



# **The Effect Of Natural Retarder On Geopolymer Concrete With Different Curing Regime**

by

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the requirements for the  
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# **CERTIFICATION OF APPROVAL**

## **The Effect Of Natural Retarder On Geopolymer Concrete With Different Curing Regime**

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**Nadia Nadzirah Binti Jamil**

A project dissertation submitted to the  
Civil Engineering Programme  
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**BACHELOR OF ENGINEERING (Hons)**  
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**UNIVERSITI TEKNOLOGI PETRONAS**  
**TRONOH, PERAK**  
**JUNE 2010**

## 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.



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NADIA NADZIRAH BINTI JAMIL



## ABSTRACT

Geopolymer concrete is an alternative environmentally friendly product to replace Ordinary Portland Cement (OPC) concrete as main construction material around the world. This eco-friendly concrete material developed because of the needs to reduce the greenhouse gases emission, minimize waste and save the non-renewable resources as the effect of Ordinary Portland Cement production. This research was proposed to obtain the optimum mix proportion and curing method for geopolymer concrete with cast in-situ applications. Fly ash has been used as a source material for this research work while the alkaline solutions used in geopolymerisation is a combination of sodium hydroxide (NaOH) and sodium silicate ( $\text{Na}_2\text{SiO}_3$ ). Different curing regimes have been applied to cure the geopolymer concrete to find the best curing method. Sucrose was utilized in the mixture to produce workable geopolymer concrete which is not too stiff and easily hardened. The compressive strength test done on the geopolymer concrete sample showed that external exposure curing provided superior performance compared to other curing method. It was also supported by the observation at microstructure level through FESEM test.

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## **ABBREVIATIONS AND NOMENCLATURES**

|                                  |   |
|----------------------------------|---|
| OPC                              | Ordinary Portland cement                    |
| UPV                              | Ultrasonic Pulse Velocity                   |
| FESEM                            | Field Emission Scanning Electron Microscopy |
| Mpa                              | Mega Pascal                                 |
| CO <sub>2</sub>                  | Carbon Dioxide                              |
| NaOH                             | Sodium Hydroxide                            |
| Na <sub>2</sub> SiO <sub>3</sub> | Sodium Silicate                             |

# **THE EFFECT OF NATURAL RETARDER ON GEOPOLYMER CONCRETE WITH DIFFERENT CURING REGIME**

## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1 BACKGROUND OF STUDY**

Ordinary Portland Cement (OPC) is the main ingredients used in the production of the concrete and the most widely used as construction material in the world. It is estimated that the production of cement will increase from about 1.5 billion tons in 1995 to 2.2 billion tons in 2010 (Malhotra, 1999).

On the other hand, the climate change due to global warming has become a major concern. The global warming was caused by the emission of greenhouse gases such as carbon dioxide ( $\text{CO}_2$ ) to the atmosphere by human activities. Among the greenhouse gases,  $\text{CO}_2$  contribute about 65% of global warming and the cement industry is held responsible for some of the  $\text{CO}_2$  emission. This is because the production of OPC requires the burning of large quantities of fuel and decomposition of limestone that result in significant emissions of  $\text{CO}_2$ . For every ton production of Portland cement emits approximately one ton of  $\text{CO}_2$  into the atmosphere (Davidovits, 1994 ; McCaffery, 2002).

Several efforts are in progress to reduce the use of Portland cement in concrete in order to address the global warming issue. These include the utilization of supplementary cement replacement material (CRM) such as fly ash, silica fume, MIRHA, granulated blast furnace slag and metakaolin in the development of alternative binders to Portland cement (Rangan, 2008).

In this respect, another form of cementitious materials called geopolymer has been developed as an alternative binder to the Portland cement. Geopolymers are the members of the family of inorganic polymers. The chemical composition of the geopolymer material is similar to the natural zeolitic materials, but the microstructure is amorphous. There are two main constituents of geopolymers, namely the source materials and the alkaline liquids (Rangan, 2008).

The source materials for geopolymers based on alumina-silicate should be rich in silicon (Si) and aluminium (Al). These could be natural mineral such as kaolite, clays and others. Alternatively, by-product materials such as fly ash, silica fume, rice husk ash, etc could be used as source materials. The choice of the source materials for making geopolymers depends on factors such as availability, cost, type of application and the specific demand of the end users (Rangan, 2008).

The alkaline liquids used are from soluble alkali metals that are usually Sodium or Potassium based. The most common alkali liquid used in geopolymerisation is a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate or potassium silicate (Rangan, 2008).

Geopolymer binder is usually made of fly ash activated with alkaline solution at low temperature and it is sometime called alkali-activated fly ash. These materials differ substantially from Portland cement, because geopolymers use a totally different reaction pathway in order to attain structural integrity. Geopolymers utilize the polycondensation of silica and alumina precursors and a high alkali content to attain structural strength (Jaarsveld, 2002).

As opposed to OPC, the manufacture of fly ash geopolymer does not consume high level of energy, as fly ash is already an industrial by-product. This geopolymer technology has the potential to reduce emission of CO<sub>2</sub> by 80% because high temperature calcining is not required (Davidovits, 1993).



Lately, numerous researches have highlighted the potential use of fly ash geopolymer in cement and concrete production. The primary difference between geopolymer concrete and Portland cement concrete is the binder. The silicon and aluminum oxide in the low calcium fly ash reacts with the alkaline liquids to form the geopolymer paste that binds coarse aggregates, fine aggregates and other un-reacted materials together to form the geopolymer concrete. The component of geopolymer concrete mixtures can be designed using the tools currently available for Portland cement concrete. The compressive strength and the workability of the geopolymer concrete are influenced by several parameters such as ratio of alkaline liquid-to-fly ash (by mass), concentration (in Molar) of NaOH solution, ratio of sodium silicate solution-to-sodium hydroxide solution (by mass), curing temperature, curing time, water content of mixture, mixing time and also age of concrete (Rangan, 2008).

Geopolymer concrete have also been investigated to have good bond strength to OPC concrete, hence it is a good repair material with superior abrasion resistance. Fly ash geopolymer composites have superior performance compared to OPC concrete in certain areas such as resistance to sulfate attack and have lower and creep and shrinkage than conventional concrete. Geopolymers concrete are generally believed to perform better than the conventional concrete in fire, due to their ceramic like properties (Sanjayan, 2008).

Currently, geopolymeric materials have a wide range of applications in the field of industries such as in the automobile and aerospace, non-ferrous and metallurgy, civil engineering and plastic industries.

| Si/Al       | Application   |
|-------------|---|
| 1           | Bricks, ceramics, fire protection   |
| 2           | Low CO <sub>2</sub> cements, concrete, radioactive and 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                              |

*Table 1.1 : Applications of Geopolymers*



## 1.2 PROBLEM OF STATEMENT

Concrete usage around the world is one of the most widely used construction materials. The conventional concrete is using Ordinary Portland Cement (OPC) as a main ingredient. The production of one ton of cement emits approximately one ton of carbon dioxide ( $\text{CO}_2$ ) to the atmosphere and can lead to the air pollution. Moreover, cement production is not only highly energy-intensive, but also consumes significant amount of natural resources.

The geopolymer concrete based on fly ash has been introduced in order to reduce the  $\text{CO}_2$  emission to the atmosphere caused by the cement production. Most of the fly ash available globally is low calcium fly ash formed from coal-burning power stations. The extent of power generated by these plants is on the increase due to the huge reserves of good quality coal available worldwide, the volume of fly ash would also increased. Therefore, the huge quantities of fly ash will be available for this industry. Since the fly ash is a waste material, the low calcium fly ash geopolymer concrete is cheaper than the Ordinary Portland cement (OPC).

In the industry, it is recommended to apply heat curing for the fly ash geopolymer concrete. The specimen will be heat cured at  $80^\circ\text{C}$  in an oven. Due to this, geopolymer concrete is only limited for precast concrete only. In order to prepare the geopolymer concrete to be cast in-situ concrete, an alternative curing regime was introduced by using the natural source, which is sunlight (external exposure) to cure the geopolymer concrete.

The fresh geopolymer concrete is easily to hardened and very stiff. In order to minimize these problems, a small amount of natural additives have been added in the mix design of geopolymer concrete to increase its setting time.

Therefore, this research was proposed to analyze the optimum mix proportion of fly ash based geopolymer concrete that involved suitable cast in-situ curing regime. Sucrose

was utilized to address the workability performance of fly ash based geopolymer concrete since the amount of water is limited regarding the segregation issue.

### **1.3 OBJECTIVE**

The objectives of this topic are :

1. To identify the effect of natural retarder (sucrose) on geopolymer concrete.
2. To ascertain the suitable curing regime for geopolymer concrete in cast in situ provision.
3. To determine the optimum mix proportion of geopolymer concrete incorporating with sucrose as an additives.
4. To determine the compressive strength and microstructure properties of geopolymer concrete.

### **1.4 SCOPE OF WORKS**

This research utilized low-calcium fly ash as the source material for making geopolymer concrete. The alkaline liquid used in geopolymerisation is sodium based which is a combination of 8M sodium hydroxide (NaOH) and sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) with the ratio of  $\text{Na}_2\text{O}$  and  $\text{SiO}_2$  is 2.0. Sucrose used as an additive in the mix design. The purpose to have sucrose is to delay the setting time of geopolymer concrete. Three different curing regimes were applied, which are ambient curing, external exposure and oven.

The concrete properties studied include the workability of fresh concrete using slump test, the quality and integrity of the concrete by determining the presence of voids, honeycomb or crack using UPV test and the compressive strength on hardened concrete at 1, 3, 7, 28 and 56 days. After 56 days, the Field Emission Scanning Electron Microscopy (FESEM) test was performed to study the microstructure of geopolymer concrete.



## **CHAPTER 2**

### **LITERATURE REVIEW**

This chapter presents the backgrounds to the needs for the developments of alternative binders to manufacture concrete and the use of fly ash in concrete. The available published literature on geopolymer technology was also briefly reviewed.

#### **2.1 CONCRETE AND ENVIRONMENTS**

The global use of concrete is second only to water. As the demand for concrete as a construction material increases, so also the demand for Portland cement. Ordinary Portland Cement (OPC) is the main ingredients used in the production of the concrete and the most widely used as construction materials in the world. Portland cement concrete industry has grown astronomically in recent years. It will continue to grow as the result of continuous urban development. However, Portland cement concrete poses problems such as durability and carbon dioxide emissions (Mehta, 1997; Hardjito, 2008).

Manufacturing of Portland cement is an energy intensive process and releases a large amount of green house to the atmosphere. It has been reported that 1.35 billion tons is produced from this process worldwide, which accounts for about 7% of the green house gas produced annually. The production of 1 tonne ordinary Portland cement consumes 4GJ energy and produces about 1 tonne of carbon dioxide to the atmosphere (Mehta 2001). About half of the carbon dioxide emissions from Portland cement production are due to calcination of limestone, while the other half are due to combustion of fossil fuel. In the year 1995, the global production of ordinary Portland cement was about 1.4 billion tonne, thus emitting about 1.4 billion tonne of carbon dioxide to the atmosphere (Malhotra, 2004).

Carbon dioxide emission trading is likely to be a critical factor for the construction industry, in particular, the cement and concrete industry. The World Earth Summits in

Rio de Janeiro, Brazil in 1992, and Kyoto, Japan in 1997 made it abundantly clear that for long-term sustainability, rate of emission of greenhouse gases to the atmosphere must be prevented from increasing. It has been speculated that one tonne of emission can have a trading value of US\$10 (Malhotra, 1999; Malhotra, 2004).

According to researchers at Oak Ridge National Laboratory, there are two very different sources of carbon dioxide emissions during cement production. Combustion of fossil fuels to operate the rotary kiln is the largest source: approximately  $\frac{3}{4}$  tons of CO<sub>2</sub> per ton of cement. But the chemical process of calcining limestone into lime in the cement kiln also produces CO<sub>2</sub>. This chemical process is responsible for roughly  $\frac{1}{2}$  ton of CO<sub>2</sub> per ton of cement. Combining these two sources, for every ton of cement produced, 1.25 tons of CO<sub>2</sub> is released into the atmosphere (News, 1993).

Besides CO<sub>2</sub>, both cement and concrete production generate considerable quantities of air-pollutant emissions. Dust is usually the most visible of these pollutants. The U.S. EPA (cited by UBC researchers) estimates total particulate (dust) emissions of 360 pounds per ton of cement produced, the majority of which is from the cement kiln. Other sources of dust from cement production are handling raw materials, grinding cement clinker, and packaging or loading finished cement, which is ground to a very fine powder-particles as small as 0.00004 of an inch (News, 1993).

Other air pollution emissions from cement and concrete production result from fossil fuel burning for process and transportation uses. Air pollutants commonly emitted from cement manufacturing plants include sulfur dioxide (SO<sub>2</sub>) and nitrous oxides. SO<sub>2</sub> emissions (and to a lesser extent SO<sub>3</sub>, sulfuric acid, and hydrogen sulfide) result from sulfur content of both the raw materials and the fuel (especially coal). Strategies to reduce sulfur emissions include use of low-sulfur raw materials, burning low-sulfur coal or other fuels, and collecting the sulfur emissions through state-of-the-art pollution control equipment. Interestingly, lime in the cement kiln acts as a scrubber and absorbs some sulfur (News, 1993).



Nitrous oxide emissions are influenced by fuel type and combustion conditions (including flame temperature, burner type, and material/exhaust gas retention in the burning zone of the kiln). Strategies to reduce nitrogen emissions include altering the burner design, modifying kiln and pre-calciner operation, using alternate fuels, and adding ammonia or urea to the process. The cement industry claims to have reduced overall pollution emissions by 90% in the last 20 years (News, 1993).

The concrete industry has recognized these issues. For example, the U.S. Concrete Industry has developed plans to address these issues in 'Vision 2030: A Vision for the U.S. Concrete Industry'. The document states that *'concrete technologists are faced with the challenge of leading future development in a way that protects environmental quality while projecting concrete as a construction material of choice. Public concern will be responsibly addressed regarding climate change resulting from the increased concentration of global warming gases.'* In this document, strategies to retain concrete as a construction material of choice for infrastructure development, and at the same time to make it an environmentally friendly material for the future have been outlined (Mehta, 2001; Plenge, 2001).

In the concrete industry, the easiest and most effective way to reduce green house gases is to increase the use of such silica rich by-products as fly ash, slag and silica fume thereby reducing the amount of cement used per cubic meter of concrete. Concrete generates about 7% of the total CO<sub>2</sub> generated worldwide. About 1625 million tons of cement was produced in 2000. If a ton of cement produces a ton of CO<sub>2</sub>, and if only 18.5% of the cement can be replaced with slag or fly ash, then the CO<sub>2</sub> reduction would be 300 million tons per year world wide. Over the past decade the average annual increase in CO<sub>2</sub> emissions is 1.3 percent or nearly 300 million tons a year worldwide (Bremner, 2001).

Our industry alone could easily reduce global warming and at the same time enhance the properties of the concrete produced. Not only can our industry greatly reduce global

warming but also we can roll it back, as 18.5% cement reduction pales in comparison to what should be used for optimum concrete properties (Bremner, 2001).

According to McCaffrey (2002), he suggested three alternatives in order to reduce the amount of carbon dioxide (CO<sub>2</sub>) by the cement industries by decreasing the amount of calcined material in cement, by decreasing the amount of cement uses in concrete and by decreasing the number of building using cement. Mehta (2002) ideas are to produce the environmentally friendly concrete. He proposed a short-term effort known as ‘*industrial ecology*’ which are reduce the use of natural resources, utilize less energy, and minimize CO<sub>2</sub> emissions. Lowering the rate of material consumptions to reduce the impact of unwanted industrial by-products is his long term view (Hardjito, 2004).

## 2.2 FLY ASH

Fly ash is comprised of the non-combustible mineral portion of coal. When coal is consumed in a power plant, it is first ground to the fineness of powder which typically finer than cement particles. Blown into the power plant’s boiler, the carbon is consumed which leaving molten particles rich in silica, alumina and calcium. These particles solidify as microscopic, glassy spheres that are collected from the power plant’s exhaust before they can “fly” away, hence the product’s name is called Fly Ash (HWR).

According to American Concrete Institute (ACI) Committee 116R, fly ash is “*the finely divided residue that results from the combustion of ground or powdered coal and that is transported by flue gases from the combustion zone to the particle removal system*” (ACI Committee 232, 2004).

Chemically, fly ash is a pozzolan. ACI 116R defines “pozzolan” as “*a siliceous or siliceous and aluminous material that in itself possesses little or no cementitious value but that will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds having*



*cementitious properties; there are both natural and artificial pozzolans” (HWR, ACI Committee 232, 2004).*

When mixed with lime (calcium hydroxide), pozzolans combine to form cementitious compounds. Concrete containing fly ash becomes stronger, more durable, and more resistant to chemical attack. Mechanically, fly ash also pays dividends for concrete production. Because fly ash particles are small, they effectively fill voids. Because fly ash particles are hard and round, they have a “ball bearing” effect that allows concrete to be produced using less water. Both characteristics contribute to enhanced concrete workability and durability (HWR).

Fly ash material solidifies while suspended in the exhaust gases and is collected by electrostatic precipitators or filter bags. Since the particles solidify while suspended in the exhaust gases, fly ash particles are generally spherical in shape and range in size from 0.5  $\mu\text{m}$  to 100  $\mu\text{m}$ . They consist mostly of silicon dioxide ( $\text{SiO}_2$ ), which is present in two forms: amorphous, which is rounded and smooth, and crystalline, which is sharp, pointed and hazardous; aluminium oxide ( $\text{Al}_2\text{O}_3$ ) and iron oxide ( $\text{Fe}_2\text{O}_3$ ). Fly ashes are generally highly heterogeneous, consisting of a mixture of glassy particles with various identifiable crystalline phases such as quartz, mullite, and various iron oxides (MCCR Committee, 2006).

The types and relative amounts of incombustible matter in the coal determine the chemical composition of fly ash. The chemical composition is mainly composed of the oxides of silicon ( $\text{SiO}_2$ ), aluminium ( $\text{Al}_2\text{O}_3$ ), iron ( $\text{Fe}_2\text{O}_3$ ), and calcium ( $\text{CaO}$ ), whereas magnesium, potassium, sodium, titanium, and sulphur are also present in a lesser amount (Malhotra and Ramezaniapour, 1994).

The major influence on the fly ash chemical composition comes from the type of coal. The combustion of sub-bituminous coal contains more calcium and less iron than fly ash from bituminous coal. The physical and chemical characteristics depend on the combustion methods, coal source and particle shape. The chemical compositions of

various fly ashes show a wide range, indicating that there is a wide variations in the coal used in power plants all over the world (Malhotra and Ramezaniapour, 1994).

There are two basic types of fly ash that are Class F and Class C. Class F fly ash or low-calcium fly ash normally produced from burning anthracite or bituminous coal. It consists of mainly an alumino-silicate glass and has less than 10 percent of CaO .Class C or high-calcium fly ash is normally produced from the burning of subbituminous coal and lignite and typically contains more than 20 percent of CaO (Ramezaniapour, 1994; ACAA, 2003; Rangan, 2005). Both types react in concrete in similar ways. Both Class F and Class C fly ashes undergo a “pozzolanic reaction” with the lime (calcium hydroxide) created by the hydration (chemical reaction) of cement and water, to create the same binder (calcium silicate hydrate) as cement. In addition, some Class C fly ashes may possess enough lime to be self-cementing, in addition to the pozzolanic reaction with lime from cement hydration (HWR).

The color of fly ash can vary from tan to gray to black, depending on the amount of unburned carbon in the ash. The lighter the color, the lower the carbon content. Lignite or subbituminous fly ashes are usually light tan to buff in color, indicating relatively low amounts of carbon as well as the presence of some lime or calcium. Bituminous fly ashes are usually some shade of gray, with the lighter shades of gray generally indicating a higher quality of ash (Anthony M, 1972).

The main benefit of fly ash in concrete is that it not only reduces the amount of non-durable calcium hydroxide (lime), but in the process converts it into calcium silicate hydrate (CSH), which is the strongest and most durable portion of the paste in concrete. The use of fly ash creates significant benefits for our environment. Fly ash use conserves natural resources and avoids landfill disposal of ash products. By making concrete more durable, life cycle costs of roads and structures are reduced. Furthermore, fly ash use partially displaces production of other concrete ingredients, resulting in significant energy savings and reductions in greenhouse gas emissions (HWR).



In addition to economic and ecological benefits, the use of fly ash in concrete improves its workability, reduces segregation, bleeding, heat evolution and permeability, inhibits alkali-aggregate reaction, and enhances sulfate resistance. Even though the use of fly ash in concrete has increased in the last 20 years, less than 20% of the fly ash collected was used in the cement and concrete industries (Helmuth, 1987). Enhanced economics and improved technologies have led to a greater use of fly ash, principally in the ready-mix concrete industry. Fly ash is now used in concrete for many reasons, including improvements in workability of fresh concrete, reduction in temperature rise during initial hydration, improved resistance to sulfates, reduced expansion due to alkali-silica reaction, and contributions to the durability and strength of hardened concrete. Development of high volume fly ash (HVFA) concrete is the most important achievement in the use of fly ash in concrete (ACI Committee 232, 2004).

## **2.3 GEOPOLYMER**

In 1978, Joseph Davidovits developed inorganic polymeric materials and coined the term “Geopolymer” for it (1990). Geopolymer has the potential to replace ordinary Portland cement concrete and produce fly ash-based Geopolymer concrete with excellent physical and mechanical properties. Geopolymer was used as the binder to completely replaced ordinary Portland cement in producing Geopolymer concrete. In order to produce Geopolymer, low-calcium fly ash needs to be activated by an alkaline solution to produce polymeric Si-O-Al bonds. Geopolymer concrete has the potential to reduce greenhouse emissions from the concrete industry by 80% (Daniel et al, 2006).

Geopolymer concrete is also known as Alkali-activated concrete or Inorganic polymer concrete. The chemical composition of geopolymer is similar to zeolite, but amorphous in microstructure (Davidovits, 1999). The silicon and the aluminium atoms in the source materials are induced by alkaline solutions to dissolve and form a gel. The polymerisation process may be assisted by applied heat, and followed by drying. The geopolymer gel binds the loose coarse aggregates, fine aggregates and other un-reacted

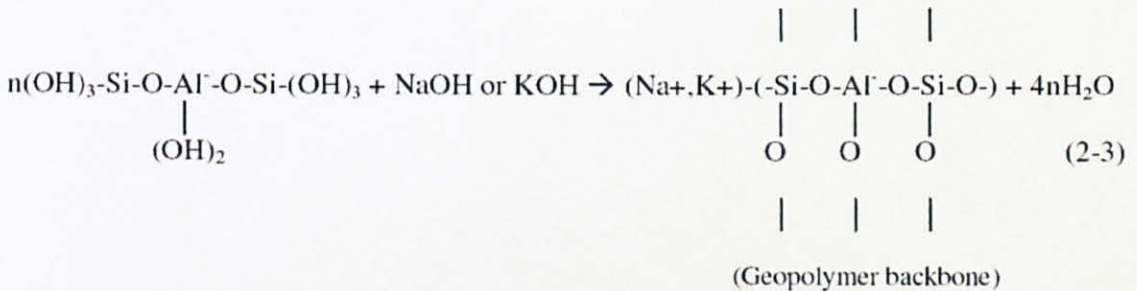
materials together to form the geopolymer concrete. The chemical reaction period is substantially fast (Hardjito, 2004).

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 (Davidovits, 1999) :



Where: M = the alkaline element or cation such a potassium, sodium or calcium; the symbol – indicates the presence of a bond, n is the degree of polycondensation or polymerisation; z is 1,2,3, or higher, up to 32.

The schematic formation of geopolymer material can be shown as described by Equations (2-2) and (2-3) (van Jaarsveld et al, 1997; Davidovits, 1999):



The chemical reaction may comprise the following steps (Davidovits, 1999; Xu and van Deventer, 2000):

- 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 (Palomo et al, 1999).

A geopolymer can take one of the three basic forms (Davidovits, 1999):

- Poly (sialate), which has  $[-\text{Si-O-Al-O-}]$  as the repeating unit.
- Poly (sialate-siloxo), which has  $[-\text{Si-O-Al-O-Si-O-}]$  as the repeating unit.
- Poly (sialate-disiloxo), which has  $[-\text{Si-O-Al-O-Si-O-Si-O-}]$  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 nanopores 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 (Rangan, 2005).

Geopolymer concrete has excellent mechanical properties, does not dissolve in acidic solutions, and does not generate any deleterious alkali-aggregate reaction even in the presence of high alkalinity (Davidovits, 1999). Some of the immediate applications of geopolymer concrete are marine structures, precast concrete products such as railway sleepers, sewer pipes, pre-fabricated units for the housing market as well as waste containment or encapsulation (Hardjito, 2004).

## 2.4 CONSTITUENTS OF GEOPOLYMER

There are two main constituents of geopolymers, namely the source materials and the alkaline liquids. The source materials for geopolymers based on alumina-silicate should be rich in silicon (Si) and aluminium (Al). These could be natural minerals such as kaolinite, clays, etc. Alternatively, by-product materials such as fly ash, silica fume, slag, rice-husk ash, red mud, etc could be used as source materials. The choice of the source materials for making geopolymers depends on factors such as availability, cost, type of application, and specific demand of the end users (Rangan, 2008).

Several researched have been investigated in the past about some minerals and industrial by product material as source material. From researched on the nature of source material, it have been found that calcined source materials, such as fly ash, slag, calcined kaolin, demonstrated a higher final compressive strength when compared to those who made using non-calcined materials such as kaolin clay, mine tailings and naturally occurring minerals (Barbosa et al. 2000). However, there also some researches shown that significant improvement in compressive strength and reduction in reaction time can be achieved using a combination of calcined (e.g fly ash) and non-calcined materials (e.g koalinite or kaolin clay) (Xu and van Deventer,2002).

Among the by-product materials, only fly ash and slag have been proved to be the potential source materials for making geopolymers. Fly ash was considered to be advantageous due to its high reactivity that comes from its finer particles size than slag. Moreover, low-calcium fly ash is most abundantly available around the world rather than slag (Rangan, 2005).

The alkaline liquids are from soluble alkali metals that are usually Sodium or Potassium based. The most common alkaline liquid used in geopolymerisation is a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate or potassium silicate (Rangan, 2008).



## CHAPTER 3

### METHODOLOGY

Three sections were reviewed under this chapter. The constituents of fly ash-based geopolymer concrete were discussed in this chapter. All the relevant methods and procedure used in order to achieve the objectives of this research have also been reviewed.

#### 3.1 Constituents of Geopolymer Concrete

##### 3.1.1 Fly Ash

The source material for this research work is low-calcium fly ash Class F obtained from the Manjung Power Plant, located at Perak. The chemical composition of the fly ash was determined by X-Ray Fluorescence (XRF) analysis are given in Table 3.1. The molar ratio of Si to Al is about 2:1 while and the most constituent in the composition of fly ash is the silicon and aluminium oxides contents. The dark color of fly ash indicates that there is  $\text{Fe}_2\text{O}_3$  in these compositions.

| Compounds               | Percentages (%) |
|-------------------------|-----------------|
| $\text{SiO}_2$          | 51.19           |
| $\text{Al}_2\text{O}_3$ | 24              |
| $\text{Fe}_2\text{O}_3$ | 6.6             |
| $\text{CaO}$            | 5.57            |
| $\text{MgO}$            | 2.4             |
| $\text{SO}_3$           | 0.88            |
| $\text{K}_2\text{O}$    | 1.14            |
| $\text{Na}_2\text{O}$   | 2.12            |

*Table 3.1 : Chemical Composition of Fly Ash*

##### 3.1.2 Alkaline Liquid

The alkaline liquid used is the combination of sodium hydroxide ( $\text{NaOH}$ ) solution and sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) solution. The sodium hydroxide pellets with 99% purity

supplied by Quicklab Sdn. Bhd was dissolved in water to obtain the sodium hydroxide solution (NaOH) with a concentration of 8M.

Sodium silicate solution used was obtained from Malay-Sino Chemical Industries Sdn Bhd located at Ipoh, Perak. The chemical composition of the sodium silicate solution was  $\text{Na}_2\text{O} = 14.73\%$ ,  $\text{SiO}_2 = 29.75\%$  and water 55.52% by mass. The used of sodium hydroxide solution as alkaline activator because it is cheap and widely available rather than potassium hydroxide solutions.

### **3.1.3 Aggregate**

Two types aggregates used are coarse and fine aggregates. The maximum size of coarse aggregates used was 20 mm while the size for fines aggregates was not more than 5 mm. Approximately, 75% - 80% of geopolymer concrete mass was occupied by these aggregates. Saturated-surface-dry (SSD) conditions were applied to both coarse and fine aggregates. The sieve analysis was also performed on sample of aggregates to determine its grading. Both types of aggregates used are well-graded type (*Appendix I*).

### **3.1.4 Water**

Generally, water in concrete consists of combination of water added to the mixture and water that held by aggregates. Mixing water in concrete should not contain any undesirable impurities such as suspended solids, organic matters or inorganic constituents in excessive proportions or hence it will give harmful to the concrete in term of its properties. In this research, tap water has been used as a source of mixing water for concrete.

### **3.1.5 Sucrose**

Sucrose has been added in the concrete mixture as a natural additive in order to enhance the workability of geopolymer concrete. Only small percentage of sucrose needed in

order to delay in the setting time of geopolymer concrete. Sucrose used in this research obtained from Prai, Pulau Pinang.

### 3.2 Sample Preparations

The mixture of geopolymer concrete in this research was prepared using the selected material as discussed in section 3.1 above :

- Low-calcium Fly Ash Class F
- Alkali liquid from combination of sodium hydroxide (NaOH) solution with sodium silicate ( $\text{Na}_2\text{SiO}_3$ )
- Coarse and fine aggregate in saturated-surface-dry (SSD) conditions
- Sucrose
- Extra water

Figure 3.1 shows the dry materials used in this research. Table 3.2 shows the mix proportions of geopolymer concrete that were applied in this research.



*Figure 3.1 : Fine, Coarse aggregates, Fly ash and Sucrose*



| Mix Code | Fly Ash Content (kg/m <sup>3</sup> ) | Coarse Aggregate (kg/m <sup>3</sup> ) | Fine Aggregate (kg/m <sup>3</sup> ) | NaOH Solution (kg/m <sup>3</sup> ) | Na <sub>2</sub> SiO <sub>3</sub> Solution (kg/m <sup>3</sup> ) | Extra Water (kg/m <sup>3</sup> ) | Sucrose % | Sucrose (kg/m <sup>3</sup> ) | Curing            |
|----------|--------------------------------------|---------------------------------------|-------------------------------------|------------------------------------|--|----------------------------------|-----------|------------------------------|-------------------|
| E1       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 35                               | 3         | 10.5                         | Ambient           |
| E2       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 35                               | 2         | 7                            |                   |
| E3       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 35                               | 1         | 3.5                          |                   |
| E4       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 35                               | 0         | 0                            |                   |
| E5       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 52.5                             | 0         | 0                            |                   |
| F1       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 35                               | 2         | 10.5                         | External Exposure |
| F2       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 35                               | 2         | 7                            |                   |
| F3       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 35                               | 1         | 3.5                          |                   |
| F4       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 35                               | 0         | 0                            |                   |
| F5       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 52.5                             | 0         | 0                            |                   |
| G1       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 35                               | 2         | 7                            | Oven              |
| G2       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 35                               | 1         | 3.5                          |                   |
| G3       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 35                               | 0         | 0                            |                   |
| G4       | 350                                  | 1200                                  | 645                                 | 41                                 | 103  | 52.5                             | 0         | 0                            |                   |

*Table 3.2 : Mix Proportions for Geopolymer Concrete*



### 3.2.1 Mixing, Sampling and Curing

The conventional method or technique used in the manufacturing of Ordinary Portland cement concrete was applied to the manufacturing of geopolymer concrete. Alkaline liquid or activator was prepared in the laboratory. Distilled water was used to dissolve NaOH pellets in order to avoid any unknown contaminants in the solutions. The alkaline activator was prepared by adding the sodium silicate solutions into sodium hydroxide solutions and mixing them together. In order to maintain the reactivity of the alkali solution, their preparation was done just before starting the mixing of geopolymer concrete.

Mixing is very important in order to obtain the uniformity of the mix. Rotating pan mixer was used in this research to mix the geopolymer concrete for about 4 minutes. To ensure the homogeneity of the mixture, fly ash, coarse and fine aggregate were first dry mixed together in the mixer for 2.5 minutes. Alkaline activator which is the combinations of sodium silicate and NaOH solutions were then poured together with the prepared amount of sucrose diluted in extra water to the mixture and mixed for another 1.5 minutes. The fresh geopolymer concrete was then immediately cast into cube mould. Figure 3.2 shows the mixer used in this research and the fresh geopolymer concrete after mixing.



*Figure 3.2 : Rotating Pan Mixer and Fresh Geopolymer Concrete*

The cube mould used for the sample is 100 x 100 x 100 mm in size and made from steel (Figure 3.3). During casting, the mould and its base was clamped together in order to prevent leakage of geopolymer mixture. A thin layer of mineral oil was applied to the top and the inside surfaces of the mould to prevent the mixture sticking to the mould when it hardened. The fresh geopolymer mixture was compacted using the electric vibrator immediately after casting to remove any air voids (Figure 3.4). The vibrations uniformly applied to the entire mixture in the mould or else there are some parts that would not be fully compacted while there were some parts might be segregated due to over-compacting. The mixtures in the mould were then prepared for curing.



*Figure 3.3 : 100 x 100 x 100 mm Steel Cube Mould*



*Figure 3.4 : Compaction using Vibrator*

In this research, there were three different curing regimes applied to the sample of geopolymer concrete in order to study its performance at different curing regime, which is ambient curing (Figure 3.5), external exposure curing (Figure 3.6) and oven curing (Figure 3.7). The temperature for ambient curing was in the range of 27°C - 32°C while



for external exposure curing, the temperature range was between 33°C - 40°C, and oven curing used 65°C temperature setting. The samples were placed at ambient curing after casting without any delay time while for oven and external exposure curing the 1 hours delay time was applied before samples can be placed.



*Figure 3.5 : Ambient Curing*



*Figure 3.6 : External Exposure Curing*





*Figure 3.7 : Oven Curing*

### **3.3 Experimental Work**

The properties and performance of the geopolymer concrete were determined by performing some sample testing on the fresh and hardened geopolymer concrete. The testing performed was based on British Standard requirement at specified age of geopolymer concrete. There are two types of testing applied on hardened geopolymer concrete which are non-destructive and destructive. The non-destructive tests done on geopolymer concrete without or slightly damaged occur to the sample while destructive test were carried out until the sample failure. Table 3.3 shows the experimental work done in this research.

| Concrete Type                               | Test Type                       | Standard           | Equipment                   | Testing Age    | Sample Size                         | Number of Test    | Measurement Unit  |
|---|---------------------------------|--------------------|-----------------------------|----------------|-------------------------------------|-------------------|-------------------|
| FRESH CONCRETE                              | Slump Test                      | BS EN 12350-2:2000 | Slump Cone                  | Fresh Concrete | -                                   | each batch        | mm                |
| HARDENED CONCRETE<br>(Destructive Test)     | Compression Strength            | BS EN 12390-3:2002 | Compression Testing Machine | 3,7,28,56 days | 100 mm <sup>3</sup> cube            | 3 cubes / mix/age | N/mm <sup>2</sup> |
| HARDENED CONCRETE<br>(Non Destructive Test) | Ultrasonic Pulse Velocity (UPV) | BS EN 12504-4:2004 | PUNDIT UPV Tester           | 3,7,28,56 days | 2 surfaces 100 mm <sup>3</sup> cube | 2 readings / cube | km/s              |

*Table 3.3 : Experimental work done in this research.*

### 3.3.1 Slump Test

The slump test is the most well-known and widely used to measure the workability of the concrete. The American Concrete Institute (ACI) describes workability as “that property of freshly mixed concrete that determines the ease with which it can be mixed, placed, consolidated and finished to a homogeneous condition. In other words, workability also defined as “the amount of useful internal work necessary to produce full compaction”.

The apparatus for the slump test consisted of the 300mm high conical mould with smaller opening at the top and a standard 16mm diameter steel rod and rounded at the end as a tamping rod. The mould is placed on a smooth surface and filled with concrete in three layers. Each layer is tamped 25 times with tamping rod. The inside of the mould and its base should be moistened at the beginning of every test in order to reduce the influence on slump of the variation in the surface friction. After filling and tamping, the mould is then lifted vertically from concrete. The decrease in the height of the slumped concrete is called slump and is measured to the nearest 5mm. The test was performed to each of the fresh geopolymer concrete. Figure 3.8 shows the apparatus of the slump test used in this research and the measurement of the slump.



*Figure 3.8 : Slump test apparatus*



### 3.3.2 Ultrasonic Pulse Velocity (UPV) Test

This non-destructive test method was performed on geopolymer concrete to determine the quality of concrete by ultrasonic pulse velocity method. The method consists of measuring the time of an ultrasonic pulse travel through geopolymer concrete. The apparatus consists of transducers which were placed in contact with the concrete horizontally, a pulse generator with a frequency of between 10 and 150 Hz, an amplifier, a time measuring circuit and a display of the time taken by the pulse of longitudinal waves to travel between transducers. Figure 3.9 shows the UPV apparatus used in this research.



*Figure 3.9 : UPV apparatus*

### 3.3.3 Compressive Strength Test

The compressive strength test on hardened fly ash geopolymer concrete were done using testing machine ELE ADR 3000kN with 3.00 kN constant load applied. The test was done on 100mm x 100mm x 100mm geopolymer concrete sample size with different curing regime and different age of sample. The test were performed on sample at age 3, 7, 28 and 56 days for ambient and sunlight curing. For oven curing, the test was performed at 1, 3, 7 and 28 days. For every compressive strength test, three samples of geopolymer concrete were used. Figure 3.10 shows the compression testing machine used in this research.



*Figure 3.10 : Compressive Strength Testing Machine*

### **3.3.4 Microstructure Analysis**

The inner microstructure conditions of geopolymer concrete samples with different curing regime studied by performing the Field Emission Scanning Electron Microscopy (FESEM) analysis. The analysis was performed on concrete samples from oven and external exposure curing after the samples was cured at 56 days. Supra 55 VP Inca X-Act Oxford FESEM Instrument was used to perform the analysis. In order to obtain quality FESEM image, concrete as a non-conductive material must be coated with gold atoms in sputter coater. Coated concrete then was placed in the vacuum chamber inside the FESEM. To facilitate the operations of filament and electron inside FESEM, the analysis must be operated in specific pressure. Figure 3.11 shows the FESEM used in this research.



*Figure 3.11: Supra 55 VP Inca X-Act Oxford FESEM Instrument*



## CHAPTER 4

### RESULT AND DISCUSSION

In this chapter, the experimental result and analysis are discussed and presented. Several test have been conducted in which three sample of geopolymer concrete with different curing regime have been used for each test. The mean values for each test data have been used to plot the figures.

#### 4.1 WORKABILITY

The workability of the fresh geopolymer concrete depends on the amount of sucrose added in the mix design. The workability test on fresh geopolymer concrete was performed immediately after mixing. The slump values for each mix proportion are given in Table 4.1 below. The characteristic for almost all the mix proportions are moderately workable and not stiff.

| Mix Proportion         | Slump Value (cm) |
|------------------------|------------------|
| 3% sucrose + 10% water | 23               |
| 2% sucrose + 10% water | 23               |
| 1% sucrose + 10% water | 22               |
| 0% sucrose + 10% water | 22               |
| 0% sucrose + 15% water | 24               |

*Table 4.1: Slump value for geopolymer mix proportions*

Sucrose added was to delay the setting time of geopolymer concrete. Setting time means the time taken for geopolymer concrete to hardened. The longer time for geopolymer concrete to harden enhanced the workability of fresh geopolymer concrete.

Sucrose added in the mix proportion also affects the water viscosity, hence affects the fresh geopolymer concrete flow ability. It can be observed that from the increasing amount of sucrose added, the higher slump value obtained.

## 4.2 ULTRASONIC PULSE VELOCITY (UPV) TEST

This non-destructive test method was performed on geopolymer concrete to determine the quality of concrete by ultrasonic pulse velocity method. The method consists of measuring the time of an ultrasonic pulse travel through 100mm geopolymer concrete sample. The correlation of velocity through geopolymer concrete and curing days of geopolymer concrete sample are given in Figures 4.1 – Figures 4.3 below.

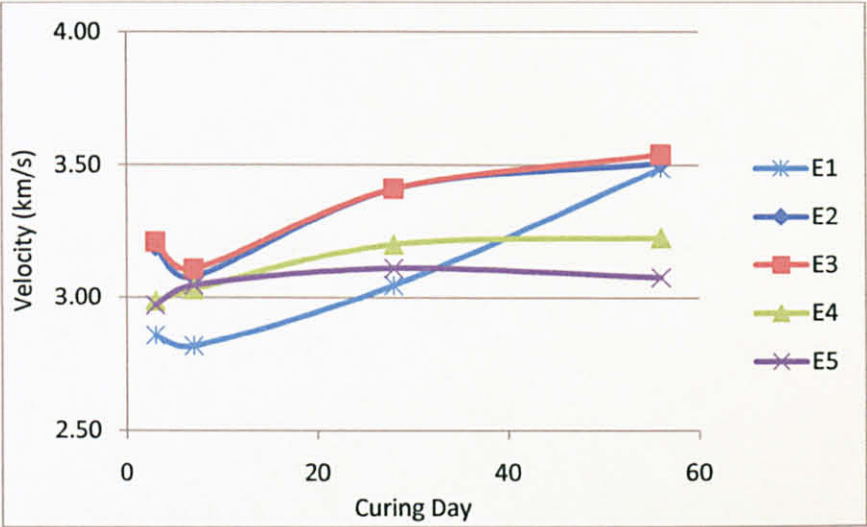


Figure 4.1: Relationship between UPV and Curing Days with Ambient Curing

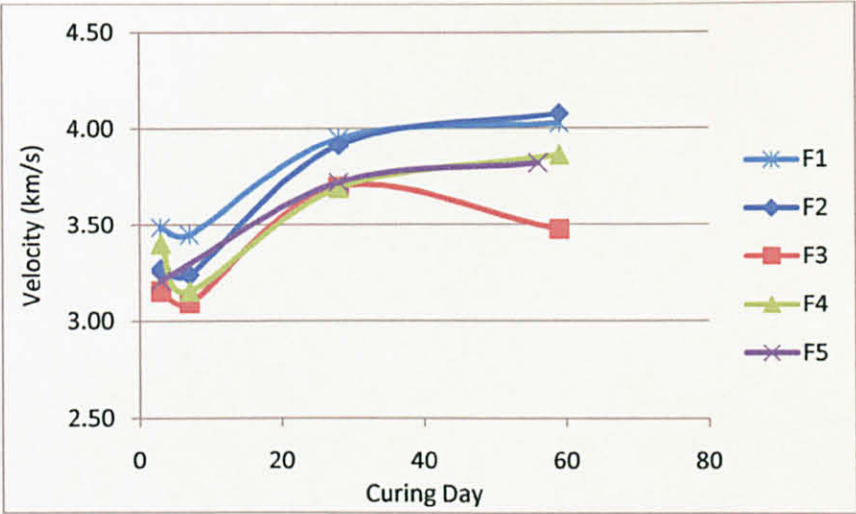
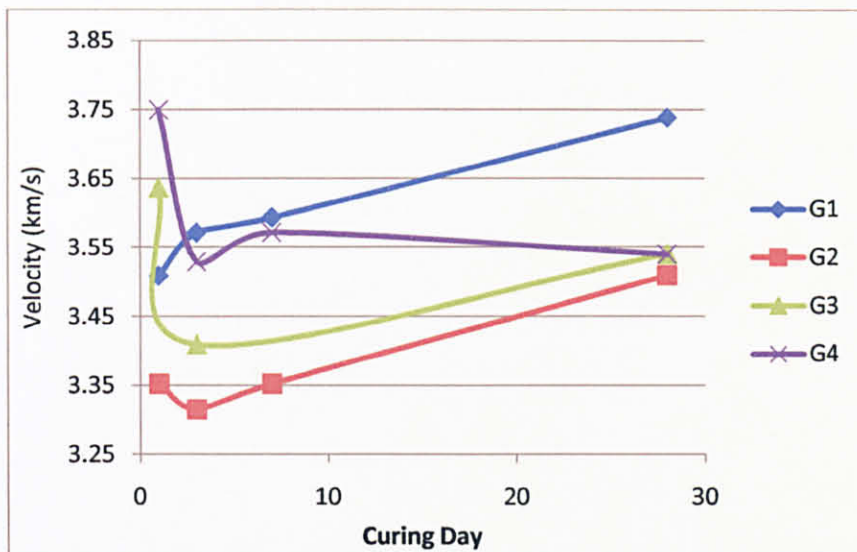


Figure 4.2: Relationship between UPV and Curing Days with External Exposure



*Figure 4.3: Relationship between UPV and Curing Days with Oven Curing*

For each curing regime, the pattern of the relationship between curing days of geopolymer concrete sample and velocity through geopolymer concrete sample are quite similar. In the first day, the value was high and then drastically decreased before it increased back until the maximum curing days at 56 days.

In the first day, there were still more water entrapped in the geopolymer concrete sample. This water will occupy some spaces in the geopolymer concrete sample. In the first week, the water will be released and evaporated and left some micropores inside the concrete. This explains why the velocity was fluctuative for the first week. After that, the polymeric reaction will take places and its end products start to fill up the micropores.

### 4.3 DENSITY

The density of concrete is a measurement of concrete solidity. To form a higher or lower density of concrete end product, the mix proportions of concrete can be modified to get the result.



The results for geopolymer concrete density are in range of 2390 kg per cubic meter ( $\text{kg/m}^3$ ) to 2460 kg per cubic meter ( $\text{kg/m}^3$ ) as showed in Table 4.2 below. It shows that the density of geopolymer concrete are quite similar to density of normal OPC which about 2400 kg per cubic meter ( $\text{kg/m}^3$ ). The concrete densities are varies depend on the amount and density of the aggregate, how much air entrapped and also the size of aggregate used.

| Curing Regime     | Mix Code | Average Weight (kg) | Concrete Volume ( $\text{m}^3$ ) | Density ( $\text{kg/m}^3$ ) |
|-------------------|----------|---------------------|----------------------------------|-----------------------------|
| Ambient           | E1       | 2.43                | 0.001                            | 2430                        |
|                   | E2       | 2.42                | 0.001                            | 2420                        |
|                   | E3       | 2.46                | 0.001                            | 2460                        |
|                   | E4       | 2.45                | 0.001                            | 2450                        |
|                   | E5       | 2.45                | 0.001                            | 2450                        |
| External Exposure | F1       | 2.42                | 0.001                            | 2420                        |
|                   | F2       | 2.40                | 0.001                            | 2400                        |
|                   | F3       | 2.39                | 0.001                            | 2390                        |
|                   | F4       | 2.44                | 0.001                            | 2440                        |
|                   | F5       | 2.39                | 0.001                            | 2390                        |
| Oven              | G1       | 2.43                | 0.001                            | 2430                        |
|                   | G2       | 2.40                | 0.001                            | 2400                        |
|                   | G3       | 2.40                | 0.001                            | 2400                        |
|                   | G4       | 2.40                | 0.001                            | 2400                        |

*Table 4.2: Density of Geopolymer Concrete*

## 4.4 COMPRESSIVE STRENGTH

Fly ash was used in the mix proportion to optimize the utilization of waste material that can be consumed by geopolymer concrete. Different percentage of sucrose and extra water was added to delay the setting time and increased the workability of geopolymer concrete. The results for each mix proportion are given in Figure 4.4 - Figure 4.9 below.

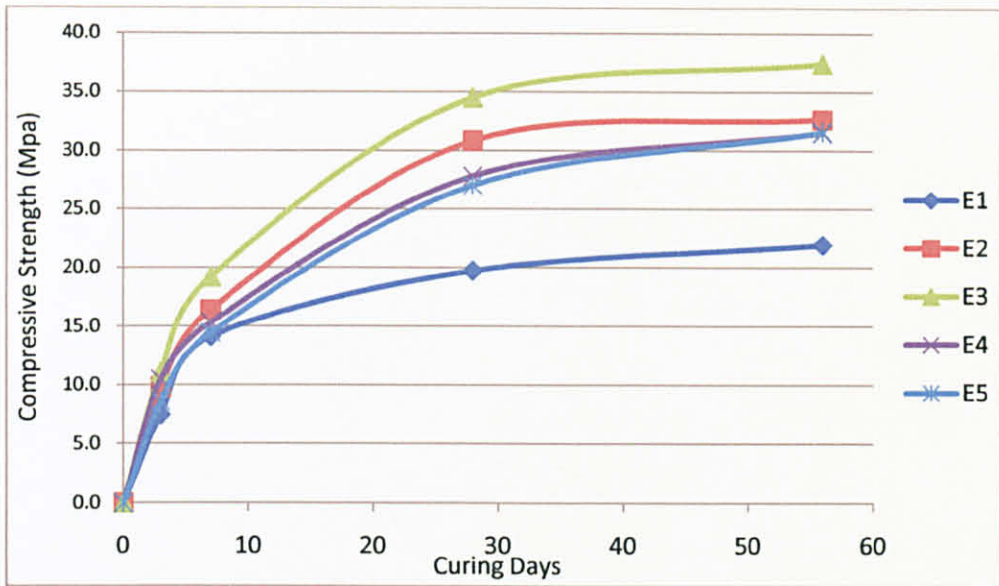


Figure 4.4: Compressive Strength in Ambient Curing

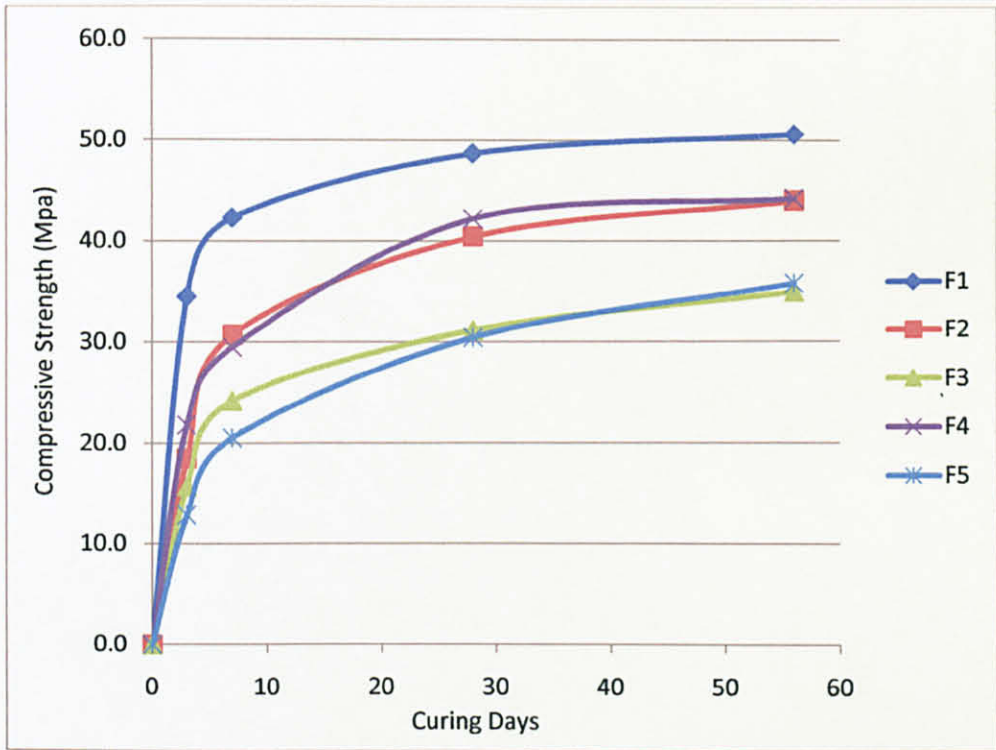
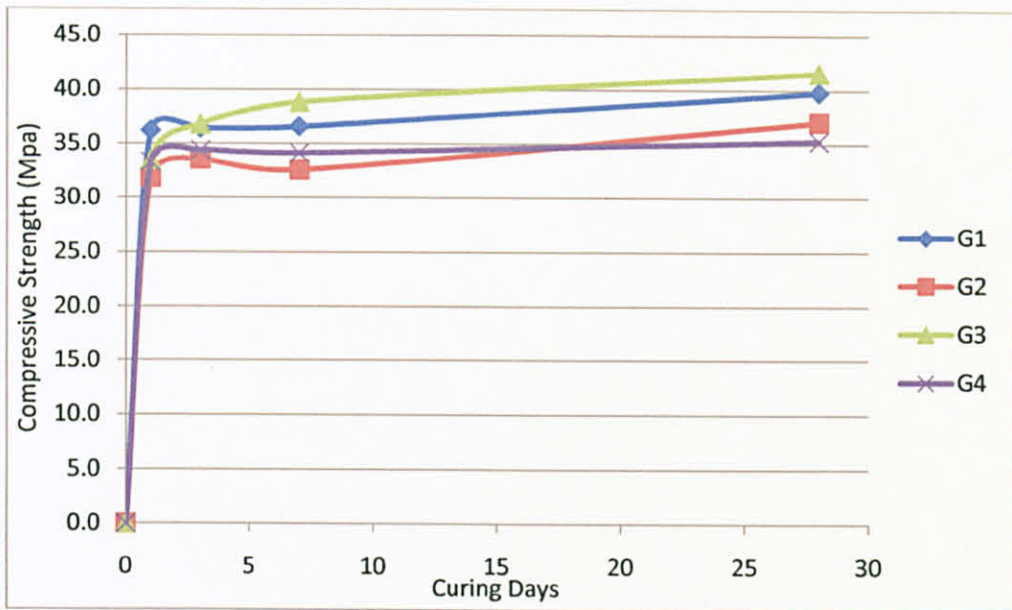


Figure 4.5: Compressive Strength in External Exposure Curing



*Figure 4.6: Compressive Strength in Oven Curing*

Figure 4.4 – Figure 4.6 shows the compressive strength obtained for each mix proportions at different curing regime. In ambient curing, the strength development of geopolymer concrete sample with 1% sucrose added are higher while in the external exposure curing, geopolymer concrete sample with 3% sucrose added show higher development strength than others. Geopolymer concrete sample cured with oven curing shows that sample with 0% sucrose added have higher development strength than others. It is because sucrose effects the polymeric reaction in ambient curing. However, in oven and external exposure curing the effects was reduced with elevated temperature.

Sucrose was added to increase the water viscosity so that water is not easily evaporated. In ambient curing, the higher amount of sucrose added increased the water viscosity of concrete sample hence prevent water from easily evaporated. Since the temperatures of ambient curing are low, so water still exist in the geopolymer concrete sample. The micelle bonding between geopolymer concrete particle are not strong due to the sample that still soft because too much water exist inside the sample hence the strength are reduced.



In external exposure curing, the higher amount of sucrose added will increase the strength of geopolymer concrete. Water can maintain the geopolymer concrete humidity due to high temperature of external exposure hence it prevent the sample from cracking. This resulted in higher strength development of geopolymer concrete sample cured with external exposure curing.

In oven curing, sample with 0% sucrose added show higher development strength than others. The water entrapped in geopolymer concrete sample will continuously evaporated since there is a constant temperature applied which is 65°C hence resulted in no significant difference of strength development between the sample cured with oven curing.

Naturally, after 28 days, the polymeric reaction in geopolymer concrete will be started to stable. In order to determine the optimum mix proportion of geopolymer concrete incorporating sucrose as an additive, the development strength of geopolymer concrete samples were observes until 28 days. Geopolymer concrete sample added with 1% sucrose was the optimum mix proportions for ambient curing, while in external exposure curing, the optimum mix proportion produced by geopolymer concrete sample with 3% sucrose added in the mixture.

Additional works of 3% sucrose mixture was added in the mix proportion of geopolymer concrete cured with external exposure and ambient curing. Different with oven curing that presented almost similar results among all its mixture, ambient and external exposure curing performed dissimilar trend in their results. 3% inclusion of sucrose in external exposure curing could increase concrete compressive strength up to 20.5% and 56.4% compared to 2% and 1% inclusion respectively, while if compared to control mixture with 0% addition of sucrose, 3% sucrose addition could improve geopolymer concrete strength up to 14.2%. As a comparison, 3% sucrose mixture in external exposure curing had compressive strength 27.25% higher than oven curing samples in average.

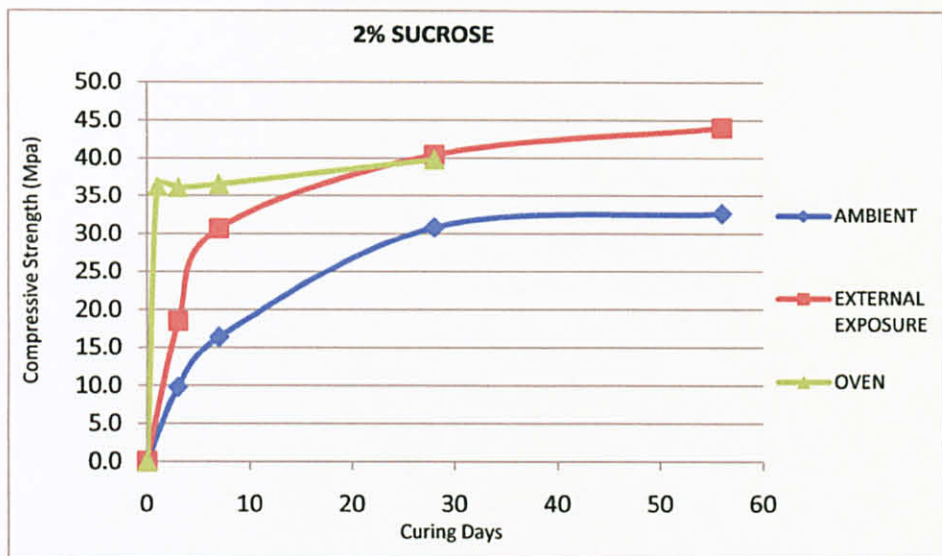


Figure 4.7: Compressive Strength with 2% Sucrose in Different Curing Regime

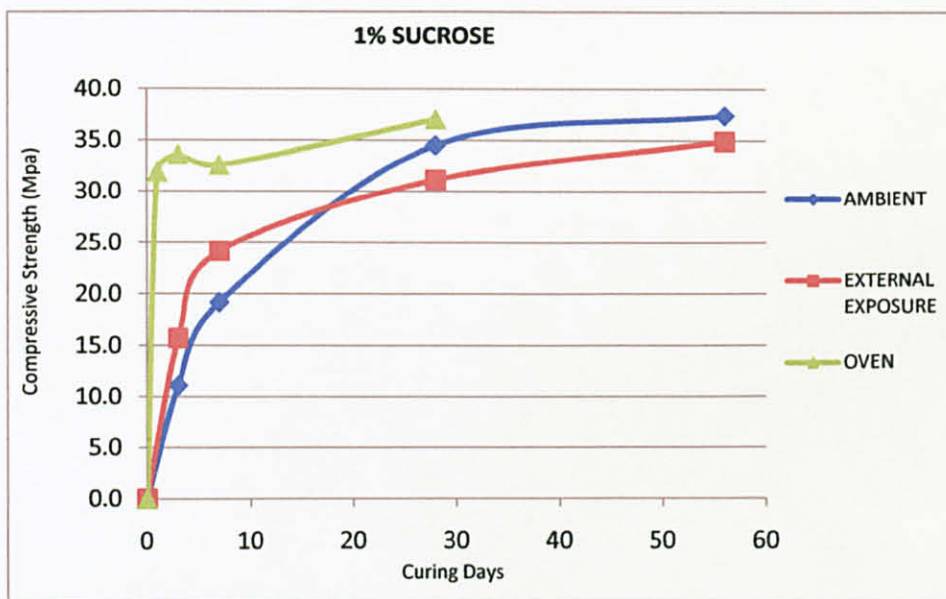


Figure 4.8: Compressive Strength with 1% Sucrose in Different Curing Regime

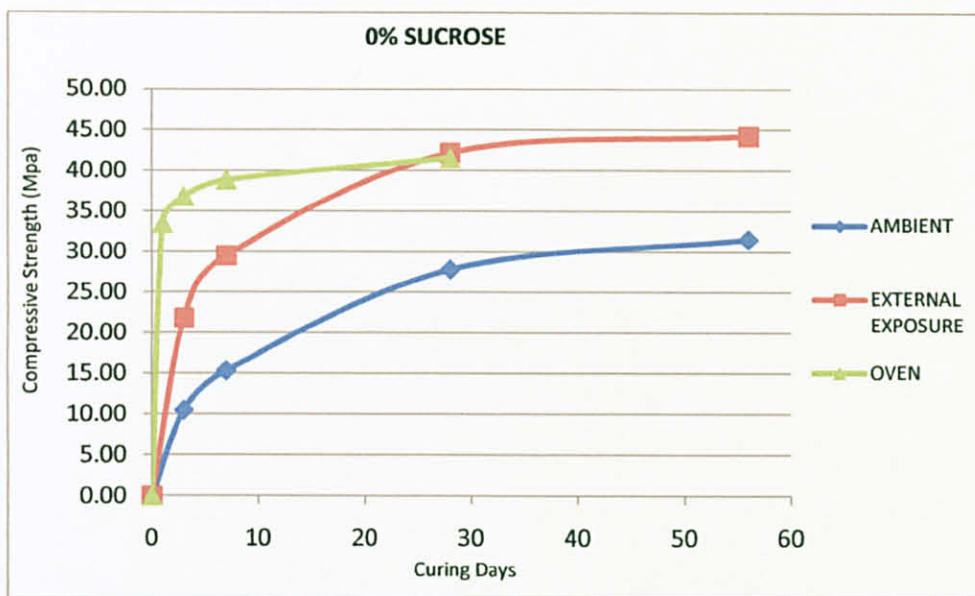


Figure 4.9: Compressive Strength with 0% Sucrose in Different Curing Regime

For geopolymer concrete sample with 2% sucrose added, external exposure curing increased the geopolymer concrete strength up to 1.56% compared to oven curing, while for geopolymer concrete sample with 0% sucrose added, external exposure curing increased the geopolymer concrete strength up to 7.9% compared to oven curing. The equipment effectiveness and availability to cure the concrete with oven has also limited the geopolymer concrete application to precast only and therefore not suitable for cast in-situ applications. Temperature in external exposure curing was not as high as oven temperature, but cyclic heating cooling by environment gives positive impact to the strength development of geopolymer concrete sample.



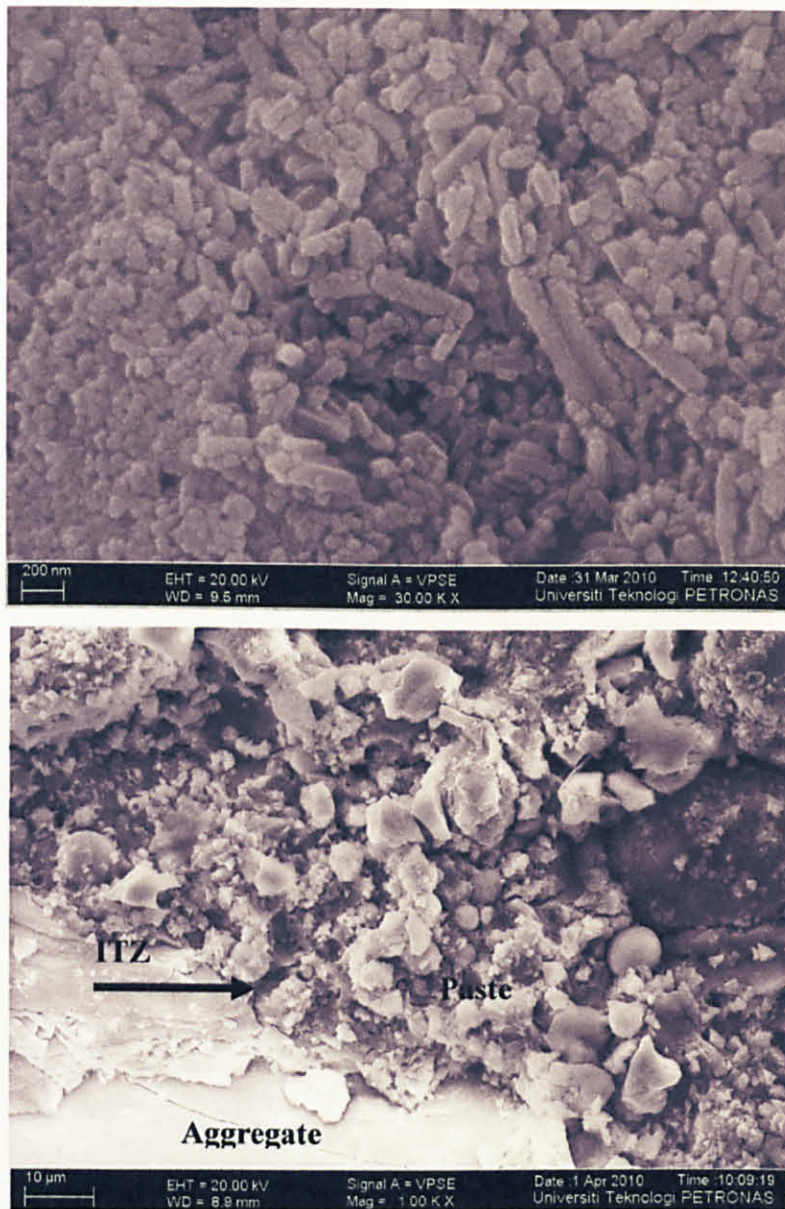
Figure 4.10: Inner Structure of Geopolymer Concrete Cured in (a) Ambient, (b) External Exposure, and (c) Oven Curing



As stated in the literature review, geopolymer concrete has excellent compressive strength and suffers very little drying shrinkage. Figure 4.10 also shows that there was no segregations occur in the geopolymer concrete sample during mixing and casting process.

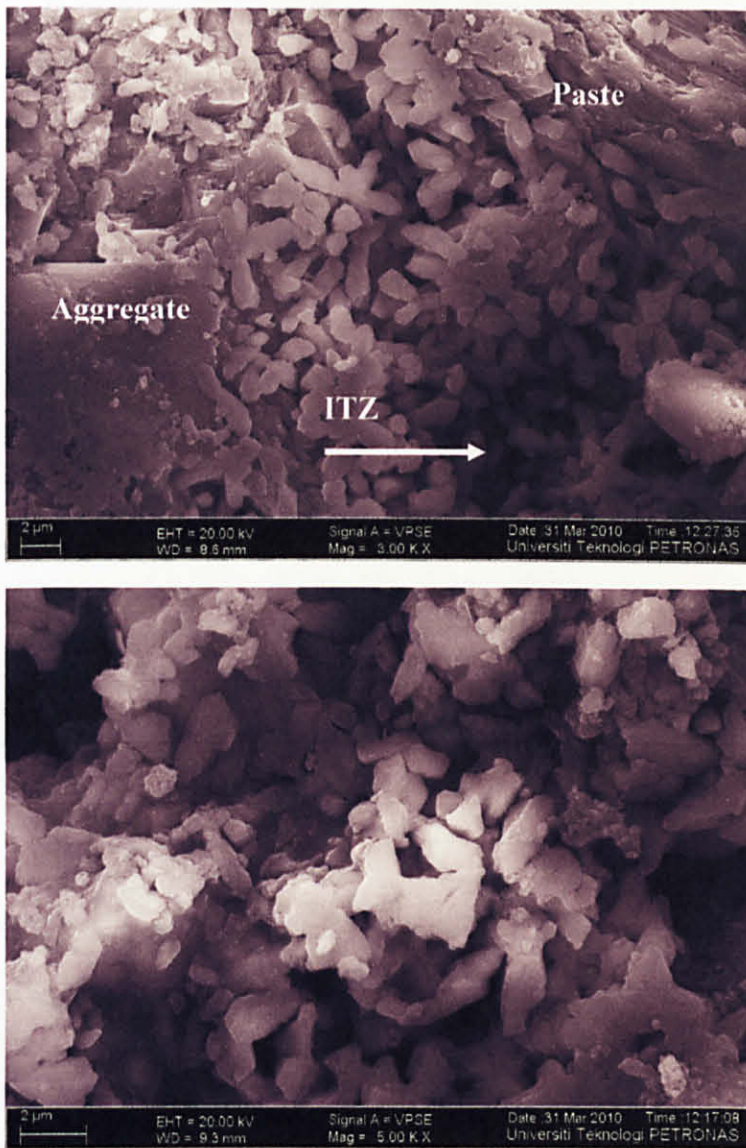
#### **4.5 FIELD EMISSIONS SCANNING ELECTRON MICROSCOPY (FESEM) ANALYSIS**

Field Emissions Scanning Electron Microscopy (FESEM) test was conducted on hardened geopolymer concrete sample with 56 days age in external exposure and oven curing. This test was performed to observe the relationship on Interfacial Transition Zone (ITZ) characteristics and microstructure properties in geopolymer concrete.



*Figure 4.11: FESEM image of geopolymer concrete with external exposure curing (above) nanostructure image and (below) microstructure image*

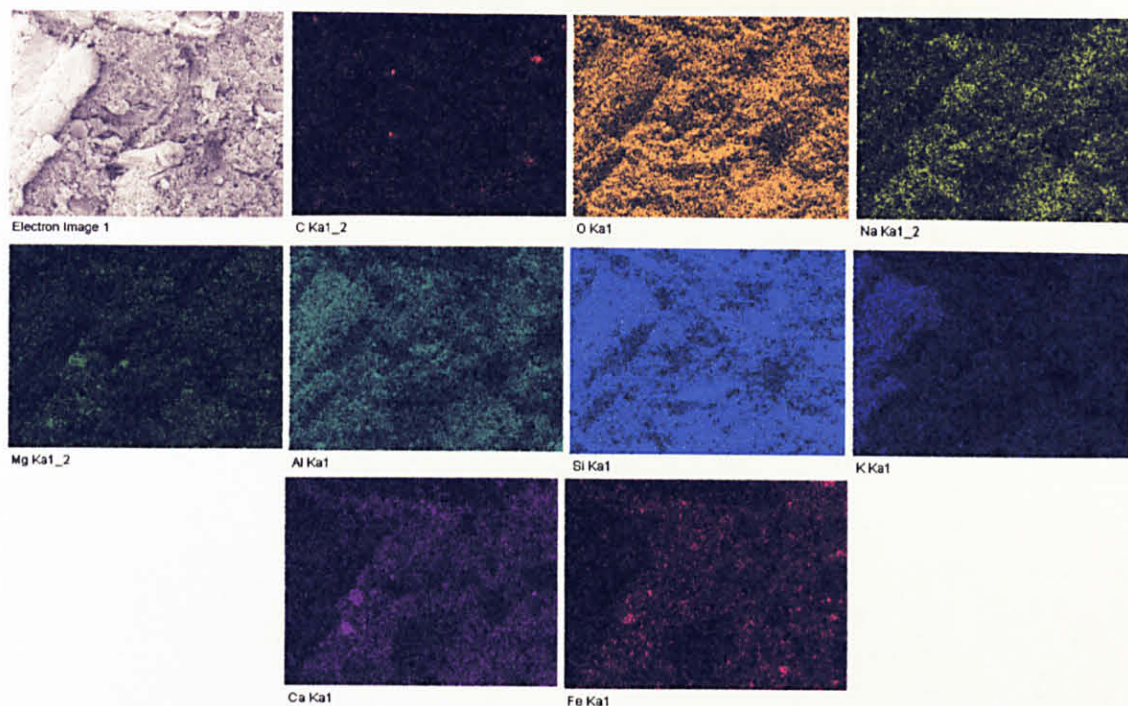
The image shows a nanostructure of geopolymer concrete matrix, which called as *micelle*. The microstructure image shows an ITZ boundary between aggregate and geopolymer paste produced from polymerization process. This paste covered some areas of ITZ in these sample hence resulted in higher compressive strength.



*Figure 4.12: FESEM image of geopolymer concrete with oven curing*

Figure 4.12 shows the presence of geopolymer micelles in occupying the ITZ to provide better performance to the geopolymer concrete. It is also showed that unlike Portland cement with its ettringite formation, geopolymer concrete microstructure shaped more like elliptical crystals.





*Figure 4.13: Image Mapping of Geopolymer Concrete Constituent*

From Figure 4.13, it can be observed that the distribution of SI elements is well distributed, not accumulated at one spot. It indicates that all geopolymeric materials were properly reacted. Low detection of Ca elements in the sample's image proved the utilization of low calcium fly ash. Since the alkaline solution used to assist the geopolymerization process are sodium (Na) based, the presence of Na was detected at the geopolymer matrix, while potassium (K) normally occurred at aggregate.

## CHAPTER 5

### CONCLUSION AND RECOMMENDATION

#### 5.1 CONCLUSION

Production of Ordinary Portland Cement (OPC) created environmental problems such as depletion of cement raw material and CO<sub>2</sub> emission to the atmosphere due to production of cement. Geopolymer concrete produced is to replace the use of OPC concrete as main construction material around the world. Currently geopolymer concrete are limited to precast application due to high temperature requirement in curing process. Several series of test were performed on fresh and hardened geopolymer concrete to obtain the optimum mix proportion and suitable curing regime for cast in-situ application. Based on the experimental result reported in the paper, it can be concluded that geopolymer concrete incorporating sucrose as an additives showed higher compressive strength than geopolymer concrete without sucrose because sucrose added can increased the water viscosity and prevent water from evaporated easily. Water inside geopolymer concrete can maintain its humidity hence prevent the sample from cracking. External exposure curing is the best curing method in the development of geopolymer concrete for cast in-situ application. Geopolymer concrete incorporating 3% sucrose is the optimum mix proportion for geopolymer concrete cured in external exposure condition. The compressive strength of geopolymer concrete incorporating 3% sucrose as an additive could reach up to 50.6 Mpa, while the appearance of ITZ was small in terms of gap size. ITZ was effectively covered by geopolymer *micelles*, hence increasing the compressive strength performance of geopolymer concrete.

## **5.2 RECOMMENDATION**

This research can be further improved through several recommendations for the future study. The amount of sucrose used as a natural retarder should be in wider range to study its effect on geopolymer concrete in terms of compressive strength because in this research the amount of sucrose used was up to 3% only due to time limitation. Test on mechanical properties can also be expanded to durability test (porosity, permeability, etc) and structural characteristics (flexural strength, push out and pull out strength, etc). The molarity of sodium hydroxide solution needs to be varies to study its effect on strength of geopolymer concrete.



## CHAPTER 6

### ECONOMIC BENEFITS

In this chapter, the cost for conducted the research are discussed and presented. The total cost of the research consists from the use of raw material to produce the product. Raw materials involved are fly ash, coarse aggregate, fine aggregate, NaOH pellets,  $\text{Na}_2\text{SiO}_3$  and sucrose.

#### 6.1 Cost of The Research

Table 6.1 shows the total raw material used during conduct this research.

| Mix Code | Fly Ash Content (kg) | Coarse Aggregate (kg) | Fine Aggregate (kg) | NaOH solution (kg) | NaSiO <sub>2</sub> solution (kg) | Extra water (kg) | Sucrose % | Sucrose (kg) |
|----------|----------------------|-----------------------|---------------------|--------------------|----------------------------------|------------------|-----------|--------------|
| E1       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.66             | 3         | 0.2          |
| E2       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.66             | 2         | 0.13         |
| E3       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.66             | 1         | 0.07         |
| E4       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.66             | 0         | 0.00         |
| E5       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.98             | 0         | 0.00         |
| F1       | 0.00                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.00             | 3         | 0.20         |
| F2       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.66             | 2         | 0.13         |
| F3       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.66             | 1         | 0.07         |
| F4       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.66             | 0         | 0.00         |
| F5       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.98             | 0         | 0.00         |
| G1       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.66             | 2         | 0.13         |
| G2       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.66             | 1         | 0.07         |
| G3       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.66             | 0         | 0.00         |
| G4       | 6.56                 | 22.50                 | 12.09               | 0.77               | 1.93                             | 0.98             | 0         | 0.00         |

*Table 6.1 : Total raw materials used*

The total cost of this prototype/research was RM 105.65 with details as follow :

| Materials                        | Quantity<br>(kg) | Unit price<br>(RM/kg) | Total<br>(RM) |
|----------------------------------|------------------|-----------------------|---------------|
| Fly Ash                          | 91.84            | -                     | -             |
| Coarse Aggregate                 | 315              | RM 0.18               | RM 56.70      |
| Fine Aggregate                   | 169.26           | RM 0.04               | RM 6.80       |
| NaOH pellets                     | 10.78            | RM 0.75               | RM 8.10       |
| Na <sub>2</sub> SiO <sub>3</sub> | 27.02            | RM 1.20               | RM 32.40      |
| Sucrose                          | 1                | RM 1.65               | RM 1.65       |
| TOTAL                            |                  |                       | RM 105.65     |

*Table 6.2 : Total cost*

This research project also has been awarded an e-science grant from Ministry of Science, Technology and Innovation (MOSTI).

This research has been beneficial to several industries for instance Chemical Industry and Tenaga Nasional Berhad (TNB). TNB has allocated some extra funding to decompose fly ash produced, so by our consumption can help to save the TNB budget annually. These products give many benefits especially in environmental perspectives by minimize the emission of carbon footprint. The productions of this product completely eliminated the use of OPC and utilize the use of industrial by-product which is fly ash. The product was applicable to cast in situ and precast with the strength was comparable to conventional concrete.

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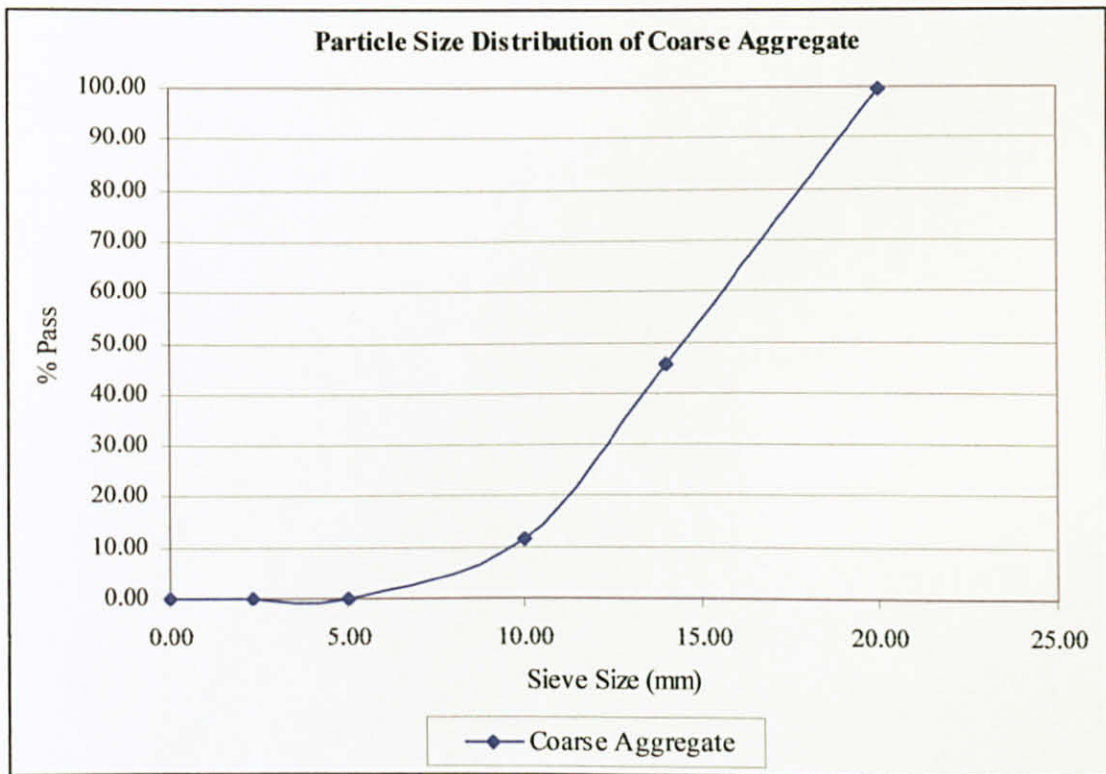
## APPENDICES

### APPENDIX I : SIEVE ANALYSIS OF COARSE AND FINE AGGREGATES

#### Sieve Analysis Results of Coarse Aggregate

| Sieve Size (mm) | Mass Retained (g) | % Mass Retained | $\Sigma$ % Mass Retained | % Finer |
|-----------------|-------------------|-----------------|--------------------------|---------|
| 20.00           | 11                | 0.50            | 0.50                     | 99.50   |
| 14.00           | 1078              | 54.00           | 54.50                    | 45.50   |
| 10.00           | 682               | 34.13           | 88.63                    | 11.37   |
| 5.00            | 224               | 11.21           | 99.84                    | 0.16    |
| 2.36            | 1                 | 0.05            | 99.89                    | 0.11    |
| 0               | 2                 | 0.10            | 99.99                    | 0.01    |

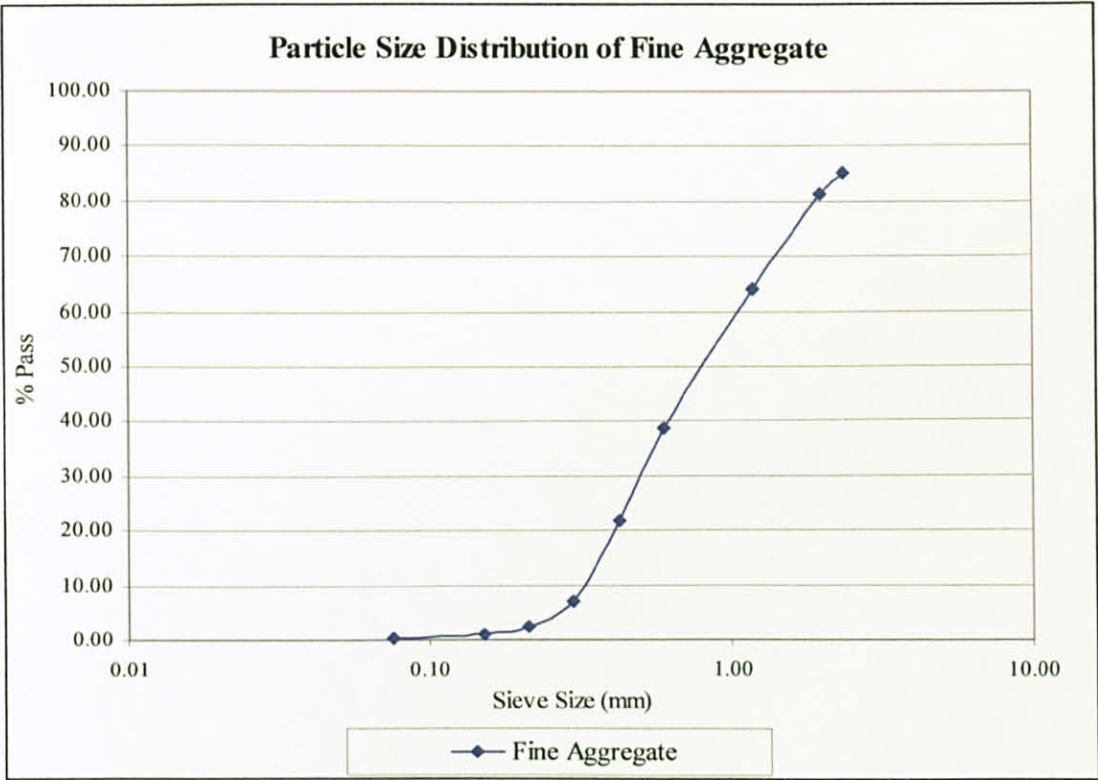
#### Particle Size Distribution Chart of Coarse Aggregate



Sieve Analysis Results of Fine Aggregate

| Sieve Size (mm) | Mass Retained (g) | % Mass Retained | $\Sigma$ % Mass Retained | % Finer |
|-----------------|-------------------|-----------------|--------------------------|---------|
| 2.36            | 74                | 14.80           | 14.8                     | 85.20   |
| 2.00            | 20                | 4.00            | 18.80                    | 81.20   |
| 1.18            | 86                | 17.20           | 36.00                    | 64.00   |
| 0.60            | 127               | 25.40           | 61.40                    | 38.60   |
| 0.43            | 84                | 16.80           | 78.20                    | 21.80   |
| 0.30            | 74                | 14.80           | 93.00                    | 7.00    |
| 0.21            | 22                | 4.40            | 97.40                    | 2.60    |
| 0.15            | 8                 | 1.60            | 99.00                    | 1.00    |
| 0.08            | 4                 | 0.80            | 99.80                    | 0.20    |
| 0.00            | 1                 | 0.20            | 100.00                   | 0.00    |

Particle Size Distribution Chart of Coarse Aggregate





**APPENDIX II : UPV VALUE**

| Mix Code | 1     | 3     | 7     | 28    | 56    |
|----------|-------|-------|-------|-------|-------|
|          | Days  |       |       |       |       |
| E1       | n/a   | 35.00 | 35.50 | 32.83 | 28.67 |
| E2       | n/a   | 31.33 | 32.50 | 29.33 | 28.50 |
| E3       | n/a   | 31.17 | 32.20 | 29.33 | 28.25 |
| E4       | n/a   | 33.50 | 33.00 | 28.38 | 31.00 |
| E5       | n/a   | 33.67 | 32.83 | 32.17 | 32.50 |
| F1       | n/a   | 28.67 | 29.17 | 25.33 | 24.84 |
| F2       | n/a   | 30.58 | 30.87 | 25.55 | 24.53 |
| F3       | n/a   | 31.68 | 32.33 | 27.05 | 28.78 |
| F4       | n/a   | 29.40 | 31.68 | 27.10 | 25.90 |
| F5       | n/a   | 31.20 | n/a   | 26.87 | 26.18 |
| G1       | 28.50 | 28.00 | 27.83 | 26.75 | n/a   |
| G2       | 29.83 | 30.17 | 29.83 | 28.50 | n/a   |
| G3       | 27.50 | 29.83 | n/a   | 28.25 | n/a   |
| G4       | 26.67 | 28.33 | 28.00 | 28.25 | n/a   |

### APPENDIX III : COMPRESSIVE STRENGTH DATA

| Mix Code | 1     | 3     | 7     | 28    | 56    |
|----------|-------|-------|-------|-------|-------|
|          | Days  |       |       |       |       |
| E1       | n/a   | 7.46  | 14.11 | 19.73 | 21.92 |
| E2       | n/a   | 9.82  | 16.40 | 30.86 | 32.65 |
| E3       | n/a   | 11.10 | 19.20 | 34.52 | 37.42 |
| E4       | n/a   | 10.47 | 15.32 | 27.80 | 31.50 |
| E5       | n/a   | 8.58  | 14.44 | 27.03 | 31.57 |
| F1       | n/a   | 34.5  | 42.3  | 48.7  | 50.6  |
| F2       | n/a   | 18.47 | 30.70 | 40.43 | 44.00 |
| F3       | n/a   | 15.69 | 24.17 | 31.13 | 34.91 |
| F4       | n/a   | 21.80 | 29.49 | 42.19 | 44.23 |
| F5       | n/a   | 12.87 | 20.52 | 30.42 | 35.76 |
| G1       | 36.2  | 36.40 | 36.58 | 39.80 | n/a   |
| G2       | 31.86 | 33.56 | 32.57 | 37.03 | n/a   |
| G3       | 33.48 | 36.78 | 38.84 | 40.34 | n/a   |
| G4       | 33.31 | 34.39 | 34.14 | 35.30 | n/a   |