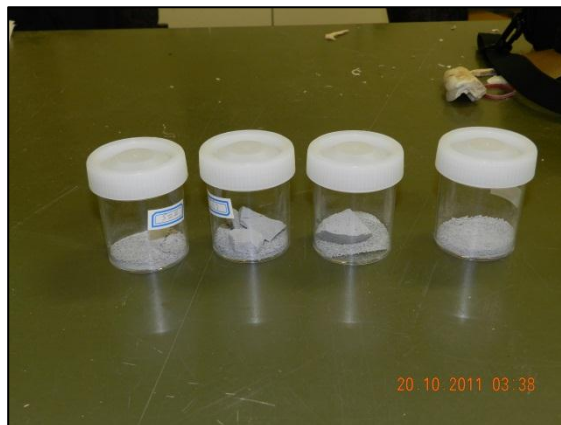


Figure 33: Specimens stored and labelled for later analysis

3.4.1 Leaching

Leaching was performed so that to measure the accurately the extent of fly ash dissolution as a result of alkali activation. Leaching was conducted on each specimen. Each sample was crushed and sieved, so that the leaching process could be accelerated. Leaching was done at intervals of 3hrs, 6hrs, 12hrs, 24hrs and 72hrs. Using the Atomic Absorption Spectrometer the level of leaching was measured for each sample.

Procedure for leaching:

1. Each specimen was crushed by hand and was passed through two sieves of size 1.66mm and later a size 1.2mm. This was the specimen size that would be used for the leaching observation.



Figure 34: Sieves used for size determination

2. After crushing the specimen, 1g was then stored in a container containing 24 ml of distilled water which was carefully labelled to avoid any confusion between specimens.

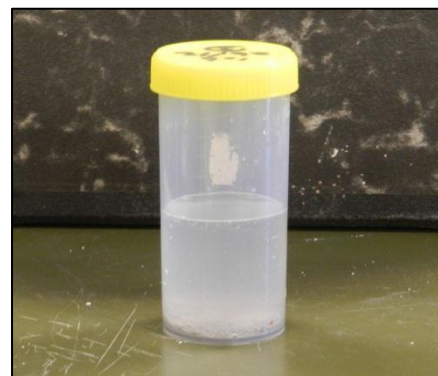
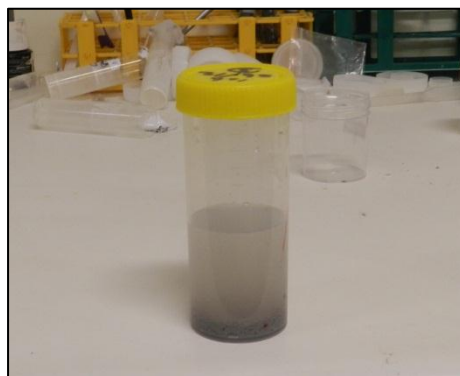


Figure 35: Sieved specimens stored in container for leaching analysis

3. After 3hours 2ml of the solution containing each of the specimens was removed and added to a container containing 50ml of distilled. Subsequently 2ml of distilled were added to the specimen container so that the 50ml volume could be maintained. This process was conducted for all of the specimens at each of the time intervals of 3hrs, 6hrs, 12hrs, 24hrs and 72hrs.

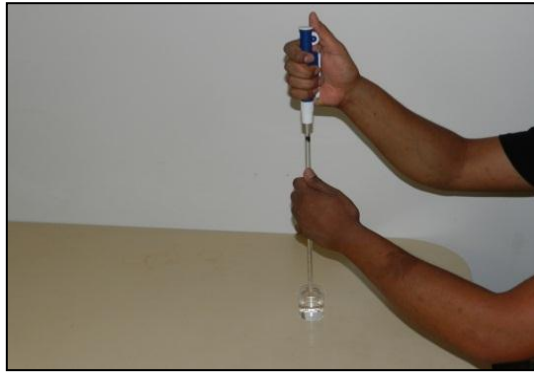


Figure 36: Leached specimens pipetted for analysis

4. After all the specimens were prepared for each it was then taken to Z1 lab to test the amount each element leached out of each specimen.

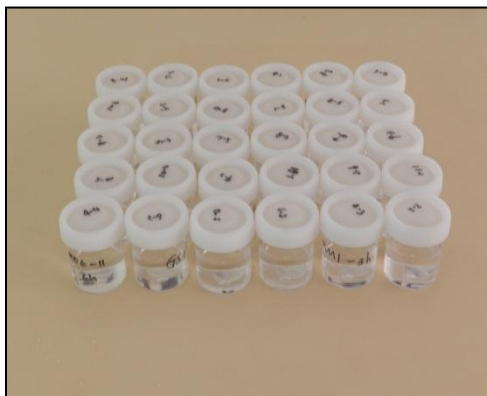


Figure 37: Leached solution labeled ready for analysis

5. The levels of leaching for Potassium, Calcium and Sodium were measured using AAS machine.

3.4.1.1 Atomic Absorption Spectrometer Testing Procedure

The Atomic absorption spectrometer is an apparatus that uses the absorption of light to measure the concentration of gas-phase atoms.

Below are the procedures for conducting the AAS test.

1. The vials are first cleaned off impurities with distilled water.



Figure 38: Vial cleaned with distilled water

- The vials are then dried using the compressed air hose. This is to ensure that all impurities are removed from the vial so as not to contaminate the test.



Figure 39: Vial being dried with compressed air

- Five vials are then filled with the standard solutions on the respective element to be measured. This process is repeated for different elements to be measured. It was made sure that the appropriate dilution was used and vials are placed into the automatic sample changer.



Figure 40: Sodium (Na) standard solutions



Figure 41: Potassium (K) standard solutions



Figure 42: Calcium (Ca) standard solutions

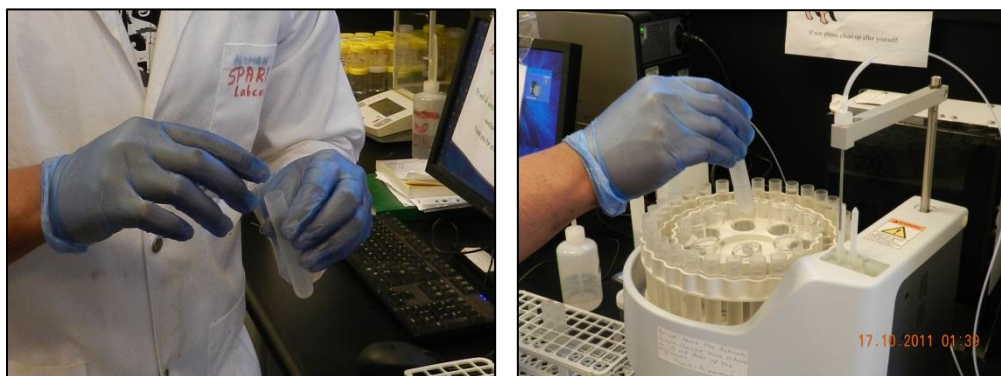


Figure 43: Standards filled and placed into slots

4. After the standards are placed in the automatic sample changer the rest of the solution to be tested are filled into the remaining vials. Once all the vials are filled then the element is selected on the screen tab. Following this the gas valves (nitrous oxide, acetylene and compressed air) are opened. Then the calibration curve is setup, once this is seen to be appropriate procedure is continued until all steps in the computer are completed.

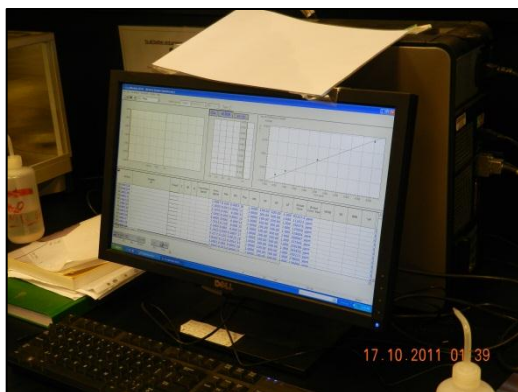


Figure 44: Calibration curve on computer

5. The AAS is ignited by pressing purge for a couple of seconds, then at the same time press ignite until a stable flame emerges.
6. After the test is completed the flame is extinguished by pressing the extinguish button and all the valves are closed. Finally all the data is saved onto the excel file for later analysis.



Figure 45: Atomic Absorption Spectrometer machine

3.4.2 pH Testing

After the AAS testing was completed the specimens were then tested for the level pH of each of the specimen.

Testing procedure

1. The specimens were laid on the work bench according to their mix design. This is carried out so that to avoid any confusion while testing the specimens.



Figure 46: Mix design specimen solutions

2. Each specimen solution was filled about 25ml into a 100ml beaker. The beaker was then set upon a magnetic stirrer which then began to stir the mixture.
3. After a few seconds when it was clear that the solution was thoroughly stirred the probe of the pH tester was then lowered and immersed into the solution.

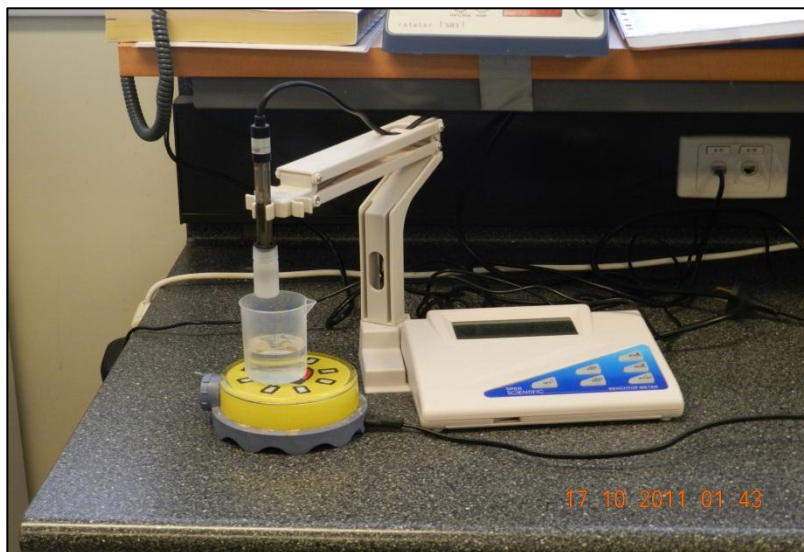


Figure 47: Measuring of specimen pH

4. While the probe was immersed in the solution the Bench top pH / MV Meter – 860031 took the reading of the pH level. When the reading stabilised at a constant value it was then taken as the pH value of the solution.
5. After the measurement of each solution was taken the probe and the beaker were then washed off using distilled water. This is to ensure no residue from the previous solution is left in the beaker or attached to the probe which will contaminate the next solution to be tested.
6. Testing continued until all specimens were finished.

4.0 Experimental Results and Discussion

In this chapter the results obtained from the various tests done to the different mix ratios will be presented. The following tests results are shown below:

- I. Compressive Test & Modulus of Elasticity
- II. Leaching
- III. pH value

4.1 Development of Stress vs. Strain Curve

The data that was obtained during the testing was extracted onto excel where the maximum load (N) tolerated by the specimen was converted to pressure (MPa) by dividing by the area in contact with the force. The strain is then calculated by dividing the change in length (ΔL) by actual length to obtain the strain. The results from the various exposure conditions are then plotted together for comparison.

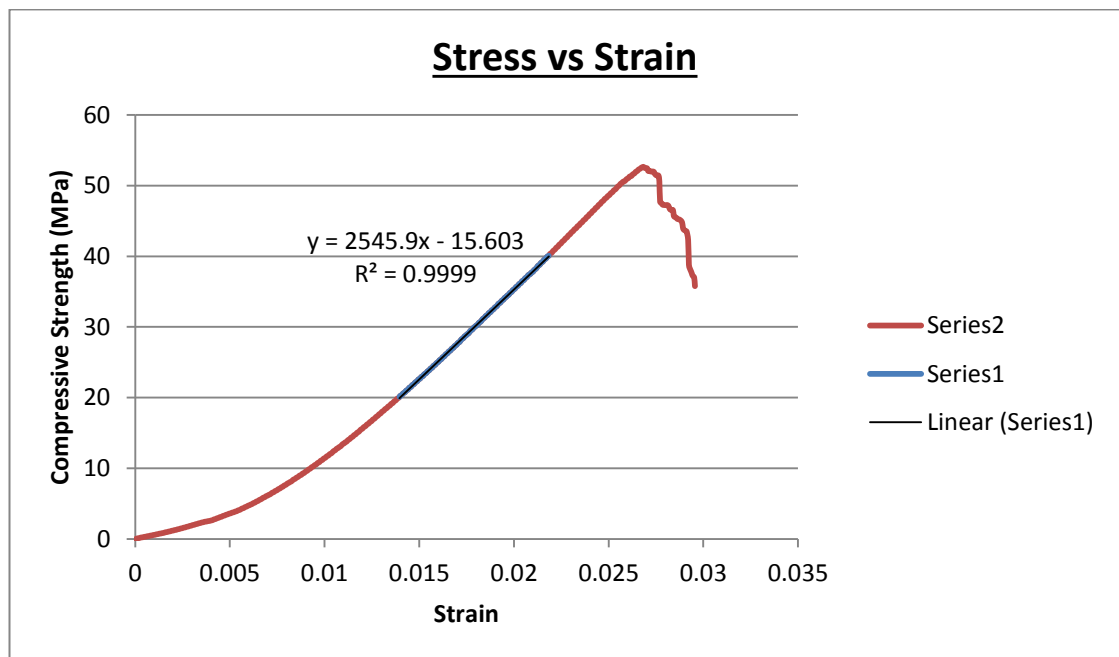


Figure 48: Example of a stress-strain curve produced for geopolymer paste specimen. A linear regression is applied to a section of the curve to determine the modulus of elasticity of the sample.

4.2 Modulus of Elasticity

The modulus of elasticity is described as the ratio of stress upon strain. Stress is the force delivered by a mass upon a surface area and this is given as force/area. The strain is the change in length of the compressed specimen upon actual length. This gives rise to the formula of:

$$E = \frac{\text{stress}}{\text{strain}} = \frac{\text{Force}/\text{Area}}{\Delta L/L}$$

The modulus of elasticity can be obtained from the graph of stress vs strain as shown below:

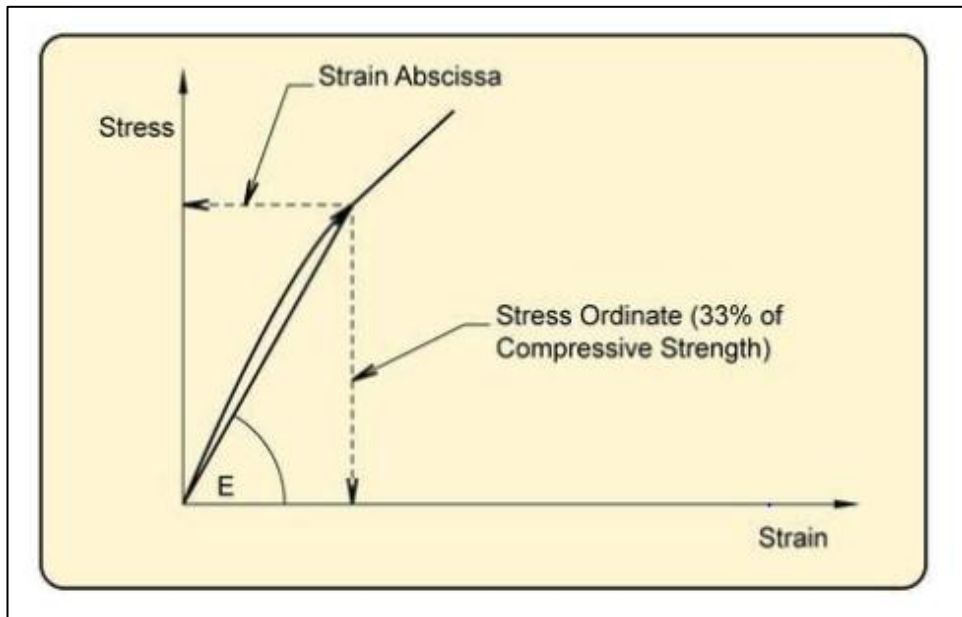


Figure 49: Stress - Strain Curve (CIV3906 2012)

To obtain the Young's Modulus (E) = Stress ordinate (MPa)/Strain abscissa

4.3 Compressive Strength and Modulus of Elasticity

4.3.1 Mix Design 1 Result

In this mix design the fly ash from Callide power station was used to produce the geopolymer paste. The alkali activator for CL1 was sodium hydroxide and the sodium silicate solution was used to activate CL2. The mix design is shown below:

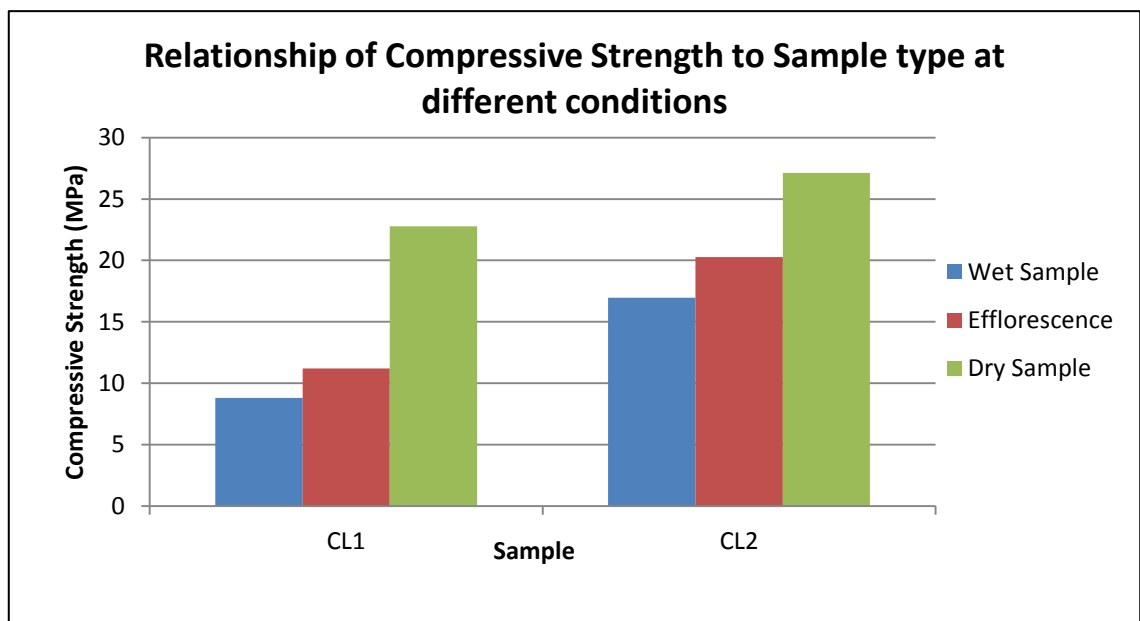


Figure 50: Compressive strength of CL1 and CL2 at different exposure conditions

Table 3: Values derived from mix design 1 stress strain curve (wet condition)

Wet Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample CL1	862.9545455	8.802666265	0.01528
Sample CL2	1228.346457	16.97075013	0.04084

Table 4: Values derived from mix design 1 stress strain curve (Dry condition)

Dry Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample CL1	1787.192982	22.76062696	0.039
Sample CL2	1671.146617	27.11863601	0.0416

Table 5: Values derived from mix design 1 stress strain curve (Efflorescence condition)

Efflorescence Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample CL1	925.7978723	11.20382556	0.0259
Sample CL2	1599	20.27648643	0.02864

4.2.4 Mix Design 1 Discussion

As can be seen in figure 46 the dry condition has a higher compressive strength for each sample. This is then followed by the efflorescence and wet condition. In this result it indicates that efflorescence has a clear effect on the compressive strength of the material. The different alkali activators used can also be compared, as can be seen in figure 46 CL2 (sodium silicate activator) has higher compressive strengths at respective conditions when compared to CL1 (sodium hydroxide). This confirms the conclusion made by Palomo, et al., (1999) that when soluble silicates are used as activators it causes higher rates of reactions than when hydroxides are used as the activators. This leads to faster mechanical strength development. It can also be noted that the compressive strength and modulus of elasticity for this design are directly proportional to each other.

4.2.5 Mix Design 2

In this mix design the fly ash from Gladstone power station was used to produce the geopolymer paste. The alkali activator for GS2 was sodium hydroxide and the sodium silicate solution was used to activate GS1. The mix design is shown below:

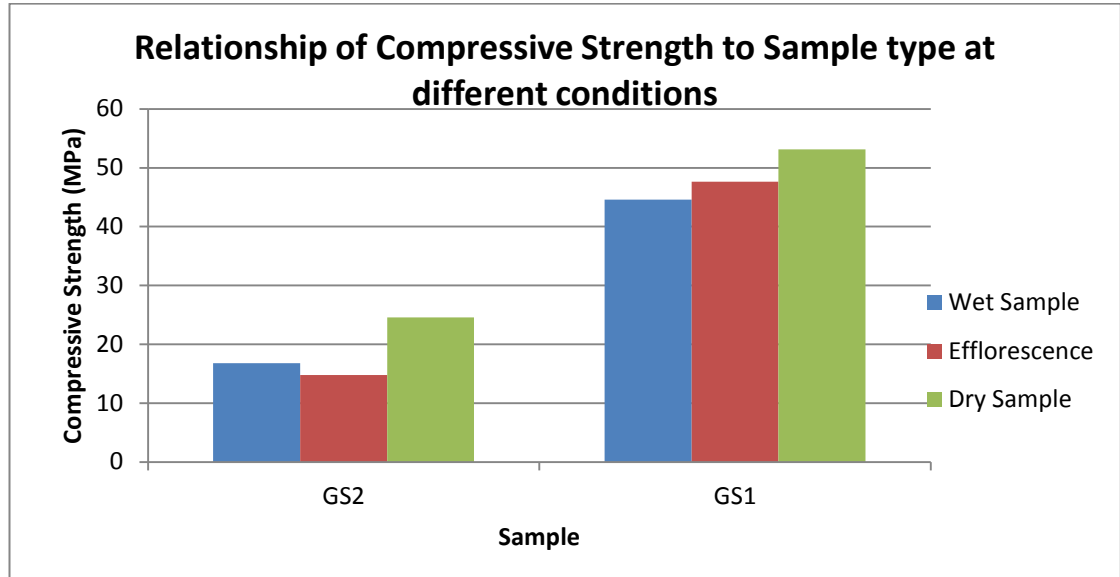


Figure 51: Compressive strength of GS1 and G2 at different exposure conditions

Table 6: Values obtained from stress strain curve of mix design 2 (wet condition)

Wet Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample GS1	2557.359307	16.81123873	0.04264
Sample GS2	1618.137255	44.5918912	0.0396

Table 7: Values obtained from stress strain curve of mix design 2 (dry condition)

Dry Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample GS1	2432.21831	24.58023851	0.02956
Sample GS2	2017.628205	53.12632841	0.05196

Table 8: Values obtained from stress strain curve for mix design 2 (efflorescence condition)

Efflorescence Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample GS1	2435.024155	14.78655079	0.04832
Sample GS2	1276.041667	47.66192546	0.02004

4.2.5 Mix Design 2 Discussion

As can be seen in figure 49 it is clear that the sample exposed to dry condition has greater compressive strength than the other two conditions. As in figure 48 the mix design that used the sodium silicate solution (GS1) as its alkali activator has greater values of compressive strength than GS2 which used sodium hydroxide. Again this confirms the conclusion made by Palomo, et al., (1999) that when soluble silicates are used as activators it causes higher rates of reactions than when hydroxides are used as the activators. This leads to faster mechanical strength development. It can also be noted that the compressive strength and modulus of elasticity for this design are directly proportional to each other.

4.2.6 Mix Design 3

In this mix design the fly ash from Millmerran power station was used to produce the geopolymer paste. The alkali activator for this sample was sodium silicate. Glass fibre and magnesium oxide (MgO) were used as an additive in this mix design.

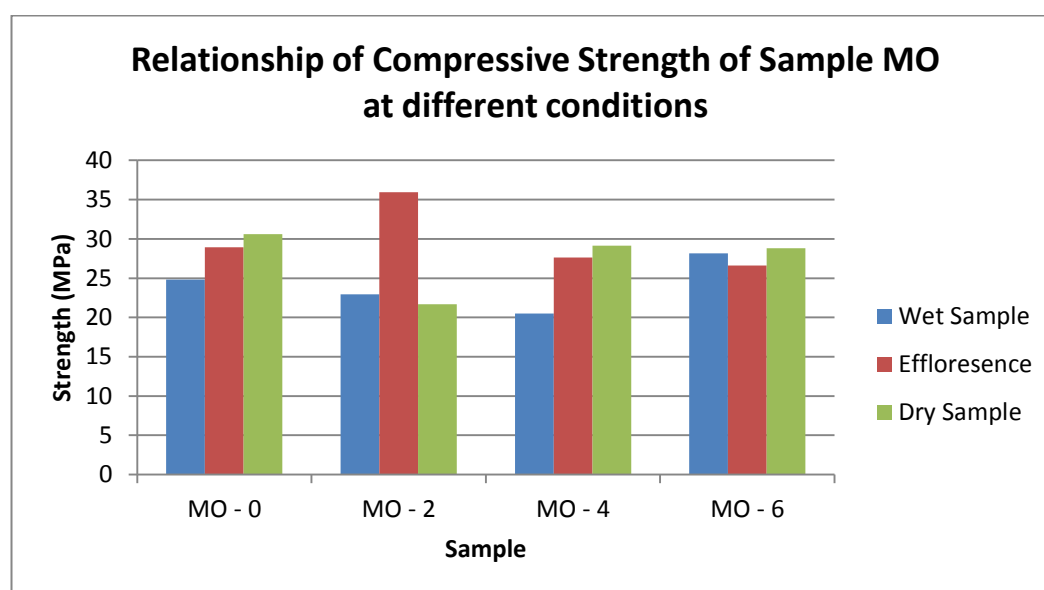


Figure 52: Compressive strength of MO – 0 to MO – 6 at different exposure conditions

Table 9: Values obtained from mix design 3 stress strain graph (wet condition)

Wet Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample MO-0	1959.259259	24.81951	0.042
Sample MO-2	1571.875	22.92442	0.0356
Sample MO-4	1472.048611	20.51545	0.0275
Sample MO-6	2146.846847	28.14979	0.042

Table 10: Values obtained from mix design 3 stress strain graph (dry condition)

Dry Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample MO-0	922.0149254	30.58779	0.0384
Sample MO-2	844.3661972	21.66951	0.026
Sample MO-4	1136.367188	29.15005	0.0352
Sample MO-6	1112.333333	28.82155	0.0372

Table 11: Values obtained from mix design 3 stress strain graph (efflorescence condition)

Efflorescence Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample MO-0	1753.413043	28.9212	0.0292
Sample MO-2	2254.086538	35.93658	0.0336
Sample MO-4	2215.327381	27.61809	0.0336
Sample MO-6	1580.182927	26.63005	0.0364

4.2.7 Mix Design 3 Discussion

It can be seen in figure 50 that the dry sample has the higher compressive strength in samples MO-0, MO-4 and MO-6. However it is interesting to note that in sample MO-2 the efflorescence sample showed remarkable compressive strength. This occurrence in the result should be further investigated. All 4 specimens of the MO-2 sample at efflorescence condition showed high compressive strengths, from these readings the average was taken and is used to calculate the compressive strength which resulted in the high compressive value for MO-2 at efflorescence condition. It can also be noted that the compressive strength and modulus of elasticity for this design are directly proportional to each other.

4.2.8 Mix Design 4

In this mix design the fly ash from Millmerran power station was used to produce the geopolymer paste. The alkali activator for this sample was sodium silicate. Magnesium oxide (MgO) was used as an additive in this mix design.

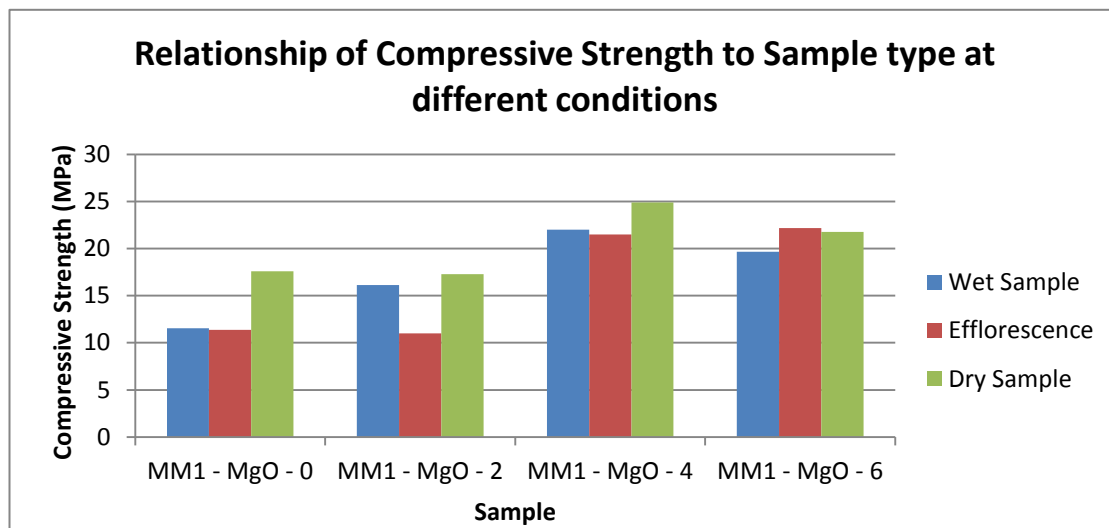


Figure 53: Compressive strength of MM1 – MgO - 0 to MM1 – MgO - 6 at different exposure conditions

Table 12: Values obtained from stress strain curve of mix design 4 (wet condition)

Wet Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample MM1-MgO-0	880.1075269	11.53606	0.0332
Sample MM1-MgO-2	1432.242991	16.1406	0.0232
Sample MM1-MgO-4	1707.075472	22.01991	0.0228
Sample MM1-MgO-6	1574.80315	19.669	0.0244

Table 13: Values obtained from stress strain curve of mix design 4 (dry condition)

Dry Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample MM1-MgO-0	438.5869565	17.58292	0.0236
Sample MM1-MgO-2	1155.952381	17.30332	0.0224
Sample MM1-MgO-4	1326.169591	24.91882	0.0244
Sample MM1-MgO-6	1315.346535	21.78359	0.0296

Table 14: Values obtained from stress strain curve of mix design (efflorescence)

Efflorescence Sample	Modulus of	Peak Stress	Strain at
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	Elasticity (Mpa)		Peak Stress
Sample MM1-MgO-0	980.861244	11.37461	0.0272
Sample MM1-MgO-2	1097.333333	11.01029	0.0364
Sample MM1-MgO-4	563.7119114	21.52436	0.0272
Sample MM1-MgO-6	1137.014563	22.1822	0.0472

4.2.8 Mix Design 4 Discussion

It can be seen in figure 53 that the dry sample has the higher compressive strength in samples MM1-MgO-0, MM1-MgO-2 and MM1-MgO-4. However it is interesting to note that for sample MM1-MgO-6 the highest compressive strength was that of the efflorescence condition. It can also be observed that the increase in the MgO added contributed to the increase in compressive strength for samples MM1-MgO-0, MM1-MgO-2 and MM1-MgO-4 at all exposure conditions. It can also be noted that increase in compressive has a direct relationship with the modulus of elasticity.

4.2.9 Mix Design 5

In this mix design the fly ash from Millmerran power station was used to produce the geopolymer paste. The alkali activator for this sample was sodium silicate. Potassium aluminium sulphate was used as an additive in this mix design.

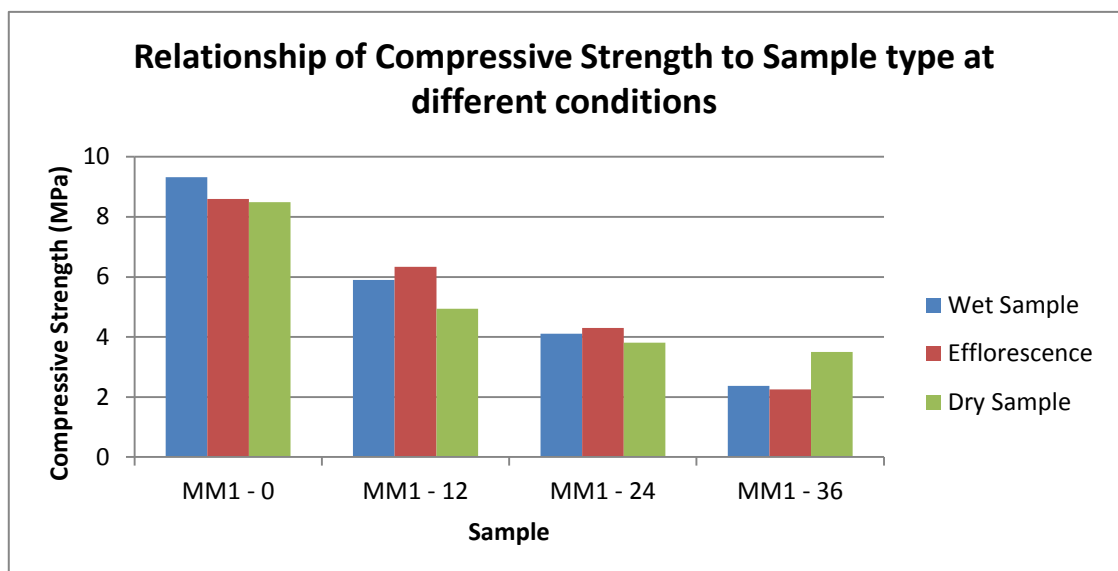


Figure 54: Compressive strength of MM1 – 0 to MM1 – 36 at different exposure conditions

Table 15: Values obtained from stress strain curve of mix design 5 (wet sample)

Wet Sample	Modulus of Elasticity (Mpa)	Peak Stress (Mpa)	Strain at Peak Stress
Sample MM1-0	1098.192771	9.31960136	0.0224
Sample MM1-12	450.7432432	5.90171816	0.0268
Sample MM1-24	384.2105263	4.11307179	0.0216
Sample MM1-36	411.3846154	2.37229921	0.0092

Table 16: Values obtained from stress strain curve of mix design 5 (efflorescence sample)

Dry Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample MM1-0	1040.53	8.49250521	0.0236
Sample MM1-12	559.697	4.93609358	0.0288
Sample MM1-24	384.2105263	3.81156875	0.0244
Sample MM1-36	408.04	3.49987981	0.0296

Table 17: Values obtained from stress strain curve of mix design 5 (efflorescence)

Efflorescence Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample MM1-0	741.087	8.59405876	0.0264
Sample MM1-12	531.004	6.34011987	0.0232
Sample MM1-24	597.5862	4.29682566	0.0128
Sample MM1-36	423.5507	2.25312402	0.0088

4.2.9 Mix Design 5 Discussion

It can be seen in figure 51 that the dry sample has the higher compressive strength in samples MO-0, MO-2 and MO-4. However it is interesting to note that in sample MO-6 the efflorescence sample showed remarkable compressive strength. This occurrence in the result should be further investigated. All 4 specimens of the MO-6 sample at efflorescence condition showed high compressive strengths, from these readings the average was taken and is used to calculate the compressive strength.

4.2.10 Mix Design 6

In this mix design the fly ash from Millmerran power station was used to produce the geopolymer paste. The alkali activator for this sample was sodium silicate. Sand was used as an additive in this mix design.

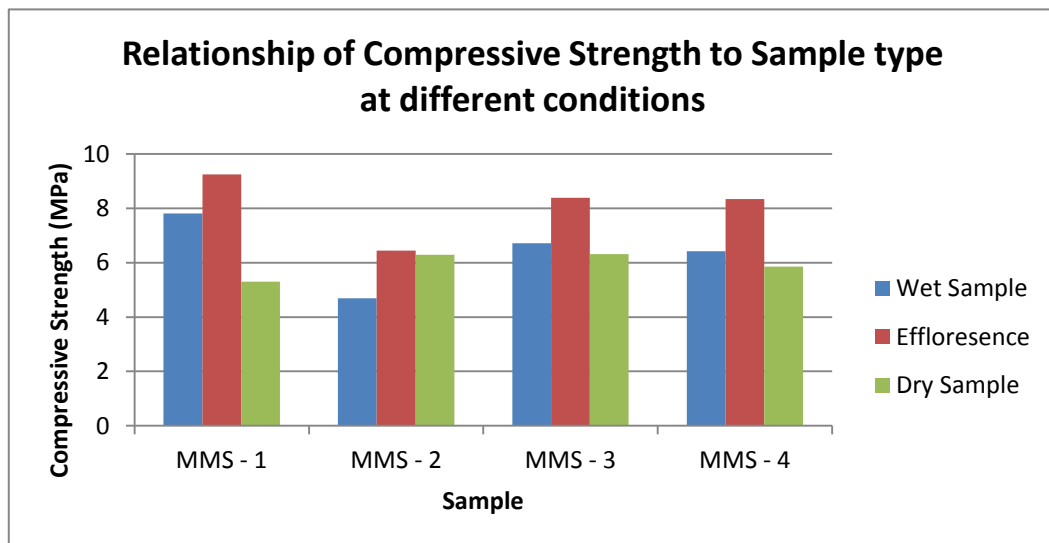


Figure 55: Compressive strength of MMS – 1 to MMS – 4 at different exposure conditions

Wet Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample MMS-1	922.0149254	7.817688	0.0364
Sample MMS-2	844.3661972	5.901718	0.0196
Sample MMS-3	1136.367188	4.113072	0.022
Sample MMS-4	1112.333333	2.372299	0.0332

Table 18: Values obtained from stress strain curve of mix design 6 (wet sample)

Dry Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample MMS-1	466.5697674	5.307879	0.0296
Sample MMS-2	553.6885246	6.295573	0.0236
Sample MMS-3	703.1862745	6.316285	0.0188
Sample MMS-4	745.9090909	5.863012	0.0244

Table 19: Values obtained from stress strain curve of mix design 6 (dry sample)

Efflorescence Sample	Modulus of Elasticity (Mpa)	Peak Stress	Strain at Peak Stress
Sample MMS-1	1616.090909	9.250167	0.0156
Sample MMS-2	832.9032258	6.448192	0.0196
Sample MMS-3	1138.80597	8.39523	0.0124
Sample MMS-4	1123.493976	8.340226	0.0136

Table 20: Values obtained from stress strain curve of mix design 6 (efflorescence sample)

4.2.10 Mix Design 6 Discussion

It can be seen and is interesting to note that the efflorescence condition records the highest compressive strength at each sample in figure 55. It can also be noted that increase in compressive has a direct relationship with the modulus of elasticity. The additive in this design mix is fine sand (fine aggregate).

4.3 Leaching Results

Leaching is carried out to accurately measure the amount of fly ash that was dissolution as the result of alkali activation. Two species (potassium and calcium) were measured for the amount which leached out; this measurement was conducted using the atomic absorption spectrophotometer. The results are presented below:

4.3.1 Potassium Leaching

4.3.1.1 Mix Design 1

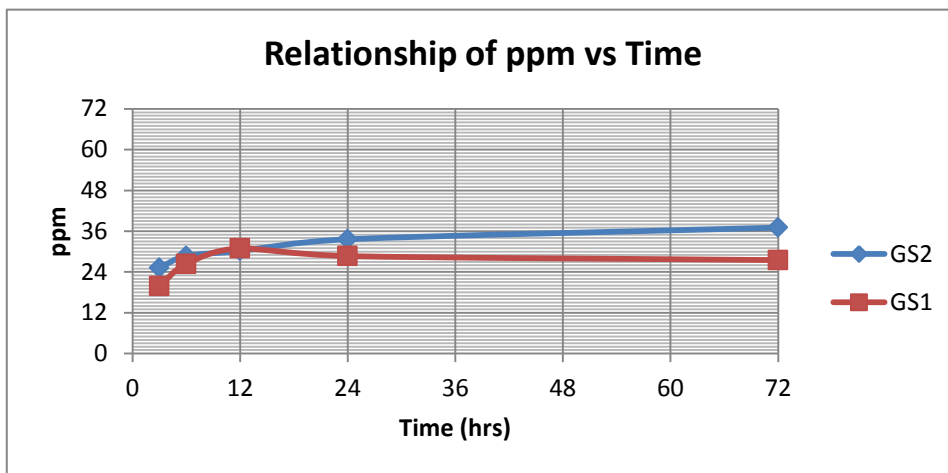


Figure 56: Mix Design 1 Leaching measurement (Potassium)

4.3.1.1 Mix Design 1 Discussion

It can be seen in figure 56 that the amount of species leaching out, increases in the first three time intervals at which the specimens are extracted. For GS2 the amount of species leaching out seems to gradually increase from the specimens taken at 24hrs and then at 72 hrs. However for the GS1 the value decreases time 12hrs to time 72 hrs.

4.3.1.2 Mix Design 2

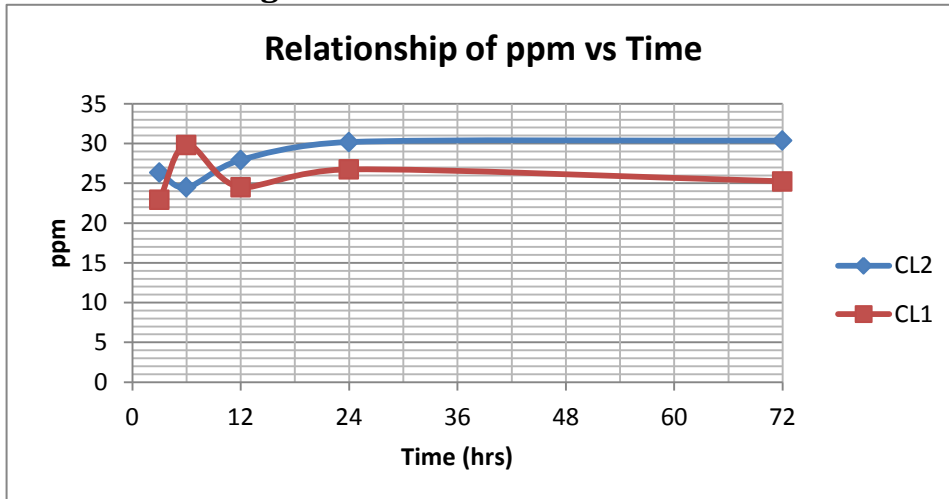


Figure 57: Mix Design 2 Leaching measurement (Potassium)

4.3.1.2 Mix Design 2 Discussion

It can be seen in figure 57 that the amount of species leaching out for CL1 increases at time 6 hrs then decreases at time 12 hrs. It again increases at time 24 hrs but again slightly decreases at time 72 hrs. For the sample CL2 the amount of species leaching out decreases at time 6hrs then it gradually increases for the rest of the time intervals.

4.3.1.3 Mix Design 3

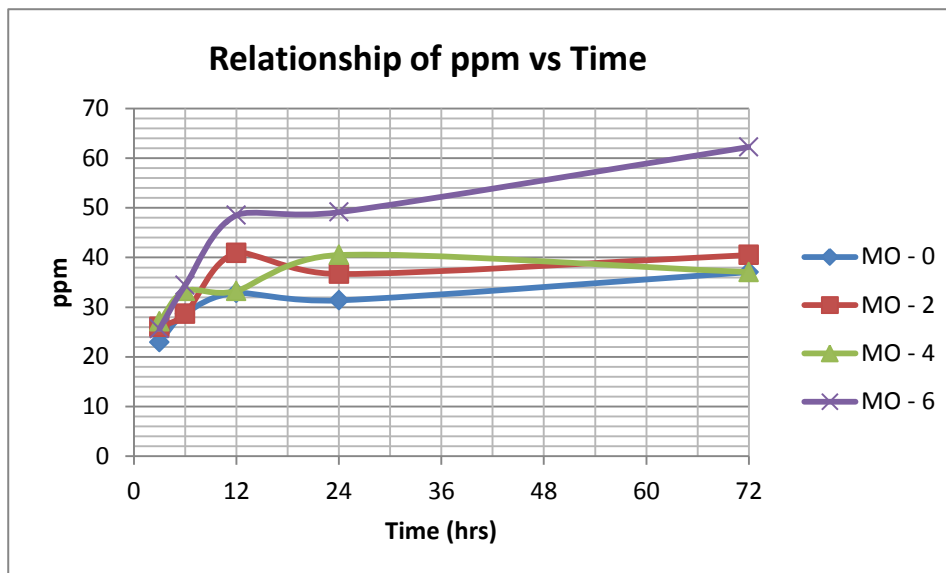


Figure 58: Mix Design 3 Leaching measurement (Potassium)

4.3.1.3 Mix Design 3 Discussion

It can be seen in figure 58 that the all samples follow a similar trend except for MO-4. The samples MO-2, MO-0 and MO-6 it increases in the first three time intervals then slightly decreases at time 24 hrs then finally increases again at time 72 hrs. The sample MO-4 decreases at time 72 hrs.

4.3.1.4 Mix Design 4

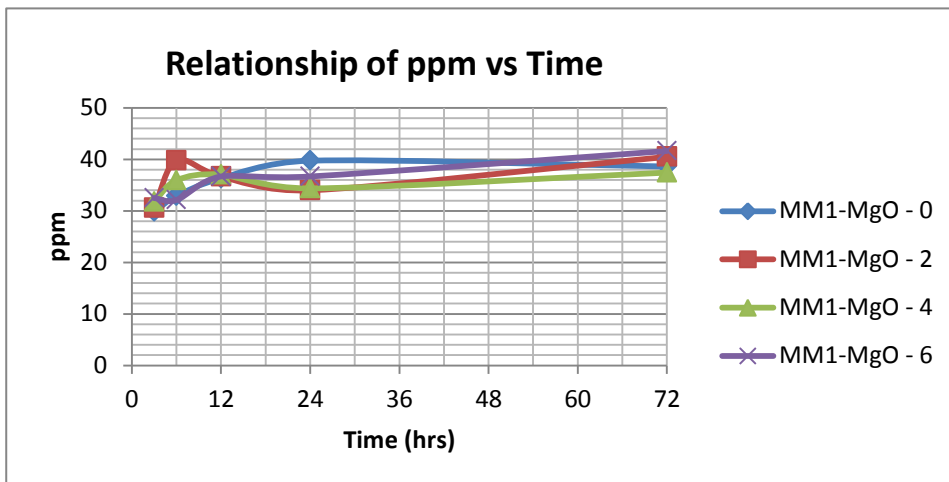


Figure 59: Mix Design leaching measurement (Potassium)

4.3.1.4 Mix Design 4 Discussion

It can be seen in figure 59 that the all samples follow a similar trend except for MM1-MgO-0. The samples MM1-MgO-2 MM1-MgO-4 MM1-MgO-6 it increases in the first two time intervals then slightly decreases at time 24 hrs then finally increases again at time 72 hrs.

4.3.1.5 Mix Design 5

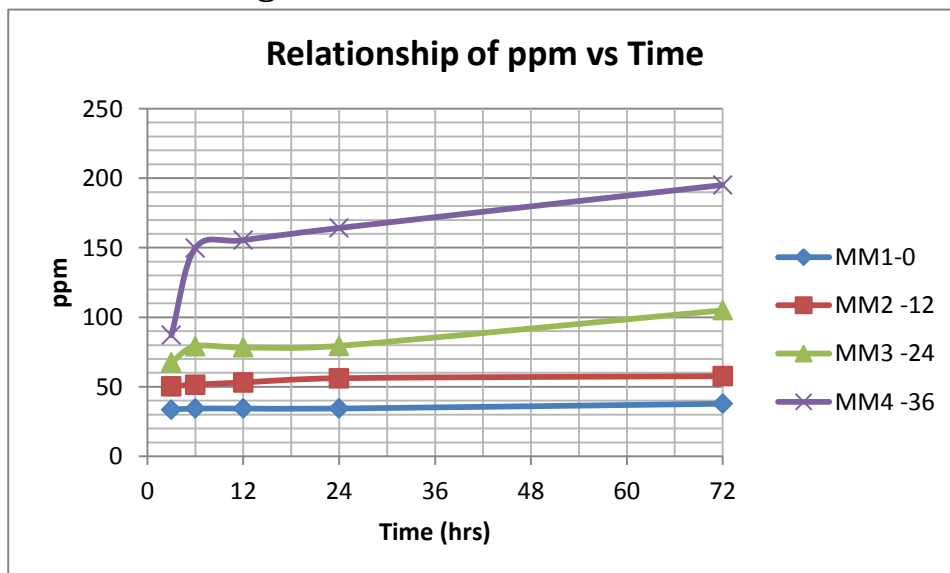


Figure 60: Mix Design 5 Leaching measurement (Potassium)

4.3.1.5 Mix Design 5 Discussion

It can be seen in figure 60 that the all samples follow a similar trend there is a sharp increase in the first time interval i.e. time 6hrs to time 12 hrs. MM2-12 and MM1-0 seem to stabilise after time 24 hrs. As for MM4-36 and MM3-24 the sample leaching species continue to increase fro time 24hrs to 72 hrs.

4.3.1.6 Mix Design 6

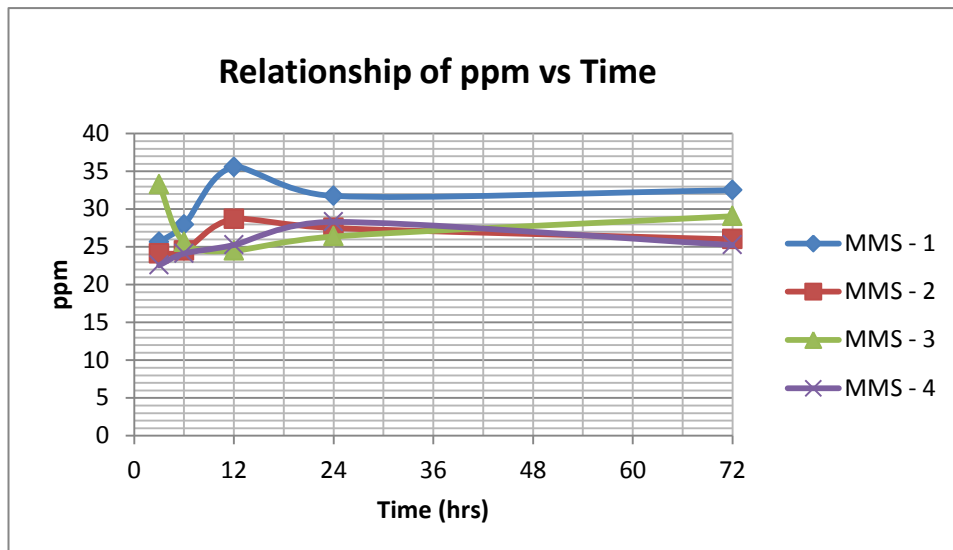


Figure 61: Mix Design 6 Leaching measurement (Potassium)

4.3.1.4 Mix Design 4 Discussion

It can be seen in figure 61 that the all samples follow a similar trend. At the first time step the values increase until the 3rd time step at 12 hrs this where MMS1 and MMS2 peak. The other two specimens peak at the 4th time step while the previous begin to decrease. At the 5th and last time step the specimens stabilise to an almost constant gradient.

4.3.2 Sodium Leaching

4.3.2.1 Mix Design 1

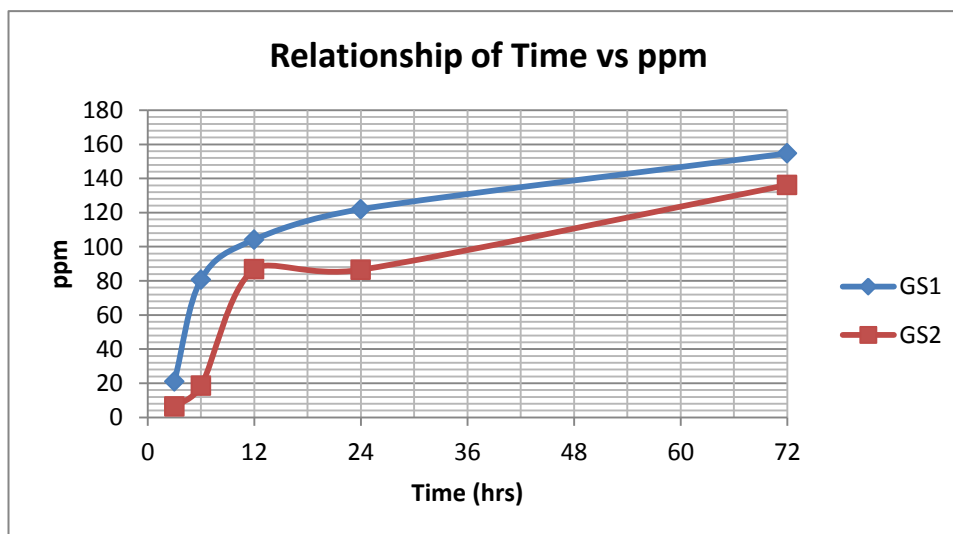


Figure 62: Mix Design 1 Leaching measurement (Sodium)

4.3.2.2 Mix Design 1 Discussion

It can be seen from figure 62 that the leaching curve of the specimen follows a typical leaching curve. This is when it is compared to leaching curve obtained from Xu & van Deventer, (2003). The leaching curve gradually increases with time as can be seen in figure 62.

4.3.2.3 Mix Design 2

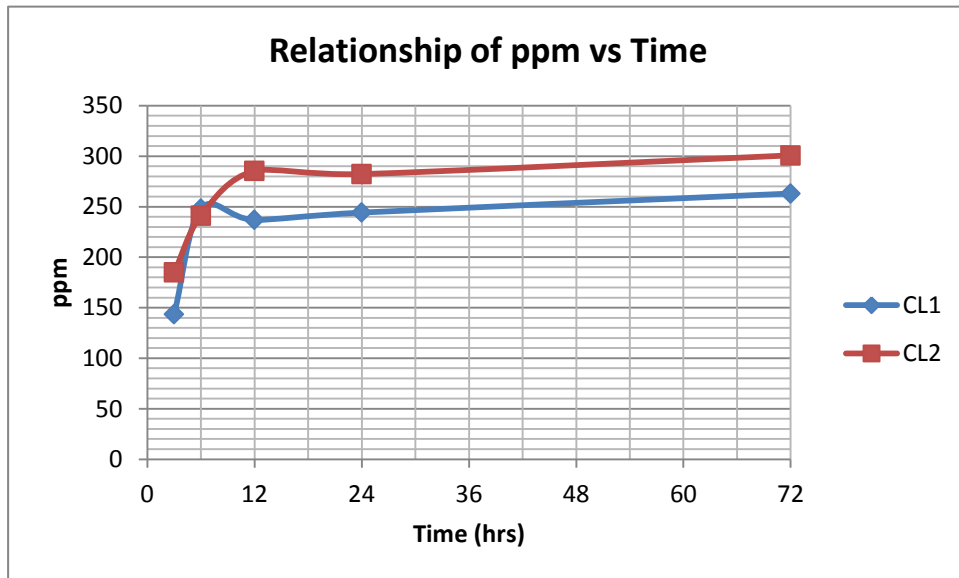


Figure 63: Mix Design 2 Leaching measurement (Sodium)

4.3.2.4 Mix Design 2 Discussion

It can be seen from figure 63 that the leaching curve of the specimen follows a typical leaching curve. This is when it is compared to leaching curve obtained from Xu & van Deventer, (2003). The leaching curve gradually increases with time as can be seen in figure 63. However there is a slight decrease after time 6hrs for CL1 then it increases again.

4.3.2.5 Mix Design 3

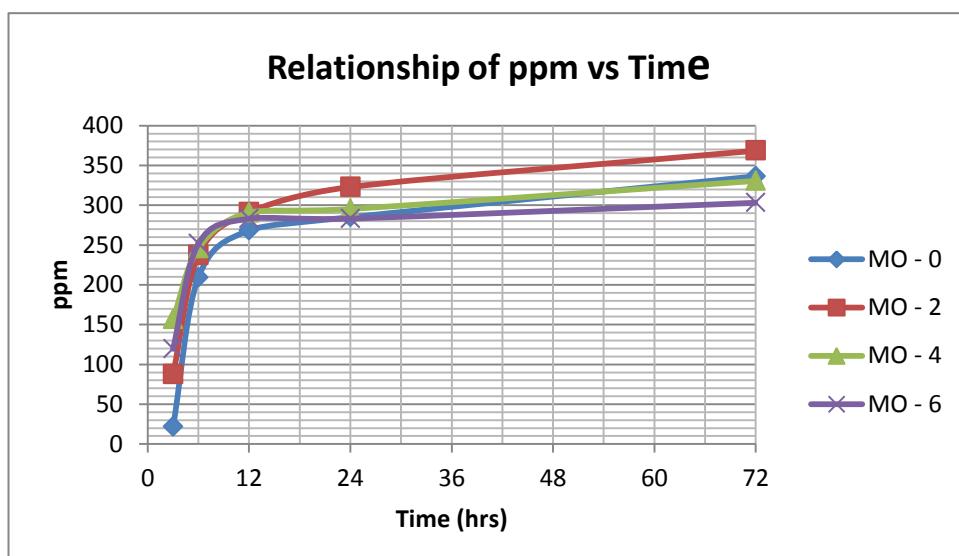


Figure 64: Mix Design 3 Leaching measurement (Sodium)

4.3.2.6 Mix Design 3 Discussion

It can be seen from figure 64 that the leaching curve of the specimen follows a typical leaching curve. This is when it is compared to leaching curve obtained from Xu & van Deventer, (2003). At the first 3 time intervals it can be seen that leaching significantly increases. After this phase the leaching continues to increase but in a more gradual fashion.

4.3.2.7 Mix Design 4

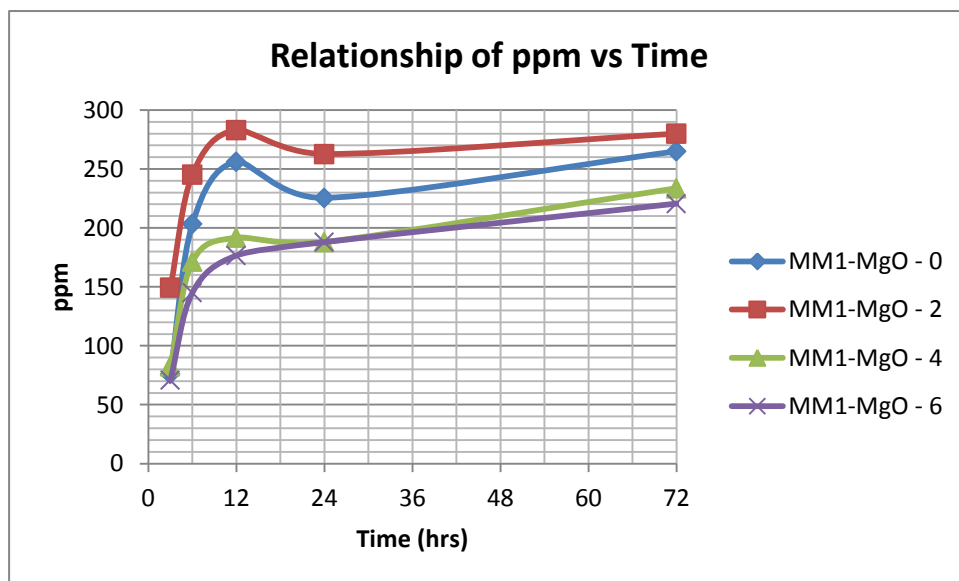


Figure 65: Mix Design Leaching measurement (Sodium)

4.3.2.8 Mix Design 4 Discussion

It can be seen from figure 65 that the leaching curve of the specimen follows a typical leaching curve. This is when it is compared to leaching curve obtained from Xu & van Deventer, (2003). Again for this mix design it can be seen that the leaching increases at a significant rate for the first 3 time intervals and then it stabilises and continues to increase gradually.

4.3.2.9 Mix Design 5

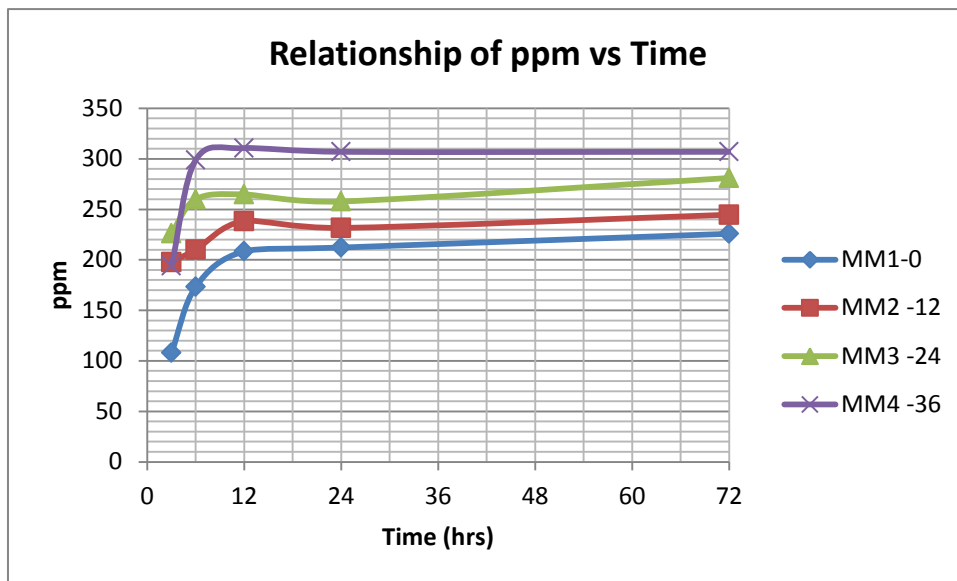


Figure 66: Mix Design 5 Leaching measurement (Sodium)

4.3.2.10 Mix Design 3 Discussion

It can be seen again from figure 66 that the leaching curve of the specimen follows a typical leaching curve. This is when it is compared to leaching curve obtained from Xu & van Deventer, (2003). Again for this mix design it can be seen that the leaching increases at a significant rate for the first 3 time intervals and then it stabilises and continues to increase gradually. From the graph above it is noted that the increase in the addition of potassium alumina sulphate increases the leaching in the specimen. Refer to design mix table pp. 42.

4.3.2.11 Mix Design 6

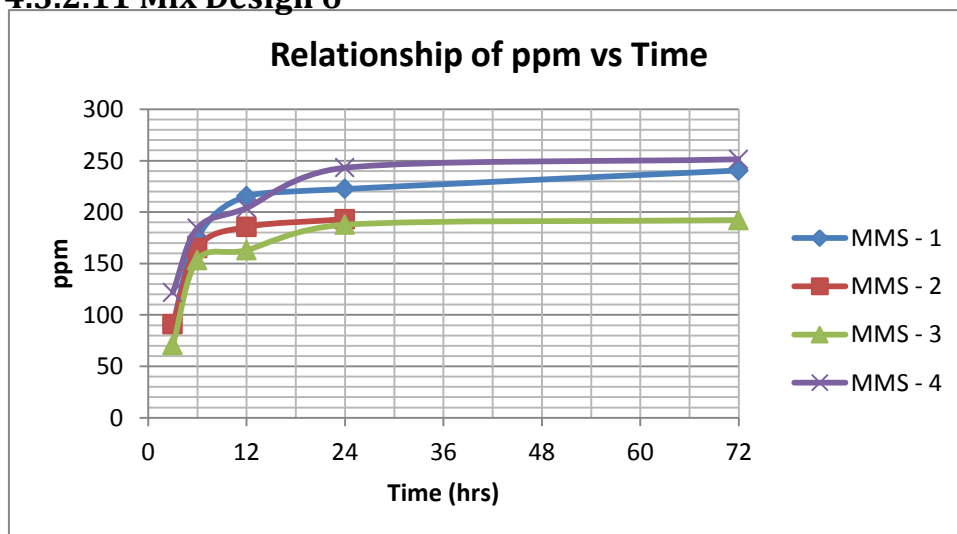


Figure 67: Mix Design 6 Leaching measurement (Sodium)

4.3.2.11 Mix Design 3 Discussion

It can be seen again from figure 67 that the leaching curve of the specimen follows a typical leaching curve. This is when it is compared to leaching curve obtained from Xu & van Deventer, (2003). Again for this mix design it can be seen that the leaching increases at a significant rate for the first 3 time intervals and then it stabilises and continues to increase gradually. Sand is used as an additive in this design mix but no relationship can be noted when comparing the addition of sand to the leaching.

4.4 pH Testing Results

In this section the results of the pH being tested for each mix design is being presented. For each sample example MM1-0 it had 5 specimens, these were the time intervals at which the specimen solution was extracted during the leaching testing process.

4.4.1 Mix Design 1

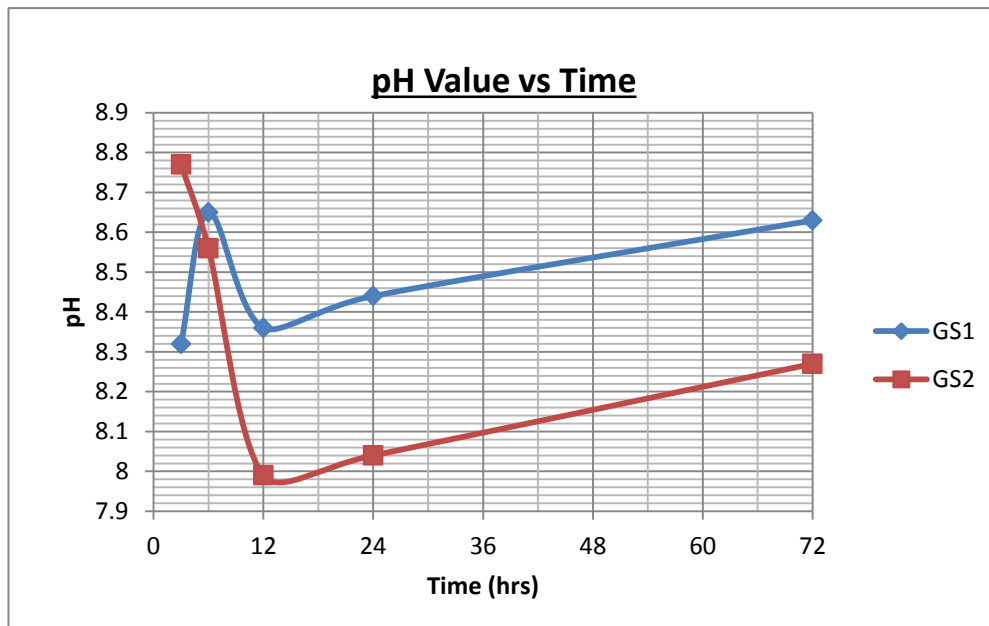


Figure 68: Mix Design 1 pH level vs Time

4.4.1.1 Mix Design 1 Discussion

From figure 68 it can be seen that the two samples have almost an identical geometry of the graph. As can be seen the pH level increases during the first time interval than decreases in the next and finally increasing again for both graphs. It can also be noted that GS1 with higher pH has greater compressive strength compared to GS2

4.4.2 Mix Design 2

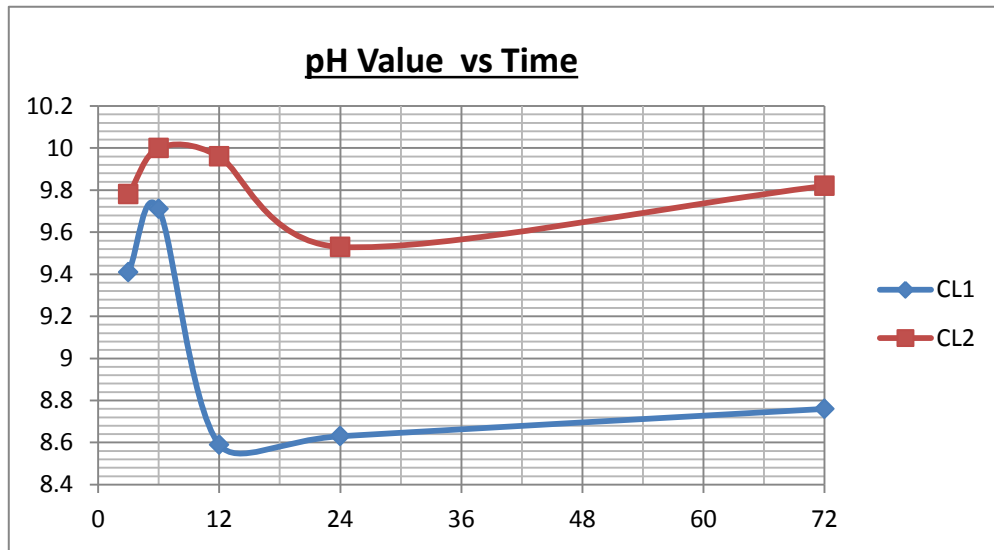


Figure 69: Mix Design 2 pH level vs Time

4.4.2.1 Mix Design 2 Discussion

Since this is an alkali activated geopolymer the graph from figure 68 can be seen to be in the range of alkali pH values of greater than 7. As can be seen the pH level increases during the first time interval than decreases in the next and finally increasing again for both graphs. It can also be noted that CL2 with higher pH has greater compressive strength when compared to CL1

4.4.3 Mix Design 3

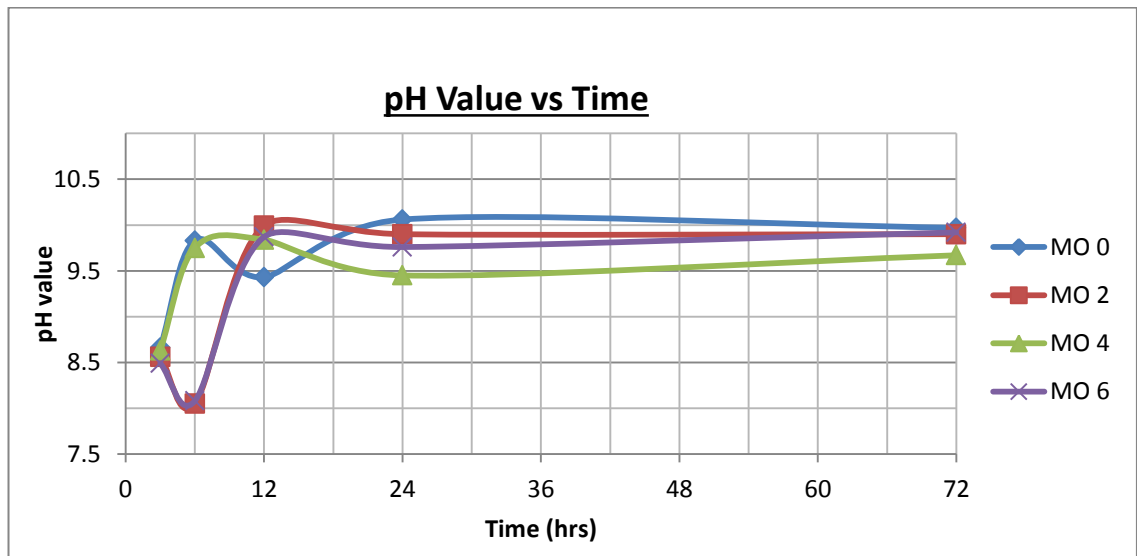


Figure 70: Mix Design 3 pH Level vs Time

4.4.3.1 Mix Design 3 Discussion

It is evident from figure 70 the material was alkali activated, this is evident in the fact that all the points plotted in the graph are all in the alkali range which is above 7. It can be seen that the graphs plotted for the sample to follow any distinct trend. It can be seen from figure 70 that MO-6 and MO-2 are almost identical while the two samples are distinct. It is also interesting to note that all samples almost converged at a point at time 72 hrs.

4.4.4 Mix Design 4

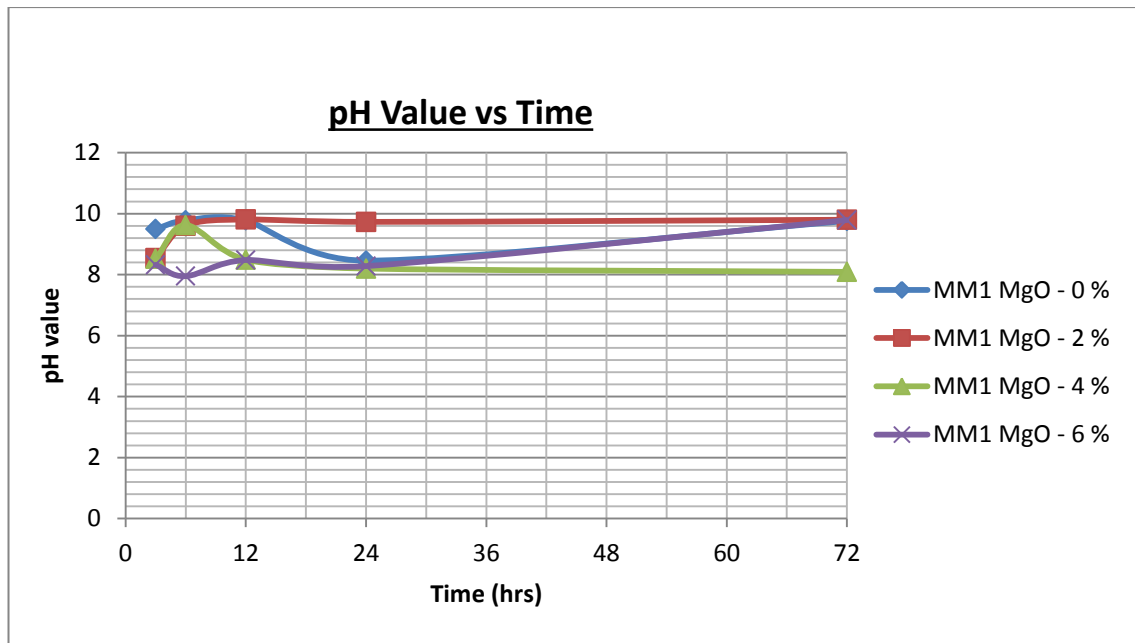


Figure 71: Mix Design 4 pH Level vs Time

4.4.4.2 Mix Design 4 Discussion

The figure above shows the pH level of the MM1-MgO-0 to MM1-MgO-6. In this mix design the additive is magnesium oxide which is added to the geopolymer at increments of 2% of the fly ash mass.

It can be seen from figure 71 that the alkali values at different time intervals are all between the pH of 9 and 10. This indicates that the solution is an alkali. As can be seen figure 71 MM1-MgO--0% and MM1-MgO--6% converges in exactly the same point.

4.4.5 Mix Design 5

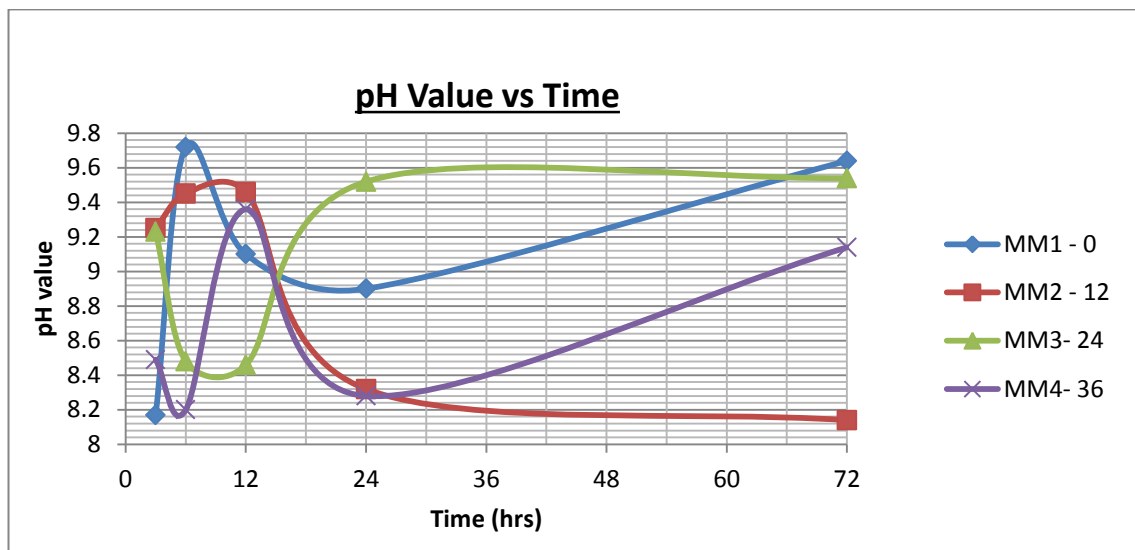


Figure 72: Mix Design 5 pH Level vs Time

The figure above shows the pH level of the MM1-0 to MM4-36. In this mix design the additive is potassium alumina sulphate is added to the geopolymers at increments of 2% to the fly ash mass. It can be seen from figure 71 that the alkali values at different time intervals are all between the pH of 9 and 10. This indicates that the solution is an alkali.

4.4.6 Mix Design 6

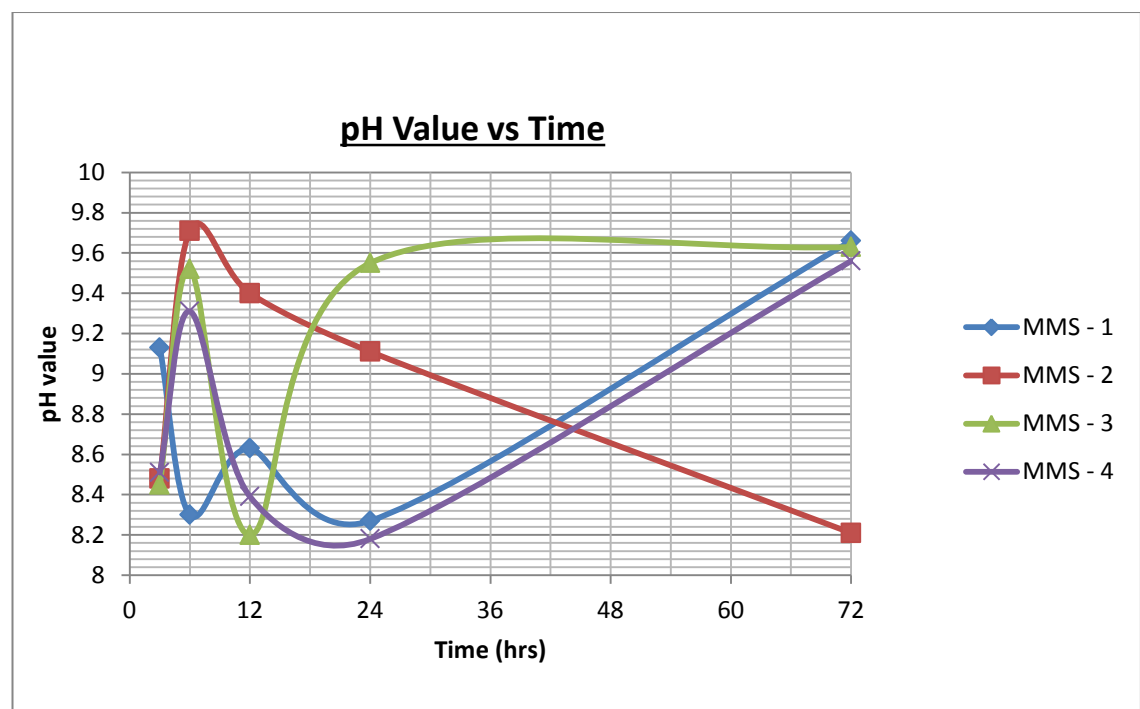


Figure 73: Mix Design 6 pH vs Time

4.4.6.1 Mix Design 6 Discussion

The figure above shows the pH level of the MMS-1 to MMS -4. In this mix design the additive is fine sand which is added to the geopolymer at increments of 10% of the fly ash mass. It should be noted that MMS -1, MMS-3, and MMS-4 are all converging near a single point. This is not only the case for this mix design but also for mix design 6, mix design 4 and mix design 3 all have three graphs converging near a point.

5.0 Conclusion and Future Work

The issue of OPC being harmful to the environment needs to be addressed. Geopolymers provide a greener and superior quality product that has benefits in that it helps in the reduction in global emissions, excellent chemical resistance (to acidic and alkaline substances), minimal drying shrinkage, cheaper and an abundance of fly ash found in Australia.

In this research project the efflorescence effect on the compressive strength, compressive modulus and leaching effect on alkali activated geopolymers was investigated. This was done on the various mix design that were cast. A total of 5 mix designs were implemented and had various additives which were added onto each mix design.

The study of efflorescence on alkali activated geopolymers is a relatively new concept and there have not been many studies carried out on the subject. In this research project efflorescence effect was simulated and its compressive strength was tested after 28 days and compared with the dry sample compressive strength. Along with this the compressive modulus was also obtained. From the investigation done it was found that efflorescence lowered the compressive strengths of various mix designs these included:

- Mix design 1: Gladstone 1
- Mix design 2: Gladstone 2
- Mix design 3: Callide 1
- Mix design 4: Callide 2
- Mix design 5: MM1-MgO

However for the rest of the various mix designs there was no conclusive evidence that showed efflorescence had a clear effect, this was because for each mix design had 5 specimens and at times the compressive strength of the efflorescence was greater than that of the dry specimen. The modulus of elasticity was shown to have a direct relationship with the compressive strength of the specimen, be it exposed to efflorescence or a dry specimen with no efflorescence. Strengths of the control or dry samples ranged between 53MPa to 4MPa while efflorescence exposed specimens ranged between 36MPa and 2.5MPa.

The leaching of all specimens was carried out. The graphs produced from the data were consistent with studies done by Xu & van Deventer, (2003). Most leaching curve followed the same trend. The pH of the leached specimen showed that the solution to

be alkali of having a pH greater than 7. This could be partially due to the use of the alkali solution to activate the geopolymer. All pH values obtained from the specimens ranged between 10 and 8.

The study of efflorescence effect on geopolymer should continue so that more data can be collected, processed and analysed. The specimens should be analysed using X-ray diffraction. In this way a better understanding of the composition and structure of each individual specimen can be achieved. Also more analysis should be done with other samples as geopolymer is a very broad topic.

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7.0 Appendix

Appendix A (table of leached specimens)

Sample Number	Sample Name	Time	Absorption	Corrected Data
Standard	0.5	n/a	n/a	
Standard	1	n/a	n/a	
Standard	2.5	n/a	n/a	
Standard	5	n/a	n/a	
Standard	10	n/a	n/a	
1	GS1	3	0.0336	21
2	GS1	6	0.1292	80.75
3	GS1	12	0.1667	104.1875
4	GS1	24	0.1952	122
5	GS1	72	0.2475	154.6875
6	GS2	3	0.0101	6.3125
7	GS2	6	0.0296	18.5
8	GS2	12	0.139	86.875
9	GS2	24	0.1384	86.5
10	GS2	72	0.2179	136.1875
11	CL1	3	0.2299	143.6875
12	CL1	6	0.3969	248.0625
13	CL1	12	0.379	236.875
14	CL1	24	0.3907	244.1875
15	CL1	72	0.4209	263.0625
16	CL2	3	0.2959	184.9375
17	CL2	6	0.386	241.25
18	CL2	12	0.4567	285.4375
19	CL2	24	0.4517	282.3125
20	CL2	72	0.4813	300.8125
21	MM1	3	0.4086	255.375
22	MM1	6	0.4478	279.875
23	MM1	12	0.4279	267.4375
24	MM1	24	0.4696	293.5
25	MM1	72	0.4989	311.8125
26	MM2	3	0.3826	239.125
27	MM2	6	0.4072	254.5
28	MM2	12	0.4173	260.8125
29	MM2	24	0.4304	269
30	MM2	72	0.4675	292.1875
31	MM3	3	0.2154	134.625
32	MM3	6	0.2962	185.125

33	MM3	12	0.316	197.5
34	MM3	24	0.3362	210.125
35	MM3	72	0.3625	226.5625
36	MO - 0	3	0.0344	21.5
37	MO - 0	6	0.3348	209.25
38	MO - 0	12	0.4293	268.3125
39	MO - 0	24	0.4559	284.9375
40	MO - 0	72	0.5381	336.3125
41	MO - 2	3	0.1404	87.75
42	MO - 2	6	0.3792	237
43	MO - 2	12	0.4657	291.0625
44	MO - 2	24	0.5166	322.875
45	MO - 2	72	0.5896	368.5
46	MO - 4	3	0.2509	156.8125
47	MO - 4	6	0.3932	245.75
48	MO - 4	12	0.4651	290.6875
49	MO - 4	24	0.4724	295.25
50	MO - 4	72	0.5289	330.5625
51	MO - 6	3	0.1913	119.5625
52	MO - 6	6	0.4025	251.5625
53	MO - 6	12	0.4531	283.1875
54	MO - 6	24	0.4531	283.1875
55	MO - 6	72	0.485	303.125
56	MM1-MgO - 0	3	0.1219	76.1875
57	MM1-MgO - 0	6	0.3253	203.3125
58	MM1-MgO - 0	12	0.4097	256.0625
59	MM1-MgO - 0	24	0.3608	225.5
60	MM1-MgO - 0	72	0.424	265
61	MM1-MgO - 2	3	0.2388	149.25
62	MM1-MgO - 2	6	0.3924	245.25
63	MM1-MgO - 2	12	0.4528	283
64	MM1-MgO - 2	24	0.4204	262.75
65	MM1-MgO - 2	72	0.448	280
66	MM1-MgO - 4	3	0.1323	82.6875
67	MM1-MgO - 4	6	0.2741	171.3125
68	MM1-MgO - 4	12	0.3068	191.75
69	MM1-MgO - 4	24	0.3009	188.0625
70	MM1-MgO - 4	72	0.3739	233.6875
71	MM1-MgO - 6	3	0.1133	70.8125
72	MM1-MgO - 6	6	0.2321	145.0625
73	MM1-MgO - 6	12	0.2825	176.5625
74	MM1-MgO - 6	24	0.3007	187.9375
75	MM1-MgO - 6	72	0.353	220.625
76	MM1-0	3	0.1728	108

77	MM1-0	6	0.2772	173.25
78	MM1-0	12	0.3334	208.375
79	MM1-0	24	0.3395	212.1875
80	MM1-0	72	0.3613	225.8125
81	MM2 -12	3	0.3166	197.875
82	MM2 -12	6	0.3362	210.125
83	MM2 -12	12	0.3815	238.4375
84	MM2 -12	24	0.3706	231.625
85	MM2 -12	72	0.3915	244.6875
86	MM3 -24	3	0.3622	226.375
87	MM3 -24	6	0.4159	259.9375
88	MM3 -24	12	0.4237	264.8125
89	MM3 -24	24	0.4125	257.8125
90	MM3 -24	72	0.4497	281.0625
91	MM4 -36	3	0.3102	193.875
92	MM4 -36	6	0.4782	298.875
93	MM4 -36	12	0.497	310.625
94	MM4 -36	24	0.4911	306.9375
95	MM4 -36	72	0.4911	306.9375
96	MMS - 1	3	0.1449	90.5625
97	MMS - 1	6	0.2802	175.125
98	MMS - 1	12	0.3446	215.375
99	MMS - 1	24	0.356	222.5
100	MMS - 1	72	0.3848	240.5
101	MMS - 2	3	0.1454	90.875
102	MMS - 2	6	0.2643	165.1875
103	MMS - 2	12	0.297	185.625
104	MMS - 2	24	0.309	193.125
105	MMS - 2	72	0.3488	218
106	MMS - 3	3	0.1133	70.8125
107	MMS - 3	6	0.245	153.125
108	MMS - 3	12	0.2607	162.9375
109	MMS - 3	24	0.3004	187.75
110	MMS - 3	72	0.3076	192.25
111	MMS - 4	3	0.1949	121.8125
112	MMS - 4	6	0.2948	184.25
113	MMS - 4	12	0.3264	204
114	MMS - 4	24	0.389	243.125
115	MMS - 4	72	0.4022	251.375

Appendix B (Table of pH data)

No#	Batch	3 hrs	6hrs	12hrs	36hrs	72hrs
1	GS1	8.32	8.65	8.36	8.44	8.63
2	GS2	8.77	8.56	7.99	8.04	8.27
3	CL1	9.41	9.71	8.59	8.63	8.76
4	CL2	9.78	10	9.96	9.53	9.82
5	MM1	8.96	9.91	10.02	9.81	8.32
6	MM2	9.73	9.81	9.85	9.86	9.73
7	MM3	9.27	8.76	8.41	8.18	9.47
8	MO 0	8.66	9.83	9.43	10.06	9.97
9	MO 2	8.56	8.05	9.992	9.9	9.9
10	MO 4	8.63	9.75	9.84	9.45	9.67
11	MO 6	8.49	8.08	9.87	9.76	9.92
12	MM1 MgO - 0 %	9.5	9.78	9.78	8.46	9.76
13	MM1 MgO - 2 %	8.54	9.6	9.81	9.73	9.8
14	MM1 MgO - 4 %	8.52	9.62	8.49	8.2	8.09
15	MM1 MgO - 6 %	8.32	7.95	8.48	8.28	9.79
16	MM1 - 0	8.17	9.72	9.1	8.9	9.64
17	MM2 - 12	9.25	9.45	9.46	8.32	8.14
18	MM3- 24	9.23	8.48	8.46	9.52	9.54
19	MM4- 36	8.49	8.2	9.36	8.28	9.14
20	MMS - 1	9.13	8.3	8.63	8.27	9.66
21	MMS - 2	8.48	9.71	9.4	9.11	8.21
22	MMS - 3	8.45	9.52	8.2	9.55	9.63
23	MMS - 4	8.51	9.31	8.39	8.18	9.56

Appendix C

Faculty of Engineering and Surveying

Courses ENG4111/112 Research Project Parts 1 & 2

Title: Effects of Efflorescence on the mechanical properties of geo-polymers Name: Savenaca Madanavosa (0061034013)

**Supervisors:
Dr Hao Wang**

Project

Description/Direction(s)/Information:

Alkali- activated geo-polymer provides a greener alternative to the use of Ordinary Portland Cement which emits harmful carbon dioxide gas into the atmosphere during production. This greener solution provides a material that can provide the same strength but also suffers the same problem of efflorescence as does its predecessor. Efflorescence in OPC concrete reduces the structural strength of the concrete. In this research project the efflorescence effect on the mechanical properties will be investigated on various samples.

The project sequence:

1. Literature review
2. Evaluate different alkali solutions on the effect of efflorescence and its mechanical properties.
 - a. Sodium Silicate, $M_s = 1.5$ ($\text{SiO}_2/\text{Na}_2\text{O} = 1.5$)
 - b. Sodium Hydroxide (12 Mol/L)
3. Evaluate the effects of fly ash sources on efflorescence and its mechanical properties
 - a. Tarong power station
 - b. Gladstone power station
 - c. Millmerran power station

only in sodium hydroxide
4. Evaluate the curing condition on efflorescences and mechanical properties
 - a. Room temperature (80°C)
 - b. Sealed container at room temperature (80°C)

At every step of the evaluation process the three samples will be taken and put into different containers after which will be tested for strength:

- a. Container 1, water just up to the base of the sample.
- b. Container 2, dry container with just sample (control)
- c. Container 3, sample submerged in water.

For all samples above pH, conductivity and leaching will be checked.

5. If time permits:
Try calcined red mud, slag and aluminates on efflorescence and its effects on the mechanical properties.
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