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Development of Chemical Resistant Geopolymer As Waste Storage Containing Hazardous Material

by

Mohamad Khairulanuar Bin Md Mustfa

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

(Chemical Engineering)

MAY 2013

Universiti Teknologi PETRONAS
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Perak Darul Ridzuan

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CERTFICATION OF APPROVAL

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A project dissertation submitted to the
Chemical Engineering Programme
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Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
MAY 2013

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, tha 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.

MOHAMAD KHAIRULANUAR BIN MD MUSTFA

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ABSTRACT

This research paper is to develop a geopolymer as as waste storage containing radioactive material. This topic is very relevant to our current world as there is still no certain solution for the storage for the radioactive material. The rate at which other environmental problems are mounting is also alarming. The toxic dumps are filled at a rapid pace, with few sites being developed. Abandoned mining waste laden with heavy metals and acidic solutions are poisoning vast acres of once-virgin land. The world water quality is worsening and our river and sea are being contaminated with this chemical wastes.

In order to fulfill the objective of this research in which to develop a sustainable radioactive waste container by using geopolymer material, number of tests and experiments need to be conducted. The tests that are need to undergone are water absorption test, sulfuric acid test on geopolymer, and also sodium sulfate test on geopolymer. The main purpose of these tests is to determine the best properties out from the geopolymer to be applied as waste storage. The length of this research is up to six months starting from March 2013 until September 2013.

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ACKNOWLEDGEMENT

First and foremost, thanks to God for His blessing and mercy in completing this one year project. It would befit to extend my outstretched gratitude to AP. Dr. Zakaria Bin Man, Chemical Engineering Department, Universiti Teknologi PETRONAS. It is a privilege to be under his supervision. Even with his tight schedules as lecturer and high commitment to Universiti Teknlogi PETRONAS, there was no moment where she fails to provide support and guidance. His advices and moral support gave a sense of strength and confidence in conducting the final year project.

Many thank to our Final Year Project Coordinators, for their unlimited contributions success in providing the students with guidelines and seminars to enlighten hopes of confidence. Not forget to thank all lab executive and technicians as their willingness to provide the facilities and entertain our demand during conducting the project.

Last but not least, thanks to all the Universiti Teknologi PETRONAS involved lecturers and students who have been contributing great efforts and ides making this final year project a success.

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CHAPTER 1
INTRODUCTION

1. INTRODUCTION

1.1 Background

This project seeks to manufacture chemical resistant geopolymeric cements primarily for the long-term containment of hazardous and toxic waste. The good durability of Portland cement compositions in normal service environments has long been recognized. However, cements and concretes made with cement binders can be attacked and, as a result, exhibit a reduced service life. Most of the adverse conditions are recognized from experience and have been the subject of numerous examinations of field concretes as well as laboratory studies. Not surprisingly, research and testing have focused on the areas of underperformance. The concept of a “service life” is not new.

The ancient world used stone, brick, tile and, from Roman times onwards, concrete, because of their permanence. Today, cement and Portland cement concrete are widely used and comprise the world's major structural material. Although modern cements are much improved in properties, the high and rising cost of construction and the economic cost and disruption associated with replacement and renewal, especially of major infrastructure facilities, placed new pressures on ensuring durable construction. Again, these pressures are not new but have intensified particularly in view of the relatively high carbon penalty associated with cement production and use. The perceived problems arising from limited performance have long been the subject of investigation. Most of this has been empirical in nature although often employing sophisticated statistical controls. We have also seen the rise of modeling, as a way of predicting durability and compressing the time factor without distortion of the underlying mechanisms. Thus the art and science of durability are in a state of great activity with the development of a variety of

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approaches. This is healthy. But we have so much of significance to report that this review can only capture selected aspects of current research.

In this research paper, author will analyze the characteristic of geopolymer cements through the experiment. The experiment need to be conducted in order to confirm that the commercialize cements which is known as Portland cements are strongly affected by the acidic medium. This test also needs to prove that exceptional good properties of geopolymeric cements.

1.2 Problem Statement

Generally the toxic and hazardous waste material released corrosive substance for an instance sulfuric acid. This corrosive acid will react with geopolymer and subsequently will cause the drop of geopolymer performance in reliability as a storage waste material. The effect of acid with time is study in detail in term of reaction kinetic. This waste may also have some others bad effect on geopolymer that to be studied further in this project.

1.3 Objective

The objective of this project is to develop a geopolymer waste storage which is chemically resistant to hazardous material. In order for the author to fulfill this objective, there are many experiments and tests need to be conducted to ensure that this geopolymer is really suitable to be used as hazardous waste storage. Apart from corrosion effect, the author needs to consider other tests such as acid effect and sulfate effect toward the geopolymer. Analysis also need to be done after each test, and evaluate whether the geopolymer sample is safe and pass the test to be used in industry.

1.4 The Relevancy of the project

The title of this project which is development of chemical resistant geopolymer as waste storage containing hazardous material is very much related with this current world. The rate at which other environmental problems are mounting is also alarming. Rivers and ground water are widely contaminated, and in many cases, contaminants already exceed water quality standards several fold. Up until today, there is still debate about the selection of a suitable toxic waste

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storage. Therefore, this topic is very much suitable and related in an effort of conserving our environment for future generation.

1.5 Feasibility of the project

The scope of this project is to understand the characteristic and properties of geopolymer in the application as the chemical resistant hazardous storage by conducting experiments and tests on the geopolymer sample. There are number of experiments that are going to be run such as test on sulphuric acid, test on sodium sulphate, and also water absorption. The time frame given is approximately about 3 months to complete the project. The author believed that the project will be completed in the given time frame. The equipment and tools needed to conduct the experiment are all available and provided, thus there will not be much issues to be completed the project if the author follow the dateline in the Gantt chart accordingly.

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CHAPTER 2
LITERATURE REVIEW

2. LITEATURE REVIEW

2.1 Classification of Radioactive Waste

Nuclear waste can be generally classified as either ‘low -level’ radioactive waste or ‘high-level’ radioactive waste.

2.1.1 Low-level radioactive waste

Basically all radioactive waste that is not high-level radioactive waste or intermediate-level waste or transuranic waste is classified as low-level radioactive waste. Volume-wise it may be larger than that of high level radioactive waste or intermediate-level radioactive waste or transuranic waste, but the radioactivity contained in the low-level radioactive waste is significantly less and made up of isotopes having much shorter half-lives than most of the isotopes in high-level radioactive waste or intermediate-level waste or transuranic waste.[1] Low-level wastes are usually defined in terms of what they are not. They are not spent fuel, milling tailings, reprocessed materials, or transuranic materials. Low-level waste includes the remainder of radioactive wastes and materials generated in power plants, such as contaminated reactor water, plus those wastes created in medical laboratories, hospitals, and industry. Wastes in this category usually, although not always, release smaller amounts of radiation for a shorter amount of time. [2]

2.1.2 High-level radioactive waste

High-level waste consists mostly of spent nuclear reactor fuel from both commerical power plants and military facilities, as well as reprocessed materials which can emit large amounts of radiation for hundreds of thousands of years. Commercial nuclear power plants in the U.S. alone produce 3,000

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tons of high-level waste each year.[2] This waste includes uranium, plutonium and other highly radioactive elements created during fission, made up of fission fragments and transuranic. These two components have different times to decay. The radioactive fission fragments decay to different stable elements via different nuclear reaction chains involving α, β and γ emission to innocuous levels of radioactivity, and this would take about 1000 years. [1]

In Malaysia there is one radioactive plant which is Lynas Corporation's Advanced Materials Plant (LAMP). This plant is designed at 11,000 tonnes of separated Rare Earths Oxide (REO) per annum. The rare earth mineral concentrate contains low levels of naturally occurring radioactive material. Lynas is absolutely confident that by-products of the LAMP will be recycled and reused in commercial applications, and will not require long-term storage. Lynas places hydrated residues in a safe, reliably engineered, elevated Residue Storage Facility that is designed so that there is no possibility of any leakage of material into the environment. This facility is monitored and regulated by both Lynas and the Atomic Energy Licensing Board to ensure full compliance within the approved conditions. This includes continuous air and water monitoring.[11]

Currently, industrial companies apply various type methods in storing the radioactive material. To begin with the radioactive waste management approach is to consider the nature of radioactive elements involved in terms of their half-lives and then choose the appropriate method of handling. If the concentrations of radioactive elements are largely short lived, then one would resort to what is referred to as '**delay and decay**' approach; that is, to hold on to such a waste for a sufficiently long time that the radioactivity will die in the meanwhile. A second approach is to '**dilute and disperse**' so that the hazard in the environment is minimized. But when the radioactivity is long-lived, the only approach that is possible is to '**concentrate and contain**' the activity. In order to carry out concentrating the waste (generally the sludge), chemical precipitation, ion exchange, reverse osmosis and natural or steam evaporation, centrifuging, etc. are resorted to. The main concern of all these 3 methods is that the radioactive waste material is store in a concrete container which is not applicable for durable usage as it can be easily leach and erosion by the

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corrosive substance from the waste material such as sulfuric acid and sulfate acid.

Therefore, presently there is new arising solution to this predicament which is the application of geopolymer as chemical and radioactive waste storage where the author will be discussing further in this report.

2.2 Geopolymer

Geopolymers are members of the family of inorganic polymers. The chemical composition of the geopolymer material is similar to natural zeolitic materials, but the microstructure is amorphous instead of crystalline[3].The polymerisation process involves a substantially fast chemical reaction under alkaline condition on Si-Al minerals, that results in a three dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds, as follows [4]:

$$M_n\,[-(SiO_2)_z\text{–}AlO_2]_n \cdot wH_2O \qquad (2\text{-}1)$$

Where**:** **M** = the alkaline element or cation such as potassium, sodium or calcium; the **symbol –** indicates the presence of a bond, **n** is the degree of polycondensation or polymerisation; z is1,2,3, or higher, up to 32.

The schematic formation of geopolymer material can be shown as described by Equations (2-2) and (2-3) [5]:

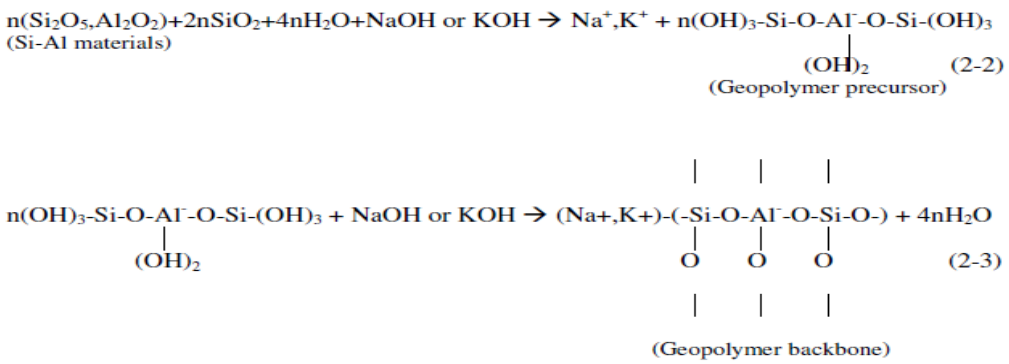

Figure 2.1 Schematic formation of geopolymer material

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The chemical reaction may comprise the following steps [3]:

• Dissolution of Si and Al atoms from the source material through the action of hydroxide ions.

• Transportation or orientation or condensation of precursor ions into monomers.

• Setting or polycondensation/polymerisation of monomers into polymeric structures.

However, these three steps can overlap with each other and occur almost simultaneously, thus making it difficult to isolate and examine each of them separately [6]. A geopolymer can take one of the three basic forms [4]:

• **Poly (sialate)**, which has [-Si-O-Al-O-] as the repeating unit.

• **Poly (sialate-siloxo)**, which has [-Si-O-Al-O-Si-O-] as the repeating unit.

• **Poly (sialate-disiloxo)**, which has [-Si-O-Al-O-Si-O-Si-O-] as the repeating unit.

Sialate is an abbreviation of silicon-oxo-aluminate. The last term in Equation 2-3 reveals that water is released during the chemical reaction that occurs in the formation of geopolymers. This water, expelled from the geopolymer matrix during the curing and further drying periods, leaves behind discontinuous nano-pores in the matrix, which provide benefits to the performance of geopolymers. The water in a geopolymer mixture, therefore, plays no role in the chemical reaction that takes place; it merely provides the workability to the mixture during handling. This is in contrast to the chemical reaction of water in a Portland cement mixture during the hydration process.

Davidovits (1999) proposed the **possible applications of the geopolymers depending on the molar ratio of Si to Al**, as given in Table 1.1. [4]

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TABLE 2.1 Applications of the geopolymers depending on the molar ratio of Si to Al

Si/Al	Application
1	Bricks, ceramics, fire protection
2	Low CO_2 cements, concrete, radioactive & toxic waste encapsulation
3	Heat resistance composites, foundry equipments, fibre glass composites
>3	Sealants for industry
20<Si/Al<35	Fire resistance and heat resistance fibre composites

2.3 Factors Affecting the Properties of Geopolymers

Several factors have been identified as important parameters affecting the properties of geopolymers. Palomo et al (1999) concluded that the **curing temperature** was a reaction accelerator in fly ash-based geopolymers, and significantly affected the mechanical strength, together with the curing time and **the type of alkaline liquid**.[6] Higher curing temperature and longer curing time were proved to result in higher compressive strength. Alkaline liquid that contained soluble silicates was proved to increase the rate of reaction compared to alkaline solutions that contained only hydroxide. Van Jaarsveld et al (2002) concluded that the water content, and the curing and calcining condition of kaolin clay affected the properties of geopolymers. However, they also stated that curing at too high temperature caused cracking and a negative effect on the properties of the material. Finally, they suggested the use of mild curing to improve the physical properties of the material.[5] In another study, van Jaarsveld et al (2003) stated that the source materials determine the properties of geopolymers, especially the CaO content, and the water-to-fly ash ratio.[7]

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Based on a statistical study of the effect of parameters on the polymerisation process of metakaolin-based geopolymers, Barbosa et al (1999; 2000) reported **the importance of the molar composition** of the oxides present in the mixture and the water content. They also confirmed that the cured Geopolymers showed an amorphous microstructure and exhibited low bulk densities between 1.3 and 1.9. [8]

Based on the study of geopolymerisation of sixteen natural Si-Al minerals, Xu and van Deventer (2000) reported that factors such as the **percentage of CaO, K2O, and the molar Si-to-Al rati**o in the source material, the type of alkali liquid, the extent of dissolution of Si, and the molar Si-to-Al ratio in solution significantly influenced the compressive strength of geopolymers.[3]

2.4 Advantages of Geopolymer Cements

Rock-based Geopolymer cements are manufactured in a different manner than Portland cement. Geopolymeric cements **do not require high temperature kilns**, **or large expenditures of fuel**, nor do they require such **a large capital investment** for the plant and equipment. Thermal processing at temperatures **not higher than 600-700°C** of naturally occurring alkali-silico-aluminates and alumino-silicates (geological resources available on all continents) provides suitable rock-based geopolymeric raw-materials.[9]

In addition, the energy consumption of manufacturing cement is lower than Portlant cements. The global introduction of these low-CO2 geopolymeric cements, for civil engineering, infrastructure and general construction purposes will **reduce the CO2 emissions created** by the cement concrete industry by 80%. This can mitigate overall Global Warming.

2.5 Application of Geopolymer in Present Industry

There exist a wide variety of potential and existing applications. Some of the geopolymer applications are still in development whereas others are already

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industrialized and commercialized. Here we discussed some of the current and present application of geopolymer in industry such as:

1. Low Temperature Geopolymeric Setting of ceramic, L.T.G.S
2. Fire-resistant wood-chipboards
3. Aviation applications

2.5.1 Low Temperature Geopolymeric Setting of Ceramic, L.T.G.S

Low Temperature Geopolymeric Setting (L.T.G.S.) takes place at drying temperatures (50°C to 250°C), in alkaline conditions, through an oligosialate precursor (-Si-O-Al-O-) (Na) in concentrations from 2 to 6% by weight of the ceramic paste. The kaolinite in clays is transformed by LTGS into a three dimensional compound of the poly(sialate) Na-PS sodalite type, stable to water and possessing high mechanical strength.

L.T.G.S. may dramatically enhance and modernize the traditional ceramic industry. Once geopolymerised into Na-polysialate (Na-PS) or K-polysialate (K-PS), at 125-250°C, ceramic bodies may be ultra rapidly fired at 1000°C-1200°C, to produce high quality ceramics.

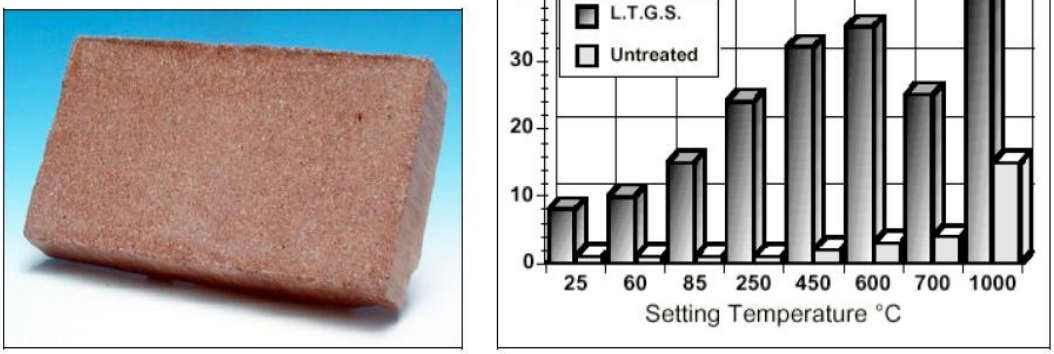

FIGURE 2.2: Brick made with L.T.G.S. on kaolinitic soils. Mechanical compressive strength in Mpa for untreated and geopolymerised kaolinitic earth (with 3% by weight equivalent Na2O). Setting temperature ranges between 20°C and 1000°C.[10]

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2.5.2 Fire-Resistant Wood-Chipboards

The first applications were building products (developed with J.J. Legrand), such as fire-resistant chip-board panels, comprised of a wooden core faced with two nanocomposite coatings, in which the entire panel was manufactured in a one-step process. An unusual feature was observed to characterize the manufacturing process: for the first time, the hardening of organic material (wood chips and organic resin) occurred simultaneously with the setting of the mineral silico-aluminate (Na-Poly(sialate)/quartz nanocomposite), when applying the same thermosetting parameters as for organic resin

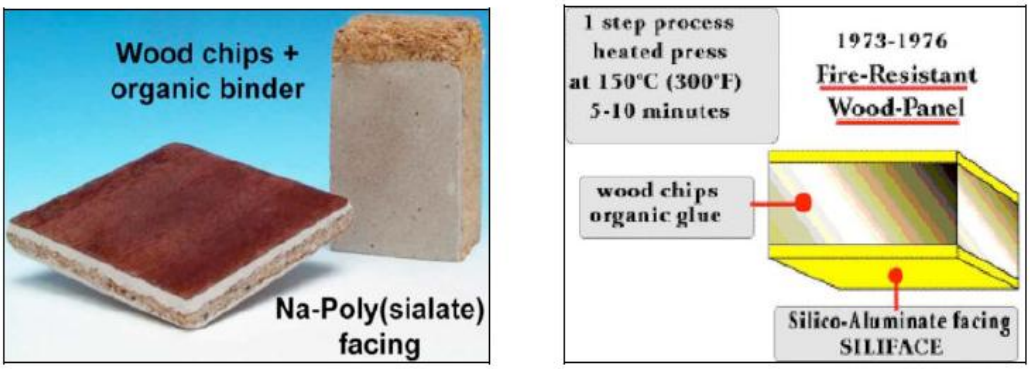

FIGURE 2.2: Manufacture of fire-resistant wood-chipboards faced with geopolymer (Na-Poly(sialate)[10]

2.5.3 Aviation Applications

Aircraft cabin materials targeted for geopolymer composite include cargo liners, ceiling, floor panels, partitions and sidewalls, stowage bins, wire insulation, yielding 2500-3000 kg. There is an increase demand for fire-resistant containers.

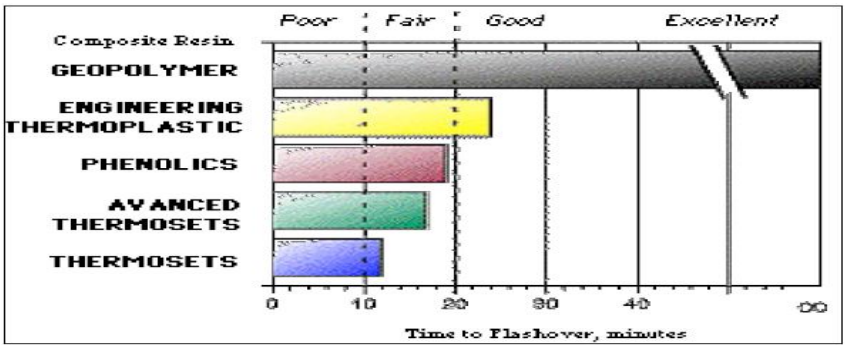

Figure 2.3:Time to flashover (minutes) for various organic resins compared to geopolymer resin.[10]

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2.6 Acid Sulfate Soils

This geopolymer container that use as a storage for these hazardous material will be placed underneath the soils. Based on the study, normally our soil contains certain amount of acid. Acid sulfate soil is the common name given to the soils and sediments and when exposed to air due to drainage or disturbance, these soils produce sulfuric acid, often releasing toxic quantities of iron, aluminium and heavy metals. [14]

Acid sulfate soils can cause acid attack and when brickwork is persistently wet as in the foundations, crystalline may occur and in time the brickwork expand and rendering to disintegrate. [15] Therefore it is so crucial to conduct a test on the geopolymer samples by immersing them into a basin fill with sulphuric acid.

2.7 Resistant of Geopolymer to Chemical

Vijaya Rangan et al,(2005) had studied the effect of various salient parameters on the low-calcium fly ash-based geopolymer concrete. The parameters considered are as follows:

- Sulfate Resistance
- Sulfuric Acid Resistance

Tests were performed to study the sulfate resistance of the low-calcium fly ash-based polymer and the normal commercialize concrete. The test specimens were immersed in 5% sodium sulfate up to one year. The result showed that there was no sign of surface erosion, cracking or spalling and there was also no significant changes in mass and length of the geopolymer concrete.[12]

In term of sulfuric acid resistance, the specimens are placed in three different concentration of sulfuric acid solution which are 2%, 1% and 0.5%. Similar to sulfate resistance test, the specimens are placed in the sulfuric acid solution for up to one year. The finding from this test is that the maximum loss of the test

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specimens of about 3% after one year. [13] The damage to the surface of the specimens increased as the concentration of the acid solution increased.

In other study conducted, the commercialize concrete is immersed in the same set of sulfuric acid solution. The result show staggering different compare to geopolymer concrete. For an instance, if both sample test with the 5% concentration of sulfuric acid solution, the acid had destroyed almost 65% of the commercialize concrete compare to geopolymer concrete which is only 10%.

Based from this finding, geopolymer is found to be the best solution in replacing the commercialize concrete as the storage for the chemical and radioactive storage because of its lasting and durable characteristic and also can withstand against the corrosive acid material. However, the current geopolymer can be upgrade by extending the research in which is going to implement in this research. In term of testing the sulfate resistance and sulfuric acid resistance, the final dissolved solution should be tested to determine the composition in the solution. Through this finding, hopefully they could determine what component of geopolymer that are reacted and dissolve in the solution. Apart from that, the auther can also improve the properties of this geopolymer cement by investigating which part or area that is affected or dissolved the most in the solution.

2.8 Water Absorption on Geopolymer

Water penetrability, namely water absorption is important measurement to control geopolymer durability. Regarding to this, pores in the geopolymer have an important role to allow the liquid/fluid move through the geopolymer. However the tendency of geopolymer to absorption and transmission of water by capillary action not only depends on the porosity but also on its pore diameter, distribution, continuity and tortuosity.

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According to Olivia, et al, (2008) the fly ash geopolymer contains higher proportion of pores in the mesopores size and this condition may lead water to penetrate easily and will affect the durability of the material.

To determine the water absorption of geopolymer specimens, after curing stage, its mass determined as initial weight. The samples were then immersed in water for 24 hours and its saturated weight was recorded as the final weight. Water absorption of a specimens is reported as the percentage increase in mass.

$$\text{Percentage of water absorption of geopolymer} = \frac{M_f - M_i}{M_i} \times 100\%$$

Where;

M_f = mass of the specimen after immersed in the water (gram)

M_i = mass of the specimen after curing stage (gram)

Many studies of water absorption on geopolymer had been done, and the author found out that there is lacking with their finding. Most of the test only had been done on the weight loss on the geopolymer. Supposedly, there is also should be a test to investigate the type of chemical that react and dissolve during the water absorption test. The method that can be used is by using simple titration on the sample of the final dissolved water. Apart from that, to determine the composition of the dissolved water, atomic absorption spectroscopy (AAS) also can be used. Through this finding, it can help to improve the composition of the geopolymer to make it more vulnerable to water.

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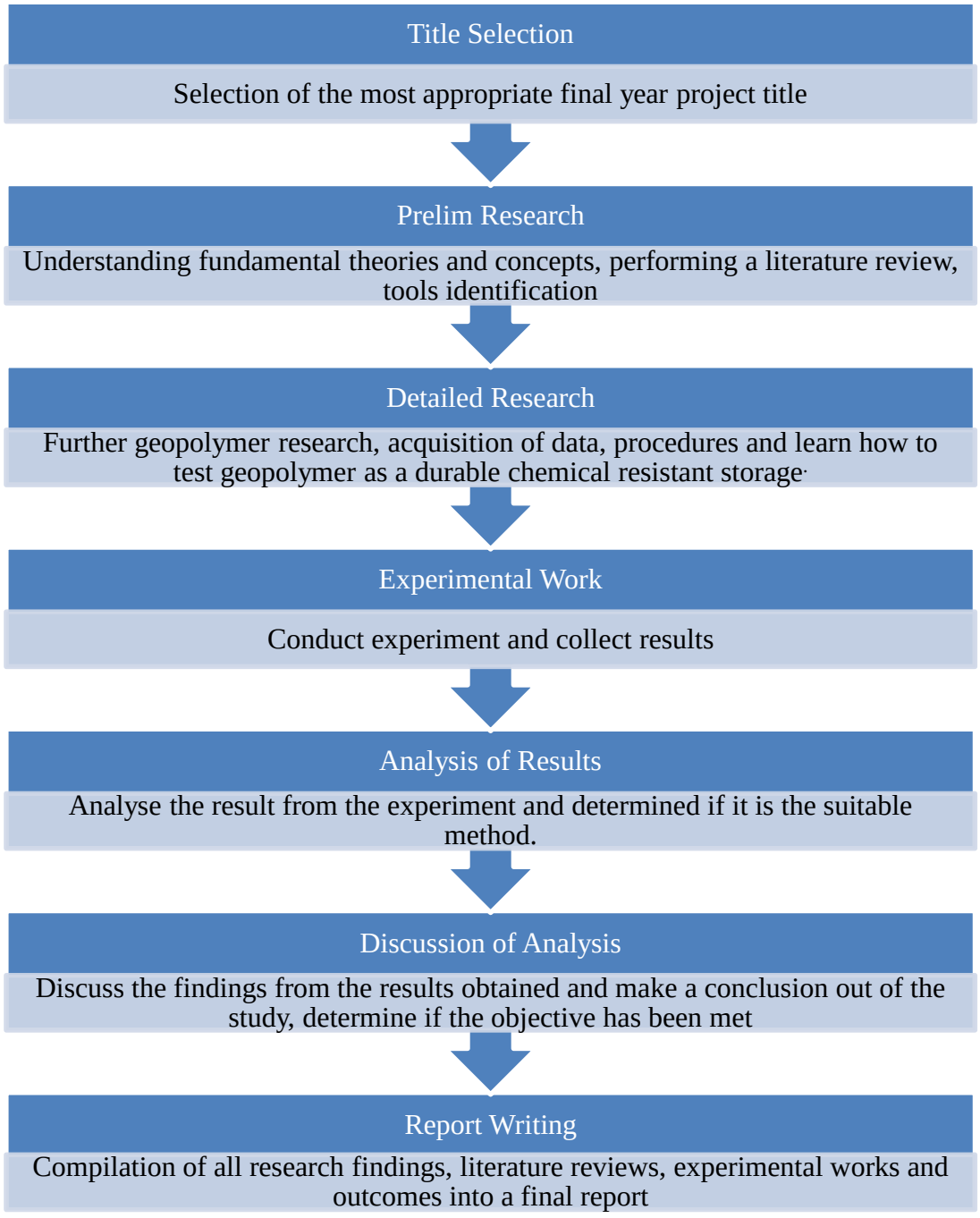
CHAPTER 3

METHODOLOGY

3.1. Flow Chart



3.2 Gantt Chart

Timelines for FYP 2

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Project Work Continues																
2	Submission of Progress Report																
3	Project Work Continues																
4	Pre-EDX																
5	Submission of Draft Report																
6	Submission of Dissertation (soft bound)																
7	Submission of Technical Paper																
8	Oral Presentation																
9	Submission of Project Dissertation (Hard Bound)																

Mid-Semester Break

Suggested milestone

Process

3.3 Raw Materials and Chemicals Needed

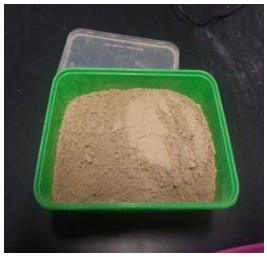
In the experiments that are going to be conducted, several raw materials and chemicals are needed. There are:

- Sulfuric Acid, H_2SO_4 (concentration 98%)
- Sodium Hydroxide, NaOH (pellet)
- Sodium sulfate (concentration 5%)
- Fly-ash

3.4 Research Procedure

3.4.1 Preparation of Geopolymer Sample

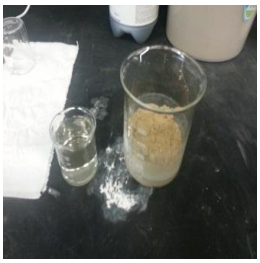
1. Pour fly-ash into a container.



2. Prepare sodium hydroxide (NaOH) solution with concentration of 12M.

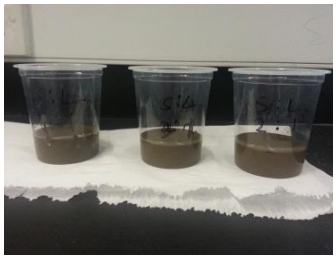
3. Weight 450g of fly-ash and 150g of sodium hydroxide solution. (Solid: Liquid=3:1)

4. Pour 150g of sodium hydroxide solution into 450g of fly-ash.



5. Stir the mixture until it is well mix.

6. Leave the sample for curing process at room temperature.



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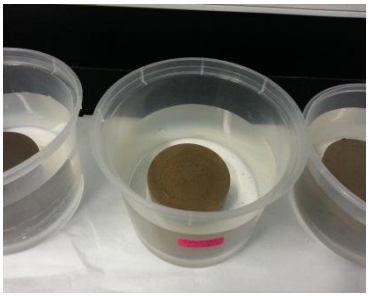
- Repeat the step 1 until step 5 by manipulating the concentration of sodium hydroxide (NaoH) solution with 10M and 8M.
- Finally repeat step 1 until 6 by curing the sample inside the oven with setting temperature of 60^{O}C (oven).
- Therefore, there will be all together 6 samples of geopolymer all together.

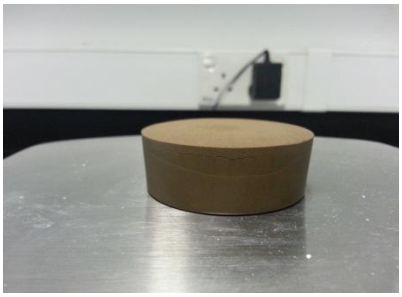
3.3.2 Test on Water Absorption

Below are procedures for water absorption testing on the geopolymer sample.

1. Slowly remove out the sample from the container → 2. Weight and record the mass of each sample

3. Immersed every sample into a separated container filled with water and label each container. → 4. Weight and record the mass of the immersed sample with the interval of 2 days until day 8. → 5. Analyses and discuss about the finding and result.





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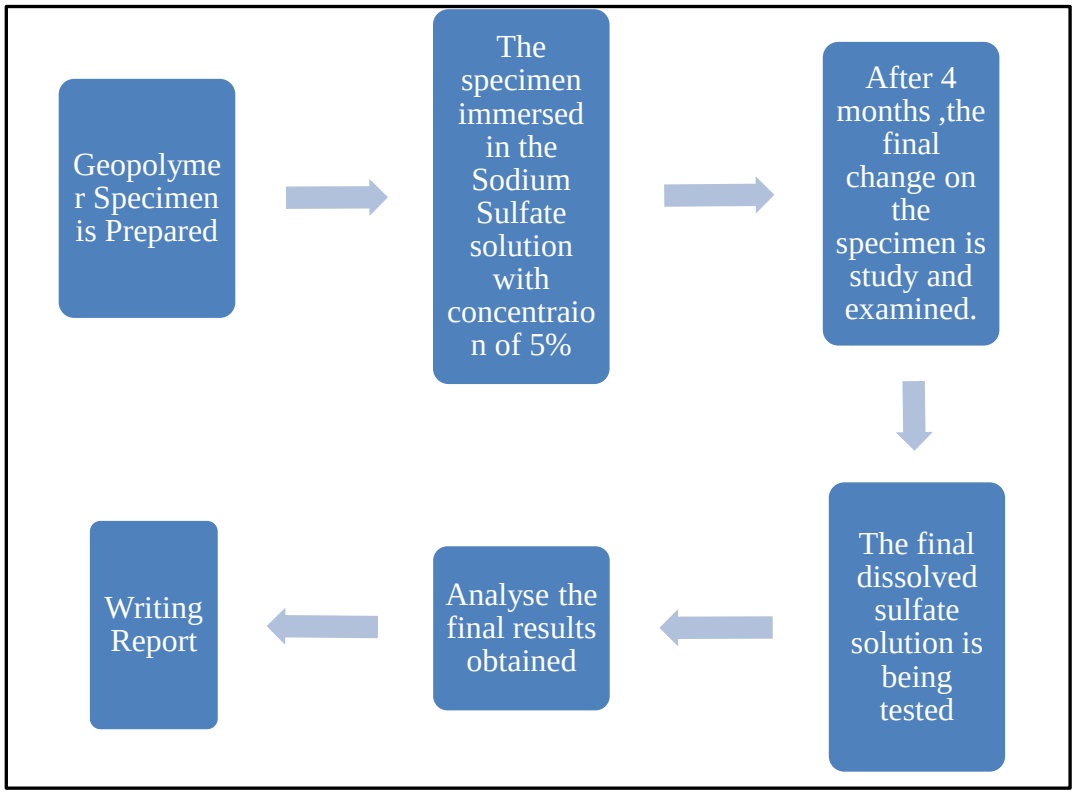
3.3.3 Test on Sodium Sulfate Resistance



Procedure:

1. Prepare the geopolymer by using raw material which are fly ash, sodium hydroxide (alkaline liquid) and water with the right proportion.
2. After preparing the specimen, fill a basin with sodium sulfate solution with concentration of 5%.
3. Record the initial mass of the geopolymer specimen.
4. Immersed the specimen in the basin and leave it for 4 months.
5. After month, the final specimen is analysed and examined. Record the final mass of the specimen and compare the value with the initial mass.
6. Test and analyse the final sodium sulfate solution with the centrifuge.

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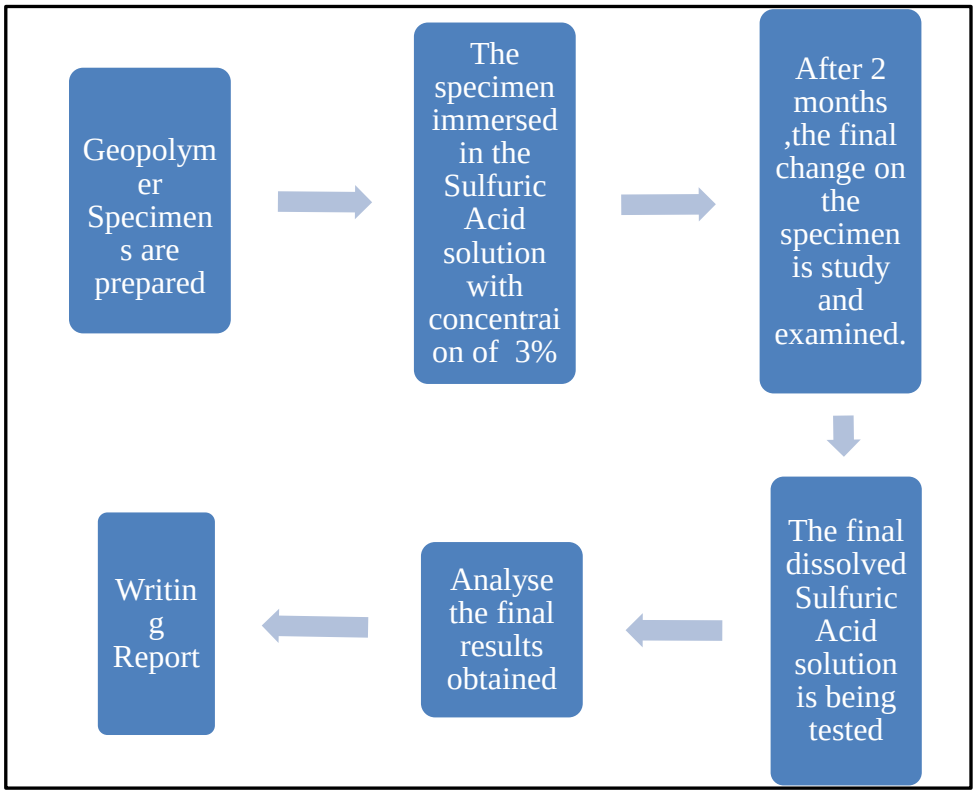
3.3.4 Test on Sulfuric Acid Resistance



Procedure:

1. Prepare geopolymer specimens by using raw material which are fly ash, sodium hydroxide (alkaline liquid) and water with the right proportion.
2. After preparing the specimens, prepare 3 basins and fill them with of sulfuric acid solution respectively.
3. Record the initial mass of all three geopolymer specimens.
4. Immersed the specimen in the basins and leave it for 4 months.
5. After month, the final specimens are analysed and examined. Record the final mass of the specimens and compare the value with the initial mass.
6. Test and analyse the final sulfuric acid solution with the centrifuge.
7. Repeat the step 1 till step 6 by changing the specimen with the the batch that curing at 60^{O}C (oven).

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CHAPTER 4
RESULT AND DISCUSSION

4.1 Water Absorption Test

Below is the results of water absorption after immerse in a basin fill with water for a period of 7 days.

In this part of experiment, there are a few factors that we keep them as constant variable which are:

1. Curing Time (8 days)
2. Volume of immersed water (300ml)
3. Concentration of Sodium Hydroxide (12M)

TABLE 4.1: Result of Water Absorption

No	Curing Temp. (^{O}C)	S:L Ratio	Initial Mass (gram)	Total Mass after 2 days (gram)	Total Mass after 4 days (gram)	Total Mass after 6 days (gram)	Total Mass after 8 days (gram)
A	26	4:1	102.10	101.17	100.87	100.61	100.45
B	26	3:1	94.05	91.80	91.30	90.87	90.73
C	60	4:1	99.30	100.13	100.50	100.63	100.88
D	60	3:1	92.14	95.32	95.70	95.75	95.69
E	60	2:1	78.20	82.77	82.67	82.33	82.15

Based from the finding of this result, a graph of mass of geopolymer versus number of days can be plotted for each of the samples. Through this graph we

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could see clearly the changes in mass of all the geopolymers endured throughout the 8 days of experiment.

According to the graph below, samples with curing temperature of 26^{O}C which are sample A and sample B, show a same pattern. Both of these samples reduce in mass after immersed in the water after 2 days. These two samples mass continue to decrease gradually until 8 days of experiment. This is mainly due to the curing temperature which is too low. The structure of both geopolymer samples are not harden enough and still have the tendency to soften and dissolve upon immersed in the basin fill with water.

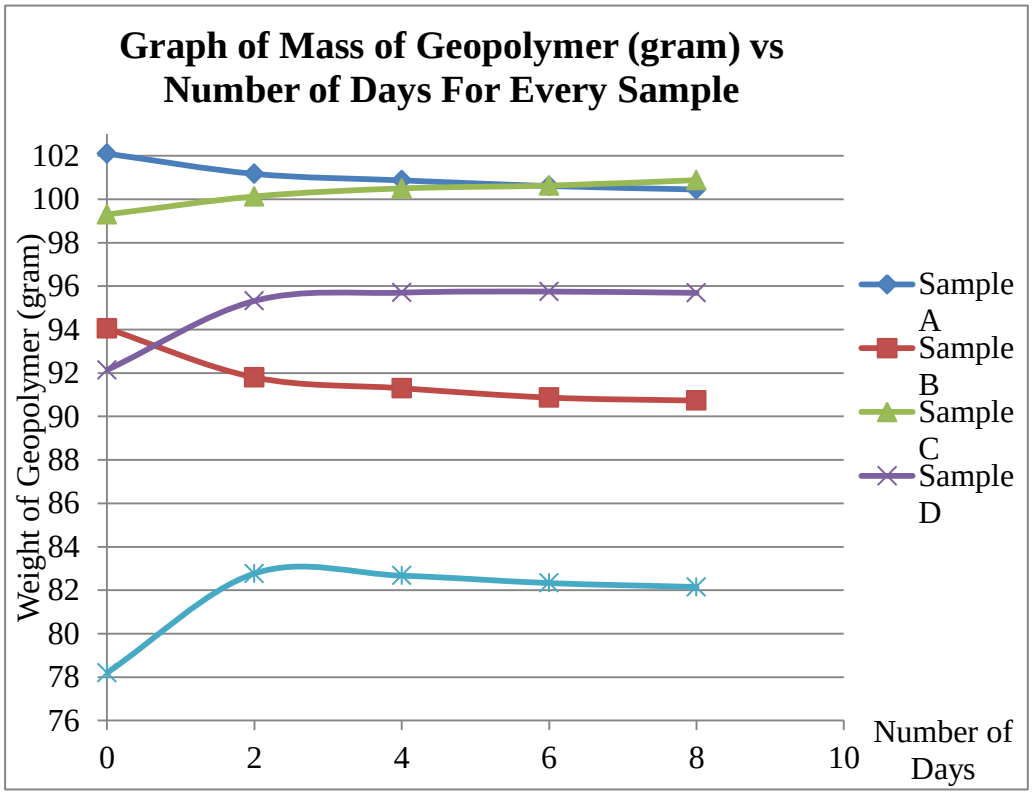

FIGURE 4.1: Graph of mass of geopolymer versus number of days for every sample

Meanwhile, sample with curing temperature of 60^{O}C which are sample A, sample B and sample C also show a same pattern among them. All of these samples increase in mass after immersed in the water after 2 days. The mass of all the three samples continues to increase gradually until 8 days of experiment except sample E in which the mass of the geopolymer sample is decrease. Sample D also start to decrease in mass on Day 8 of the experiment. However the obvious outcomes show that the sample with high curing temperature

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increase in mass and sample with low curing temperature show the vice versa pattern.

TABLE 4.2: Percentage of water absorption comparison between sample with 60^{O}C curing temperature

Sample	Initial Weight (gram)	Total weight after 2 days	Weight different (gram)	Water absorption (%)
C	99.30	100.13	0.83	0.84
D	92.14	95.32	3.18	3.45
E	78.20	82.77	4.57	5.84

The comparison between samples of high curing temperature can obviously determine by calculating the percentage of water absorption. Sample E show the highest percentage of water absorption followed by sample D and sample C. Sample E display the highest percentage of water absorption because it has higher water content and this will lead to higher porosity. Consequently higher porosity, would lead to more penetration of water through pores.

4.2 Sulphuric Acid Test

4.2.1 Mass Changes

Tests were performed to study the sulphuric acid resistance of the low-calcium fly ash-based geopolymer concrete. The concentration of sulfuric acid is 3%. The sulphuric acid resistance of geopolymer concrete was evaluate based on the mass loss and the residual compressive strength of the test specimens after immersed in the basin filled with acid up to 56 days with is approximately 2 months. The test specimens, 100 x 100 mm cubics, were made using mixture based on the table below. The curing period of all these geopolymers specimen is up to 7 days and the solid to liquid ratio is keep constant throughout this testing because solid to liquid ratio 3:1 is chosen because they has the best compressive strength. [12]

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TABLE 4.3: Geopolymer Specimen Mixture for Sulphuric Acid Test

Specimen	Solid:Liquid	Curing Temperature (℃)	NaOH (M)
1	3:1	26	8
2			10
3			12
4		60	8
5			10
6			12

The mass changes of all the specimens are recorded with the interval of 7 days until 56 days 21 months). Below is the table shows the mass changes of all the geopolymer samples:

TABLE 4.4 : Mass Changes of Geopolymers Samples Curing at 26°C

Curing Temper-ature	26°C								
Concen-tration of NaOH	8M			10M			12M		
Day	Mass Sample 1 (g)	Mass Sample 2 (g)	Avg. Mass (g)	Mass Sample 1 (g)	Mass Sample 2 (g)	Avg. Mass (g)	Mass Sample 1 (g)	Mass Sample 2 (g)	Avg. Mass (g)
Day 1	249.06	248.75	248.91	260.29	257.56	258.93	264.04	263.42	263.73
Day 7	246.57	243.55	245.06	258.35	251.20	254.78	256.39	262.40	259.40
Day 14	245.95	241.44	243.70	257.79	248.26	253.03	252.30	262.98	257.64
Day 21	247.85	241.35	244.60	256.90	247.25	252.08	247.66	263.08	255.37
Day 28	235.77	230.79	232.77	250.93	236.43	243.68	240.62	256.35	248.49

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Day 35	234.33	229.45	231.89	250.02	236.74	243.38	251.62	255.91	253.77
Day 42	233.85	228.87	231.36	249.43	236.13	242.78	251.17	255.23	253.20
Day 49	233.11	228.21	230.66	248.75	235.71	242.23	250.76	254.74	252.75
Day 56	232.56	227.73	230.15	248.21	235.08	241.65	249.95	253.97	251.96

TABLE 4.5 : Mass Changes of Geopolymers Samples Curing at 60°C

Curing Temp e-rature	60°C								
Conc en-tratio n of NaO H	8M			10M			12M		
Day	Mass Sam ple 1 (g)	Mass Sam ple 2 (g)	Avg. Mas s (g)	Mass Sam ple 1 (g)	Mass Sam ple 2 (g)	Avg.M ass (g)	Mass Sam ple 1 (g)	Mass Sam ple 2 (g)	Avg.M ass (g)
Day 1	232. 90	231. 91	232. 41	235. 98	237. 51	236.75	242. 67	238. 5	240.59
Day 7	240. 84	249. 41	245. 13	247. 39	251. 04	249.22	254. 31	249. 6	251.96
Day 14	243. 98	251. 47	247. 73	250. 97	254. 67	252.82	259. 55	254. 97	257.26
Day 21	244. 28	251. 76	248. 02	251. 42	255. 33	253.38	259. 72	255. 32	257.52
Day 28	245. 55	253. 83	249. 69	255. 01	260. 77	257.89	261. 21	256. 85	259.03
Day 35	245. 55	252. 09	248. 82	249. 95	253. 71	251.83	259. 55	254. 97	257.26
Day 42	246.	252.	249.	251.	255.	253.49	259.	255.	257.52

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	14	78	46	35	63		72	32	
Day 49	246.56	253.21	249.89	252.01	256.77	254.39	261.21	256.85	259.03
Day 56	247.11	253.87	250.49	252.93	257.42	255.18	262.25	257.54	259.90

Meanwhile below are 2 separate graphs showing the comparison of mass changes of all geopolymer sample at curing temperature of 26°C and 60°C respectively.

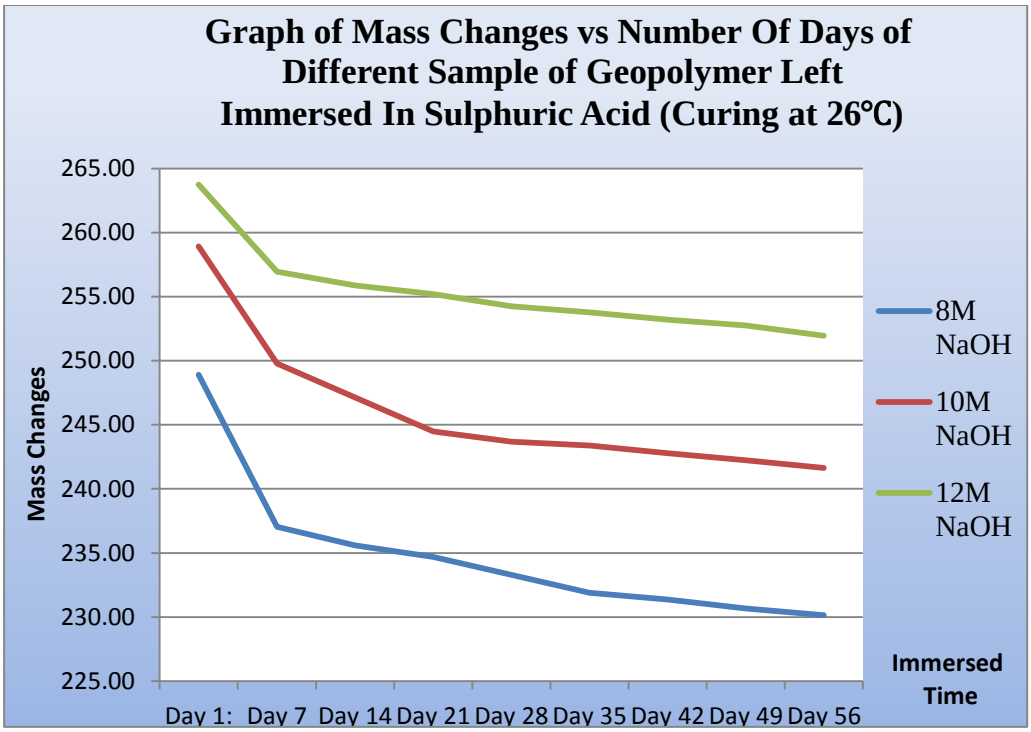

FIGURE 4.2 :Graph of Mass Changes vs Number Of Days of Different Sample of Geopolymer Left Immersed In Sulphuric Acid (Curing at 26℃)

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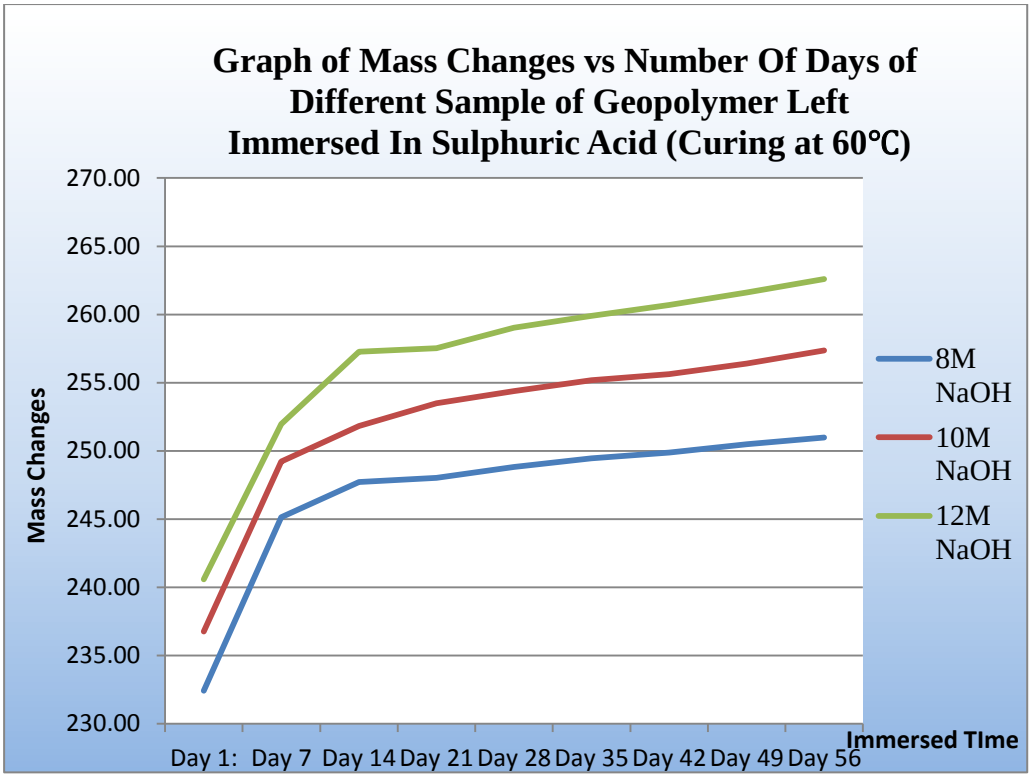

FIGURE 4.3 :Graph of Mass Changes vs Number Of Days of Different Sample of Geopolymer Left Immersed In Sulphuric Acid (Curing at 60℃)

The value for the both graphs above are taken from the average value of respective group of sample. The percentage different of each group of geopolymer specimen can be calculated based on the formula below:

$$Percentage\ Different\ (\%) = \left|\frac{Inital\ mass - Final\ mass}{Final\ mass}\right| \times 100\%$$

Geopolymer Curing at 26℃:

$$Percentage\ Different\ \mathbf{8M}\ (\%) = \left|\frac{248.91 - 230.15}{248.91}\right| \times 100\%$$

$$= \mathbf{7.54}\ \%$$

$$Percentage\ Different\ \mathbf{10M}\ (\%) = \left|\frac{258.93 - 241.65}{258.93}\right| \times 100\%$$

$$= \mathbf{6.67}\%$$

$$Percentage\ Different\ \mathbf{12M}\ (\%) = \left|\frac{263.73 - 251.96}{263.73}\right| \times 100\%$$

$$= \mathbf{4.46}\%$$

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Geopolymer Curing at 60℃:

$$Percentage\ Different\ \mathbf{8M}\ (\%) = \left|\frac{232.41\ -250.98}{232.41}\right| \times 100\%$$

$$= \mathbf{7.44}\ \%$$

$$Percentage\ Different\ \mathbf{10M}\ (\%) = \left|\frac{236.75 - 257.37}{236.75}\right| \times 100\%$$

$$= \mathbf{8.71}\%$$

$$Percentage\ Different\ \mathbf{12M}\ (\%) = \left|\frac{240.59\ -262.59}{240.59}\right| \times 100\%$$

$$= \mathbf{9.14}\ \%$$

Based on the graphs and calculation of the percentages different, it shows two different pattern between two geopolymer samples that curing at 26℃ and 60℃. The geopolymer specimens that curing at 26℃ show a mass reduction after immersing in the sulphuric acid. The mixtures with lower concentration of NaOH tend to loss more mass compare to mixture with higher concentration of NaOH. This is mainly due to the high concentration of NaOH that make the structure more hard and dense therefore the geopolymer can withstand acid attack. This outcome also shows that, the sample with higher concentration of NaOH and higher curing temperature can have better completion of the reaction during the mixing.

This pattern totally opposite displays by the samples that curing at 60℃ in which their mass is increasing after expose to acid. The samples with higher concentration of NaOH tend to increase more mass compare to samples with lower concentration. This is because the when the sample is being cure at 60℃ and with the aid of high concentration of NaOH, it will cause the sample to have more porosity for the acid to soak into the samples.

The visual appearance of specimens after being immersed in sulphuric acid solution after 56 days showed that acid attack slightly damaged the surface of the specimens. Figures below compare the visual appearance of the geopolymer samples after immersed with acid and the sample without

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exposing to the acid. It can be seen that the specimens being immersed undergoes erosion of the surface. The damage to the surface of the sample increase as the concentration of NaOH for geopolymer mixing is decrease.

A. Curing at 26℃

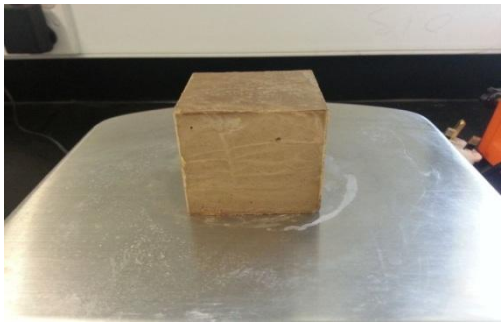

FIGURE 4.4 Geopolymer Sample Without Immersed in Sulphuric Acid

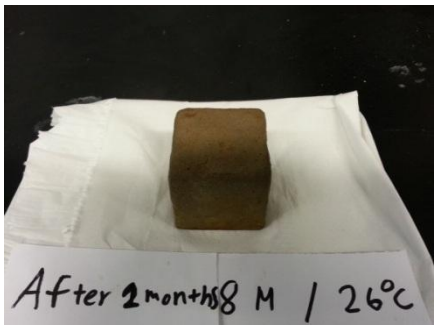

FIGURE 4.5 Geopolymer Sample (8M) after Immersed in Sulphuric Acid

FIGURE 4.6 Geopolymer Sample (12M) after Immersed in Sulphuric Acid

FIGURE 4.7 Geopolymer Sample (10M) after Immersed in Sulphuric Acid

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B. Curing at 60℃

FIGURE 4.8 Geopolymer Sample Without Immersed in Sulphuric Acid

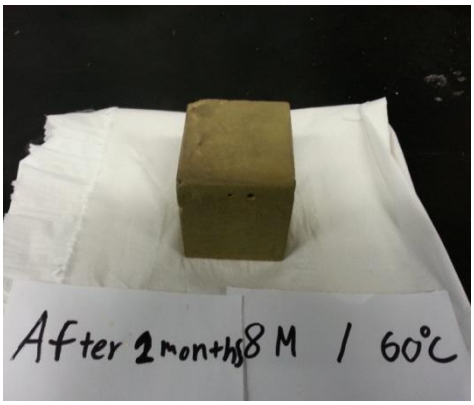

FIGURE 4.9 Geopolymer Sample (8M) after Immersed in Sulphuric Acid

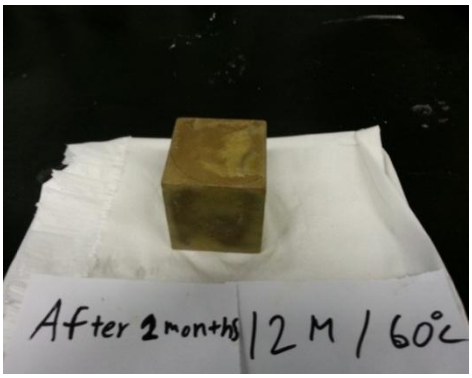

FIGURE 4.11 Geopolymer Sample (12M) after Immersed in Sulphuric Acid

FIGURE 4.10 Geopolymer Sample (10M) after Immersed in Sulphuric Acid

4.2.2 Compressive Strength Test

After immersing the geopolymer samples in a basin filled with sulphuric acid of 3% concentration for 56 days, all of these specimens need to test their compressive test by using 3000KN Compression Machine.

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FIGURE 4.12: 3000KN Compression Machine

The visual appearance of the specimens after being compress showed that the geopolymer cubic block at completely crack and broken into pieces. Below are pictures show the final result after being compress:

A. Curing at 26℃:

FIGURE 4.13: 8M NaOH Curing at 26℃

FIGURE 4.14: 10M NaOH Curing at 26℃

FIGURE 4.15: 12M NaOH Curing at 26℃

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B. Curing at 60℃:

FIGURE 4.16: 8M NaOH Curing at 60℃

FIGURE 4.17: 10M Na Curing at 60℃

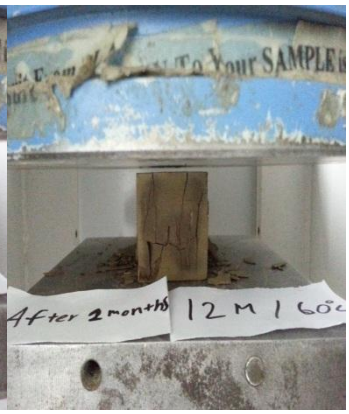

FIGURE 4.18: 12M NaOH Curing at 60℃

After recording all the compressive reading for all the geopolymer specimens, comparison was made by plotting a line graph. Below is the graph comparing the Sample Stress of the geopolymer samples:

TABLE 4.6: Result for the compressive test

Curing Temperature (°C)	NaOH Concentration (M)	No Sample	Sample Peak Load (KN)	Sample Stress (MPa)
26	8	1	28.90	11.57
		2	29.90	11.94
		Average:	29.40	11.76
	10	1	34.50	13.80
		2	32.70	13.07
		Average:	33.60	13.44
	12	1	42.30	16.93
		2	41.70	16.70
		Average:	42.00	16.82
60	8	1	67.60	27.05
		2	66.80	26.73
		Average:	67.20	26.89
	10	1	71.50	28.60
		2	76.40	30.56
		Average:	73.95	29.58
	12	1	82.10	32.84
		2	83.40	33.36
		Average:	82.75	33.10

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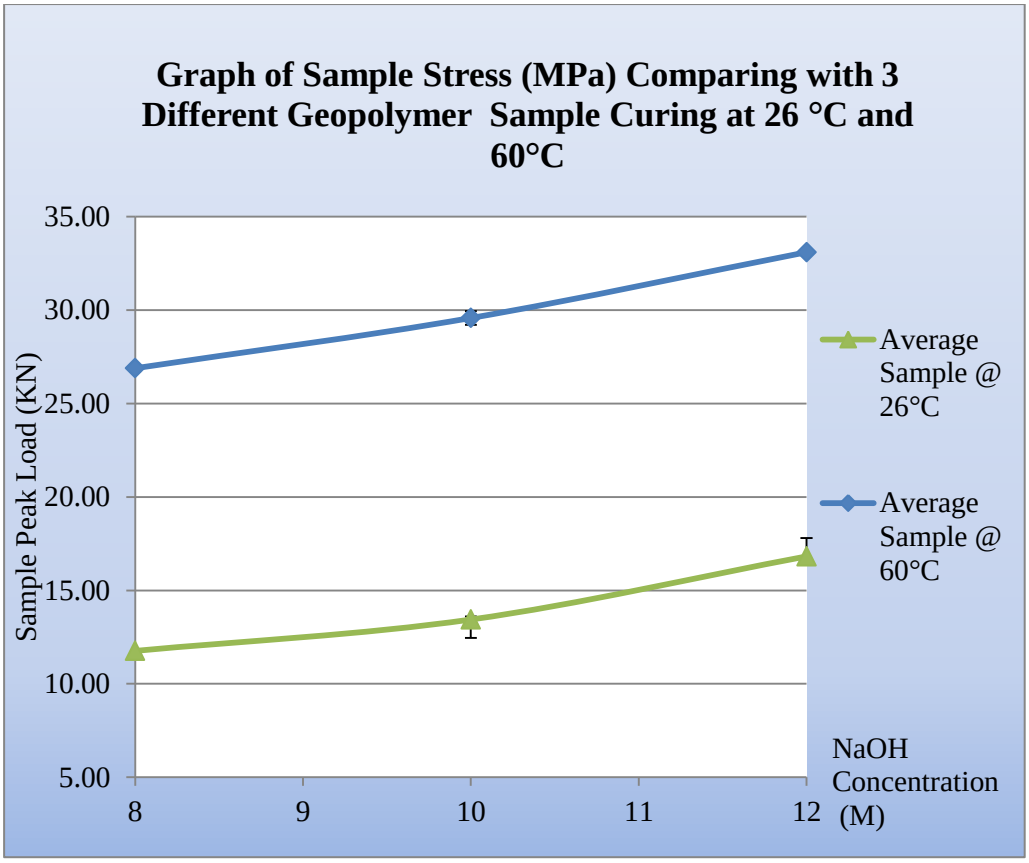

FIGURE 4.19: Graph of Sample Stress (MPa) Comparing with 3 Different Geopolymer Sample Curing at 26 °C and 60°C

Based on Figure 4.11, the graph show that the geopolymer samples that cure at 60°C is much stronger and can withstand more stress compare to the samples that cure at 26°C. The sample can increase its hardness by mix the fly ashes with higher concentration of NaOH. Curing at higher temperature with high concentration of NaOH can help to form a strong structure and make the sample more dense compare to the samples that are cure at 26°C and lower concentration of NaOH.

4.2.3 Characterisation Of Geopolymer Fly-Ashes.

After doing the compressive test, the leftover samples were being sent to do the Variable Pressure Field Emission Scanning Electron Microscope (VPRESEM) test. The main purpose of this test is to see closer the structure and the properties of the samples.

Figures below show the result from the test, based from the figures, we can make comparison between the samples that being cure at 60°C and 26°C.

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A. Curing at 26°C

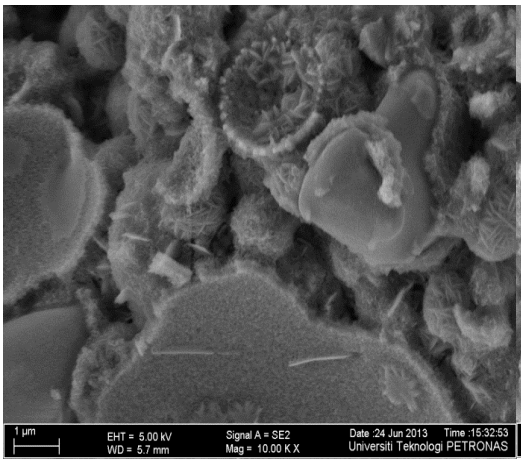

FIGURE 4.20: VPFESEM magnified Geopolymer sample (8M NaOH)

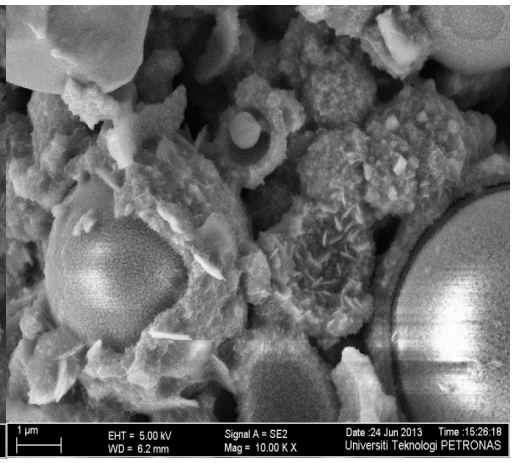

FIGURE 4.21: VPFESEM magnified Geopolymer sample (12M NaOH)

B. Curing at 60°C

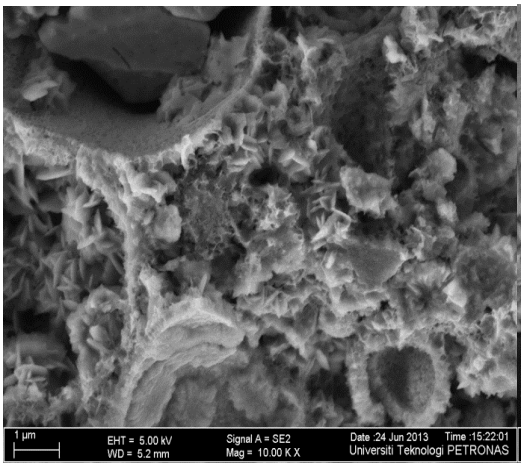

FIGURE 4.22: VPFESEM magnified Geopolymer sample (8M NaOH)

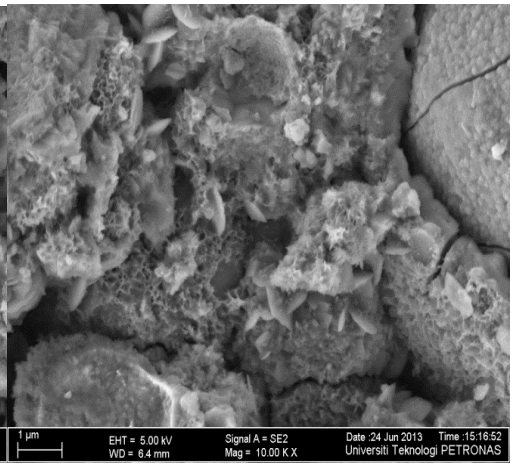

FIGURE 4.23: VPFESEM magnified Geopolymer sample (12M NaOH)

Based on the figures above, it show that the structure for samples that being cure at 60°C is more dense compare to sample cure at 26°C. Subsequently it cause the samples that cure at 26°C to have a lower compressive strength compare to samples cure at 60°C.Crystalline also begin to form at the samples that cure at 60°C. These figures also show that all the samples are not well mix as there is round shape in size of fly-ashes can be seen. This problem can take in account for future study in tackling this problem by improving the mixing.

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4.3 Sodium Sulphate Test

4.3.1 Mass Changes

Tests were conducted to study the sodium sulphate resistance of the low-calcium fly ash-based geopolymer concrete. After curing, the samples are immersed in the containers filled with sodium sulphate with the concentration of 5%. The sodium sulphate resistance of geopolymer concrete was evaluate based on the mass loss and the residual compressive strength of the test specimens after immersed in the basin filled with acid up to 56 days with is approximately 2 months. The test specimens, 100 x 100 mm cubics, were made using mixture based on the table below. The curing period of all these geopolymers specimen is up to 7 days and the solid to liquid ratio is keep constant throughout this testing because solid to liquid ratio 3:1 is chosen because they has the best compressive strength.

TABLE 4.7: Geopolymer Specimen Mixture for Sodium Sulphate Test

Specimen	Solid:Liquid	Curing Temperature (℃)	NaOH (M)
1	3:1	26	8
2			10
3			12
4		60	8
5			10
6			12

The mass changes of all the specimens are recorded with the interval of 7 days until 56 days 21 months). Below is the table shows the mass changes of all the geopolymer samples:

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Table 4.8 : Mass Changes of Geopolymers Samples Curing at 26°C

Curing Temper-ature	26°C								
Concen-tration of NaOH	8M			10M			12M		
Day	Mass Sample 1 (g)	Mass Sample 2 (g)	Avg. Mass (g)	Mass Sample 1 (g)	Mass Sample 2 (g)	Avg. Mass (g)	Mass Sample 1 (g)	Mass Sample 2 (g)	Avg. Mass (g)
Day 1	259.26	258.77	259.02	263.39	260.56	261.98	266.24	265.43	265.84
Day 7	249.35	244.87	247.11	258.24	247.34	252.79	256.75	261.75	259.25
Day 14	248.21	242.97	245.59	255.35	245.12	250.24	255.46	260.11	257.79
Day 21	247.84	241.56	244.70	254.64	240.33	247.49	254.72	259.65	257.19
Day 28	245.74	240.83	243.29	253.83	239.53	246.68	254.19	258.37	256.28
Day 35	244.36	239.45	241.91	253.02	239.54	246.28	253.52	257.94	255.73
Day 42	243.75	238.87	241.31	252.33	239.33	245.83	253.14	257.33	255.24
Day 49	243.13	238.01	240.57	251.65	238.61	245.13	252.73	256.76	254.75
Day 56	242.58	237.71	240.15	251.24	238.08	244.66	251.85	255.98	253.92

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TABLE 4.9 : Mass Changes of Geopolymers Samples Curing at 60°C

Curing Tempe-rature	60°C								
Concen-tration of NaOH	8M			10M			12M		
Day	Mass Sample 1 (g)	Mass Sample 2 (g)	Avg. Mass (g)	Mass Sample 1 (g)	Mass Sample 2 (g)	Avg. Mass (g)	Mass Sample 1 (g)	Mass Sample 2 (g)	Avg. Mass (g)
Day 1	266.24	265.43	265.84	245.98	257.51	251.75	252.65	241.50	247.08
Day 7	250.84	259.44	255.14	257.35	261.05	259.20	264.33	259.65	261.99
Day 14	253.93	261.48	257.71	259.96	263.74	261.85	269.55	264.93	267.24
Day 21	254.29	261.79	258.04	261.45	265.64	263.55	269.72	265.35	267.54
Day 28	255.57	262.13	258.85	262.03	266.75	264.39	271.21	266.83	269.02
Day 35	256.15	262.79	259.47	262.94	267.45	265.20	272.26	267.64	269.95
Day 42	256.55	263.25	259.90	263.35	267.95	265.65	272.96	268.48	270.72
Day 49	257.15	263.88	260.52	264.45	268.56	266.51	273.79	269.42	271.61
Day 56	257.77	264.42	261.10	265.07	269.69	267.38	274.86	270.34	272.60

Meanwhile below are 2 separate graphs showing the comparison of mass changes of all geopolymer sample at curing temperature of 26°C and 60°C respectively.

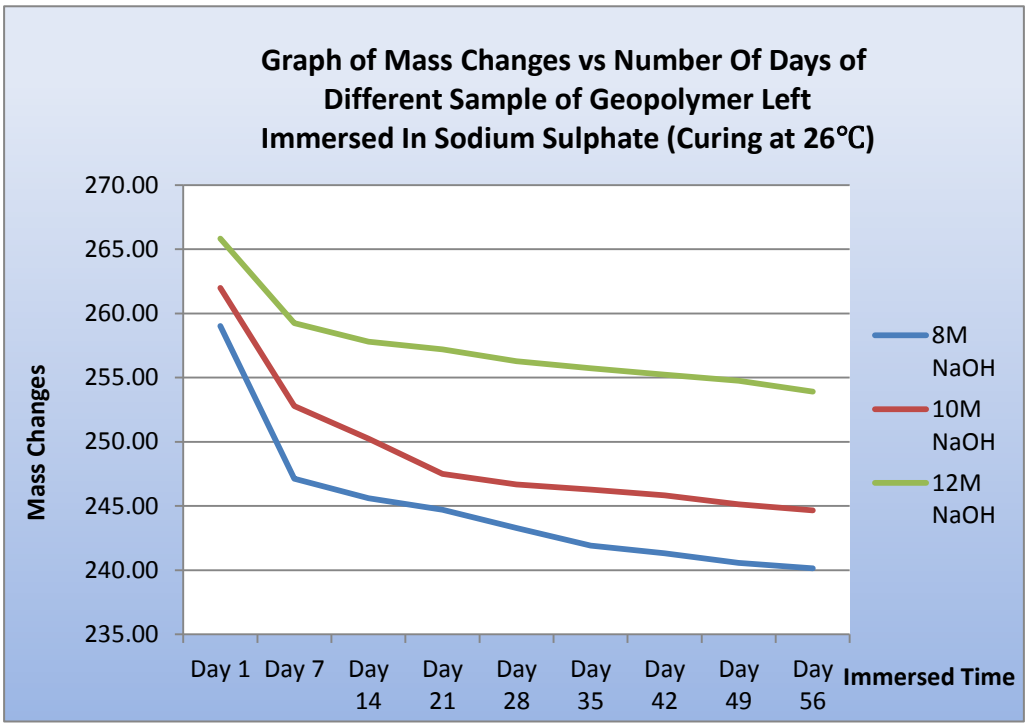

FIGURE 4.24 :Graph of Mass Changes vs Number Of Days of Different Sample of Geopolymer Left Immersed In Sodium Sulphate (Curing at 26℃)

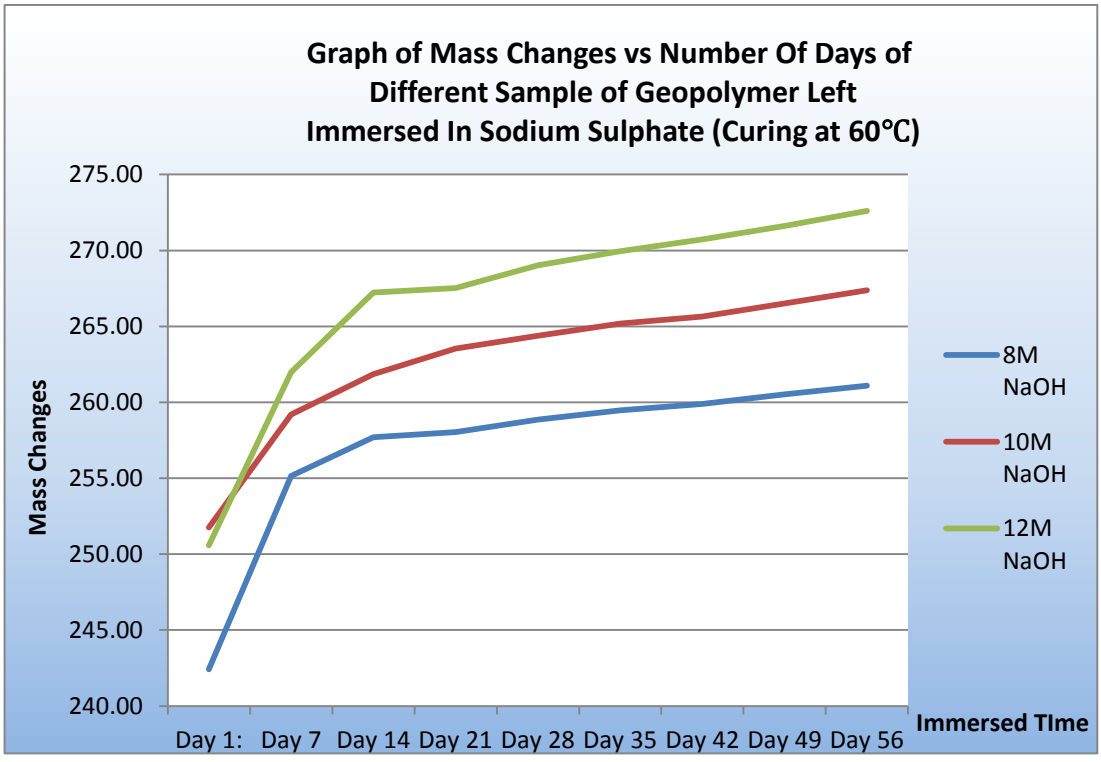

FIGURE 4.25 :Graph of Mass Changes vs Number Of Days of Different Sample of Geopolymer Left Immersed In Sodium Sulphate (Curing at 60℃)

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The value for the both graphs above are taken from the average value of respective group of sample. The percentage different of each group of geopolymer specimen can be calculated based on the formula below:

$$Percentage\ Different\ (\%) = \left|\frac{Inital\ mass - Final\ mass}{Final\ mass}\right| \times 100\%$$

Geopolymer Curing at 26℃:

$$Percentage\ Different\ \mathbf{8M}\ (\%) = \left|\frac{259.02\ -240.15}{259.02}\right| \times 100\%$$
$$= \mathbf{7.29}\ \%$$

$$Percentage\ Different\ \mathbf{10M}\ (\%) = \left|\frac{261.98\ -244.66}{261.98}\right| \times 100\%$$
$$= \mathbf{6.23}\%$$

$$Percentage\ Different\ \mathbf{12M}\ (\%) = \left|\frac{265.84\ -253.92}{265.84}\right| \times 100\%$$
$$= \mathbf{4.48}\%$$

Geopolymer Curing at 60℃:

$$Percentage\ Different\ \mathbf{8M}\ (\%) = \left|\frac{249.43\ -261.10}{249.43}\right| \times 100\%$$
$$= \mathbf{4.68}\ \%$$
$$Percentage\ Different\ \mathbf{10M}\ (\%) = \left|\frac{251.75 - 267.38}{251.75}\right| \times 100\%$$
$$= \mathbf{6.21}\%$$
$$Percentage\ Different\ \mathbf{12M}\ (\%) = \left|\frac{247.08\ -272.60}{247.08}\right| \times 100\%$$
$$= \mathbf{10.32}\ \%$$

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Based on the graphs and calculation of the percentages different, it shows two different pattern between two geopolymer samples that curing at 26℃ and 60℃. The geopolymer specimens that curing at 60℃ show a mass increment after immersing in the sulphuric acid which is totally opposite with the sample that curing at 26℃. Samples that curing at 60℃ tends to absorb and soak more solution and meanwhile the sample that curing at 26℃ are not resistance enough toward acid attack. The reason behind this outcome is that the sample that curing at higher temperature will have better completion in term of reaction.

Based on the result, it also shows that the mixtures with lower concentration of NaOH tend to loss more mass compare to mixture with higher concentration of NaOH. This is mainly due to the high concentration of NaOH that make the structure more hard and dense therefore the geopolymer can withstand acid attack.

4.2.2 Compressive Strength Test

After immersing the geopolymer samples in a basin filled with sodium sulphate of 5% concentration for 56 days, all of these specimens need to test their compressive test by using 3000KN Compression Machine.

After recording all the compressive reading for all the geopolymer specimens, comparison was made by plotting a line graph. Below is the graph comparing the Sample Stress of the geopolymer samples:

TABLE 4.9: Result for the compressive test

Curing Temperature (°C)	NaOH Concentration (M)	No Sample	Sample Peak Load (KN)	Sample Stress (MPa)
26	8	1	27.90	10.57
		2	28.80	10.84
		Average:	28.35	10.71
	10	1	33.50	13.11

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		2	31.70	12.07
		Average:	32.60	12.59
	12	1	41.30	16.13
		2	41.10	15.70
		Average:	41.20	15.92
60	8	1	66.60	26.06
		2	65.80	25.63
		Average:	66.20	25.85
	10	1	71.10	28.01
		2	76.00	29.56
		Average:	73.55	28.79
	12	1	81.10	31.84
		2	82.40	32.96
		Average:	81.75	32.40

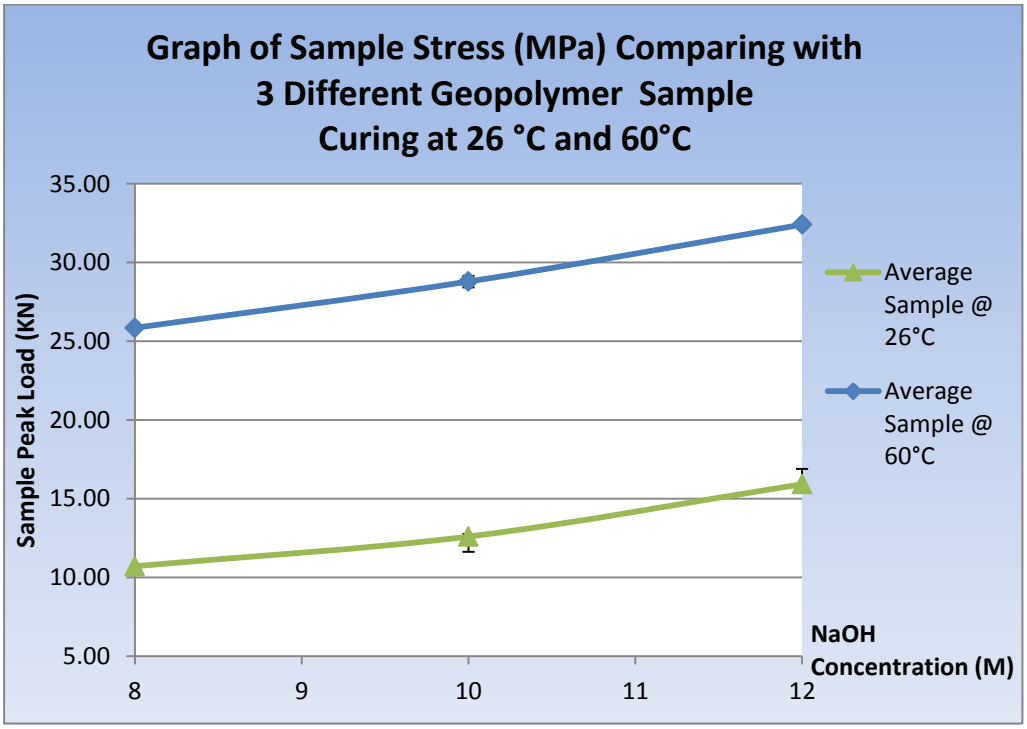

FIGURE 4.26: Graph of Sample Stress (MPa) Comparing with 3 Different Geopolymer Sample Curing at 26 °C and 60°C

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Based on Figure 4.26, the graph show that the geopolymer samples that cure at 60°C is much stronger and can withstand more stress compare to the samples that cure at 26°C. This pattern is roughly the same with the samples that are being immersed in the sulphuric acid.. Curing at higher temperature with high concentration of NaOH can help to form a strong structure and make the sample more dense compare to the samples that are cure at 26°C and lower concentration of NaOH.

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CHAPTER 5
CONCLUSION

5. CONCLUSION

In conclusion, the study of chemical properties of geopolymer using experimental study is important to understand chemical behavior of geopolymer. The knowledge and theory learnt in the class throughout this project. The project is feasible and practical to be done in given time frame. The preliminary methodologies have been outlined and all the chemical, tools and equipments are available.

5.1 Relevancy to Objective

Recall the priority objective of this project which is to develop a geopolymer waste storage which is chemically resistant to hazardous material is achieved. In order to fulfill this objective, number of tests had been conducted. The scope of the tests have meet and related with the objective. Based on the result, geopolymer are very stable upon reaction with sulphuric acid and sodium sulphate. In the future, geopolymer has a massive potential in replacing the commercial cement as the storage for the hazardous material.

5.2 Future Work

The future works of this project are stated as below:

- Continue conducting experiment and analyze the results
  The results gained from the experiments will give better idea on which the best properties of the geopolymer that can be applied to be used as a hazardous waste container. Comparison also can be made by construction of graph based on the collection of data from the experiment. Other

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parameter that can be continue to be tested are creep and drying shrinkage and also leaching test.

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CHAPTER 6: REFERENCES

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