FLY ASH BASED GEOPOLYMER CONCRETE FOR MARINE STRUCTURE APPLICATION RESISTANCE TO SULPHATE ATTACK

by

Ilyas Nurhadi
17024

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

SEPTEMBER 2015

Universiti Teknologi PETRONAS
32610 Bandar Seri Iskandar
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

Fly ash based geopolymer concrete

For marine structure application: Resistance to sulphate attack

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A project dissertation submitted to the

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Approved by,

____

(Ir. Abdul Aziz bin Omar)

UNIVERSITI TEKNOLOGI PETRONAS

BANDAR SERI ISKANDAR, PERAK

September 2015

CERTIFICATION OF ORIGINALITY

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

ILYAS NURHADI

ABSTRACT

The world becomes increasingly ocean-oriented for energy and other resources. It is predicted that concrete sea structures will dominated construction activity for the next century. Marine environment can be considered as harsh environment for concrete durability. One of the most serious problems concerning the durability of concrete is sulphate attack. Ordinary Portland Cement (OPC) has dominated the world of construction as most used material due to its economical and durability reasons. However, cement industry contributes 5% of global carbon dioxide emissions. Therefore, in order to reduce CO_2 emission hence slowing the global warming, an alternative has been developed which has stronger durability, utilized industrial waste, and environmental friendly during its production. The alternative is called fly ash based geopolymer concrete. In this project the potential of geopolymer replacing OPC is tested by determining its durability in marine condition. NaOH and KOH are the activating materials used with different concentration including 4M, 6M, 8M,10M, and 12 M. The geopolymer also cured in two different temperature 25°C and 60°C. The specimens are exposed to 5% sodium sulphate (represent the marine environment) for 14 days. Compressive strength and change in weight of the specimen is observed. Based on the result obtained, it can be concluded that specimens activated with 8M alkali activator and cured in 60°C has superior compressive strength which suitable for construction. In addition, based on the change in mass specimens cured in higher temperature gain weight while specimens cured in lower temperature loss weight. It can be observed that higher alkali content specimens have lower change in mass which is more suitable for construction purposes.

ACKNOWLEDGEMENT

First praise is to Allah, the Almighty, on whom ultimately we depend for sustenance and guidance. With His blessing I able to complete my final year project.

I am using this opportunity to express my deepest gratitude and special thanks to the Ir. Abdul Aziz bin Omar who in spite of being extraordinarily busy with his duties, took time out to hear, guide and keep me on the correct path and allowing me to carry out my project.

I express my deepest thanks to Mrs. Tia Rahmiati, Post Graduate Chemical Engineering student, for taking part in useful decision and giving necessary advices and guidance and arranged all facilities to make life easier. I choose this moment to acknowledge her contribution gratefully.

I also would like to extend my appreciation to my family and all my friends whose name has not been mentioned. As they have encouraged me to go through all the hard work and difficult situations I faced.

I perceive as this opportunity as a big milestone in my career development. I will strive to use gained skills and knowledge in the best possible way, and I will continue to work on their improvement, in order to attain desired life objectives. Hope to continue cooperation with all of you in the future.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The world becomes increasingly ocean-oriented for energy and other resources. It is predicted that concrete sea structures will dominated construction activity for the next century. Concrete is one of the three basic marine structural materials, namely timber, steel and concrete. Reinforced concrete has become the most widely used material for building marine structure such as offshore drilling platforms, superspan bridges, and undersea tunnels (Mehta, 1991). When compared to other building material, concrete shows better resistance to exposure of marine environment.

Engineers must consider marine environment in order to design structure that durable and long-lasting. Mostly seawaters are similar with respect to the types and amounts of dissolved salts. The typical salt content is 3.5% by weight and the major ions are Na^{+}, Mg^{+2}, Cl^{-}, and SO_4^{-2}. Dissolved salts have important role in the chemical and electrochemical phenomena influencing concrete durability. One of the most serious problems concerning the durability of concrete is sulphate attack (Karacoc, et al., 2015).

Ordinary Portland Cement (OPC) has dominated the world of construction as most used material due to its economical and durability reasons. However, since the invention of Geopolymer, a revolutionary change in construction materials technology began. Geopolymer has emerged as a powerful alternative of OPC since it provides technological advantages (Van Deventer & Provis, 2009)

It is claimed by Karacoc (2015) geopolymer concrete offers benefits as construction material under the sulphate attack.

Another competitive edge of geopolymer is its technology utilizes industrial waste, Alumino-silicates source which usually being used in formation of geopolymer includes metakaolin, fly ash, and ash. Fly ash has advantage for being cheaply available (Temuujin, et al., 2010). By the year 2010, The total of fly ash worldwide production is approximately 750 million. The main contributors are China and India. A significant effort has been made in utilization of fly ashes about 20% of the fly ash generated is being used in concrete production. However, it is not sufficient enough as large amount of fly ash waste are still discharged into ash ponds, lagoons or landfills (Yao, et al., 2015).

In addition, replacing OPC with geopolymer also means decrease CO_2 emission. Cement production is a highly energy intensive production process. About 2% of global energy consumption or almost 5% of the total global industry energy consumption is contributed by cement industry. Due to dominant use of fuel concrete making process, Carbon dioxide emitted trough calcination process (shown in the equation 1) and fuel use, concrete industry also a major emitter of CO_2. Hendriks, et al. (2004) reported that cement industry contribute 5% of global carbon dioxide emissions. While 900 kg CO2 is emitted to the atmosphere for producing one ton of cement.

$$CaCO_3 \rightarrow CaO + CO_2 \tag{1}$$

While OPC is still dominating as the most type of concrete used in construction industry, geopolymer is growing rapidly. Davidovits (2010) stated in state of geopolymer annual conference that scientific papers published during last decades exponentially raised. It can be understood however, one of the major setbacks of using geopolymer is the uncertainty of the based material due to variety of composition depend on the silica-alumina source. Moreover, geopolymerization process is a sensitive process. Many studies have been conducted on factors influencing the formation of geopolymer and how can it affect the mechanical characteristic. The purpose of this study is to analyse and understand how certain parameters play roles in the geopolymerization to its resistance against marine environment.

1.2 Problem Statement

Ocean has been used for fishing, commercial navigation, and waste disposal since long time ago and with increasing human population there has been a corresponding increase in coastal and offshore construction. Marine structure is designed to be durable to its so called aggressive environment. Seawater is responsible to chemical attacks which happened to concrete. High content of sodium sulphate intensifies the probability of one of the most serious attack, sulphate attack.

Use of geopolymer concrete as material for the marine structure benefits the environment due to utilization of waste such as fly ash and replacement of OPC will reduce CO_2 emission. However, since the geopolymerization process is sensitive, a right measurement should be done in order to produce geopolymer which suitable to marine environment.

1.3 Objective

The objective of this report is to investigate the proper combination of factors influencing the geopolymerization process name, alkali activator, alkali concentration, and curing temperature which eventually give the best resistance to sulphate attack. This project contributes as follows:

- To synthesize fly ash based geopolymer.
- To find suitable set of parameters to produce geopolymer concrete which possess high resistance and suitable in marine environment.
- To test geopolymer concrete durability prior to sodium sulphate exposure.

1.4 Scope of Study

This project focused on the preparation of geopolymer and testing its durability against sulphate attack. Fly-ash based geopolymer was exposed to 5% $NaSO_4$ solution and its compressive strength and weight change examined and compared afterward. The geopolymer synthesis parameters such as curing temperature, type of alkali activator, and alkali concentration were varied.

CHAPTER 2

LITERATURE REVIEW

2.1 Geopolymer and Geopolymerization

2.1.1 The origins of geopolymer

The term ‘Geopolymer’ is applied to a class of solid materials synthesized by the reaction of an aluminosilicate powder with an alkaline solution and originated by Davidovits in 1970s. The characteristic that represent geopolymer is that the binding phase comprises an alkali aluminosilicate gel, with aluminium and silicon linked in a three-dimensional tetrahedral that is relatively resistant to dissolution in water (Van Deventer & Provis, 2009).

In the past 10 years, there was a significant increase in geopolymer research which most has been linked with geopolymers’ potential as fire resistance material, decorative stone artefacts, thermal insulation, low-tech building materials, low energy ceramic tiles, refractory items, thermal shock refractories, bio-technologies, foundry industry, cements and concretes, composites for infrastructure repairing and strengthening, high-tech resin systems, radioactive and waste containment, arts and decoration, cultural heritage, and archaeology (Davidovits 2011).

However, the primary application for geopolymer binders has since shifted to uses in construction. The primary area of application of geopolymer technology is currently in the development of reduced-CO_2 construction materials as an alternative to Portland-based cements. Mechanism of Geopolymerization

In general, geopolymerization which an exothermic reaction consists of three steps including dissolution, reorientation, and solidification as shown in figure 2.1.

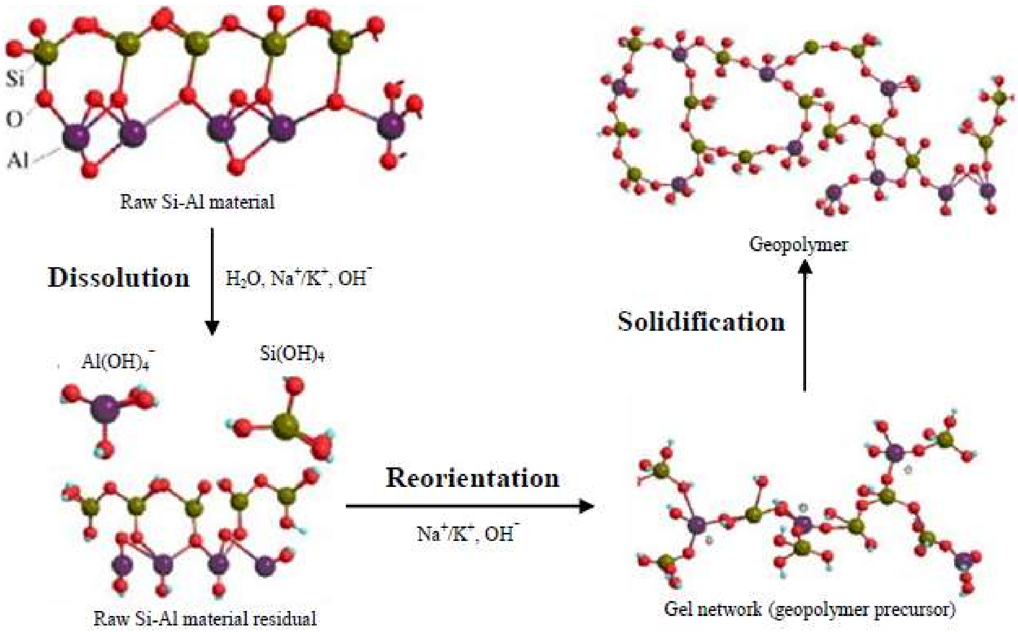


Figure 2.1 Geopolymerization Scheme

The first step, dissolution, is where Si-Al raw materials come in contact with activating solution which results in Si and Al species are produced. According to Xu (2002) the formation of Si and Al species influenced by the concentration of the alkaline solution as the activating solution, the type of metal cation presence in the solution (e.g., Na^+, K^+), mixing rate and time, as well as intrinsic properties (e.g., structure and composition) of Si-Al raw materials. Among these factors, concentration of alkaline solutions and the intrinsic properties are dominant.

In the reorientation step, dissolved Si and Al species are diffused into oligomers. When formed, the oligomers are in aqueous phase forming large network by condensation resulting in formation of a gel. Immediately upon the dissolved Al^{3+} and Si^{4+} are removed from the surface of Si-Al materials, further leaching of reactive

Al and Si species from the raw materials is occurring. During this step, time and intensity of stirring are essential factors for this step. Intensive stirring and longer leaching period maximize the removal of dissolved Si and Al species from the surface of raw materials and kinetically break the boundary between the Si-Al particle surface and the gel phase. Hence accelerate the reorientation of Al and Si species (Xu, 2002).

Next step, solidification, the gel network continues to rearrange and reorganize. As the connectivity of the gel network increases, amorphous or semi-crystalline three-dimensional alumina silicate network formed and called as geopolymer. In this step, temperature and air circulation are two primary factors which play roles in determining the properties of the geopolymer formed. It needs to be notified, there are no order for these 3 steps. Each steps occur simultaneously (Xu, 2002).

2.2 Basic Materials

2.2.1 Fly Ash

Fly ash is waste produced from the combustion of finely ground coal and collected using electrostatic precipitation (Tishmack, 1996). According to U.S Environmental Protection Agency (2010) fly ash can also be removed using scrubber system. Fly ash has very fine form physically, spherical in shape, and range in size from 0.5 µm to 100 µm. It consists mostly of SiO_2, Al_2O_3, and Fe_2O_3.

Figure 2.2 Fly Ash

The particle size distribution and chemical composition of fly ash, as well as shape and available at low cost, make fly ash an ideal material for use as a supplementary cementitious material in concrete. (Keyte, 2009).

According to American Coal Ash Association (ACAA) (2003), the utilization of fly ash particularly in concrete industry has significant environmental benefits and superior mechanical properties which are explained further below:

- Improved workability imparted by the spherical ash particles and the associated water reduction that minimizes separation of water from the cement mixture.
- Improved compressive strength and other mechanical properties as a result of the reduced water demand.
- Reduced concrete cost as the value of coal fly ash is lower than that of cement.
- Reduced CO_2 emission as less cement is required.
- Improved durability, and in some cases improved strength, in hardened concrete due to the pozzolanic reaction with calcium hydroxide generated during cement hydration increasing the volume of calcium silicate hydrate binder, which helps fill the reduced water voids and thus creates a more durable and less permeable concrete.

Chemical composition of fly ash depends on the type of the coal. Fly ash from Sub-bituminous coal contains more calcium but less iron compared to bituminous coal. In addition, the combustion methods also contribute in physical and chemical characteristics of fly ash (Malhotra & Ramezanianpour, 1994). Three classes of fly ash are defined by ASTM C618: Class N fly ash, Class F fly ash, and Class C fly ash. Amount of calcium, silica, alumina, and iron in the fly ash distinguish between these classes. Coal fly ash with low calcium content is typically referred to as class F fly ash, although the actual definition of a class F fly ash is when the sum of the silica, alumina and iron oxide is greater than 70% (Keyte, 2009). The chemical content requirement for each class is summarize in table 2-1.

Table 2.1 Chemical Requirements of Fly Ash (ASTM C 618 2005)

	Class N	Class F	Class C
Silicon dioxide (SiO_2) + aluminium oxide (Al_2O_3) + iron oxide (Fe_2O_3), min %	70	70	50
Sulphur trioxide (SO_3), max %	4	5	5
Moisture content, max %	3	3	3
Loss on ignition, max %	10	6	6

2.2.2 Alkali Hydroxide as Activating Solution

In order to activate or dissolve the amorphous, reactive silica and alumina, high alkalinity is needed in geopolymerization process. Stronger alkalinity cause in faster and more extensive dissolution of the source material as well as induces more reactive silica and alumina species it depresses ettringite and carbon-hydrogen formation during formation of binder. Hence the higher extent and degree of geopolymerization obtained, which means more generation of geopolymer binder. Hardjito et al. (2004) stated the most significant factor for geopolymerization is alkali concentration, with higher concentration yields a higher compressive strength. Karacoc et al. (2015) added higher concentration promotes higher strengths at early stages of reaction, but the strength of aged materials was compromised due to excessive OH^- in solution causing unwanted morphology and non-uniformity of the geopolymer.

Various different types of solutions have been used as activators in geopolymer synthesis, and the chemical and physical properties of each will play a role in determining the properties and value of geopolymers synthesized using them. Sodium and potassium silicate and hydroxide (NaOH, KOH, $NaSiO_3$, $KSiO_3$) are the most commonly used activating solutions, with sodium-based solutions being less expensive, but potassium-based solutions displaying more favourable phase behaviour and theology. Alternative activating solutions include carbonates, aluminates and even water, but none of these have seen widespread use in the synthesis of aluminosilicate geopolymers to date. (Van Deventer & Provis, 2009) (Duxson, 2009).

In a processing context and aside from their obviously high corrosivity, the most important properties of concentrated hydroxide solutions that must be considered are viscosity and heat of dissolution. Figure 2.3 shows the variation of viscosity with concentration at 25°C for each of the alkali hydroxides from Van Deventer & Provis (2009). And Figure 2.4 shows the heat of dissolution for common used hydroxide solution.

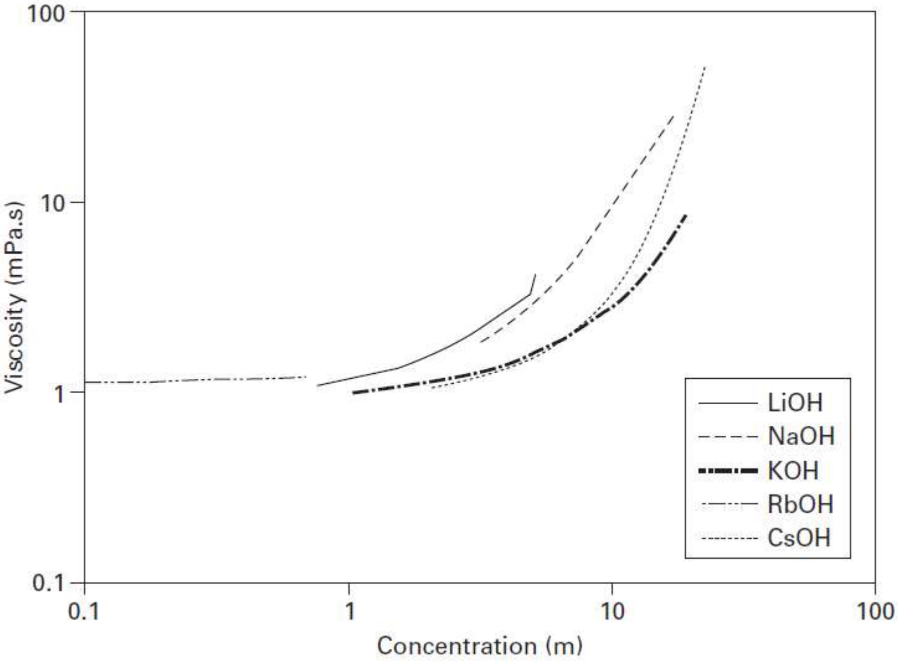


Figure 2.3 Viscosities of alkali hydroxide solutions as a function of molality.

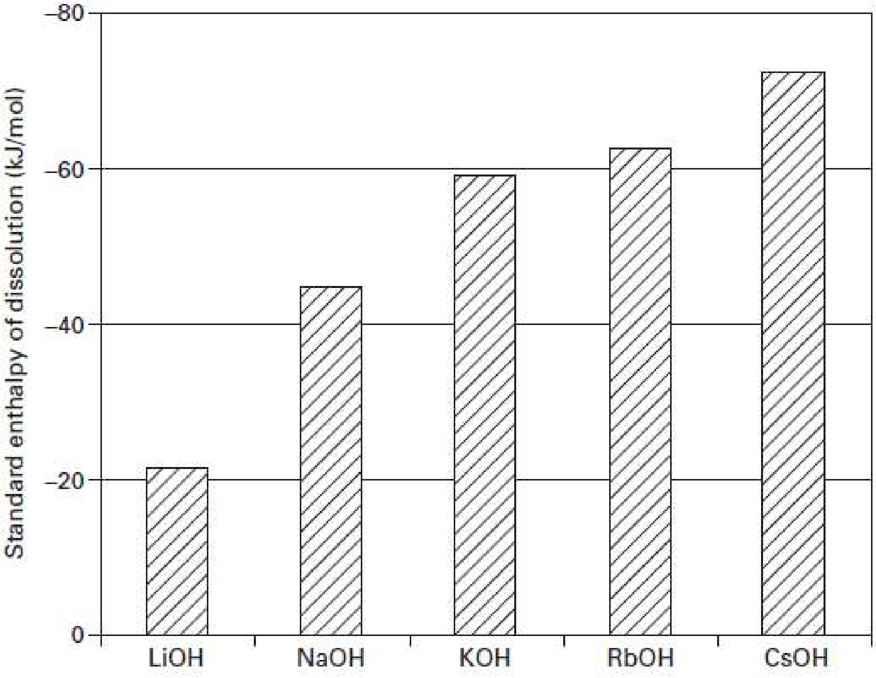


Figure 2.4 Standard enthalpies of dissolution

In term of microstructure appearance, Mustafa et al. (2011) stated that reacting NaOH with fly ash particles resulted in the roughness of surface as shown in figure 2.5

and 2.6. SEM was used to investigate the surface of fly ash before and after reacting with NaOH.

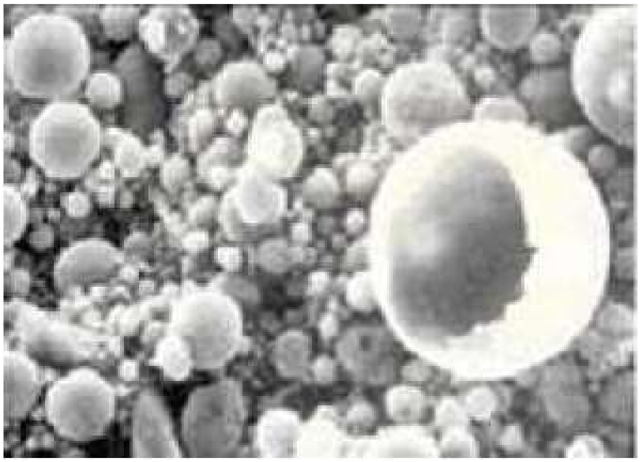


Figure 2.5 Fly Ash before reacting with NaOH

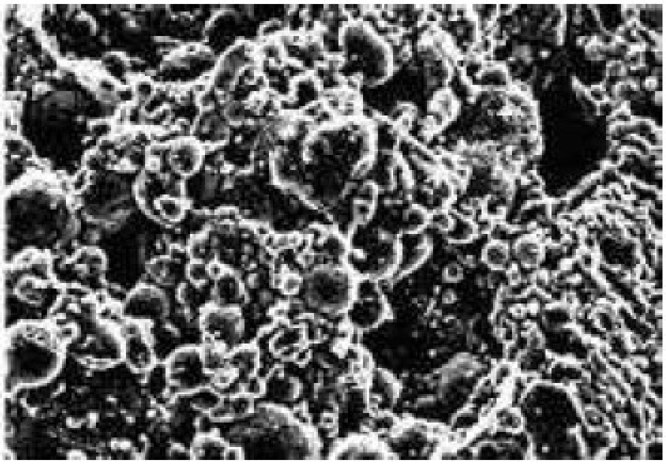


Figure 2.6 Fly Ash after reacting with NaOH

2.3 Geopolymer Synthesis Parameter

There are factors which affecting the formation of geopolymer including Al/Si ratio, curing temperature, curing time, Solid/Liquid ratio. This project focused on the curing temperature as well as solid/liquid ratio. Khale & Chaudhary (2007) reviewed all of the factors influencing the development of geopolymer. Interest parameters in this project are explained based on Khale & Chaudhary review.

2.3.1 Curing Temperature and curing time

Important factors in development of engineering properties of fly ash based geopolymer are curing temperature and time. Generally, higher temperature increases geopolymerization reaction rate. Increase in temperature also lower the water content as well as total pore volume and surface area (Bhowmick, 2012) (Sindunatha et al., 2006). The geopolymer cured at lower temperature is more porous.

Geopolymerization reactions become faster if occurring in higher temperature environment. At room temperature the reaction is extremely slow. It is reported increase in temperature results in increase in compressive strength of the final product. Prolonged curing period improve product the compressive strength, however for curing time beyond 48 hours the difference in strength is not significant (Khale & Chaudhary, 2007).

2.3.2 Solid-Liquid ratio

Though hydration is not taking place during geopolymerization, water is responsible to increases the cohesiveness and flow ability of the mix and as a result compactness increases which lead to increase in the strength. However, ratio beyond 3 to 1 affects the specimen become more porous cause decrease in strength (Bhowmick, 2012).

Strength increases as the ratio of solid to liquid increases. This trend is shared with cement to water ratio in OPC. Although chemical processes involved in the formation of binders of both are entirely different. Geopolymerization will still occur even in very high solid/liquid ratio. One of the main factor inducing crystallization is the excess amount of the water (Khale & Chaudhary, 2007).

2.3.3 Summary

The table 2.2 is the summary of recent studies done with Fly Ash as the main component and Na as metal activator:

Table 2.2 Summary of recent studies on reaction parameters

S.No	NaOH (M)	Curing in Oven		Curing (days)	Water/Solid Ratio	Compressive Strength (MPa)	Reference
		Temp (°C)	Time (h)				
1	10	80°C	4	14	0.35	6.28	(Adam & Horianto, 2014)
2	10	80°C	6	14	0.35	8.64	(Adam & Horianto, 2014)
4	10	80	4	7	0.35	3.16	(Adam & Horianto, 2014)
8	NA	50	24	1	NA	75	(Khale & Chaudhary, 2007)

2.4 Geopolymer Resistance to Chemical Attack

Concrete durability has become a critical issue. There are many concrete structure in urban and coastal environments deteriorate 20-30 years earlier than their design life. The environment has deteriorated due to acid, sulphate, chloride and carbon monoxide. Duan et al. (2015) reported that geopolymer concrete has a superior chemical attack resistance compared to OPC.

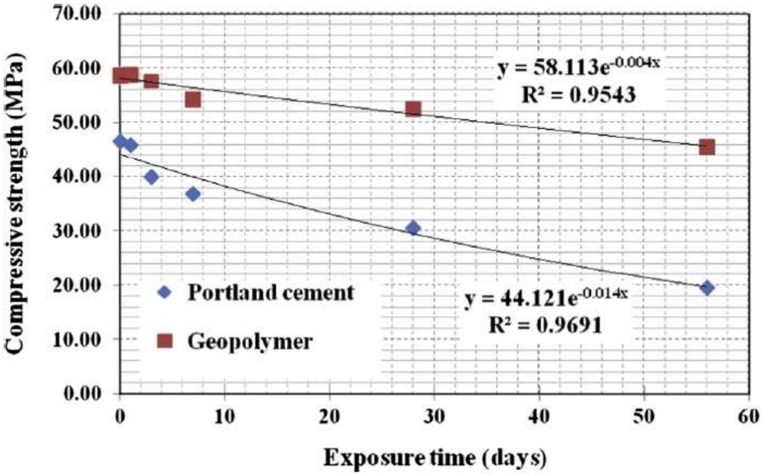


Figure 2.7 Compressive Strength comparison between OPC and Geopolymer after 2% Acid Exposure (Duan et al., 2015)

Thokchom et al (2010) claimed that fly ash based geopolymer have shown good performance when exposed to different acids with varying concentration and exposure duration. Geopolymer activated with NaOH develops greater crystallinity hence has better stability in aggressive environment such as sulphates.

Another most concerning the durability of the concrete structure is sulphate attack. A sulphate attack can cause a cement paste undergoes deterioration which results in expansion, spalling and softening (Karacoc et al, 2015).

Studies on OPC show the sulphate attack is a complicated mechanism where reactions between cement hydration product and sulphate bearing solutions has variety of ways. The external sulphate attack on OPC concrete show that the reaction involves C-H, C-S-H and the aluminate component of hardened paste.

CHAPTER 3

METHODOLOGY

3.1 Project Sequence

This project is done under a sequence shown in the figure 3-1. The sequence was followed strictly in order to produce high quality of paper.

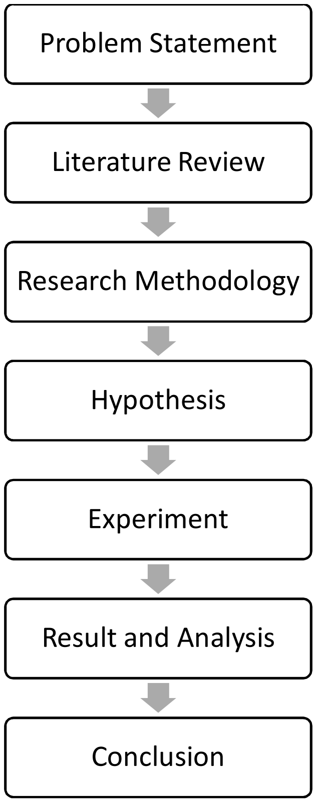
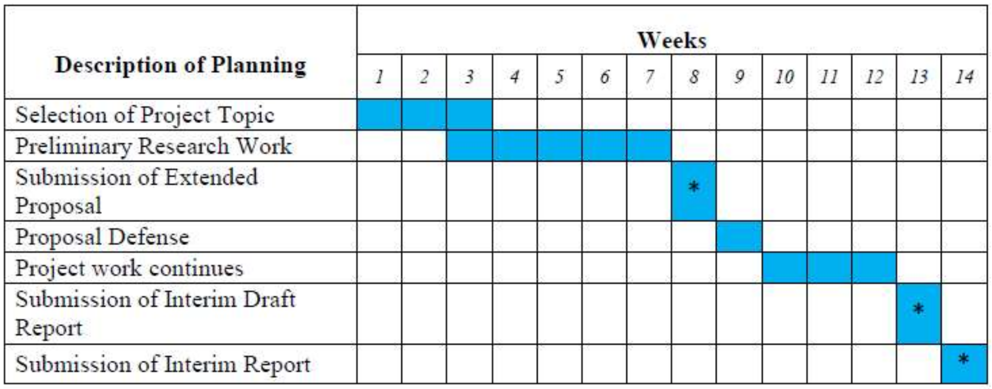


Figure 3.1 the Sequence of Work

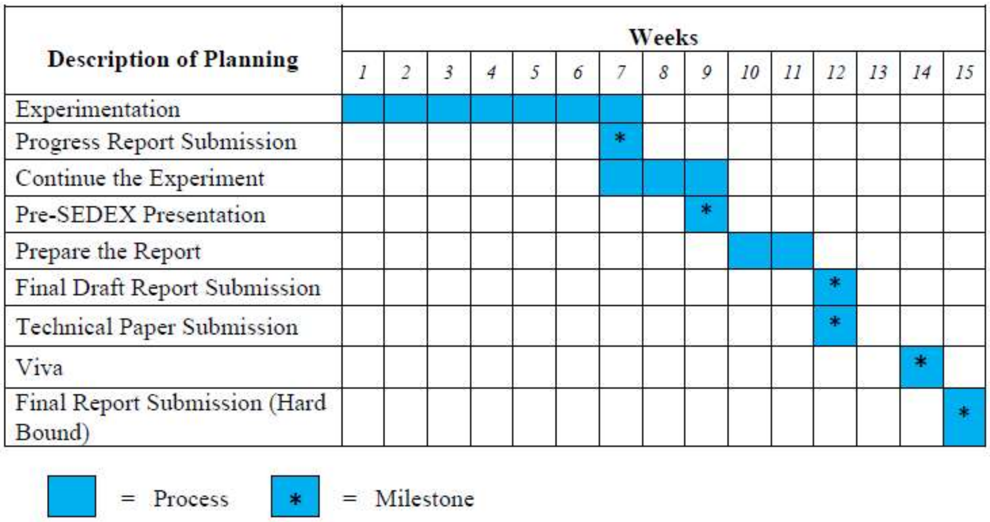
3.2 Gantt Chart/Key Milestone

Final year project is expected to finish within two semesters (8 months) and consist of two phase, final year project I and final year project II. This project started on May 2015 semester and completed on September 2015 semester. Project Gantt chart and key milestone for FYP I and FYP II are shown in table 3-2 and 3-3 respectively.



Description of Planning	Weeks													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Selection of Project Topic	🟦	🟦	🟦											
Preliminary Research Work			🟦	🟦	🟦	🟦	🟦							
Submission of Extended Proposal								*						
Proposal Defense									🟦					
Project work continues										🟦	🟦	🟦		
Submission of Interim Draft Report													*	
Submission of Interim Report														*

Table 3.1 Gantt chart & Milestones for FYP I



Description of Planning	Weeks														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Experimentation	🟦	🟦	🟦	🟦	🟦	🟦	🟦								
Progress Report Submission							*								
Continue the Experiment							🟦	🟦	🟦						
Pre-SEDEX Presentation									*						
Prepare the Report										🟦	🟦				
Final Draft Report Submission												*			
Technical Paper Submission												*			
Viva														*	
Final Report Submission (Hard Bound)															*

🟦 = Process * = Milestone

Table 3.2 Gantt chart & Milestones for FYP II

3.3 Materials and Tools

Materials and tools used in this project are listed below. All of the materials and tools are provided by UTP.

3.3.1 Materials

- Fly-ash
- Sodium Hydroxide (NaOH)
- Potassium Hydroxide (KOH)
- Sodium Sulphate ($NaSO_4$)
- Distilled Water

3.3.2 Tools

- Cement Moulds 50mm x 50mm x 50mm
- Mixer
- Compressive Strength Tester
- Oven
- Weighing Machine
- X-Ray Fluorescene (XRF) Analyzer
- Scanning Electron Microscope (SEM)
- Fourier Transformed Infrared (FTIR) Spectrometer

3.4 Experimental Procedures

This research was done in several stages as outlined in figure 3-4. First stage is preparation of the raw material, fly ash & alkaline activator. Next is geopolymer preparation by mixing raw materials. After mixing geopolymer is allowed to settle, this stage is called curing stage. Subsequently cured geopolymer tested to determine the mechanical properties reference state. Later another geopolymer is prepared to undergo treatment which is $NaSO_4$ exposure. Finally, compressive strength and change in mass of exposed geopolymer are analysed.

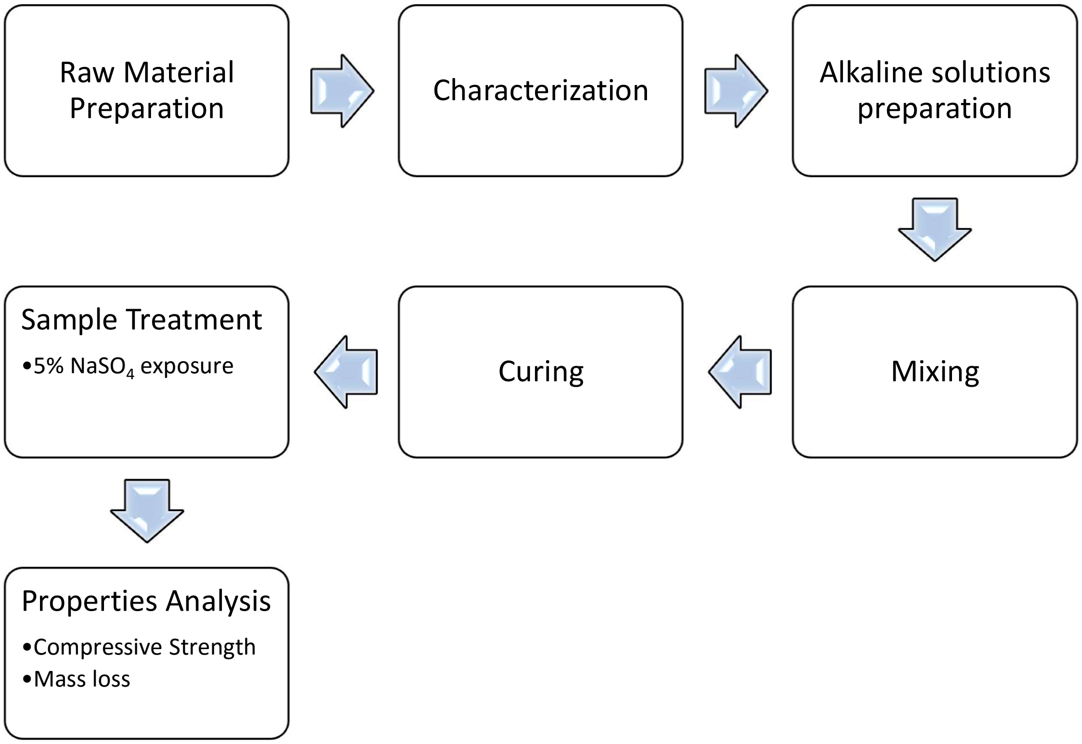


Figure 3.2 Research Procedure

3.4.1 Raw Material and Alkaline Solutions Preparation

Fly ash, used as a raw material, provides both alumina and silica which are basic component for geopolymerization. NaOH and KOH were used as alkaline activators. For mixing purpose amount of fly ash and alkaline activator ratio is kept constant 3:1 by mass (Bhowmick, 2012). 500 gram of fly ash mixed with 167 gram of specific concentration alkaline activator. The alkaline activator concentrations used are 12, 10, 8, 6 and 4 M for each type of alkaline activator.

Both NaOH and KOH were available on solid form. To prepare solution with specific concentration, Equation 2 is used to determine how much grams of NaOH and KOH.

$$m\ (gram) = M\left(\frac{mol}{L}\right) \times V\ (L) \times Mr\ \left(\frac{gram}{mol}\right) \tag{2}$$

Where;

m = Mass of required alkaline

M = Molarity of expected solution

V = Volume of the solution

Mr = Molecular Mass of the solution

Basic properties of alkaline activator NaOH and KOH and example of the calculation is available on the appendix.

3.4.2 Characterization of Raw Material

Characteristic such as chemical composition and component structure of fly ash was predetermined prior to geopolymerization. XRF analyzer are used to analyse chemical component of fly ash. Scanning result is shown in the table 3.3.

Table 3.3 Fly Ash Components

Formula	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	SO_3	Na_2O	TiO_2
Concentration (%)	43.34	20.77	12.41	11.13	3.75	1.98	1.45	0.95	0.88

Formula	P_2O_5	BaO	SrO	MnO	V_2O_5	ZrO_2	ZnO	CuO	Cr_2O_3
Concentration (%)	0.32	0.17	0.12	0.11	0.04	0.04	0.02	0.01	0.01

Based on the table 2.1 and due to high calcium content (8%-20%), it can be concluded that fly ash used can be classified as class C (ASTM C618, 2005).

3.4.3 Mixing and Curing

Geopolymerization started by mixing prepared material using mechanical mixer for 5 minutes with constant speed in the mixing vessel as shown in figure 3.5. The mixtures later were quickly casted in 50 mm x 50 mm mould (figure 3.6). After that the geopolymers were cured in different temperature of 25°C and 60°C for 24 hours. After geopolymer cured, it mass was recorded.

Figure 3.3 Mixing Vessel

Figure 3.4 Geopolymer Mould

3.4.4 Na_2SO_4 exposure

Cured geopolymer then exposed to 5% Na_2SO_4 solution for 14 days. Na_2SO_4 solution serves the purpose of representing marine environment. The specimens were put in the container filled with Na_2SO_4 solution as shown in figure 3-7. After 14 days of exposure the specimens were dried to remove remaining solution and then weighted.

Figure 3.5 Geopolymer exposed to sodium sulphate

3.4.5 Experiment parameters summary

Synthesis parameters which become our concern are curing temperature, curing time, type of alkaline activator, alkaline concentration, and solid liquid ratio. The curing temperature, type of alkaline activator and alkaline concentration become the variable parameters while the rest are fixed parameters.

The table 3.4 summarize the synthesis parameters used in this project.

Table 3.4 Summary of the parameters used

Parameters	Parameters Description
Curing Temperature	25°C, 60°C
Curing Time	24 hours
Type of Alkaline Activator	NaOH, KOH
Alkaline Concentration	4, 6, 8, 10, 12 M
Solid-Liquid Ratio	3:1

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Experimentation Design

The experiment was focused on preparing geopolymer concrete at various curing temperature, type of alkaline activator and alkaline activator concentration. Remaining parameters such as curing time and solid/liquid ratio were kept constant. Based on the type of alkaline activator, formulation of geopolymer can be divided into two: NaOH based and KOH based. Detailed formulation can be observed in table below.

Table 4.1 NaOH activated formulation

Fly Ash	NaOH	No of sample
500 grams	167 grams 4M	2 sample
	167 grams 6M	
	167 grams 8M	
	167 grams 10M	
	167 grams 12M	

Table 4.2 KOH activated formulation

Fly Ash	KOH	No of sample
500 grams	167 grams 4M	2 sample
	167 grams 6M	
	167 grams 8M	
	167 grams 10M	
	167 grams 12M	

4.2 Results

4.2.1 Compressive Strength

The compressive strength of the concrete samples was measured using a ASTM C39 test method. The compressive strength was determined after specimen exposed to sulphate solution with 14 days.

In figure 4.1 and figure 4.2 compressive strength between specimens cured in different temperature is shown. Compressive strength of specimens cured in 60°C is higher than specimens cured in 25°C regardless its type or concentration of activating solution. Insufficient geopolymerization in curing temperature 25°C compared to 60°C left the specimens more porous. Porous geopolymer is more susceptible to sulphate attack.

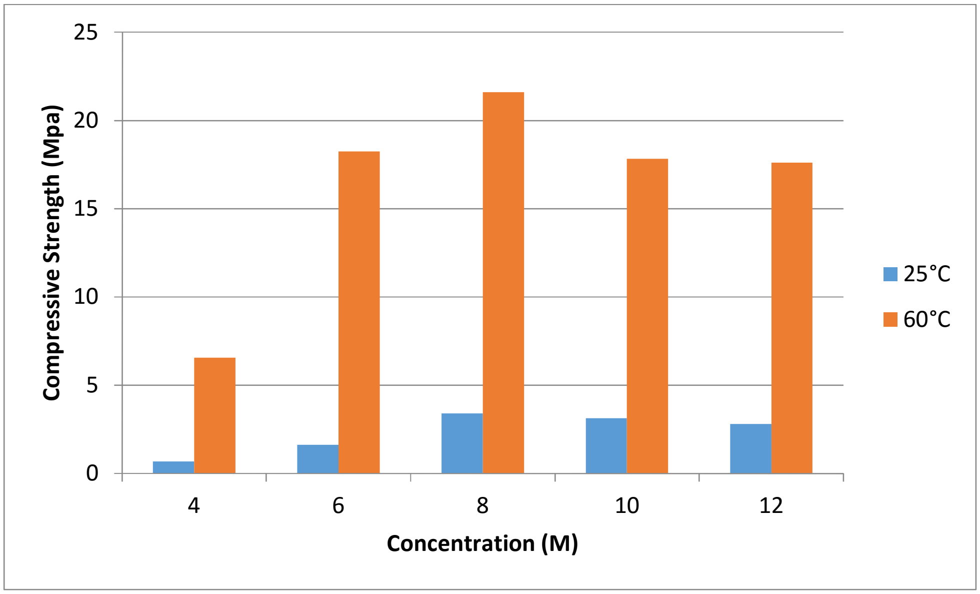


Figure 4.1 Compressive strength of NaOH activated geopolymer

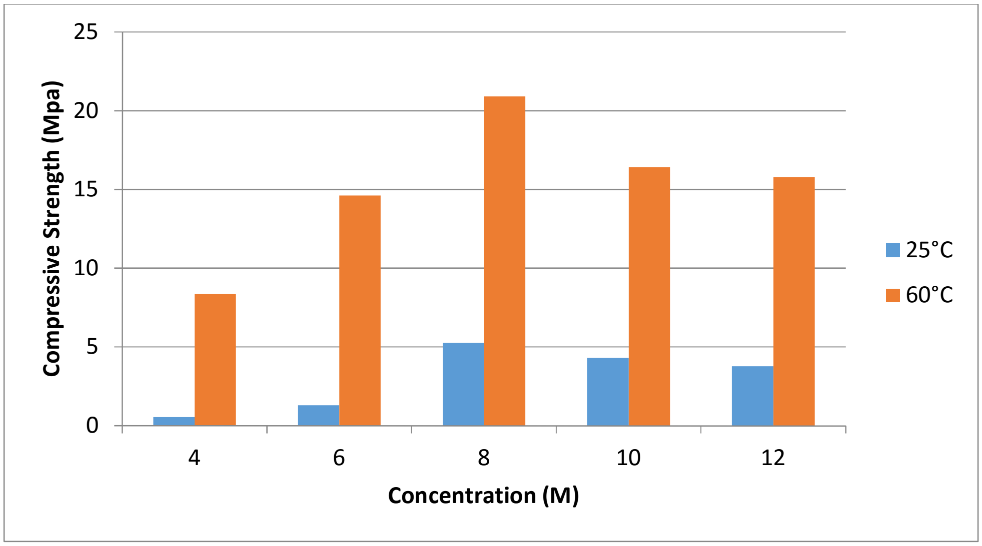


Figure 4.2 Compressive strength of KOH activated geopolymer

The performance of NaOH activated and KOH activated geopolymer in each temperature can be observed in figure 4.3 and figure 4.4. Both NaOH activated and KOH activated have same pattern in both of the temperature condition. The graph begins increasing in compressive strength as the concentration increase. And then the compressive strength has its peak in 8M concentration and is decreasing as the concentration increase. Stronger concentration of alkali activator results in faster and more extensive dissolution hence more generation of geopolymer, however up to 8M increasing excessive OH^- in solution results in non-uniformity of the geopolymer.

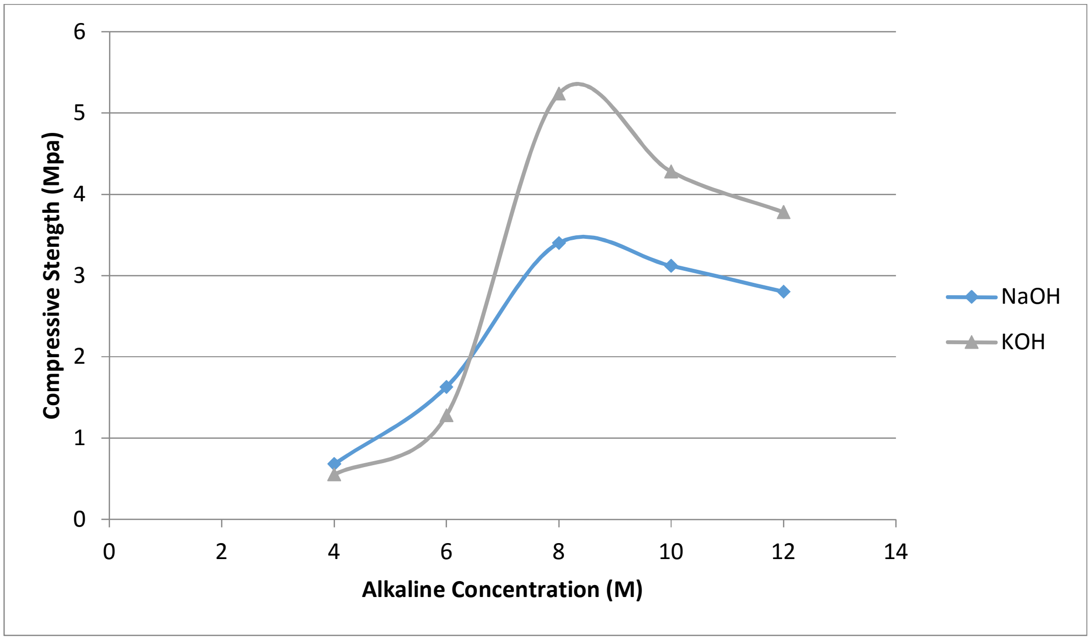


Figure 4.3 Compressive Strength in 25°C

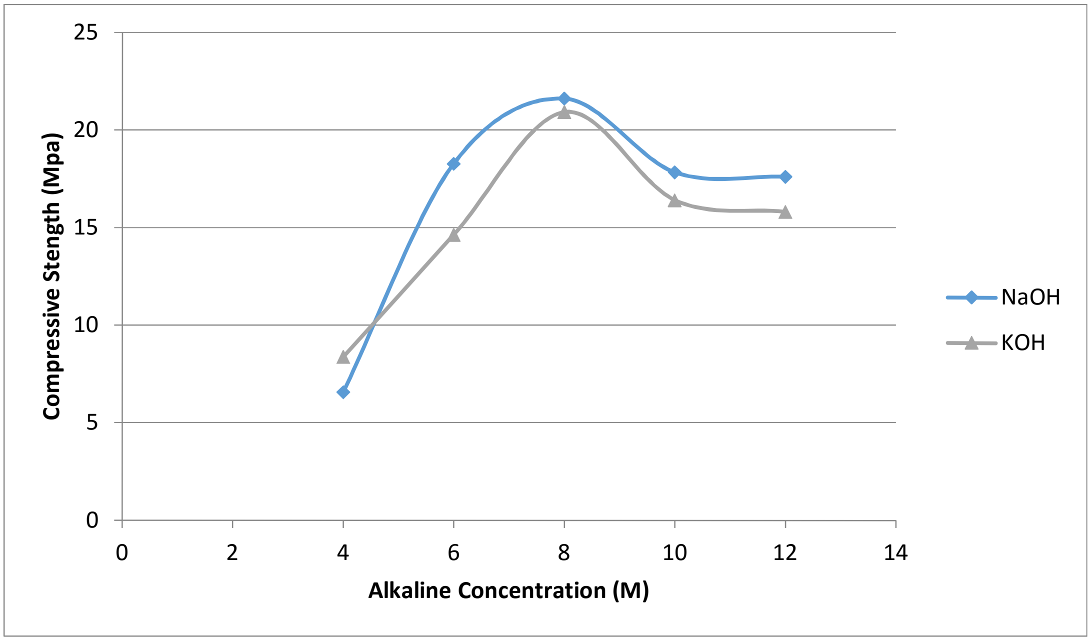


Figure 4.4 Compressive Strength in 60°C

4.2.2 Change in weight

Change in weight of specimen was measured after 14 days of sulphate exposure. Change in weight of each specimen is shown in table 4.3 and table 4.4. It can be observed that specimens cured in 25°C response in different manner compared to specimens cured in 60°C.

Table 4.3 Specimens cured in 25°C change in mass after exposure

Samples	Concentration (M)	Before exposure	After Exposure	Change in weight (%)
NaOH activated Geopolymer	4	240.60	234.13	2.69
	6	247.08	241.30	2.34
	8	258.48	250.91	2.93
	10	252.42	243.27	3.62
	12	257.29	250.00	2.83
KOH activated Geopolymer	4	251.01	249.31	0.68
	6	250.54	248.62	0.77
	8	263.08	254.48	3.27
	10	250.35	246.26	1.63
	12	251.57	245.60	2.37

Specimens cured in 25°C suffer a weight loss after sodium sulphate exposure. The weight loss results obtained in the study showed the specimen with higher alkali content to lose more weight than specimens with lower alkali content. Suffer of weight loss happens due to incomplete geopolymerization process leaving the specimens relatively big porous. Exposed to the sulphate attack, the specimens quickly eroded in just 14 days of exposure.

Table 4.4 Specimens cured in 60°C change in mass after exposure

Samples	Concentration (M)	Before exposure	After Exposure	Change in weight (%)
NaOH activated Geopolymer	4	234.69	244.39	4.13
	6	244.73	253.48	3.58
	8	252.69	257.17	1.77
	10	251.52	255.77	1.69
	12	253.43	258.29	1.92
KOH activated Geopolymer	4	247.38	255.30	3.20
	6	251.94	256.24	1.71
	8	256.75	259.76	1.16
	10	256.98	257.05	0.03
	12	257.2	259.14	0.55

For specimens cured in 60°C, increase in mass after sodium sulphate exposure was observed. The slight increase in the mass of specimens is due to the absorption of the exposed liquid. Geopolymer with lower content of alkali absorb more liquid compared to higher content of alkali geopolymer. Porous inside the geopolymer filled with the liquid, which expected to erode and damage the geopolymer in longer exposure time. In addition, for the construction purposes change in mass should be minimum as possible to make sure stability of the construction.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Relevancy to the Objective

In conclusion, the objective of this project is to find a suitable synthesize parameter for geopolymerization which have high resistance to marine environment represented by sodium sulphate solution. This project utilized fly ash as the silica-alumina source for geopolymerization due to its low cost and availability, environmental friendly, and superior end products. Based on the result obtained, it can be concluded as follow:

1. Geopolymer cured in 60°C have superior resistance to sulphate attack shows by its compressive strength compared to geopolymer cured in 25°C regardless its type and concentration of the alkali activator.

2. Both NaOH and KOH activated geopolymer have compressive strength peak at 8M concentration after the exposure. It increases in strength as the concentration approach 8 M and decrease in larger concentration than 8M. High compressive strength is more favourable for construction purposes. Therefore, 8M is the most suitable parameter to be used.

3. When exposed to sulphate solution, geopolymer cured in 25°C eroded and its mass decreased as a result. While geopolymer cured in 60°C increases in its own mass.

Therefore, for the marine construction the best set of parameter to form geopolymer is using 60°C as curing temperature and 8M of alkali activator.

5.2 Suggested Future Work for Expansion and Continuation

There are a lot of factors affecting the geopolymerization. Moreover, there are also a lot more condition that may represent marine environment. It is recommended for future work to continue the project as follow:

1. investigating the effect of parameters during geopolymerization to its resistance to marine environment. Curing temperature can be varying in order to find the optimum temperature.
2. Addition of sodium silicate to activating solution is also interesting parameter.
3. Change source of silica-alumina material.
4. Test another chemical attack that may occur in marine environment.
5. Another qualitative and quantitative approach to determine superior specimens is encouraged such as XRF or SEM analysis and change in compressive strength before and after exposure.

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APPENDICES

Calculation of NaOH and KOH required to make specific concentration of alkali solution

The formula is as the following:

$$\mathrm{m\ (gram)} = \mathrm{M}\left(\frac{\mathrm{mol}}{\mathrm{L}}\right) \times \mathrm{V\ (L)} \times \mathrm{Mr}\left(\frac{\mathrm{gram}}{\mathrm{mol}}\right) \tag{4}$$

Where;

m = Mass of required alkaline

M = Molarity of expected solution

V = Volume of the solution

Mr = Molecular Mass of the solution

Let’s say, 1 L of 12 M NaOH and KOH is required:

Mr of NaOH and KOH is equal to 40 and 56 gram/mol respectively

For NaOH 12 M

$$\mathrm{m\ (gram)} = 12\left(\frac{\mathrm{mol}}{\mathrm{L}}\right) \times 1\ \mathrm{(L)} \times 40\left(\frac{\mathrm{gram}}{\mathrm{mol}}\right) \tag{5}$$

$$\mathrm{m\ (gram)} = 480\ \mathrm{gram} \tag{6}$$

For KOH 12M

$$\mathrm{m(gram)} = 12\left(\frac{\mathrm{mol}}{\mathrm{L}}\right) \times 1\ \mathrm{(L)} \times 56\left(\frac{\mathrm{gram}}{\mathrm{mol}}\right) \tag{7}$$

$$\mathrm{(gram)} = 672\ \mathrm{gram} \tag{8}$$

Since dilution of high amount of alkali produces a lot of heat, therefore it should be avoided. It is recommended to start with medium concentration such as 8 M then add a bit more NaOH or KOH to create higher concentration and add distilled water to create lower concentration. It can be done by following the equation as follows:

For creating lower concentration of solution or dilution

$$V_1 \times M_1 = V_2 \times M_2 \tag{9}$$

$$V = V_2 - V_1 \tag{10}$$

Where;

V_1 = volume of the solution before dilution (mL)

M_1 = Molarity of the solution before dilution (M)

V_2 = volume of the solution after dilution (mL)

M_2 = Molarity of the solution after dilution (M)

V= volume of the distilled water to be added to the solution (mL)

For creating higher concentration of solution

$$M_2 = \frac{(M_1 \times V_1) + (\frac{m}{Mr})}{V_1} \tag{11}$$

Where;

V_1 = volume of the solution before adding (mL)

M_1 = Molarity of the solution before adding (M)

M_2 = Molarity of the solution after adding (M)

m = Mass of the NaOH or KOH to be added (gram)

Mr = Molecular weight of NaOH or KOH (gram/mol)

Sample of geopolymer 50mmx50mmx50mm cube