DISSERTATION

Study on the Effect of Fly Ash in Geopolymer Cement

by

Mohamed Saifullah Bin Sirajudin

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Petroleum Engineering)

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CERTIFICATION OF APPROVAL

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Petroleum Engineering Programme
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BACHELOR OF ENGINEERING (Hons)
(PETROLEUM ENGINEERING)

Approved by,

____
(Raja Rajeswary Suppiah)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2013

CERTIFICATION OF ORIGINALITY

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

MOHAMED SAIFULLAH BIN SIRAJUDIN 12661

Abstract

The utilization of by-product and solid waste of the industry in attempt as a replacement for the ordinary Portland cement (OPC) have been rapidly researched and investigated in recent years. The resultant product, also known as geopolymer cement, proves to be of significantly better than the OPC in various aspects. The quest for finding a replacement binder was mainly triggered by the great consumption of raw materials and disturbingly high emmision of CO2 in the production of OPC. Hence, the use of fly ash in geopolymer cement is duely investigated to find a more efficient binder in the casing annulus. Fly ash based geopolymer are environment friendly and are expected to solve many problems of disposing these by-products of coal at various landfills in the World. Fly ash is also much more resistant than OPC against chemical or acidic reaction and fire, have longer durability, higher pumpability and cost effective. This paper will present the work carried out to study the effect of fly ash in geopolymer cement. Fly ash is rich in alumina and silica, thus when dissolved in alkaline solution form an aluminosilicate gel that binds the elements together to form a good geopolymer cement. Fly ash is mixed with the alkaline solution, preferably NaOH, and Na_2SiO_3 solution to form a geopolymer paste, before which several tests done to investigate its advantages. Fly ash based geopolymer cement is an excellent alternative to OPC, with proven advantages.

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LIST OF ABBREVIATIONS

Forms	**Meaning**
NaOH	Sodium Hydroxide
Na_2SiO_3	Sodium Silicate
OPC	Ordinary Portland Cement
FA	Fly Ash
CaO	Calcium Oxide
M	Molarity
H_2O	Water

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Geopolymers or geopolymerization technology has been around for ages as a cementitious material, its usage as a pozzolan being tracked to the Great Pyramid of Egypt and Roman architectures. It was not until recently, that “geopolymers” have been reintroduced by Professor Joseph Davidovits (1978), triggered by the numerous catastrophic fires in France in early 1970s due to organic polymers or plastics. The geopolymer potrayed as an effective alternative to various current materials. We, as the topic underlines, will be looking at its effective binding properties as an alternative for cement.

The quest to find a “greener” or a more environment friendly cementitious material as a viable substitute for the ordinary Portland cement (OPC) has recently been initiated, mainly due to its inefficient production. High carbon dioxide (CO_2) emission leading to greenhouse effect, depletion of raw materials ie limestone, release of toxic gases leading to environment pollution, burning of large quantities of fuel are among others, the many adverse effect of the conventional cement production (Kong and Sanjayan 2008). These disadvantages of the OPC will be explained further in this paper.

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

There has been many research on the source of geopolymers especially on the utilization of the waste and by-products that contain silicates and aluminates that could undergo geopolymerization process (Khale and Chaudhary 2007). These research which are usually done by waste management groups and environmentalist are mostly concerned about the safe disposal of the huge amount or quantity of hazardous waste to the environment. These include studies on waste disposal such as fly ash, rice husk, palm font, and slag among others (Nuruddin et al. 2008)

These materials have so far been engineered to be used together with OPC in creating an improved version of the present cement. This has successfully developed a stronger and more durable concrete, more resistance to aggressive environment. Recent technologies includes up to 60% of cement replacement by fly ash known as High Volume Fly Ash (HVFA) without reducing the concrete performance (Malhotra and Mehta 2002). However, the production of OPC is still relatively high, amassing pollution and depleting raw materials from the Earth. Hence, the need to urgently find a complete replacement binder (Nuruddin et al. 2010).

This have led us to focus on the solution of using fly ash based geopolymer cement. Fly ash are the by-products from the combustion of coal (Khale and Chaudhary 2007). The content of aluminates and silicates in fly ash, as well as its properties make it an ideal geopolymerization base. Use of fly ash based geopolymer cement might bring great advantage over OPC, due to its dynamic functionality, environmental friendly and economically save application. Therefore, this research is focused on utilization of fly ash in geopolymer cement.

1.2 Problem Statement

Conserving energy and the environment have always been on the top of the list in the mission to attain a greener and healthier Earth. However, this is not the case with the current cement production. The production of one ton of Portland cement requires around 2.8 ton of raw materials, including fuel, water, lime and various other materials, generating 5-10% of dusts in the process (Khale and Chaudhary 2007). Furthermore, around the same amount of CO2 is produced during the process, making it an extremely resourceful and energy tedious process (Zongjin et al. 2002). There are many other disadvantages of using the OPC. They include concrete quality deterioration when exposed to aggressive environments, such as cracking and corrosion effect. It is also vulnerable to cracking, due to its volume contraction during drying. The drying shrinkage of concrete is directly influenced by the amount and the quality of the cement paste present The higher the water content and cement paste-to-aggregate ratio in the concrete mixture, the higher the shrinkage. Moreover, OPC production consumes large amount of water, due to the presence of electric charge on the surface that tends to form flocculants that trap volumes of the mixing water (Mehta 2002). Other weakness compared to fly ash based geopolymer includes very low early strength and reduced resistance to acid (Silverstrim et al. 1997). As 5-8% of human generated CO2 comes from concrete sector,it is obvious that production of Portland cement pose major environmental hazards. Therefore, in driving the World to adopt a greener cement alternative, the fly ash based geopolymer might be the suitable solution. It is hoped that by utilizing this waste also, we are able to save and conserve hundreds of thousands of acres currently used for landfilling these coal combustion products.

1.3 Objective and Scope of Study

The objectives of this study are:

- To study the properties of fly ash based geopolymer..
- To investigate the advantages and disadvantages of fly ash based geopolymer cement based on several conditions.

The scope of study includes:

- Conducting research on the theory and definition of terms related to the study.
- Conducting research on previous works and research done on the impact of fly ash usage in the industry.
- Conducting a research on the best conditions for the optimum production of fly ash based geopolymer.
- Establishing a laboratory procedure for conducting lab testing and experiments which includes compressive strength testing.

CHAPTER 2

LITERATURE REVIEW

2.0 Literature Review

This chapter focuses on the literature review concerning the development of fly ash based geopolymer. We will also go into details the conditions of the geopolymerization process in the following sections.

2.1 Introduction of Fly Ash

Fly ash are produced from the combustion of coal used for electricity generation. Therefore, fly ash are produced in large quantities, estimates amounting up to 780 million tons annually (Hardjito et al. 2004). They consist of finely divided ashes produced by burning pulverized coal in power stations (Khale and Chaudhary 2007). Most of these ash is disposed in landfills worldwide (Baldwin et al.). Unfortunately, more than 75% of this waste is disposed of in unmonitored onsite landfills and surface impoundments (Cambridge 2008). Landfilling, is not a desirable option since it not only causes huge financial burden to the foundries, but also makes them liable for future environmental costs and problems associated with landfilling regulations (Wiles 1988). Furthermore, these increasing loads of toxic metals in the landfill potentially increase the threat to ground water contamination (Khale and Chaudhary 2007). Hence, proper disposal and utilization of these ashes are urgently needed to preserve the ecosystem from severely or permanently damaged by the uncontrolled coal plant waste disposal (Nuruddin et al. 2010). Increasing economic factor also triggers the industry to look on recycling reuse of waste material, in the same time cheaply handling these large quantities of waste containing heavy metals as an alternatives to OPC, therefore triggering this research (Khale and Chaudhary 2007).

2.2 Types of Fly ash

Two classes of fly ash have been defined by ASTM C618: Class F fly ash and Class C fly ash. The main difference between these classes is the amount of calcium, silica, alumina, and iron content in the ash. These chemical properties of the fly ash are largely influenced by the chemical content of the coal burned (i.e., anthracite, bituminous, and lignite) (Cockrell and Leonard 1970)

2.2.1 Class C Fly ash

Class C fly ash are produced from the burning of younger lignite or subbituminous coal, in addition to having pozzolanic properties, they also have some self-cementing properties. Class C fly ash generally contains more than 20% lime or calcium oxide (CaO). Hence, they do not require an activator. Alkali and sulfate (SO_4) contents are generally higher in Class C fly ashes. In the presence of water, these type of fly ash will harden and gain strength over time (Halstead 1986).

2.2.2 Class F Fly ash

The burning of harder, older anthracite and bituminous coal typically produces Class F fly ash. This fly ash is pozzolanic in nature, and contains less than 20% lime or calcium oxide (CaO). Due to its pozzolanic properties, the glassy silica and alumina of Class F fly ash requires a cementing agent, such as Portland cement, quicklime, or hydrated lime, with the presence of water in order to react and produce cementitious compounds. Class F fly ash are typically used for geopolymerization. The addition of a chemical activator or high alkaline solution such as sodium silicate is required to induce the silicon and aluminium atom in this source for the formation of a geopolymeric paste (Halstead 1986).

2.3 Chemical Composition of Fly Ash

The chemical composition of fly ash depends on the mineral composition of the coal gangue (the inorganic part of the coal) .SiO_2, Al_2O_3, Fe_2O_3 and occasionally CaO are usually the main chemical components present in fly ashes. The iron content varies widely, and the alkalis are present in appreciable amount, with potassium prevailing over sodium (Hewlette 1998).

'Table 2.1 shows the chemical composition of Fly Ash

Component (%)	Bituminous	Subbituminous	Lignite
SiO_2	20-60	40-60	15-45
Al_2O_3	5-35	20-30	20-25
Fe_2O_3	10-40	4-10	4-15
CaO	1-12	5-30	15-40
LOI	0-15	0-3	0-5

2.4 Fly Ash based Geopolymer

2.4.1 Alkaline Activation Process (Geopolymerization)

Fly ash based geopolymer could be activated only by strong alkaline activators (geopolymerization process of fly ash). An alkaline liquid could be used to react with silicon (Si) and aluminum (Al) in a source material (in this case fly ash) of natural minerals or in by-product materials to produce binders (Davidovits 1994). The alkaline activation of materials can be defined as a chemical process that provides a rapid change of some specific structures, partially or totally amorphous, into compact cemented frameworks (Palomo et al.) Alkali activation of fly ash is a process that differs widely from Portland cement hydration and is very similar to the chemistry involved in the synthesis of large groups of zeolites. The most used alkaline activators are a mixture of sodium or potassium hydroxide (NaOH, KOH) and sodium silicate or potassium silicate (Rangan 2008).

The overall process of geopolymerization of fly ash could be described in four steps below (Xu et al. 2001).

1. The dissolution of aluminosilicate in alkaline environment occurs first. When aluminosilicate minerals are subjected to a high pH environment, the bonds between interlinked silicate and aluminate tetrahedral are broken,
2. The dissolved aluminium and silicon complexes diffuse from the solid aluminosilicate surface to the interparticle space,
3. A gel phase is formed, resulting from polymerisation between an added silicate solution and aluminium and silicon complexes,
4. The gel phase hardens due to the exclusion of spare water to form a Geopolymer product/ cement.

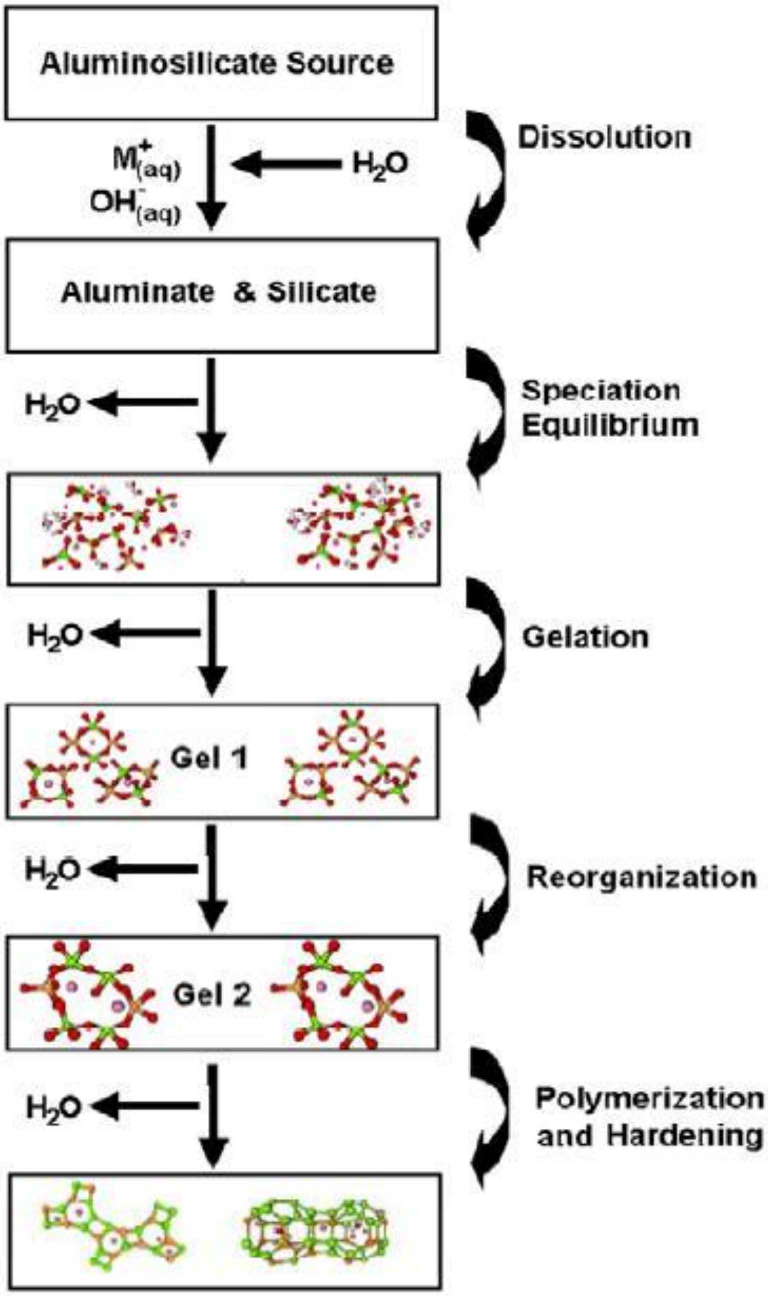


Figure 2.1. Conceptual Model of Alkaline Activation Process (Courtesy of Palomo 2011)

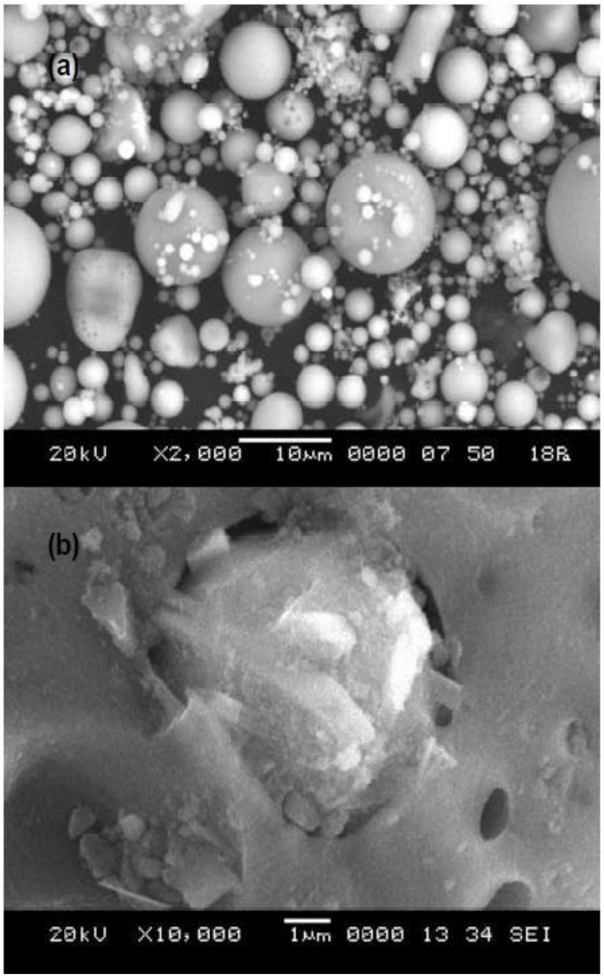


Figure 2.2 Scanning Electron Micrograph (SEM) image of (a) Fly Ash (b) Alkaline activated Fly Ash (Courtesy of Bakri 2011)

2.4.2 Mixing Process and the Factors affecting them

(Rattanasak et al. 2009) proposed two types of mixing – separate mixing and normal mixing-and mixed fly ash with NaOH solution to prepare a geopolymer paste. For separate mixing, the NaOH solution was mixed with fly ash for the first 10 minutes; a sodium silicate solution was subsequently added to the mixture. However, for normal mixing, fly ash, sodium hydroxide, and sodium silicate solution were mixed at the same time. From the results produced, separate mixing produced a slightly stronger mortar than normal mixing. Some authors believe that the optimum mixing order for alkali-activated binders is as follows. First, solids are mixed (fly ash and/or aggregates). The prepared activator is mixed with the solids, and the mixture is placed in molds (Swanepol and Strydom 2002). The samples in molds are then compacted in three layers of equal weight with standard compaction using a rod and vibrating table (Kong and Sanjayan 2010). Therefore, it is important to confirm whether the mixing order remains the same for other prime materials as well as how it is influenced by the type of alkaline activator (Jalali et al. 2008).

(Rattanasak and Chindaprasirt 2009) also proposed for separate mixing, fly ash was mixed with NaOH for 10 min to allow leaching of ions. Sodium silicate solution was then added to the mixture and mixed for 1 min. Only short mixing time was required here since the mixes were relatively fluid. For normal mixing (N), fly ash, sodium silicate solution, and 10 M NaOH were mixed together for 1 min as this corresponds to the time of exposure to sodium silicate solution for separate mixing.

2.4.3 Various Molarity of NaOH

The various molarity of NaOH is one of the factor taken into account. According to (Rangan 2008), mixing both solutions together at least 24 hours prior to use for the alkaline liquid is recommended. The use of the sodium silicate solution with an SiO2-to-Na2O ratio by mass of approximately 2 and sodium hydroxide with 97-98% purity is also recommended. The concentrations of the sodium hydroxide solution that can be used range from 8 to 16 M. (Al Bakri et al. 2011) found that the 12M NaOH solution gives the highest compressive strength. This result is supported by past studies (Palomo et al. 1999) that also found that a 12 M NaOH solution produced better results than the corresponding 18M NaOH solution. However, other researchers (Harditjo 2004) found that increasing NaOH molarity increases the compressive strength of the geopolymer. According to findings (Alonso and Palomo 2001) reported in such studies, when the activator concentration is above a 10 M NaOH solution, a lower rate of polymer formation is produced due to the high concentration of NaOH, resulting in a decreased strength. However, the study carried out by Tushar *et al*. stated that the highest compressive strength of geopolymer was affected when a 16 M NaOH solution is used.

2.4.4 Various Fly Ash / Alkaline activator ratio and Na_2SiO_3/NaOH ratio

Furthermore, the various ratio of fly ash/ alkaline activator ratio was also taken into consideration during mixing process. Previous researchers (Sathia et al. 2008) have stated that the compressive strength increases as the fly ash content and activator solution increase. Some researchers (Palomo et al. 1999) have stated that geopolymer fly ash with a fly ash/alkaline activator ratio of 3.3-4.0 can be used. However, other researchers (Sathia et al. 2008) have stated that the fly ash/alkaline activator ratio is not a relevant parameter influencing the compressive strength. Previous research (Rattanasak 2009) has concluded that the use of an Na_2SiO_3/NaOH ratio of 1.0 gives a strength of up to 70 MPa. One study indicated that the use of an Na_2SiO_3/NaOH ratio of 2.5 gives the highest compressive strength compared to the use of an Na_2SiO_3/NaOH ratio of 0.4. Sathonawaphak *et al* further stated that geopolymer with a fly ash/alkaline activator ratio of 1.4-2.3 showed a high compressive strength of 42-52 MPa. In addition, the optimum Na_2SiO_3/NaOH ratio was 1.5 while the maximum strength of 48MPa was obtained in their study. However, in their study, the highest compressive strength - up to 71 MPa - was observed at the 2.0 fly ash/alkaline activator ratio and 2.5 Na_2SiO_3/NaOH ratio.

2.4.5 Curing Process

The curing temperature is the most important factor for the geopolymer. When the curing temperature increases, the setting time of the concrete decreases (Chanh et al. 2008). During the curing process, the geopolymer concrete experiences the polymerization process. Due to the increased temperature, polymerization becomes more rapid, and the concrete can gain 70% of its strength within 3 to 4 hours of curing (Kong and Sanjayan 2008). Generally, heat-curing is recommended for flyash- based polymers. Rangan stated that heat curing (steam curing or dry curing) can assist in the chemical reaction that occurs in the geopolymer paste. The results demonstrate that the compressive strength of dry-cured geopolymer concrete is 15% higher than that of steam-cured geopolymer concrete. Nuruddin *et al* found that externally exposed curing is better than hot gunny and ambient curing. Both curing time and curing temperature influence the results for compressive strength of geopolymer concrete. A higher curing temperature results in a higher compressic strength. However, a curing temperature beyond 60ºC does not increase compressive strength (Rangan 2008). Our research found that the optimum curing temperature of 60ºC gives the highest compressive strength. Moreover, the 60ºC curing temperature was also recommended for manufacturing kaolinite and and fly ash geopolymer (Swanepol and Strydom 2002).

2.4.6 Compressive Strength

Compressive strength is an essential property for all concrete as it also depends on curing time and curing temperature. When the curing time and temperature increase, the compressive strength also increases. With curing temperatures ranging from 60ºC to 90ºC within a time ranging from 24 to 72 hours, the compressive strength of concrete can be obtained at approximately 400 to 500 kg/cm2 (Chanh et al. 2008). In addition, the compressive strength of geopolymers also depend primarily on the content of fine particles of fly ash (smaller than 43mm). The compressive strength increases as the finest of fly ash increases. Hence, the nature and concentration of activators are dominant factors in the reaction of alkali activation. The highest compressive strength was obtained using a solution of sodium silicate as an activator (n = 1.5; 10%Na_2O). Sodium silicate is most suitable as an alkaline activator since it contains dissolved and partially polymerized silicon, which reacts easily, incorporates into the reaction products, and significantly contributes to improving the mortar characteristics (Komljenovic et al. 2010).

The most significant factor controlling the compressive strength is pH. The setting time of cement decreased as the pH of the activating solution increased (Khale and Chaudhary 2007). At lower pH values the geopolymeric mix remained viscous and behaves like cement while at higher pH, the mix attained a more fluid gel composition, which was less viscous and is more workable (Phair and Deventer 2001). Strength at pH 14 was 50 times larger than those at pH 12 (less than 10 MPa at pH 12, 50 MPa at pH 14) of geopolymeric matrix utilizing cement as setting additive. Higher solubility of monomers was expected by KOH than NaOH because of higher alkalinity (Fig. 3). With increasing pH there was a predominance of smaller chain oligomers and monomeric silicate available to react with soluble aluminum. Further with increase in pH soluble aluminum increases and reacts with calcium available for reaction (Phair and Deventer

2001). Lower pH-value of the solution leads to lower monomer concentration. Figure 3 reveals the pH-value of the single alkaline solution, varying in concentration and kind of alkali ions (Kaps and Buchwald 2002). From the above observations it is clear that pH range 13–14 is most suitable for the formation of the geopolymers with better mechanical strength.

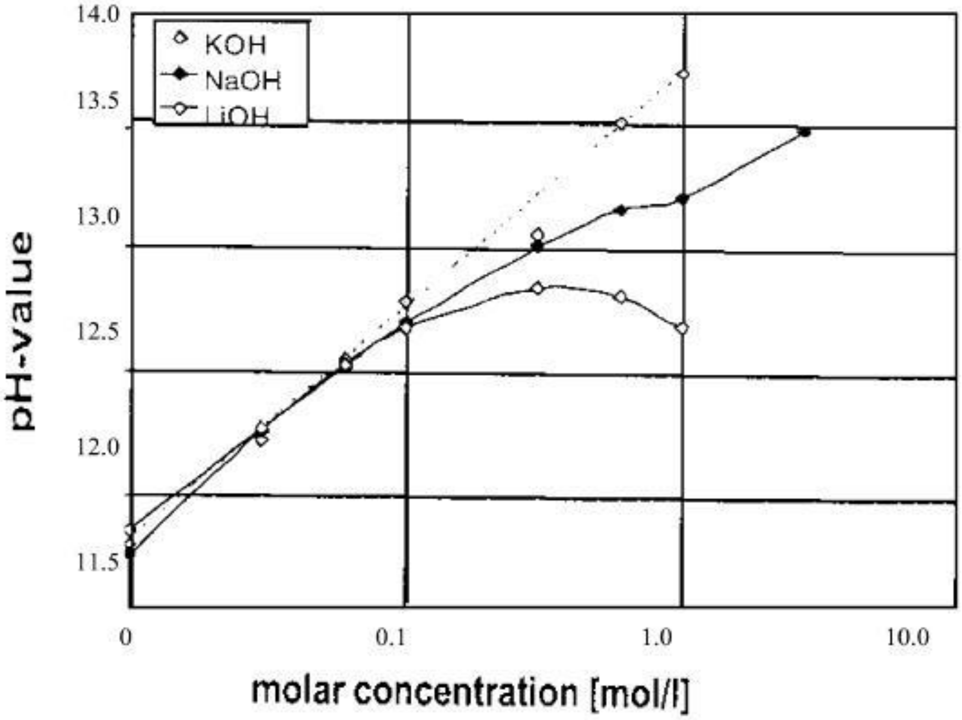


Figure 2.3. Influence of alkaline concentration on the pH value (Courtesy of Khale and Chaudhary 2007)

2.4.7 Effect of water and superplasticizer

Water plays as an important role in geopolymer concrete as it does in normal concrete. Water is used in geopolymer to improve the workability, but it increases the porosity in concrete due to the evaporation of water during the curing process at the elevated temperature (Sathia et al. 2008). Chindaprasirt *et al.* discovered that an increase in sodium hydroxide and sodium silicate concentration reduces the flow of mortar.

The workable flow of geopolymer mortar occurred in the 110+-5 to 135+-5% range. To improve the workability of mortar, superplasticiser or extra water can be added. However, the use of superplasticiser has an adverse effect on the strength of geopolymer. As such, extra water gives greater strength than the addition of superplasticiser. Reddy *et al* stated that, as the molarity of the NaOH solution increases, the workability of concrete decreases.

2.5 Advantages of Fly Ash based Geopolymer

Geopolymerization is an emerging technology for utilization of by-products like fly ash, which provides a mature and cost-effective solution to many problems where hazardous residues must be treated and stored under critical environmental conditions. Geopolymer based materials are environmentally friendly and need only moderate energy to produce. CO2 emission is reduced about 80% compared to that of ordinary Portland cement, hence making it a more feasible alternatives to Portland cement (Khale and Chaudhary 2007).

2.5.1 Lower Greenhouse Effect

The manufacture of Portland cement is always accompanied by the emission of CO2. The production of one tonne of Portland cement directly generates 0.55 tonnes of CO2 and yields an additional 0.40 tonnes due to the combustion of carbon-fuel.(Song 2007)

To simplify:

1 tonne of Portland cement = 1 tonne of CO2 (Davidovits 1994).

Portland cement manufacture is becoming less acceptable, due to the growing concerns related to resource use, biodiversity preservation and global climate change. A BaU scenario (Business-As-Usual) predicted (Davidovits 1994) that the world Portland cement requirement in 2015 would be 3500 million tonnes. If the CO2 emission were capped at 1990 levels, then only 1850 million tonnes of blended cement (the sum of Portland cement, slag and fly ash in Figure 4 could be made available to meet world demand. The remaining 1650 million tonnes would have to depend on new low-CO2 cementitious materials (Song 2007).

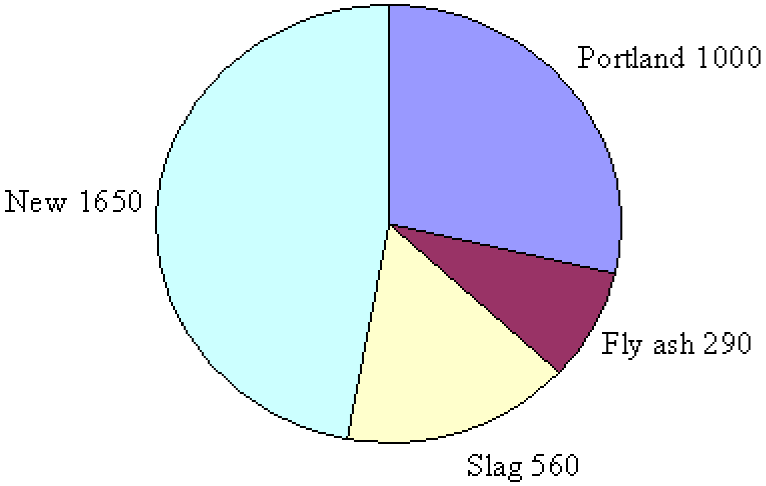


Figure 2.4. Distribution of BaU world cement in 2015 (Unit: million tonnes) (Courtesy of Song 2007)

Geopolymer has been targeted as one type of potential low-CO2 cementing system (Gartner 2004). When an industrial by-product, like fly ash, is used, then no supplementary CO2 will be released. Moreover, although the manufacture of the activator itself (i.e. alkali silicate) will have CO2 emission similar to the making of ordinary "bottle" glass, the activator addition in a Geopolymer concrete mix is very low. So in terms of CO2 emission per unit volume of concrete, the total CO2 emission level in Geopolymer concrete is theoretical 10 times lower than pure Portland cement concrete (Gartner 2004). Given the relatively low greenhouse intensity of Geopolymer in producing cementitious materials, it is a potential environmentally friendly binder for building materials (Spannagle 2002).

2.5.2 High Resistance to Aggressive Environment (Chemical/ Acidic surrounding)

Concrete durability is becoming critical issues for the future (Mehta 1997) because many concrete structures in urban and coastal environment start to deteriorate in 20 to 30 years, though their design life is at least 50 years. By contrast, the Pyramids in Egypt remain unaffected displaying extreme durability. It is found that the long-term durability in those ancient structures lies in the silico-aluminosilicate structure (Davidovits 1987).

Fly ash-based geopolymer has been shown by many studies to provide better resistance to an aggressive environment. As such, this advantage can be used to construct a structure exposed to a marine environment (Chanh et al.2008). The exposure of the geopolymer in the acid solution shows that the weight loss due to the exposure is only 0.5% compared to normal concrete when immersed in 3% sulfuric acid (Sathia et al. 2008). According to Bakharev, in acidic exposure, high-performance geopolymer materials deteriorate with the formation of fissures in an amorphous polymer matrix whereas low performance geopolymers deteriorate through the crystallization of zeolites and the formation of fragile grainy structures. The formation of aluminosilicate gel is important for determining the stability of the geopolymer. Crystalline geopolymer material prepared with sodium hydroxide is more stable in the aggressive environment of sulfuric and acetic acid solutions than amorphous geopolymers prepared with a sodiumsilicate activator.

Thokchom *et al* exposed the geopolymer mortar to 10% sulfuric acid and found specimens that were still intact and did not show any recognizable change in color after 18 weeks. When observed under an optical microscope, the exposed surface revealed a corroded structure that progressed with exposure over time. In addition, the weight loss results obtained in this study showed better performance than OPC; specimens with a higher alkali content were observed to lose more weight than specimens with a lower alkali content. At 18 weeks, the specimens were fully dealkalized by the sulfuric acid, but they still had substantial residual compressive strength to prove the higher resistance against acid exposure (Thokchom et al. 2009).

2.5.3 Excellent Fire Resistance

The fire resistance is another beneficial characteristic of Geopolymer. There were various catastrophic fires in France in 1970/73. The serious consequence pushed the government to investigate a new fire resistant binder. Geopolymer was the result of this research (Davidovits 1991). The fusion temperature for Geopolymer is 1050-1250°C (Davidovits and Davidovics 1998). Such a property makes Geopolymer promising in tunnels and high-rise structures as a fire-resistant material.

Cheng and Chin reported the fire resistance property of geopolymers. When a 10 mm thick panel of geopolymer is exposed to 1100°C flame; the measured reverse-side temperature reached 240–283°C after 35 min. Authors also observed that the fire characteristics could be improved by increasing the KOH or the alkali concentration and amount of metakaolin. It was concluded that geopolymers could be fabricated for construction purpose and have great potential for engineering application.

2.5.4 Summary of Geopolymer's Advantages

Wide advantages of the applications of geopolymers could be listed as follows (Khale and Chaudhary 2007):

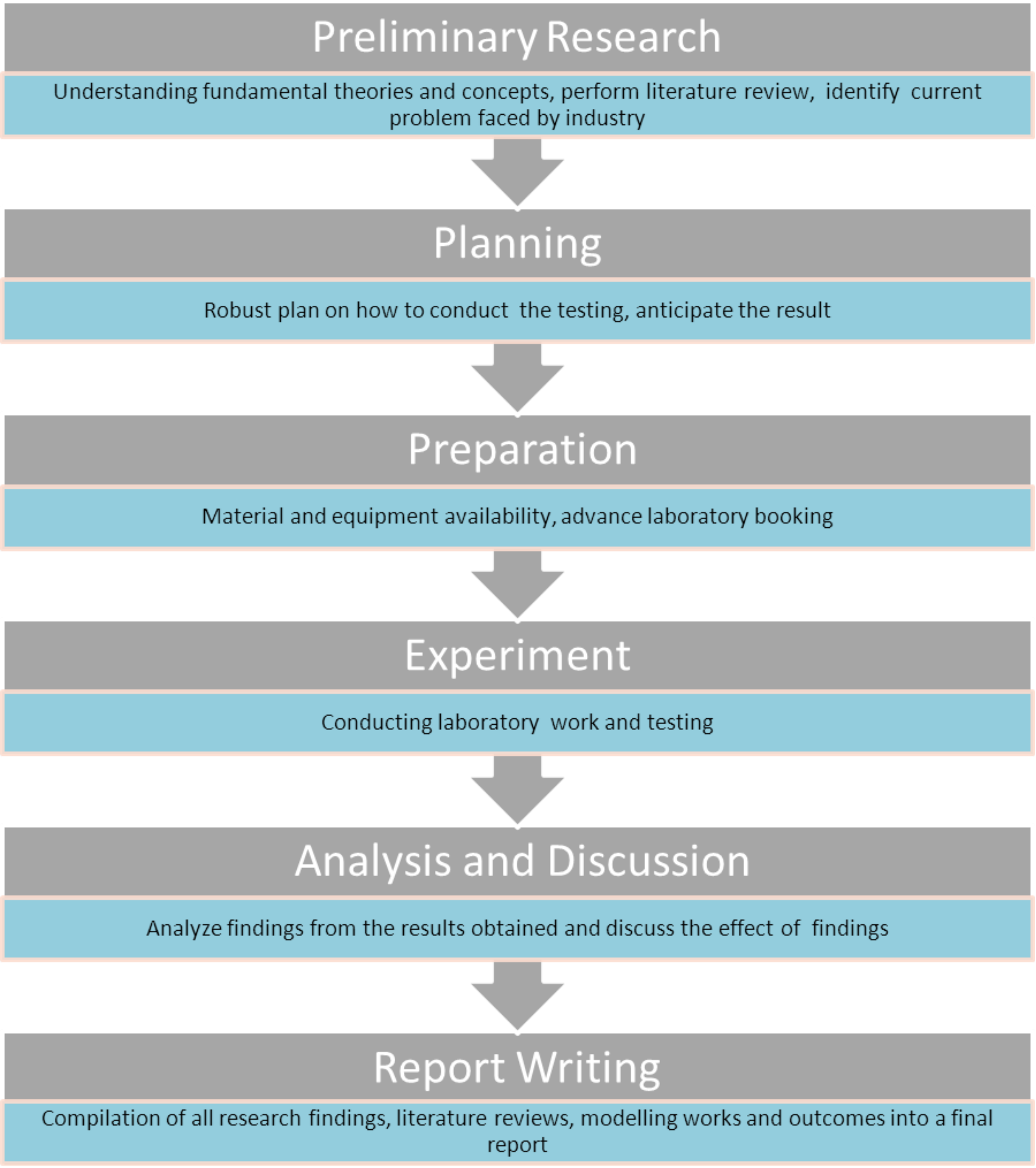
1) Geopolymers possess excellent mechanical strength due to high degree of Polycondensation.
2) Long-term durability: Geopolymer concrete or mortars withdraw thousands of years weathering attack without much function loss.
3) Unique high temperature properties.
4) Easily recycled, adjustable coefficient of thermal expansion.

5) Hazardous waste disposal binder for the heavy metal fixation especially for nuclear waste solidification.
6) Fire resistant: Geopolymer can withstand 1000°C to 1200°C without losing function.
7) It is also known as ‘‘Green material’’ for its low energy consumption and low waste gas emission during manufacture. Thermal processing of natural alumino-silicates at relative low temperature provides suitable geopolymeric raw material, resulting in 3/5 less energy assumption than Portland cement. In addition less CO2 is emitted (Hardjito et al. 2004).
8) Fast setting: Geopolymer obtain 70% of the final compressive strength in the first 4 h of setting.

CHAPTER 3

METHODOLOGY

3.1 Project Methodology



3.2 Key Milestone

Table 3.2. Key Milestone for Project

January 2013 Semester		
Week	**Date**	**Objective**
1	14 January - 18 January	
2	21 January - 25 January	
3	28 January - 1 February	
4	4 February - 8 February	
5	11 February - 15 February	Completion of Preliminary Research Work
6	18 February - 22 February	
7	25 February - 1 March	1) Submission of Extended Proposal 2) Mid Semester Break (28 February - 3 March)
8	4 March - 8 March	
9	11 March - 15 March	Completion of Proposal Defense
10	18 March - 22 March	
11	25 March - 29 March	
12	1 April - 5 April	
13	8 April - 12 April	Submission of Interim Draft Report
14	15 April - 19 April	Submission of Interim Report
15	22 April - 26 April	Examination Week (25 April - 5 May)
16	29 April - 3 May	
17	6 May - 10 May	End of Semester Break (6 May - 12 May)

July 2013 Semester		
Week	**Date**	**Note**
1	13 May - 17 May	
2	20 May - 24 May	
3	27 May - 31 May	
4	3 June - 7 June	
5	10 June - 14 June	
6	17 June - 21 June	
7	24 June - 28 June	1) Submission of Progress Report 2) Mid Semester Break (27 June - 30 June)
8	1 July - 5 July	
9	8 July - 12 July	
10	15 July - 19 July	Pre-SEDEX
11	22 July - 26 July	Submission of Draft Report
12	29 July - 2 August	1) Submission of Dissertation (Soft bound) 2) Submission of Technical Paper
13	5 August - 9 August	Oral Presentation
14	12 August - 16 August	
15	19 August - 23 August	1) Submission of Project Dissertation (Hard Bound) 2)Examination Week (22 August - 1 September)
16	26 August - 30 August	
17	2 September - 6 September	End of Semester Break (2 September - 8 September)

3.3 Gantt Chart

Table 3. Proposed Gantt chart for the project implementation for both FYP I and FYP II.

PROJECT ACTIVITIES	WEEKS																												
	Final Year Project 1															Final Year Project 2													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Project Scope Validation	■	■	■																										
Project Introduction			■	■	■	■																							
Submission of Extended Proposal							■																						
Identify material and equipment							■	■																					
Learning on how to conduct experiment							■	■																					
Proposal Defence								■	■																				
Conduct experiment									■	■	■	■	■	■															
Submission of Interim Report (Draft & Final)													■	■															
Result analysis and discussion															■	■	■	■	■	■									
Submission of progress report																					■								
Preparation for Pre-SEDEX																					■	■	■						
Pre-SEDEX																								■					
Submission of draft report																									■				
Submission of technical paper and dissertation																										■			
Oral presentation																											■		
Submission of project dissertation Hard bound																													■

Proposed Gant chart for the project implementation for both FYP I and FYP II. Based on the Gant Chart, the project is feasible to be completed within the given amount of time.

3.4 Materials and Testing Procedure

3.4.1 Materials used in the experiment;

1. 2 types of Fly ash. (Type I obtained from Charcoal factory in Kuala Sepetang, Taiping, consists of unsieved raw fly ash. Type II obtained from the Lafarge Cement, Klang, Selangor. Consisting of very fine and sieved Class F fly ash).
2. Alkaline activator
   - Sodium Hydroxide Solution of 12M and 15M.
   - Sodium Silicate Solution
3. Water

3.4.2 Testing Procedure

Table 3.41 shows the method conducted for each objective

Objectives	Experiment Method
To study the properties of fly ash based geopolymer.	Prepare geopolymer paste / cement sample of different variables
To investigate the advantages and disadvantages of fly ash based geopolymer cement based on several conditions	Test for compressive strength of these various geopolymer samples on different curing times

3.4.2.1 Preparation of geopolymer paste of different variables to study the properties of fly ash based geopolymer.

Since we are going for normal mixing…

1. The acquired fly ash is dried in the oven and then sieved according to ASTM C136 standard.
2. Prepare a suitable amount of sieved Fly Ash that would fill 3x50 mm^3 cement moulds, in this case, we have fixed it at 200g per 50 mm^3 mould.
3. For the ratio of fly ash / alkaline solution of 2.5 and sodium silicate (Na2Sio3) / sodium hydroxide NaOH of 2.5, or in other words ratio of fly ash to alkaline

activator of 1:0.4 and sodium silicate (Na2Sio3) to sodium hydroxide NaOH solution of 1:0.4, the following values of alkaline solutions are used.

4. Prepare 22.86g* of 12M sodium hydroxide NaOH solution and 57.14g* of sodium silicate solution Na_2Sio_3 to obtain a total of 80g* of alkaline activator solution per mould.
5. Mix the Fly Ash with the NaOH and Na_2Sio_3 solution together for 3 minute in the in the constant speed mixer at 4000 rpm.
6. Observe and stir the mixture manually to check if the mixture has evolved into a paste. If it has not, prepare to add water.
7. Add a little water, in this case 10% of water (20g) with fly ash weight as reference. Then mix the mixture again in the constant speed mixer for 1 minute at 4000rpm.
8. Observe and stir the mixture again to check if the mixture has evolved into a paste. Step 6 and 7 are repeated until we obtain a geopolymer paste.
9. Fill up the cement mould with the geopolymer paste.
10. Repeat steps 2 to 9 for the other two geopolymer cube samples to obtain three samples per trial for an average calculation.
11. The moulds are then placed in the oven for curing at 60˚C for 24 hours.
12. After 24 hours, the moulds should then be taken out from the oven for exposed curing for 3 days. Steps 2 to 12 are repeated to obtain for 7 days and 14 days curing time.
13. Repeat steps 2 to 12 for the 15M NaOH solution, for the different water concentration, different type of fly ash and also different ratio of fly ash / alkaline solution.

* The value changes according to the ratio of fly ash / alkaline solution used. The values for 200g of fly ash per mould are tabulated as follows. Note that the optimum ratio of sodium silicate to sodium hydroxide is used throughout this experiment.

Table 3.42 shows the weight of NaOH and Na_2SiO_3 that is calculated based on different fly ash / alkaline ratio

Ratio of Fly Ash to Alkaline Solution	Ratio of Sodium Silicate to Sodium Hydroxide	Weight of Sodium Hydroxide	Weight of Sodium Silicate
1:0.4	1:2.5	22.86g	57.14g
1:0.5	1:2.5	28.57g	71.43g
1:1	1:2.5	57.14g	142.86g

Figure 3.1 shows the oven used for drying the fly ash before sieving.

Figure 3.2 shows the remnants wood from the sieved fly ash in the sieving cylindrical pan and accumulated fine fly ash in the black plastic bag on the top.

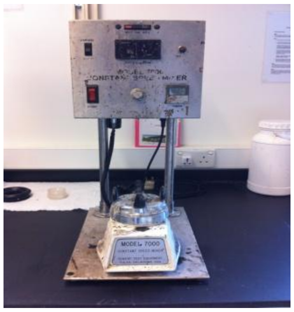


Figure 3.3 shows the constant speed mixer used in the experiment.

3.4.2.2 <u>Test for compressive strength</u>

1. Place the cured cube sample in the compressive digital testing machine. Make sure the adjustable surface above the sample is evenly touched and adjust the nut tightly.
2. Switch on the pump by pressing the ‘pump on’ button on the equipment software.
3. Apply load uniformly until the mould fails. This is done by pressing the ‘start testing’ button on the software. Do not release the mouse press until the cube fails. The results are recorded automatically on the software.
4. Repeat the steps for the other 2 sample. The average value from these three samples are taken as the final compressive strength.

Figure 3.4 shows the strength tester equipment used.

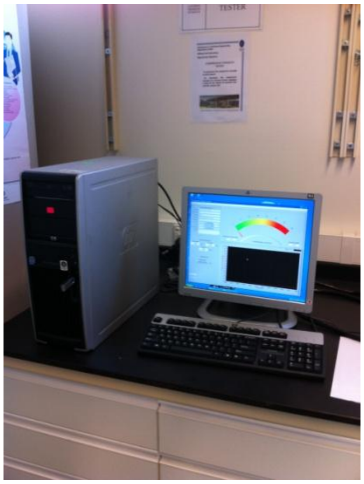


Figure 3.5 shows the computer software attached to the compressive strength tester.

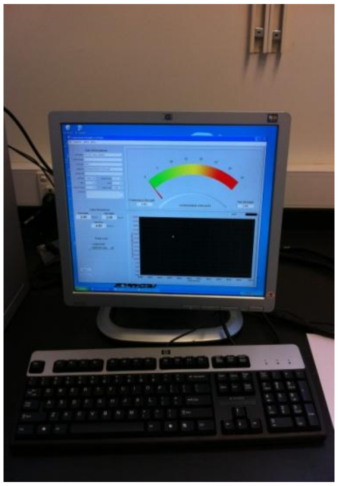


Figure 3.6 shows close-up on the software. The 'Pump On' and 'Start Testing' button situated bottom left hand corner.

Figure 3.7 shows the geopolymer paste soon after mixing and nicely slotted into the cube moulds.

Figure 3.8 shows another mould ready to be cured in the oven for 24 hours.

Figure 3.9 shows another sample of the triple cube mould.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Investigating the properties of fly ash geopolymer of different variables and identifying its advantages and disadvantages effect on the geopolymer cement.

The experiment was conducted in labs in Block 14 and 15, using several lab equipments such as compressive strength tester, the sieving machine, and the constant speed mixer. The properties of geopolymer cement of different variables were observed during the experiment. It is found out during the experiment that using different types of fly ash have a profound effect on the results, resulting in major difference in the outcome of the experiment compared to other variables. Two types of fly ash variable was used, type I was obtained from a charcoal factory in Kuala Sepetang, Taiping. The second type, the type II fly ash was obtained from Lafarge Cement, Klang, Selangor. The other variables include, different curing times for both types of fly ash, different molarity for type I fly ash, and different ratio of fly ash to alkaline solution for the type II fly ash. We will also compare the two types of fly ash, by keeping all other variables constant. From here, we will observe their differences, and have a detailed look from the results of their compressive strengths.

Figure 4.00 shows the side view of
the cube sample after curing.

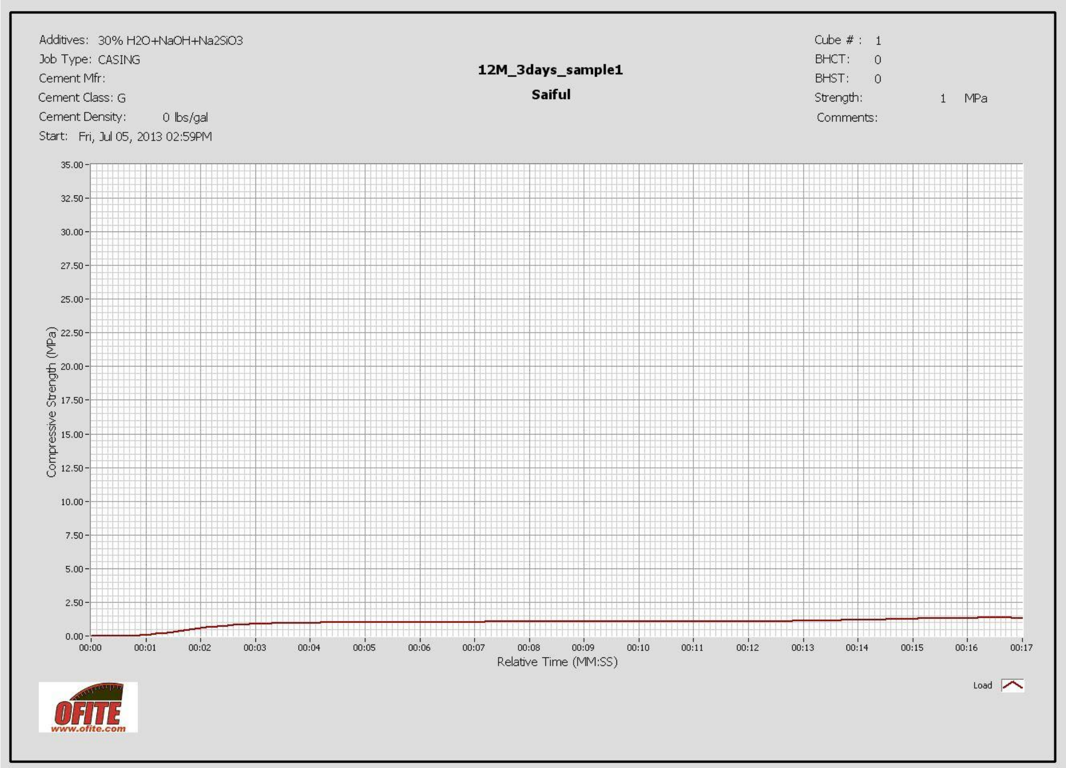
4.2 Type I Fly Ash

For the Type I fly ash, it is observed that the mix required addition of water during the mixing process to evolve into a workable paste. Hence, the concentration of water was manipulated to give us extra variety of result. For the initial run, 30% of water was added during the mixing process, with fly ash weight as reference. The variable was molarity of NaOH, with 12M and 15M variant and different curing times, of 3, 7 and 14 days. For the succeeding experiment, we reduced the water concentration to a minimum of 25% during the mix, below which we will not obtain a workable paste. The outcome of this two water concentration was compared based on their compressive strength on different curing times.

4.2.1 Compressive Test Results for 30% H_2O Concentration

Attached are the graph results from the compressive strength obtained for the 30% H_2O concentration variant. It is observed that, in this variant, the paste are more workable than those of 25% H_2O variant. However, the higher water concentration compensates for a lower strength, than the reduced water variant. We will discuss futher in detail in the succeeding sections.

i) 12M + 3 Days Sample 1



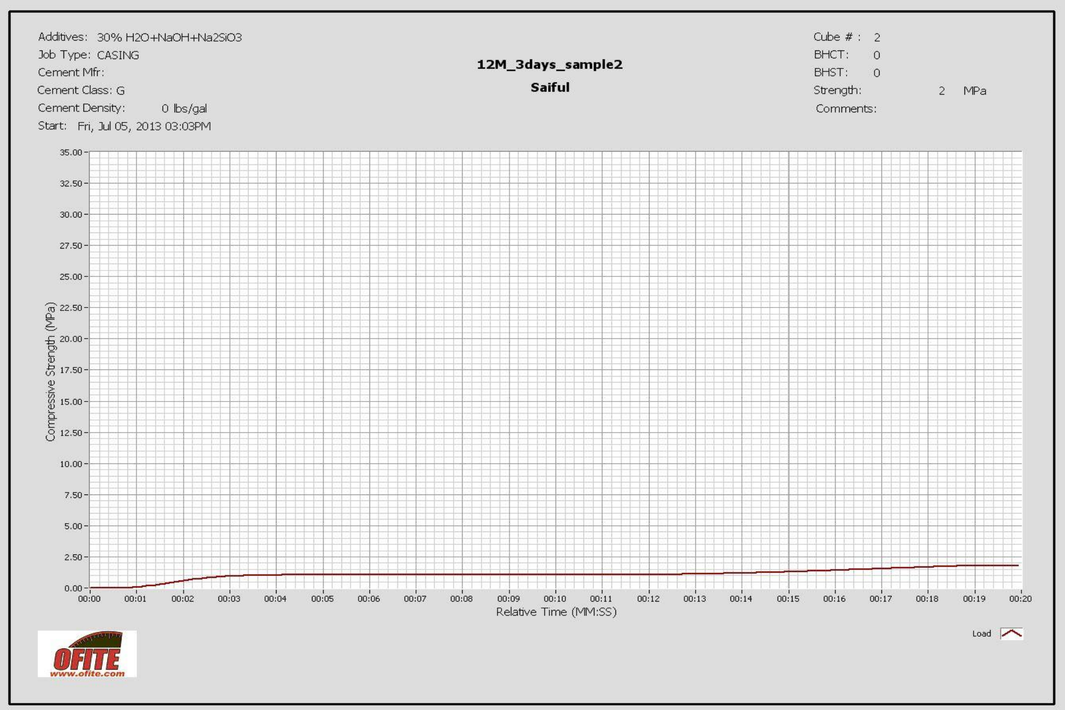
Additives: 30% H2O+NaOH+Na2SiO3
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Fri, Jul 05, 2013 02:59PM

12M_3days_sample1
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 1 MPa
Comments:

Figure 4.01 shows the compressive strength result for the 12M NaOH variant @ 30% added water concentration and 3 days curing time, for cube sample 1. The maximum compressive strength was achieved at 1.35 MPa.

ii) 12M + 3 Days Sample 2



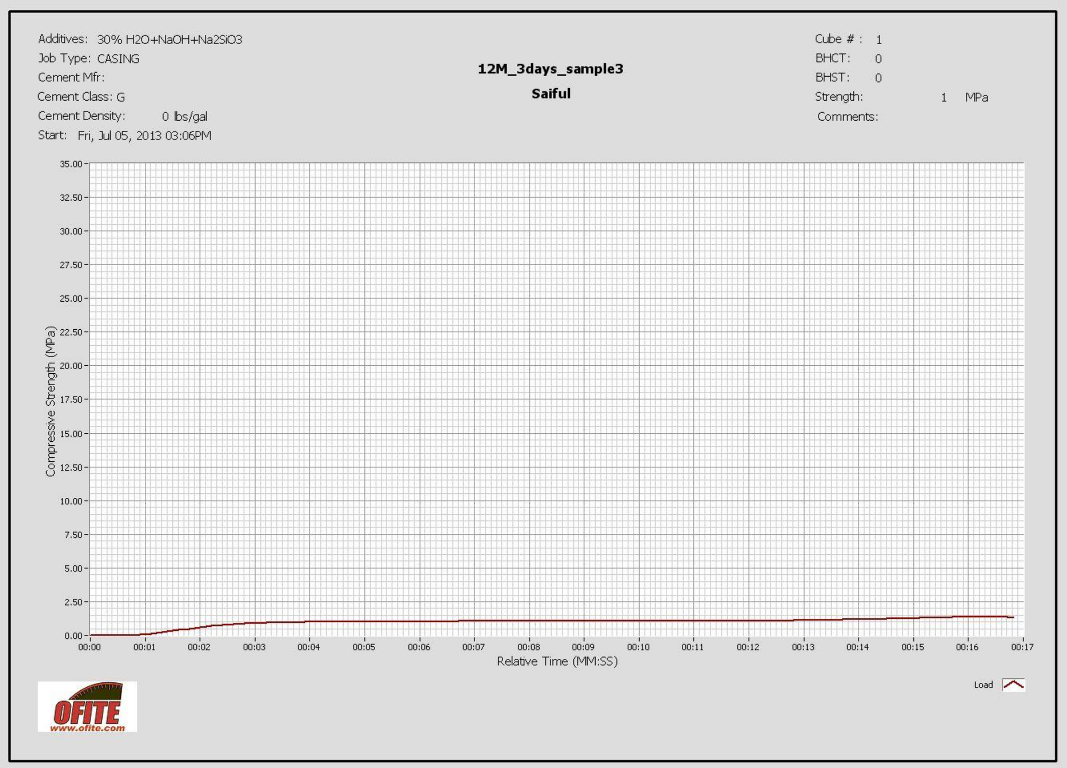
Additives: 30% H2O+NaOH+Na2SiO3
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Fri, Jul 05, 2013 03:03PM

12M_3days_sample2
Saiful

Cube # : 2
BHCT: 0
BHST: 0
Strength: 2 MPa
Comments:

Figure 4.02 shows the compressive strength result for the 12M NaOH variant @ 30% added water concentration and 3 days curing time, for cube sample 2. The maximum compressive strength was achieved at 1.81 MPa.

iii) 12M + 3 Days Sample 3



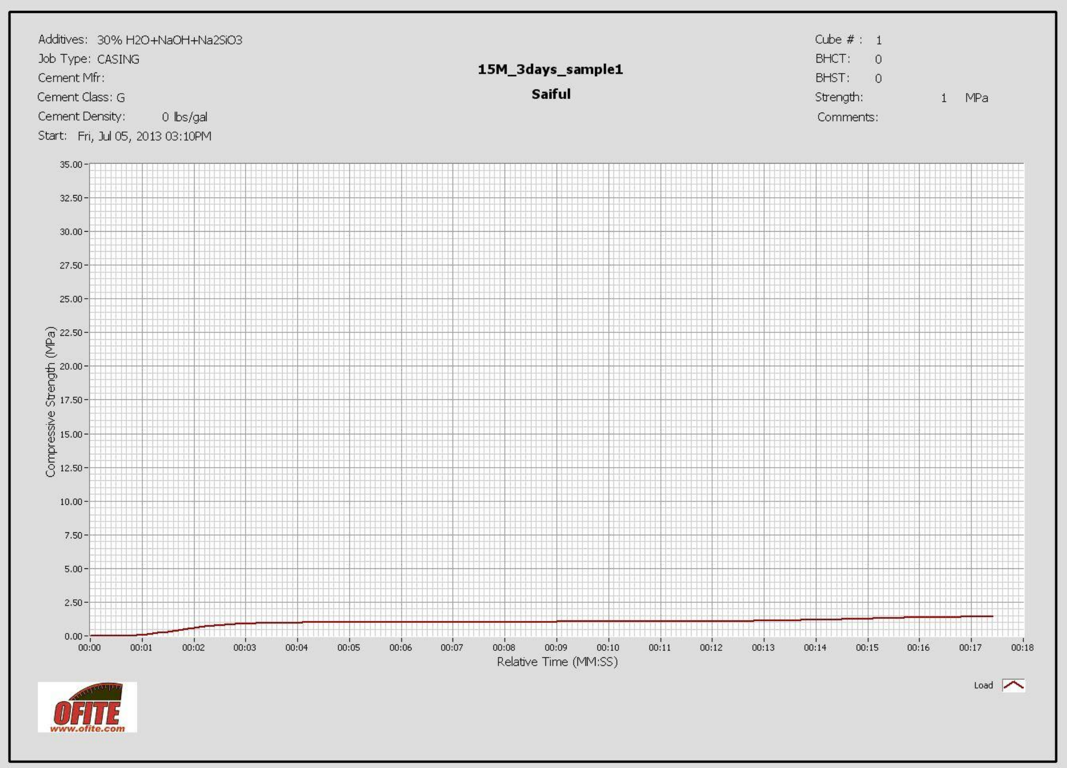
Additives: 30% H2O+NaOH+Na2SiO3
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Fri, Jul 05, 2013 03:06PM

12M_3days_sample3
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 1 MPa
Comments:

Figure 4.03 shows the compressive strength result for the 12M NaOH variant @ 30% added water concentration and 3 days curing time, for cube sample 3. The maximum compressive strength was achieved at 1.38 MPa.

iv) 15M + 3 Days Sample 1


Additives: 30% H2O+NaOH+Na2SiO3
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Fri, Jul 05, 2013 03:10PM

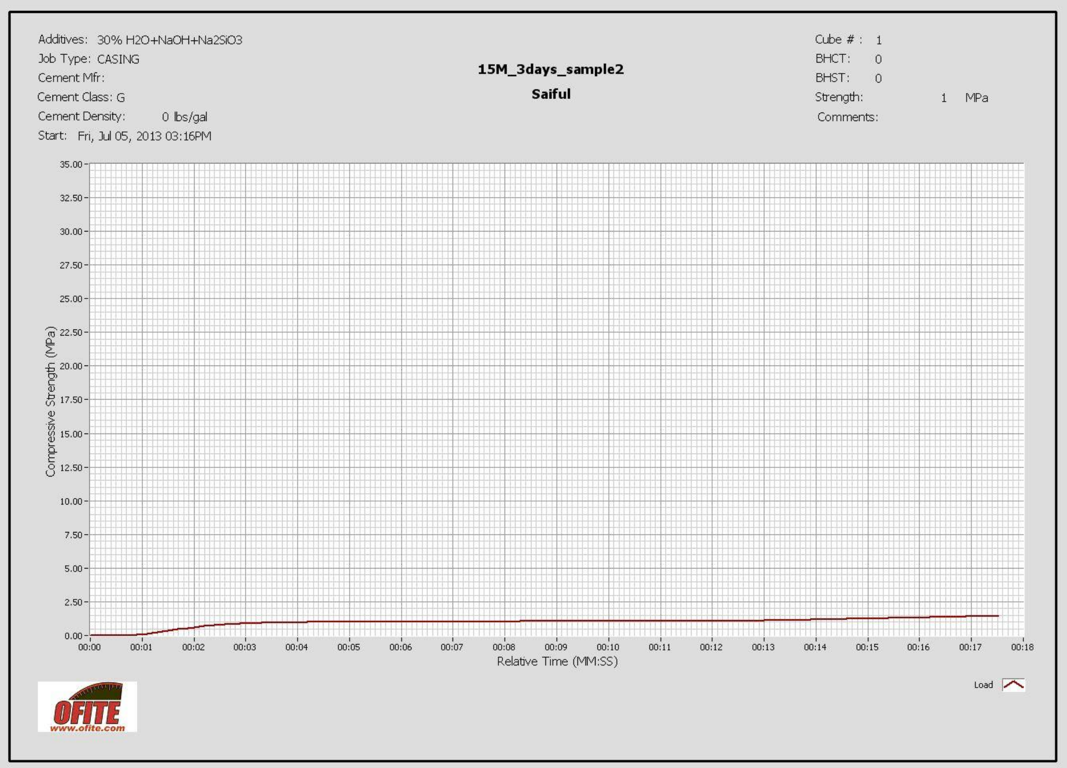
15M_3days_sample1

Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 1 MPa
Comments:

Figure 4.04 shows the compressive strength result for the 15M NaOH variant @ 30% added water concentration and 3 days curing time, for cube sample 1. The maximum compressive strength was achieved at 1.42 MPa.

v) 15M + 3 Days Sample 2



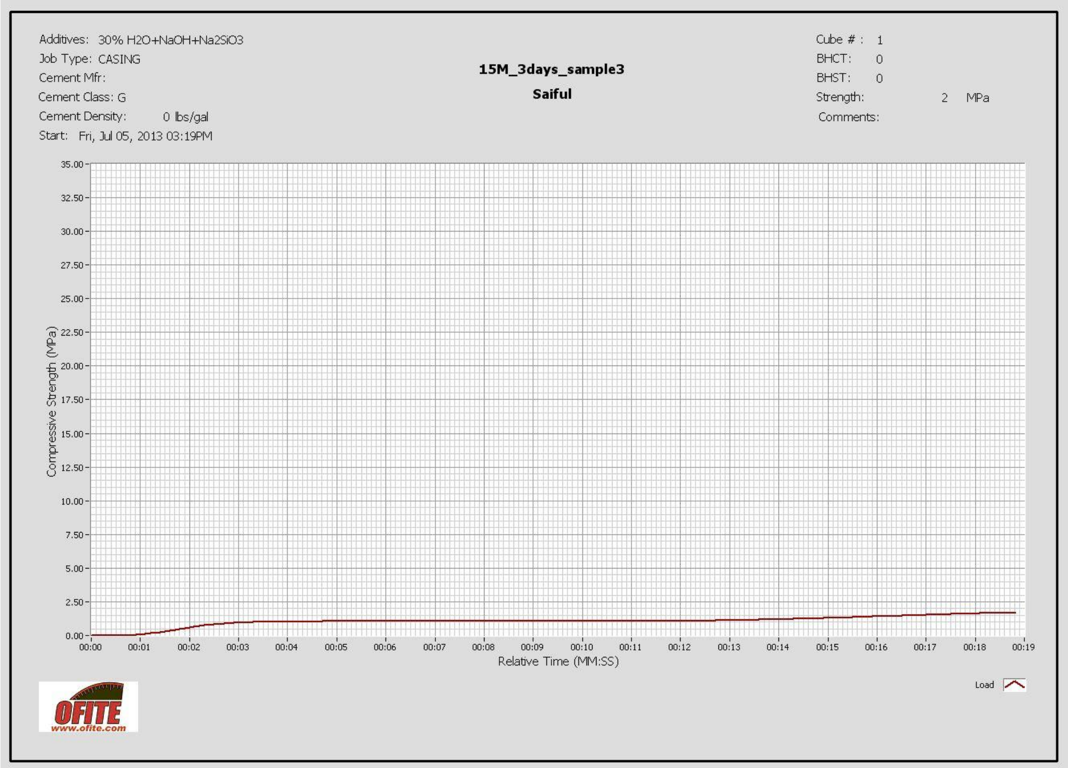
Additives: 30% H2O+NaOH+Na2SiO3
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Fri, Jul 05, 2013 03:16PM

15M_3days_sample2
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 1 MPa
Comments:

Figure 4.05 shows the compressive strength result for the 15M NaOH variant @ 30% added water concentration and 3 days curing time, for cube sample 2. The maximum compressive strength was achieved at 1.42 MPa.

vi) 15M + 3 Days Sample 3



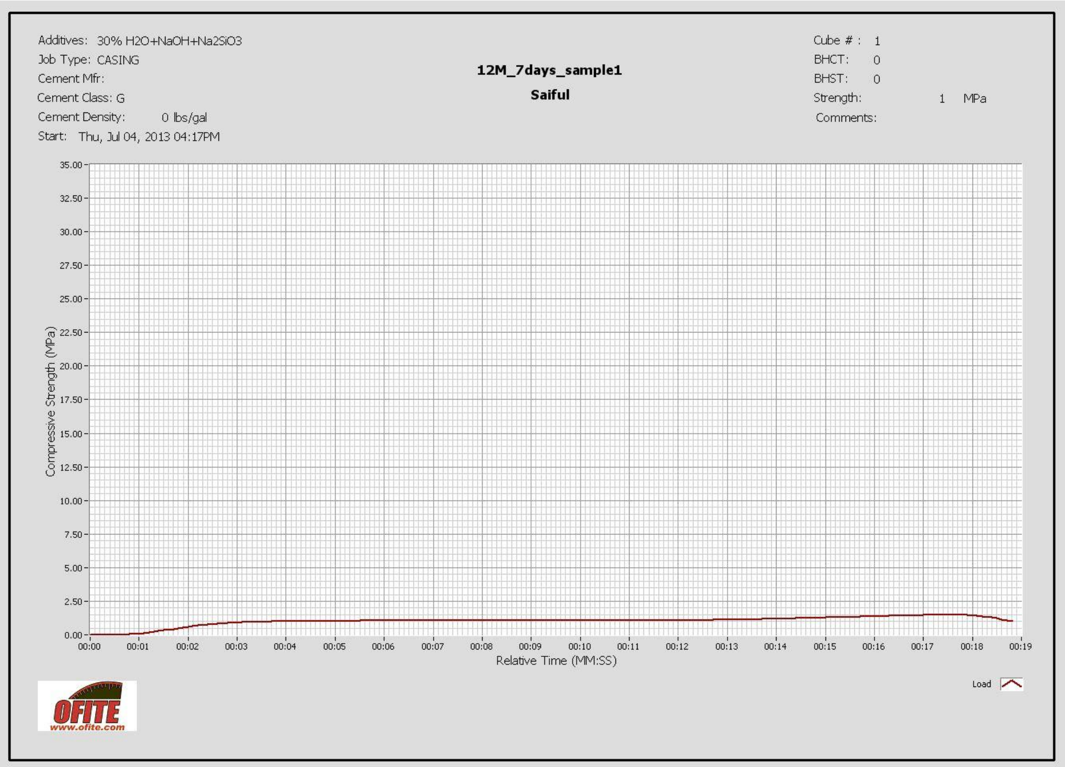
Additives: 30% H2O+NaOH+Na2SiO3
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Fri, Jul 05, 2013 03:19PM

15M_3days_sample3
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 2 MPa
Comments:

Figure 4.06 shows the compressive strength result for the 15M NaOH variant @ 30% added water concentration and 3 days curing time, for cube sample 3. The maximum compressive strength was achieved at 1.66 MPa.

vii) 12M + 7 Days Sample 1



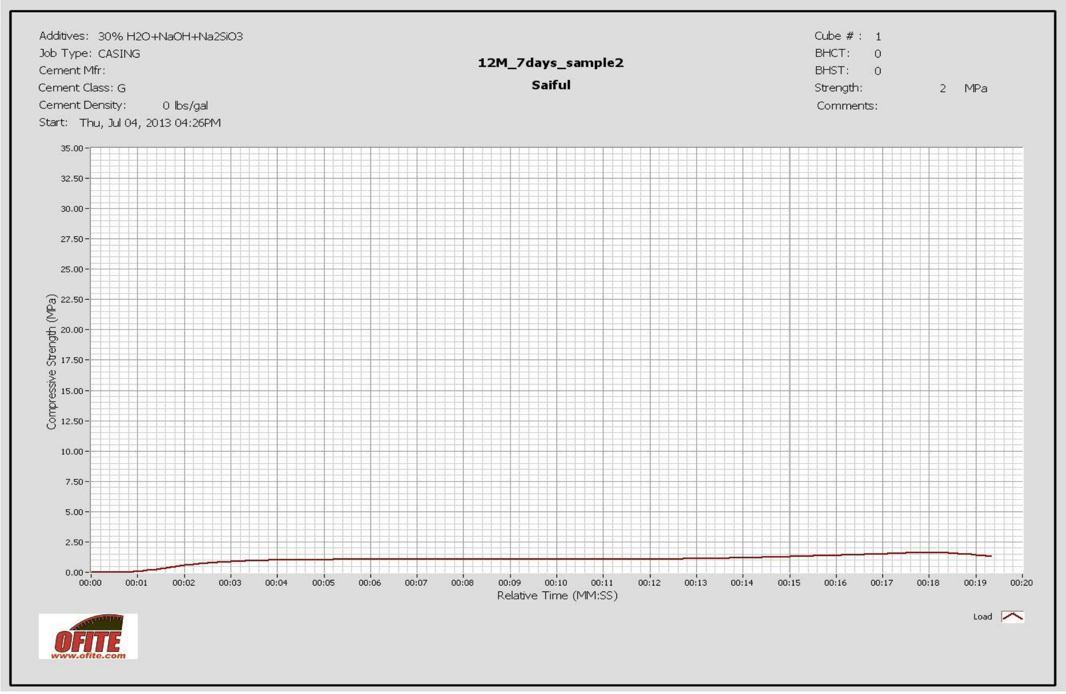
Additives: 30% H2O+NaOH+Na2SiO3
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Thu, Jul 04, 2013 04:17PM

12M_7days_sample1
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 1 MPa
Comments:

Figure 4.07 shows the compressive strength result for the 12M NaOH variant @ 30% added water concentration and 7 days curing time, for cube sample 1. The maximum compressive strength was achieved at 1.50 MPa.

viii) 12M + 7 Days Sample 2



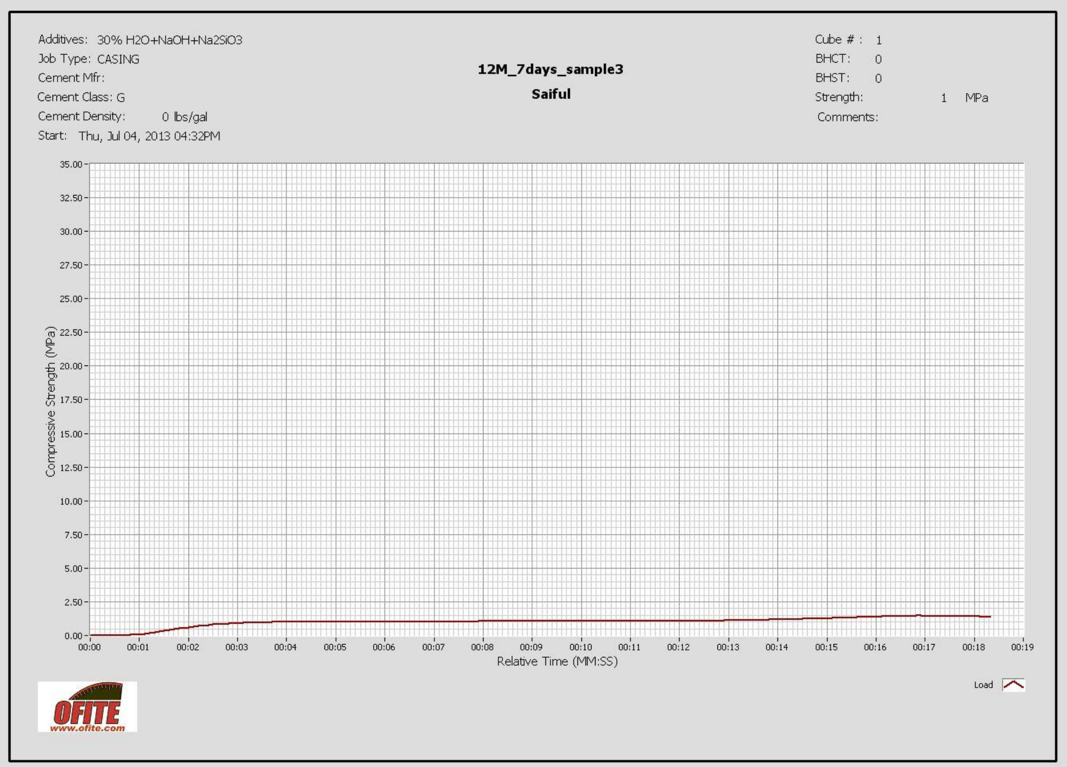
Additives: 30% H2O+NaOH+Na2SiO3
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Thu, Jul 04, 2013 04:26PM

12M_7days_sample2
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 2 MPa
Comments:

Figure 4.08 shows the compressive strength result for the 12M NaOH variant @ 30% added water concentration and 7 days curing time, for cube sample 2. The maximum compressive strength was achieved at 1.61 MPa.

ix) 12M + 7 Days Sample 3


Additives: 30% H2O+NaOH+Na2SiO3
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Thu, Jul 04, 2013 04:32PM

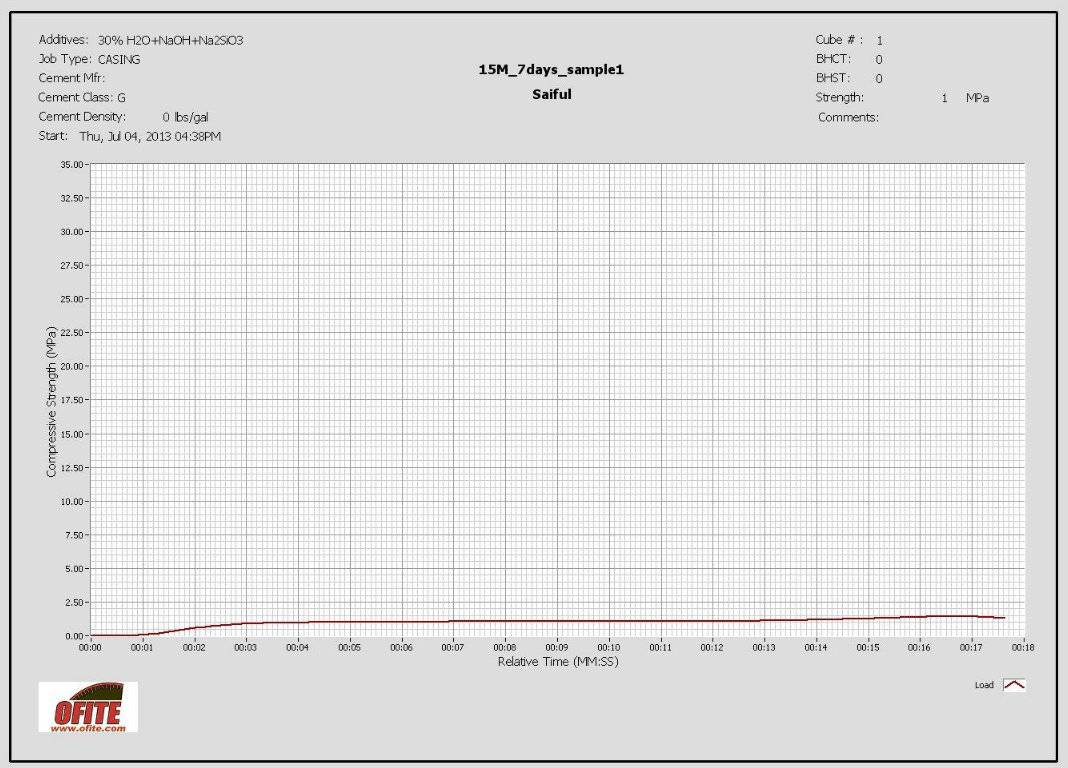
12M_7days_sample3

Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 1 MPa
Comments:

Figure 4.09 shows the compressive strength result for the 12M NaOH variant @ 30% added water concentration and 7 days curing time, for cube sample 3. The maximum compressive strength was achieved at 1.46 MPa.

x) 15M + 7 Days Sample 1



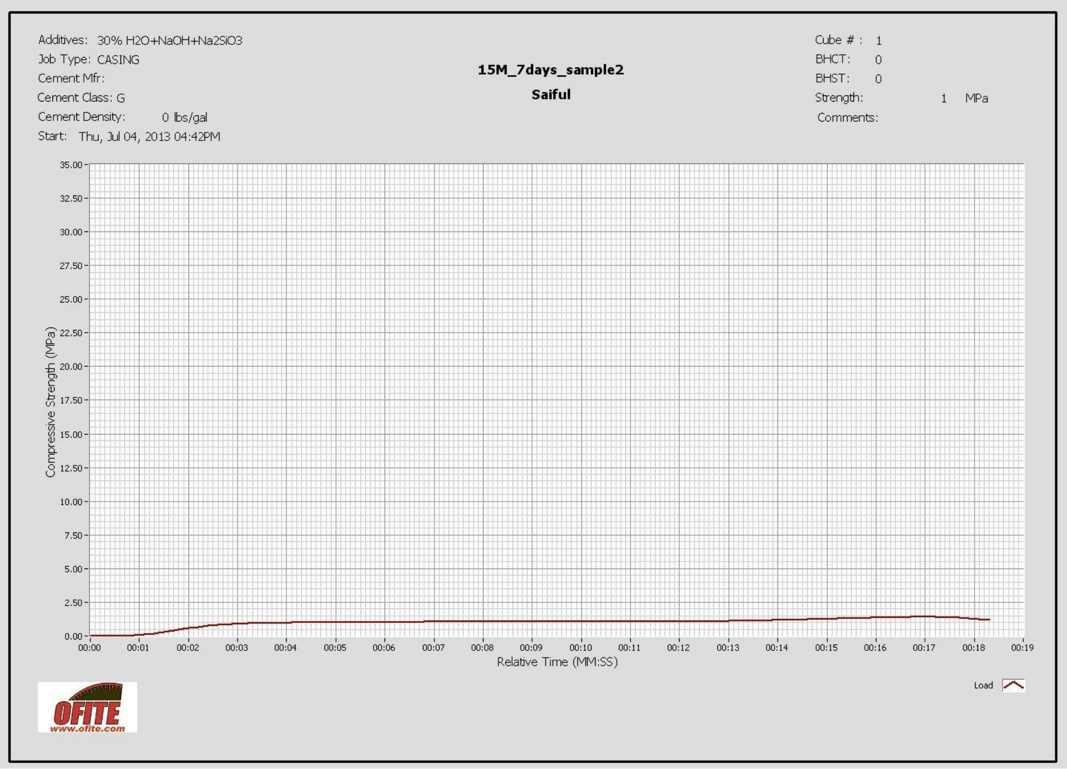
Additives: 30% H2O+NaOH+Na2SiO3
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Thu, Jul 04, 2013 04:38PM

15M_7days_sample1
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 1 MPa
Comments:

Figure 4.10 shows the compressive strength result for the 15M NaOH variant @ 30% added water concentration and 7 days curing time, for cube sample 1. The maximum compressive strength was achieved at 1.44 MPa.

xi) 15M + 7 Days Sample 2



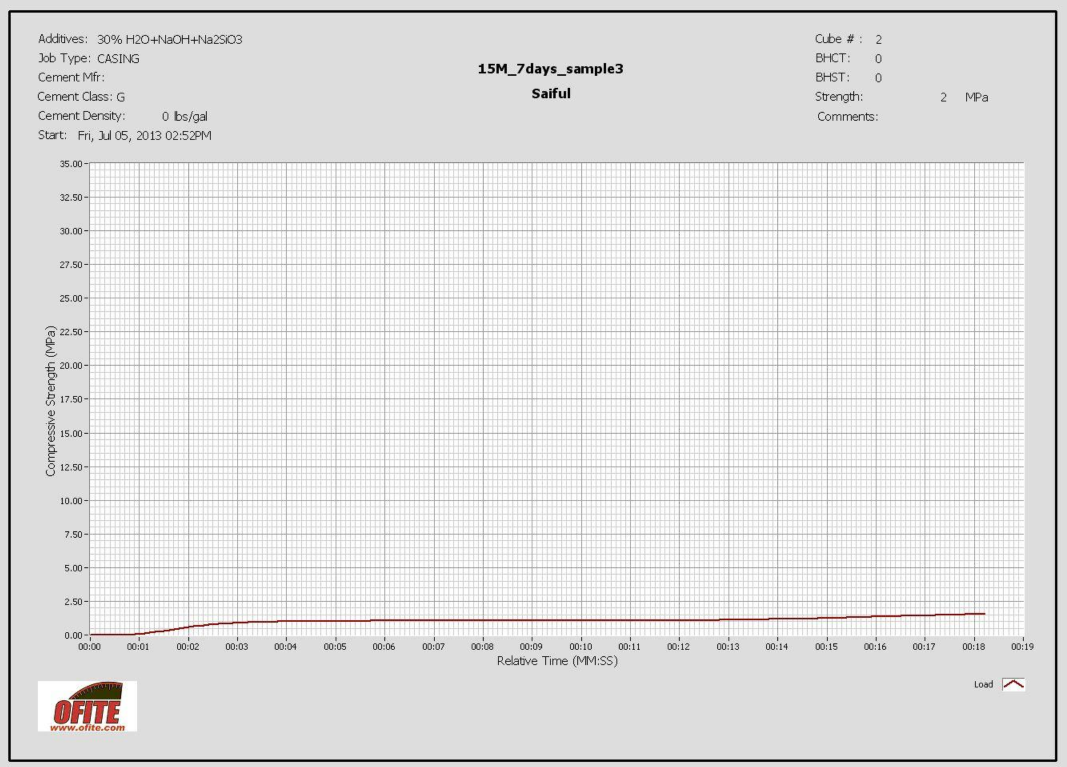
Additives: 30% H2O+NaOH+Na2SiO3
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Thu, Jul 04, 2013 04:42PM

15M_7days_sample2
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 1 MPa
Comments:

Figure 4.11 shows the compressive strength result for the 15M NaOH variant @ 30% added water concentration and 7 days curing time, for cube sample 2. The maximum compressive strength was achieved at 1.41 MPa.

xii) 15M + 7 Days Sample 3


Additives: 30% H2O+NaOH+Na2SiO3
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Fri, Jul 05, 2013 02:52PM

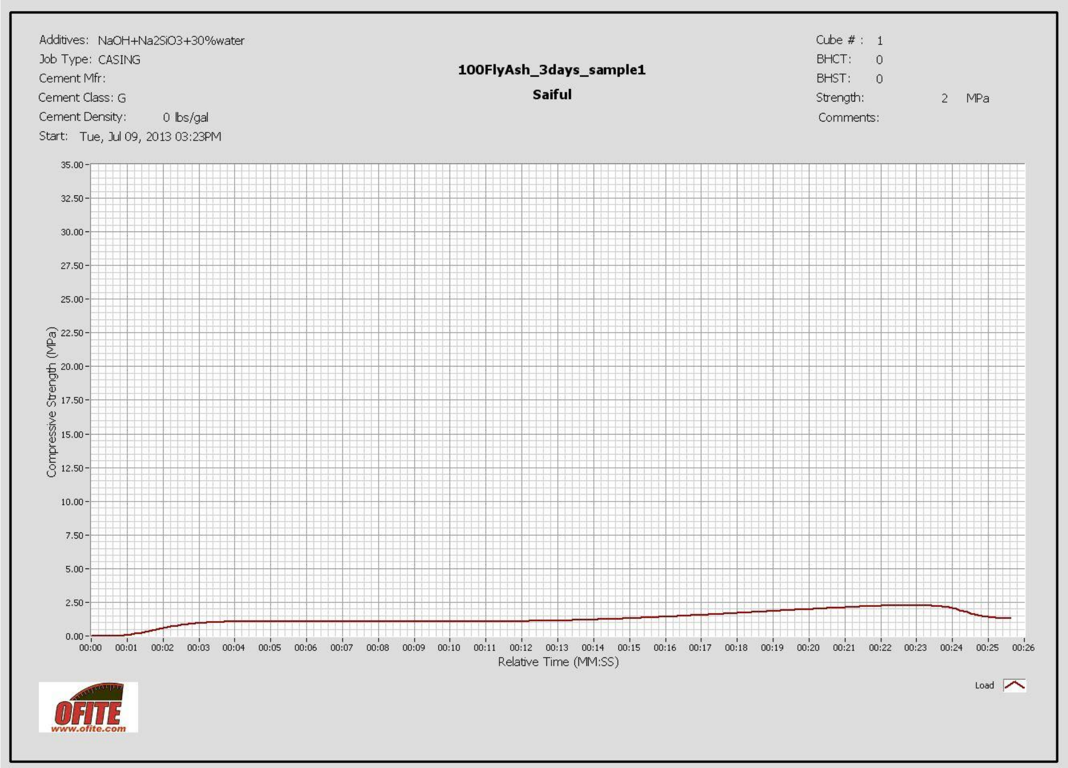
Cube # : 2
BHCT: 0
BHST: 0
Strength: 2 MPa
Comments:

Figure 4.12 shows the compressive strength result for the 15M NaOH variant @ 30% added water concentration and 7 days curing time, for cube sample 3. The maximum compressive strength was achieved at 1.55 MPa.

4.2.2 Compressive Test Results for 25% H_2O Concentration

For the 25% H_2O concentration, it is observed that the paste is very thick, and less workable than the 30% H_2O variant. However, the compressive strength are higher as could be seen in the graphs below.

i) 12M + 3 Days Sample 1



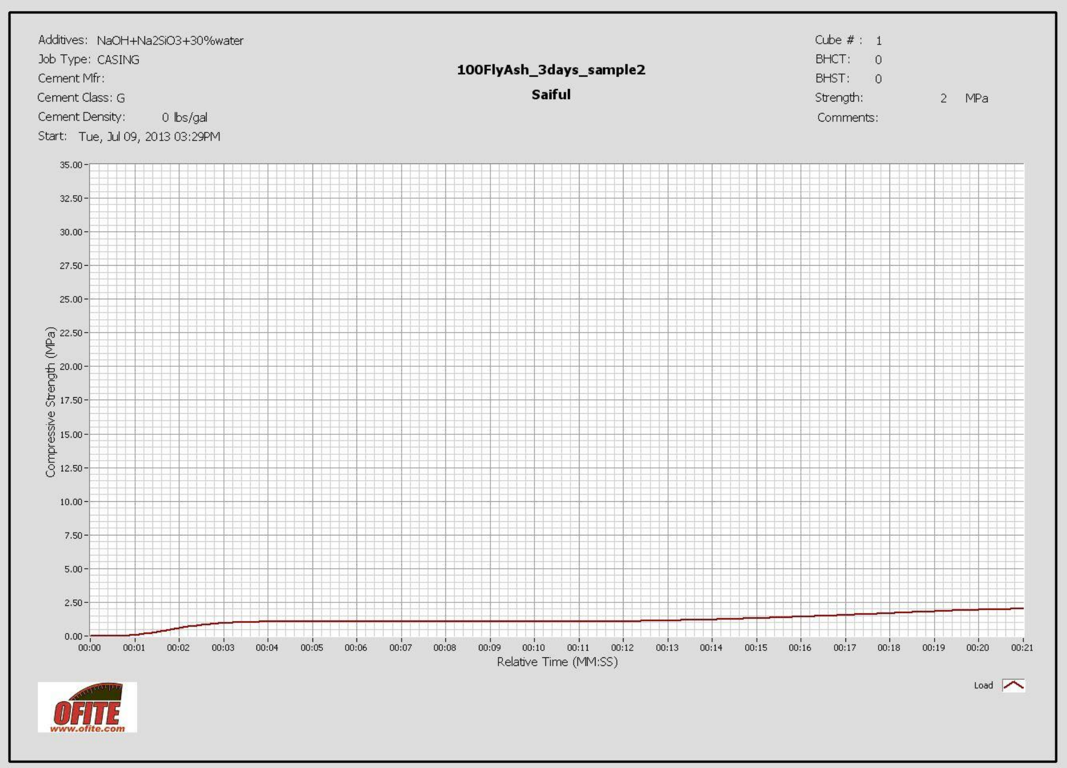
Additives: NaOH+Na2SiO3+30%water
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Tue, Jul 09, 2013 03:23PM

100FlyAsh_3days_sample1
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 2 MPa
Comments:

Figure 4.13 shows the compressive strength result for the 12M NaOH variant @ 25% added water concentration and 3 days curing time, for cube sample 1. The maximum compressive strength was achieved at 2.27 MPa.

ii) 12M + 3 Days Sample 2



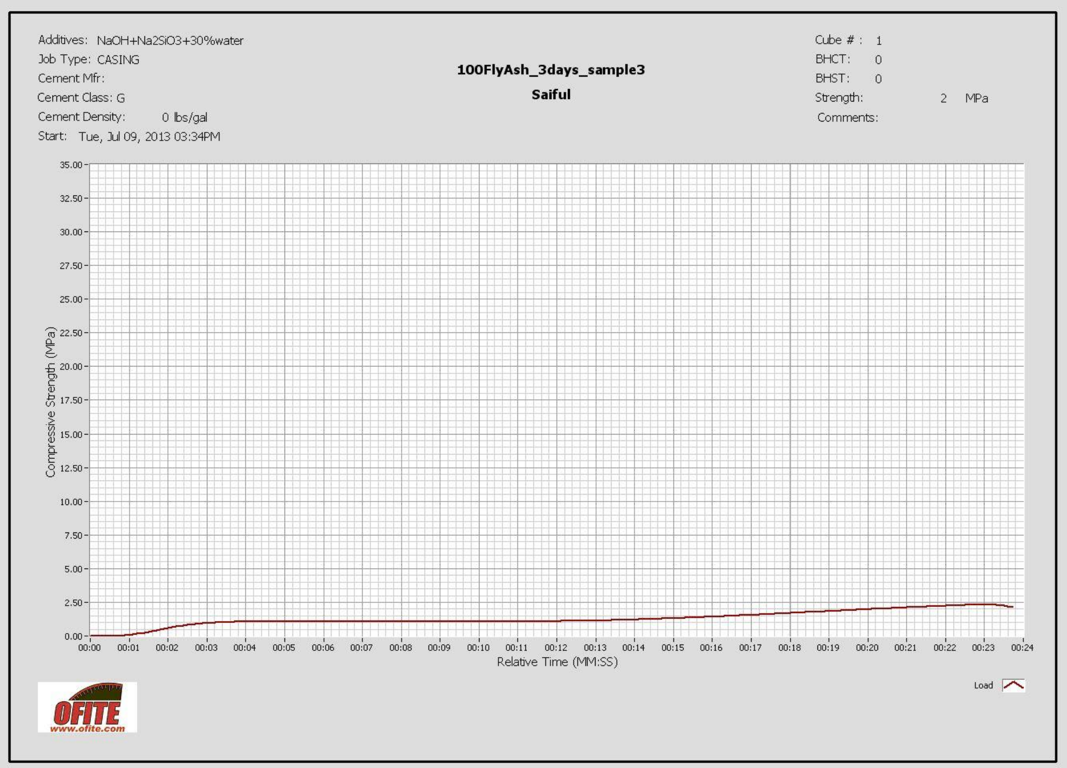
Additives: NaOH+Na2SiO3+30%water
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Tue, Jul 09, 2013 03:29PM

100FlyAsh_3days_sample2
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 2 MPa
Comments:

Figure 4.14 shows the compressive strength result for the 12M NaOH variant @ 25% added water concentration and 3 days curing time, for cube sample 2. The maximum compressive strength was achieved at 2 MPa.

iii) 12M + 3 Days Sample 3



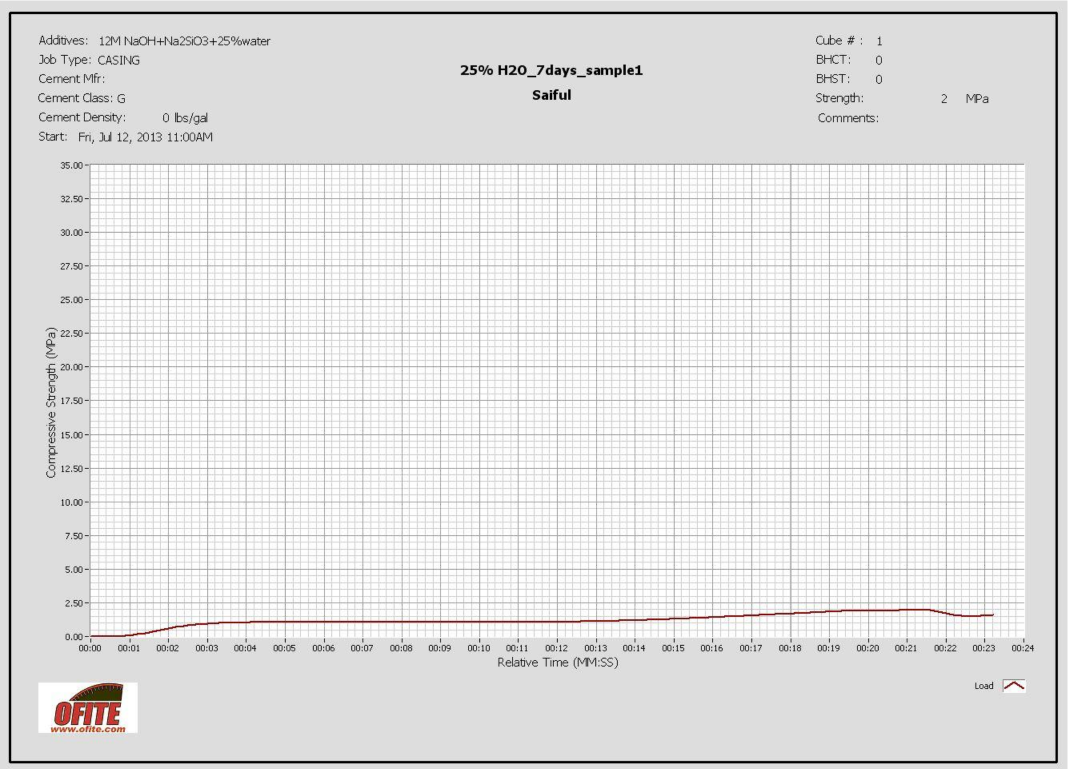
Additives: NaOH+Na2SiO3+30%water
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Tue, Jul 09, 2013 03:34PM

100FlyAsh_3days_sample3
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 2 MPa
Comments:

Figure 4.15 shows the compressive strength result for the 12M NaOH variant @ 25% added water concentration and 3 days curing time, for cube sample 3. The maximum compressive strength was achieved at 2.32 MPa.

iv) 12M + 7 Days Sample 1



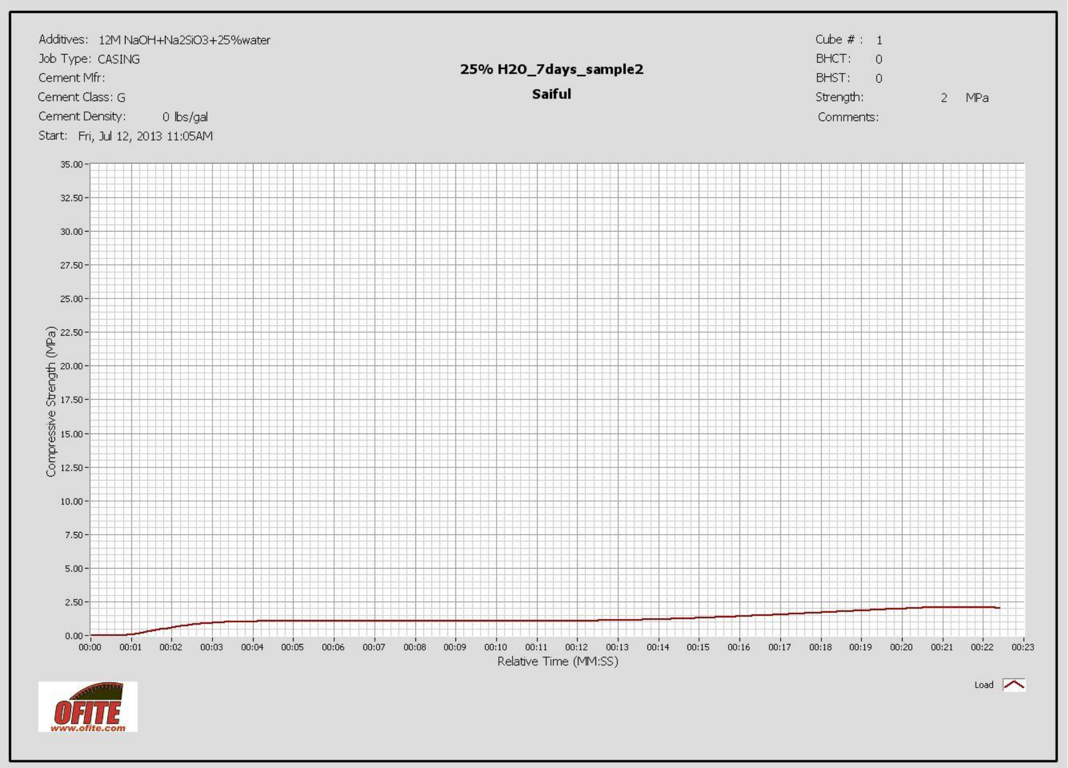
Additives: 12M NaOH+Na2SiO3+25%water
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Fri, Jul 12, 2013 11:00AM

25% H2O_7days_sample1
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 2 MPa
Comments:

Figure 4.16 shows the compressive strength result for the 12M NaOH variant @ 25% added water concentration and 7 days curing time, for cube sample 1. The maximum compressive strength was achieved at 1.97 MPa.

v) 15M + 7 Days Sample 2



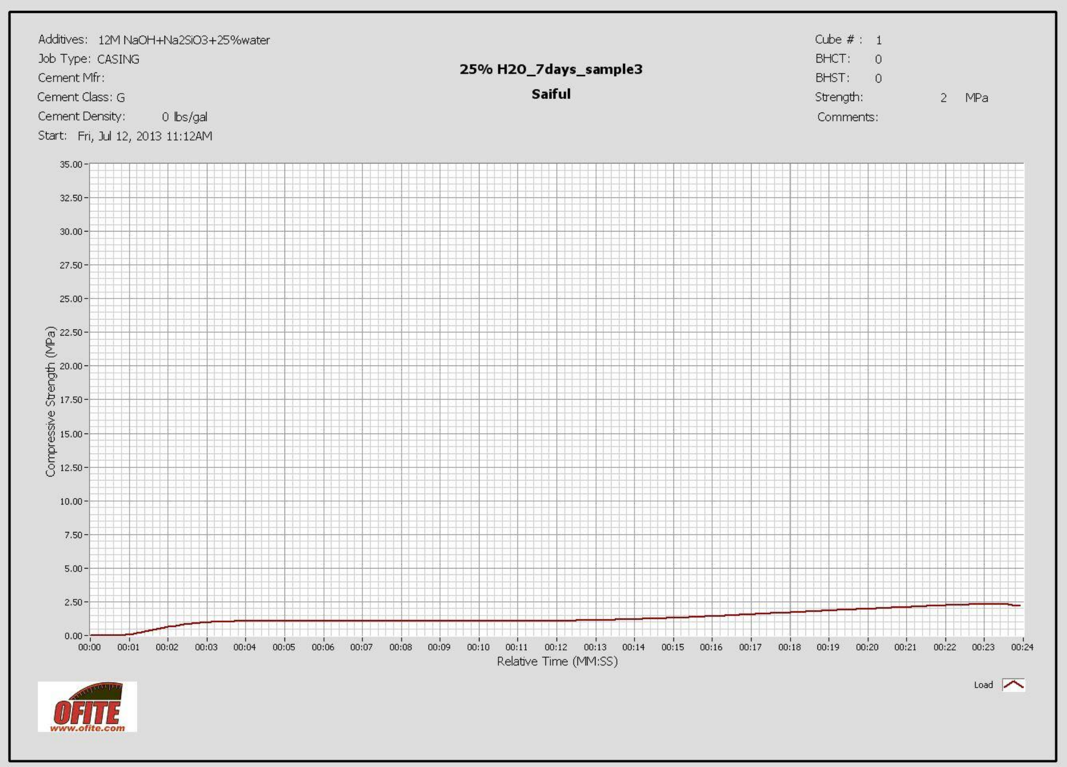
Additives: 12M NaOH+Na2SiO3+25%water
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Fri, Jul 12, 2013 11:05AM

25% H20_7days_sample2
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 2 MPa
Comments:

Figure 4.17 shows the compressive strength result for the 12M NaOH variant @ 25% added water concentration and 7 days curing time, for cube sample 2. The maximum compressive strength was achieved at 2.08 MPa.

vi) 15M + 7 Days Sample 3



Additives: 12M NaOH+Na2SiO3+25%water
Job Type: CASING
Cement Mfr:
Cement Class: G
Cement Density: 0 lbs/gal
Start: Fri, Jul 12, 2013 11:12AM

25% H20_7days_sample3
Saiful

Cube # : 1
BHCT: 0
BHST: 0
Strength: 2 MPa
Comments:

Figure 4.18 shows the compressive strength result for the 12M NaOH variant @ 25% added water concentration and 7 days curing time, for cube sample 3. The maximum compressive strength was achieved at 2.32 MPa.

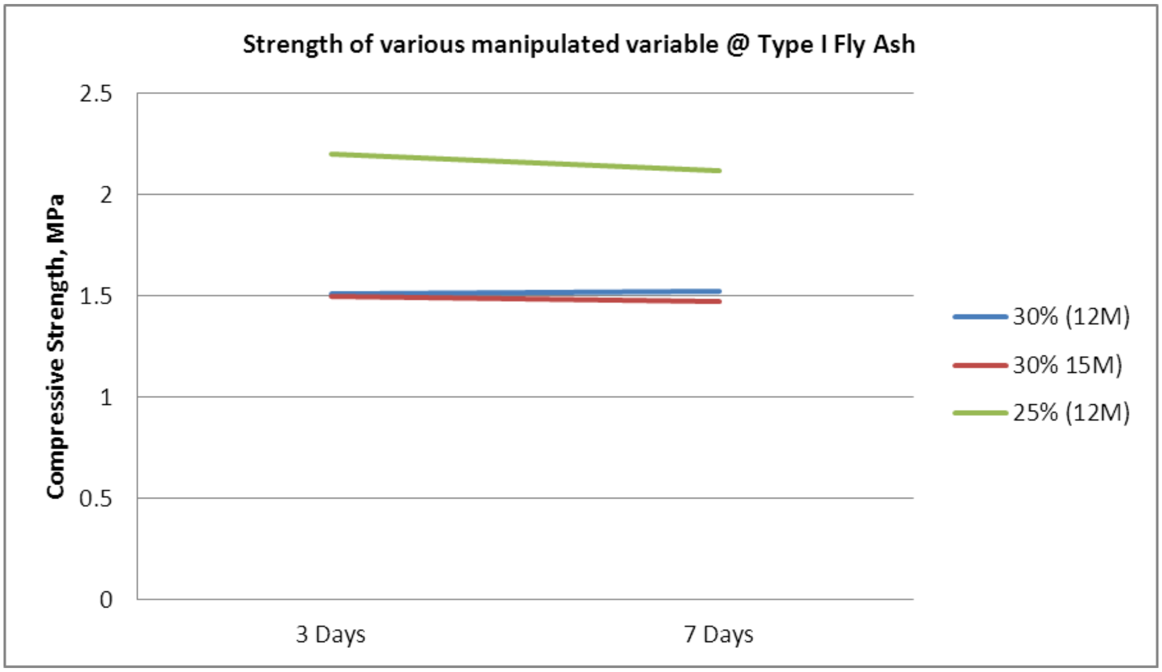


Figure 4.19 shows the graph of compressive strength of various manipulated variable @ Type I Fly Ash. The manipulated variable was the curing time, molarity of NaOH at 12M and 15M at 30% of added water (H_2O), and the difference in concentration of added water of 30% and 25% H_2O, at a fixed molarity of 12M. The result show the manipulation of water concentration (reduction in water concentration added) in the geopolymer gives a relatively higher effect to the compressive strength of the geopolymer cement (higher strength) compared to the other variables.

Table 4.1 shows the compressive strength of various manipulated variables @ Type I Fly Ash. The compressive strength was averaged to get one relative value to be compared amongst the manipulated variables.

Concentration of H_2O added	Curing Time	Sodium Hydroxide (NaOH) Molarity (M)	Maximum Compressive Strength (MPa)			Average Compressive Strength (MPa)
			Sample 1	Sample 2	Sample 3	
30%	3 Days	12M	1.35	1.81	1.38	1.51
		15M	1.42	1.42	1.66	1.50
	7 Days	12M	1.50	1.61	1.46	1.52
		15M	1.44	1.41	1.55	1.47
25%	3 Days	12M	2.27	2	2.32	2.20
	7 Days	12M	1.97	2.08	2.32	2.12

As we could see from the graph and the tables presented for type I fly ash, the various molarity and curing time had little effect on the compressive strength of the geopolymer, compared to the manipulation of water in the experiment. Reduction of 5% of H20 greatly influenced the compressive strength of the geopolymer mould, for example, in the 3 days curing time, the 5% reduction resulted in an increase from 1.51 MPa to 2.20 MPa for the highlighted 12M variant. However, the workability of the 12M was observed to have been reduced form the 15M variant. This corresponds to work carried out by (Sathia et al. 2008), who discovered that the increase in water in geopolymer improves the workability, but increases the porosity in concrete due to the evaporation at elevated tempreratures. Relatively, however, the increase in compressive strength of the sample from the 5% reduction in H_2O, is a small value. (Rangan et al. 2006) explained that water is not involved in the polymeric reaction and will be expelled during curing process. This is further confirmed by (Nuruddin et al. 2010) whose compressive strength did not vary much from the two samples of 10% extra water, and 15% extra water. Hence, it is safe to assume that the higher the amount of water, the lower the compressive strength of the geopolymer.

The other variables, such as different molarity of NaOH, for instance did not have much difference from one sample to the next, with the 12M giving a slightly higher strength than the 15M.. (Chindaprasirt et al. 2007) found that the concentration of NaOH from 10 to 20M gives a small effect on the strength. (Al Bakri et al. 2011) also found that the optimum molarity of 12M gave the highest compressive strength. This is further supported by past studies by (Palomo et al. 1999) who found the 12M NaOH solution produced better results than the 18M NaOH solution.

Also note that, for the curing process, the curing temperature was set at 60^oC for the first day (24 hours) in the oven, and subsequently taken out for externally exposed curing for the rest of the curing time. This is due to the optimum temperature found in most studies that found that the 60^oC was the optimum temperature for curing. Xiujiang Song 2007) states that the best curing condition is reported as being 60^oC for 24 hours. He further states that, a number researchers have studied the influence of curing conditions on fly ash based geopolymer, however there is no clear statement as to which temperature and heating hours are the best, since the curing conditions varies from one to another. But it is clear that the elevated temperature curing is a general trend in geopolymer. Our curing process is also further supported from other works. (Rangan 2008) states that heat curing assist in chemical reaction that occurs in geopolymer paste, and that dry curing gives 15% higher compressive strength than steam curing. He also found that increasing temperature result in increase in compressive strength up till $60^{o}C$. However, any higher curing temperature than $60^{o}C$ did not increase the compressive strength. (Nuruddin et al. 2010) also found that externally exposed curing is better than hot gunny and ambient curing. While (Bakri et al. 2011) found that the optimum curing temperature of $60^{o}C$ gave the highest compressive strength. Prolonged curing at elevated temperatures was also not recommended, as could break down the gel structure in the geopolymer matrix, resulting in lower strength (Van Jaarsveld et al. 2002)

(Rattanasak et al. 2009) states that the separate mixing produced slightly stronger mortar than the normal mixing, however, many researchers have found that the optimum mixing process is the normal mixing. We have used the normal mixing process for this experiment.

Last but not least, the fly ash to alkaline ratio of 1:0.4 or in other words fly ash/ alkaline solution of 2.5 was used for all the experiment using type I fly ash. 2.5 ratio value was also used on the Na_2SiO_3 / NaOH solution. There has been previous research that concluded that Na_2SiO_3 / NaOH ratio of 1.0 gives the highest compressive strength. However, (Hardjito 2004) found that the Na_2SiO_3 / NaOH ratio of 2.5 gives the highest compressive strength compared to value of 0.4. This is further supported by (Bakri et al 2011) who found that the highest compressive strength was obtained at Na_2SiO_3 / NaOH ratio of 2.5.

Finally, the slight reduction in compressive strength of the geopolymer from 3 days to the 7 days curing time in the 15M molarity @ 30% H_2O variant and 25% H2O variant poses a questionable doubt, hence leading us to doubt the quality of the fly ash obtained earlier. Hence, work was continued with another manipulated variable to obtain a concrete judgment, where we used another source of Fly Ash, with proven Class F constituents. This fly ash will be called type II fly ash.

4.3 Type II Fly Ash

The type II Class F fly ash was obtained from Lafarge Cement, Klang, Selangor. The fly ash was initially mixed with the same scope of the earlier type I fly ash, to get a comparison between this fly ash and the earlier one. The experiment was further conducted to find the effect of various fly ash / alkaline solution ratio on the geopolymer cement at different curing times, by manipulating its ratio. The result was measured via compressive strength and tabulated in the following sections. It is initially found that the type I fly ash was of unfavorable quality. The following results will prove this statement. Furthermore, the type II fly ash also, did not require any addition of water, since the paste was formed instantly after undergoing normal mixing process. However, due to constraints of time and molds, the type II fly ash only uses one sample cube per variant instead of three cubes as in earlier experiments.

4.3.1 Compressive Test Results of Type II Fly Ash

All experiment was conducted with a fixed 12M molarity of NaOH, with no addition of water. The curing time was manipulated at 3 and 7 days. The fly ash / alkaline solution ratio was manipulated at value of 1, 2, and 2.5 for both curing time period.

i) Fly ash / alkaline solution ratio of 2.5 + 3 Days

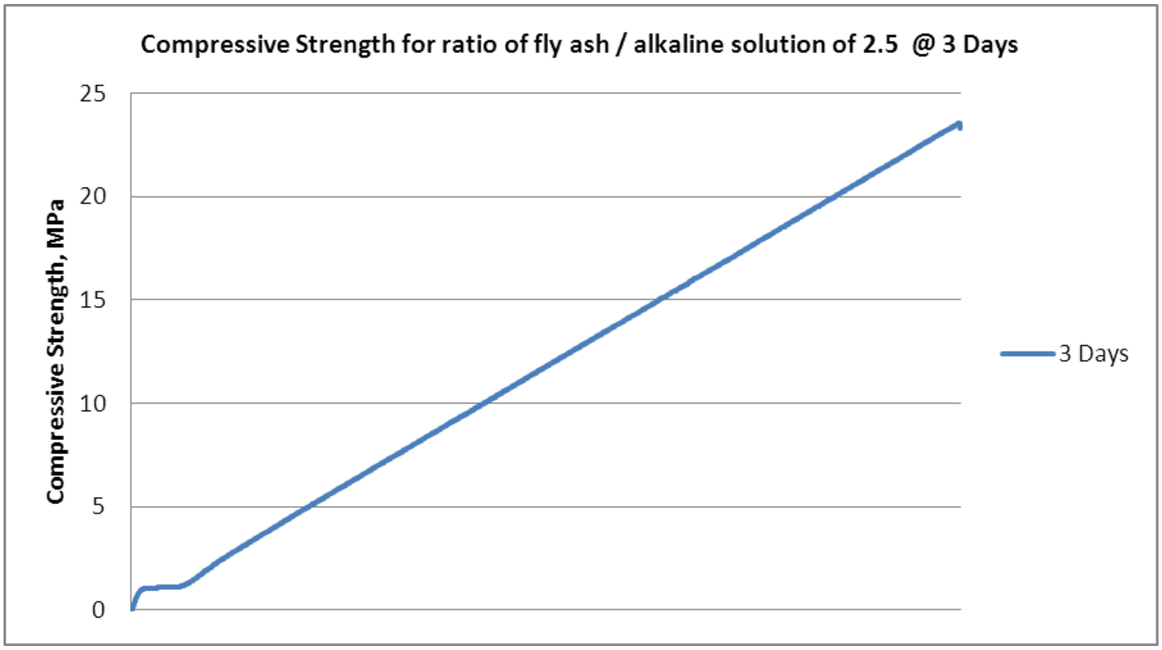


Figure 4.20 shows the compressive strength result for the fly ash / alkaline solution ratio of 2.5 @ 3 days curing time. The maximum compressive strength was achieved at a high value of 23.54 MPa.

ii) **Fly ash / alkaline solution ratio of 2.5 + 7 Days**

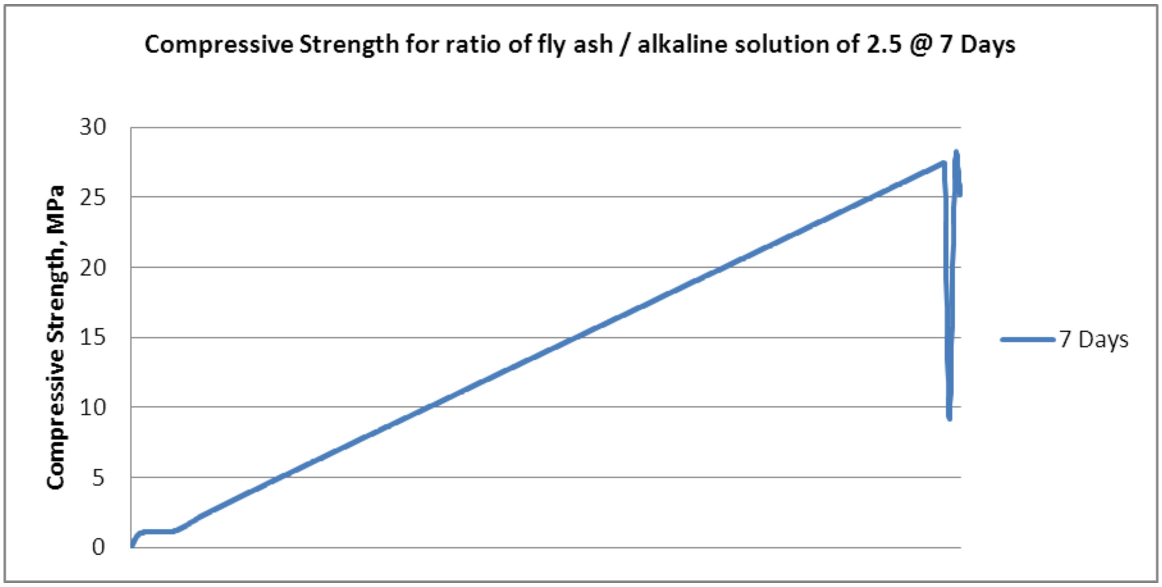


Figure 4.21 shows the compressive strength result for the fly ash / alkaline solution ratio of 2.5 @ 7 days curing time. The sharp decline on the far right shows that the pump has reached its maximum capacity and is unable to test for compressive strength above that value. The maximum compressive strength was achieved at more than 28.29 MPa. The compressive strength for this sample was the highest in the project.

iii) Fly ash / alkaline solution ratio of 2 + 3 Days

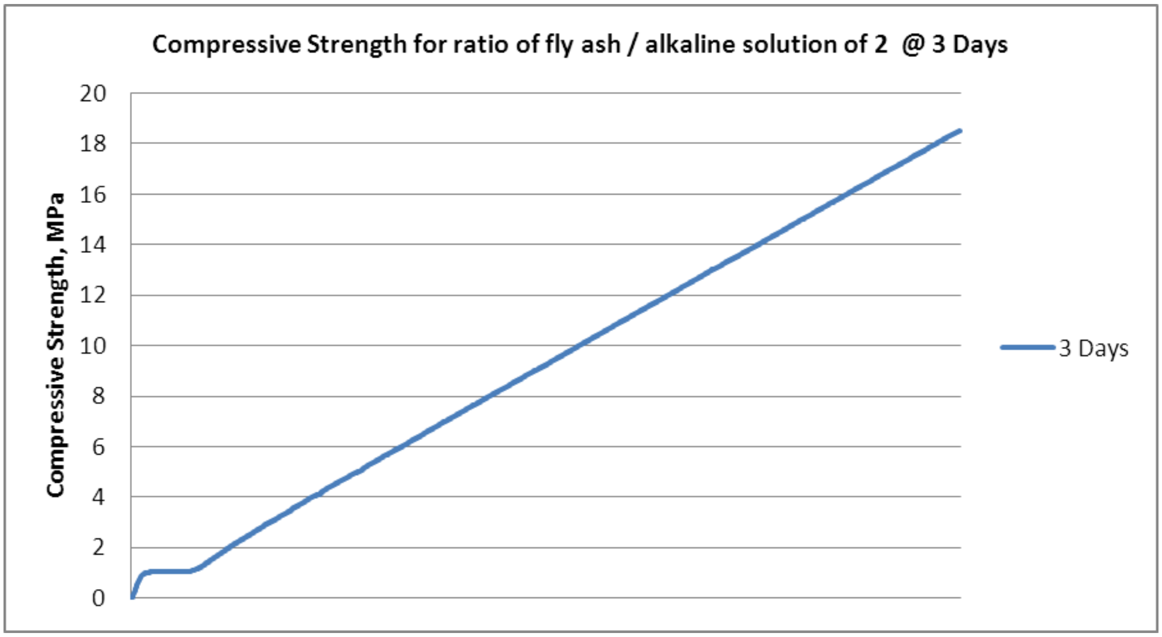


Figure 4.22 shows the compressive strength result for the fly ash / alkaline solution ratio of 2 @ 3 days curing time. The maximum compressive strength was achieved at 18.52 MPa.

iv) Fly ash / alkaline solution ratio of 2 + 7 Days

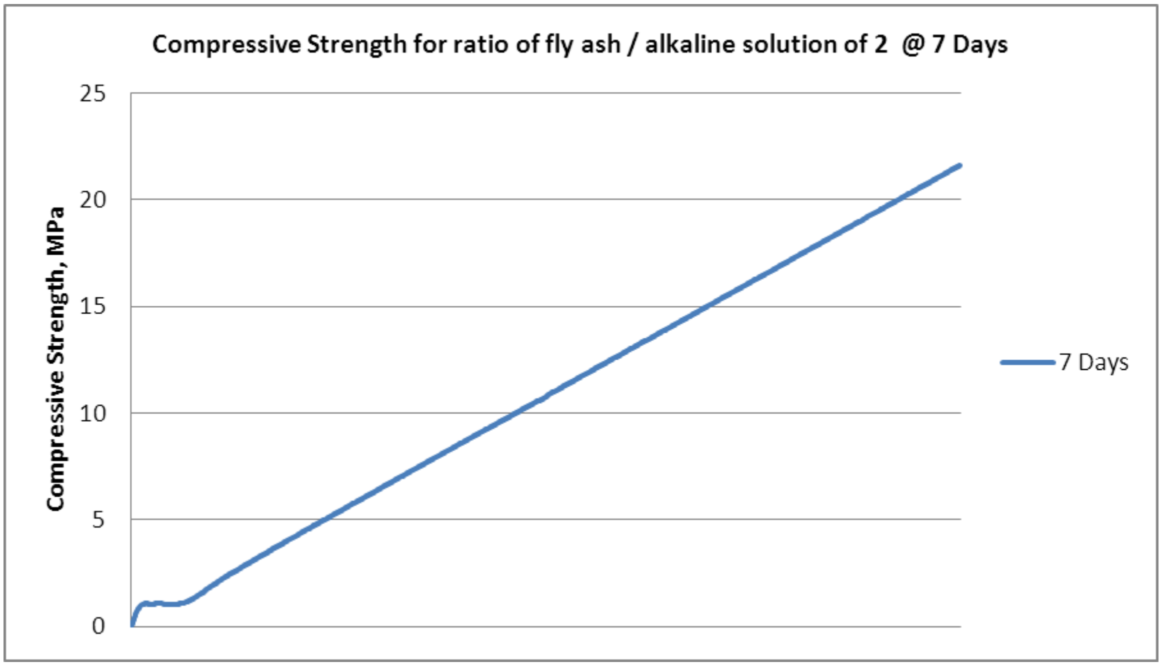


Figure 4.23 shows the compressive strength result for the fly ash / alkaline solution ratio of 2 @ 7 days curing time. The maximum compressive strength was achieved at 21.62 MPa.

v) Fly ash / alkaline solution ratio of 1 + 3 Days

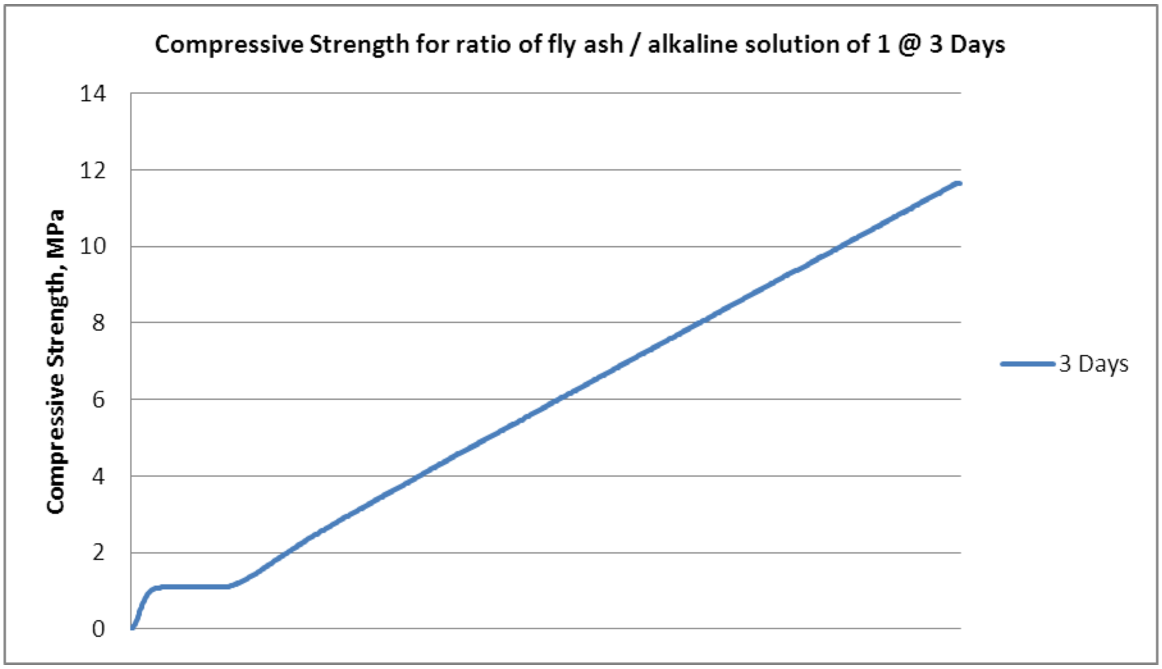


Figure 4.24 shows the compressive strength result for the fly ash / alkaline solution ratio of 1 @ 3 days curing time. The maximum compressive strength was achieved at 11.64 MPa.

vi) Fly ash / alkaline solution ratio of 1 + 7 days

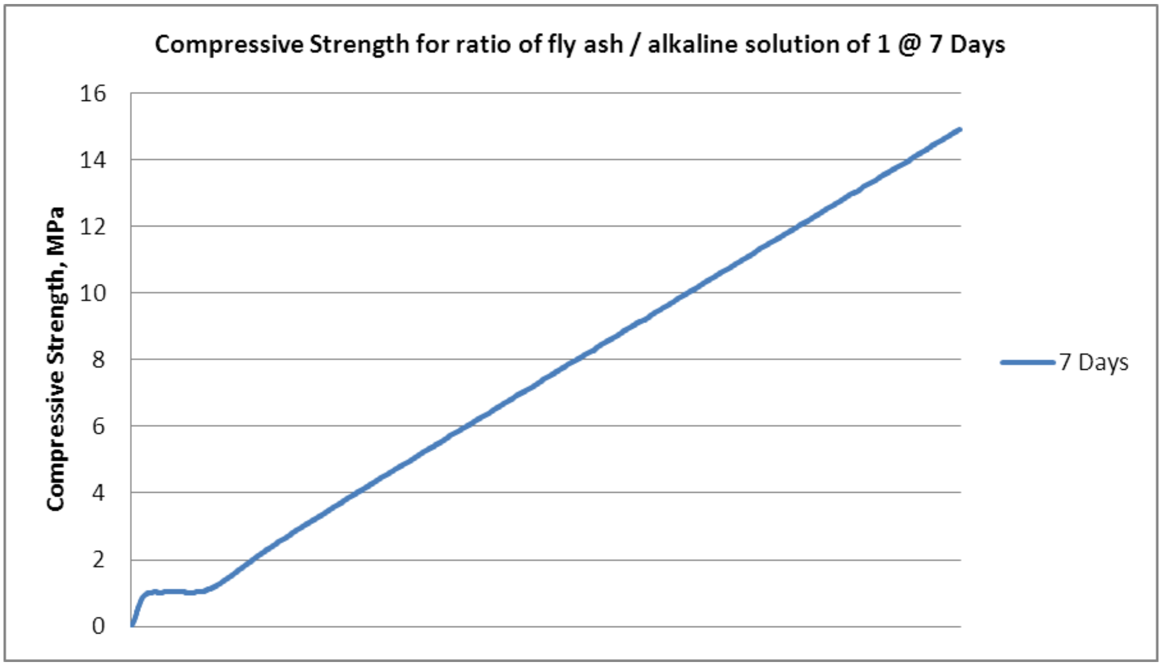


Figure 4.25 shows the compressive strength result for the fly ash / alkaline solution ratio of 1 @ 7 days curing time. The maximum compressive strength was achieved at 14.91 MPa.

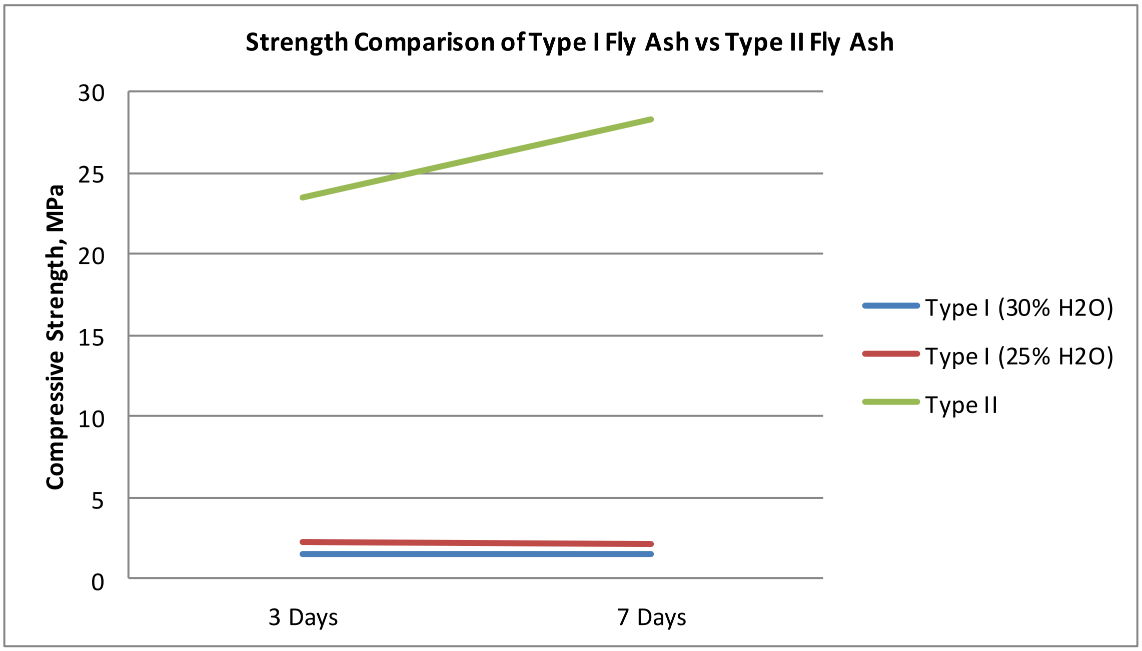


Figure 4.26 shows the comparison of compressive strength for type I and type II fly ash. The type I fly ash (at 25% and 30% of added H_20) was compared to the type I fly ash with no addition of water required. The type II fly ash was far more superior than the type I fly ash, in terms of compressive strength. The type II fly ash also had a more realistic curve of higher compressive strength at 7 days compared to the 3 days.

Table 4.2 shows the comparison of compressive strength for type I and type II fly ash.

Type of Fly Ash	Concentration of H_2O added	Curing Time	Ratio of Fly Ash / alkaline solution	Ratio of Na_2SiO_3 / NaOH solution	Sodium Hydroxide (NaOH) Molarity (M)	Average Compressive Strength (MPa)
Type I	30%	3 Days	2.5	2.5	12M	1.51
		7 Days				1.52
	25%	3 Days				2.20
		7 Days				2.12
Type II	-	3 Days				23.54
		7 Days				>28.29*

* The value shows that the original compressive strength is higher than the listed value. However the compressive strength tester pump utilized has reached its maximum capacity and is unable to read values higher than the listed in the table.

As we could see from the results and the comparison table, the type II fly ash proved far superior to type I fly ash in quality. The results from the compressive strength is also much higher than type I fly ash. The mixing process of type II fly ash did not require any addition of water, since the mixture turned into workable paste during the mixing process. This might have contributed somehow to the high compressive strength of the type II fly ash. However, as we have discussed earlier, the effect of reduction of water is relatively small, and hence would not have been the sole cause for the high strength of the fly ash.

This indirectly proves that the type I fly ash used earlier was of unfavorable quality, that might have been of undesirably high calcium content, among other factors that caused its low strength capacity. (Song 2007) states that since fly ash is produced from coal-fired power stations, it differs from one station to the next because each station uses different sources of coal and the processing procedure might not be the same. Therefore, properties of fly ash such as its chemical composition, the particle size distribution, the thermal history and calcium content as well as the loss on ignition significantly affect the final properties of the resultant geopolymer matrix. Furthemore, the type II fly ash used had low calcium content, and very fine composition, desirable for a high strength geopolymer as stated in the following statement. The potential high strength and usable fly ash to be alkali-activated for geopolymer should have such characteristics such as percentage of unburned material lower than 5%, ferric oxide content not higher than 10%, low content of calcium oxide, percentage of particles with size lower than 45 microns and also high content of glassy phase (Fernandez-Jimenez and Palomo 2003).

As you could see from the results, experiment was also conducted to find the effect of various fly ash to alkaline ratio. The manipulated variable, fly ash / alkaline ratio which was initially set to 2.5, is reduced to 1, which means increasing the amount of alkaline solution. The other properties are kept constant for this experiment. Molarity of

NaOH kept contant at 12M and ratio of Na_2SiO_3 / NaOH solution at 2.5. The result was evaluated based on the compressive strength of the samples. The comparison is tabulated as follows.

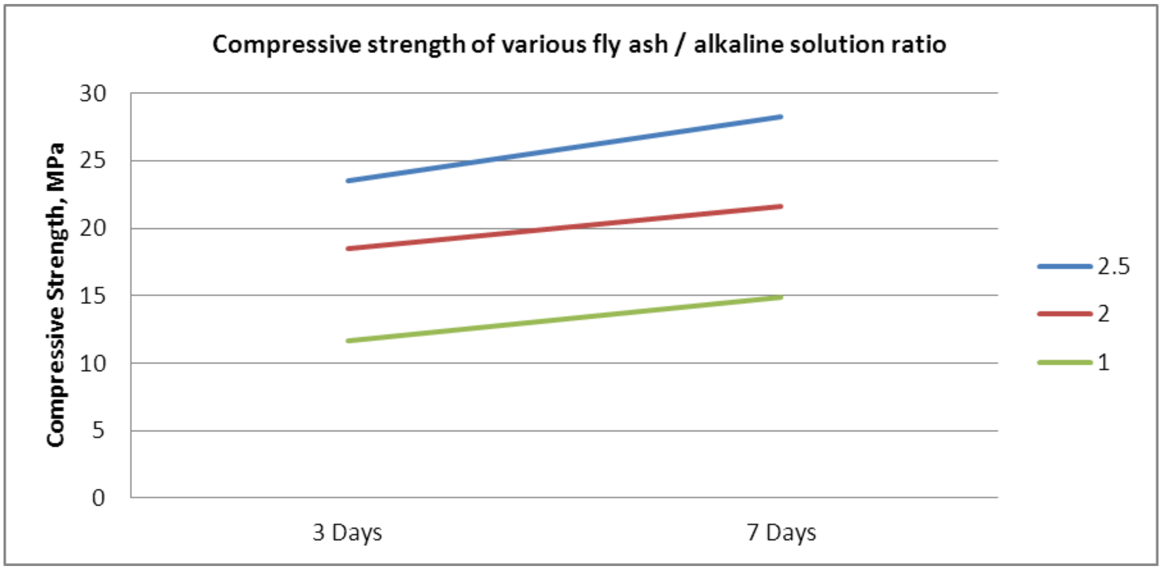


Figure 4.27 shows the comparison of compressive strength for different values of ratio of fly ash / alkaline activators. The different values of fly ash / alkaline ratio used was 1, 2, and 2.5. The fly ash/ alkaline ratio of 2.5 gives the highest compressive strength, with the least ratio of 1 giving the lowest compressive strength.

Table 4.3 shows the comparison of compressive strength for different values of ratio of fly ash / alkaline activators

Ratio of Fly Ash / alkaline solution	Curing Time	Average Compressive Strength (MPa)
2.5	3 Days	23.54
	7 Days	>28.29
2	3 Days	18.52
	7 Days	21.62
1	3 Days	11.64
	7 Days	14.91

From the table, it is apparent that the compressive strength for the ratio of 2.5 fly ash / alkaline solution gives us the highest compressive strength compared to the rest. The value at 7 days is much higher than 3 days, giving us a maximum value of higher than 28.29 MPa. We were unable to continue the measuring the compressive strength above this value, due to the incapability of the pump to go any higher since it has reached its maximum capacity or power. (Al Bakri et al. 2011) obtained the highest compressive strength at the fly ash / alkaline solution of 2, while (Sathonawaphak et al. 2009) states that the geopolymer with a fly ash/alkaline activator ratio of 1.4-2.3 showed a high compressive strength of 42-52 MPa. However, in our study, we obtained the highest compressive strength at fly ash / alkaline ratio of 2.5.

The curing time also have a profound effect here, with all the samples having a higher compressive strength after 7 days, compared to the 3 days sample. It has been discussed earlier that there has not been clear statement as to which curing hours are the best (Song 2007), however in our experiment, it is found that the most externally prolonged curing provides a better compressive strength.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The ultimate objective of this projective of this project is to study the properties of fly ash as a geopolymer cement and identify its advantages. Through the manipulated variables, we have identified several conditions of the geopolymer properties. For instance, the molarity of NaOH and water concentration had little influence on the compressive strength of the geopolymer compared to the types of fly ash. All in all, the observations and results promises a potential OPC replacement.It is deeply hoped that more investigation of fly ash based geopolymer cement will successfully lead us to a potential cement replacement for OPC in the upstream industry. The fly ash based geopolymer offers a holistic solution to increasing demands of cement in the oil and gas sector in a sustainable manner, at majorly reduced cost, and at the same time reducing the environmental impact of both the cement industry and the coal-fired power industry.

5.2 Suggestions and Recommendations

Due to the inexperience in working with geopolymer cementing prior to this experiment, there might have been some overlooked procedures and method of conducting the experiment. Hence, it is recommended to sought more advise from those experienced. It is also highly recommended that the experiment is continued at various other manipulation of variables and test against other factors in addition to compressive strength. The sample should be tested against acidic environment, especially sulfuric acid which is representative of the downhole environment. The samples should also be tested against carbonic acid to find a solution for carbon dioxide sequestration. Furthemore, the experiment can be expanded to include gel strength, additives etc to be tested as an actual downhole cement. Hence, with more time and work, it is deeply hoped that the fly ash will be a more green and economical substitute for OPC.

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