THE EFFECT OF VARIOUS FLY ASH CONTENT ON GEOPOLYMER CONCRETE WITH DIFFERENT CURING REGIME

NURUL SYAZWANI BINTI MOHAMED

CIVIL ENGINEERING
UNIVERSITI TEKNOLOGI PETRONAS
JUNE 2010

CERTIFICATION OF APPROVAL

The Effect of Various Fly Ash Content on Geopolymer Concrete with Different Curing Regime

By

Nurul Syazwani Binti Mohamed

A project dissertation submitted to the
Civil Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfillment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(CIVIL ENGINEERING)

Approved by,

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(AP. Ir. Dr. Muhd Fadhil bin Nuruddin)

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.

NURUL SYAZWANI BINTI MOHAMED

ASBTRACT

Environmental problems associated with cement production had long been recognized and concerns of reducing the impact from cement industry had arisen globally. Therefore, the urge to replace usage of cement in concrete with other materials has become the main focus in construction industry nowadays. Fly ash as a waste material resulted from combustion of coal in the electrical power plant, had also contributed to environmental problems due to abundance of fly ash that was disposed to the landfills. However, fly ash is a pozzolanic material that contain high amount of aluminium and silicon which has high potential to replace cement. In this research, Geopolymer Concrete had been developed by incorporating fly ash as the main binder and completely eliminated the used of cement. Various fly ash densities was used in this research which are 250, 300, 350, 400 and 450 kg/m^3, in order to identify the optimum proportion of fly ash in Geopolymer Concrete. The Geopolymer Concrete was cured under 3 different curing regimes which are ambient, external exposure and oven curing, to identify the effect of curing regime on concrete strength. The compressive strength were tested on 3, 7, 28 and 56 days for ambient and external exposure curing, while 1, 3, 7, and 28 days for oven curing. Besides, the inner properties of Geopolymer Concrete was also studied. The other materials used to develop this concrete are 8M sodium hydroxide, sodium silicate, aggregates and extra water. In the manufacturing process, all the solid components were dry mixed for 2.5 minutes and continued with wet mixed for another 1.5 minutes by added all the liquid components. The Geopolymer Concrete was manufactured by adopting the same equipment as OPC Concrete. It is concluded that the optimum mix proportion of fly ash in Geopolymer Concrete were recorded as 300, 350 and 400 kg/m^3 for ambient, external exposure and oven curing respectively. The results showed that curing regime had significant impact on concrete compressive strength. Oven curing concrete had the highest compressive strength compared to other concretes. The inner properties of Geopolymer Concrete was studied by conducting Field Emission Scanning Electron Microscopy (FESEM) Analysis. From the images of concrete obtained, the relation between the Interfacial Transition Zone (ITZ) with compressive strength was verified.

ACKNOWLEDGEMENT

First and foremost, praised be to Allah, the Most Merciful and the Most Beneficent, who grants the author to complete her research within the given time. The author would like to express her sincere thanks to her supervisor Associate Professor Ir. Dr. Muhd Fadhil bin Nuruddin, for his excellent guidance and support throughout this research.

The author would also like to thank her family namely her parents Mohamed bin Dollah and Pos Payatee binti Mohamed Amin for their endless love and support. Not to forget, the beloved siblings Nurul Ain binti Mohamed, Nurul Najwa binti Mohamed, Ahmad Farhan bin Mohamed, Ahmad Hazwan bin Mohamed and Nur Ainaa Marjan bin Mohamed for their love and inspiration. They contributed significantly to the author's studies.

Special thanks to PETRONAS who provide financial support for author to complete her studies. Special thanks are extended to the following people and institution for their help in conducting this research: Mr. Johan Ariff bin Mohamed and Mr. Muhammad Hafiz b Baharun@Baharuddin, Laboratory Technologist UTP Concrete Laboratory, for their assistance and material procurement, Manjung Power Station for providing fly ash, Andri Kusbiantoro, Postgraduate Student of Universiti Teknologi PETRONAS, for his continuous guidance and knowledge shared through out this research, Nadia Nadzirah binti Jamil, Adilah binti Mohd Zawawi, Nor Hafizah binti Tajudin and Ainil Husna binti Mohd Sharir, for their continuous help and encouragement.

Finally, the author would like to express her deepest gratitude to all parties who supported her throughout the study and to Universiti Teknologi PETRONAS for providing her a chance and financial support for this research.

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CHAPTER 1
INTRODUCTION

1. INTRODUCTION

In the ancient civilisation, there are enormous structures that have been build such as Pyramids, Coliseum, Basilica of Constantine and etc. The construction of these structures proved the existence of certain materials that can be use to bind stones into a bigger mass. Lime and gypsum mortar are used for the construction of Pyramids. The construction of Coliseum and Basilica of Constantine used slaked lime and volcanic ash as binder. The use of volcanic ash and slaked lime produced cement that was capable to harden under water. Many years later, natural cement was started to be used in structures. The natural cement was produced by burned the mixture of lime and clay. In 1824, Joseph Aspdin discovered the 'Portland Cement' which used limestone and clay. Until then, the Portland cement is used as the dominant cement in concrete production.

Nowadays, the rapid development of the countries in all around the world has become significant. This can be seen from the construction of numerous massive structures which are build to show the growth and prosperity of the country. For example in Malaysia itself, many massive structures have been constructed such as PETRONAS Twin Towers which was once the tallest tower in the world before being surpassed by Taipei 101. As the world continues to develop, the demand for concrete in the construction field is currently boosting.

Ordinary Portland Cement (OPC) is widely used in the concrete as primary binder. OPC is used to bind the aggregates in the concrete by undergo hydration process after addition of water. As the demand for concrete increase, the demand for OPC also increases. On the other hand, the manufacturing of OPC has caused environmental problem. This is due to the emission of greenhouse gas, which is carbon dioxide (CO_2), to the atmosphere. Malhotra, 2002 and McCaffrey, 2002 stated that, there are approximately 1.35 billion tonnes or 7% of greenhouse gas emission is contributed by cement industry [1, 2]. The production of CO_2 gas is estimated as 1 tonne for every tonne of cement produced [2, 3]. In the cement production processes, the CO_2 is emitted during the decomposition of limestone and from kiln fuel combustion. CO_2 is also produced by vehicles use as the transportation but it only contributes in small fraction compared to others. Besides, it also consume significant amount of natural resources such as limestone and use high energy during the production process. Therefore, this has become the major issue for cement industry and immediate action need to be established in order to protect and to conserve the environment.

In order to reduce the greenhouse effect due to cement industry, McCaffrey, 2002 has proposed three alternatives. The alternatives proposed are to decrease the amount of calcined material in cement, to decrease the amount of cement in concrete and to decrease the number of buildings constructed by using cement [2]. Regarding to Mehta, 2002, both short term efforts and long term efforts should be seriously considered to develop environmental friendly concrete. The short term efforts are; utilize fewer natural resources, consume less energy and minimize the CO_2 emission. While from the long term point of view, by lowering the rate of material consumption, the impact of unwanted environmental by-products can be reduced [4].

Environmental preservation has become a driving force on the research for new environmental friendly concrete to replace OPC concrete. In 1978, Davidovits has introduced the term 'geopolymer' as an alternative binder which exhibit cementitous properties. The geopolymer is produced by polymeric reaction of alkaline liquid with silicon and aluminium in source materials [5]. The source materials can be geological origin or by-product material which is rich in silicon and aluminium. Fly ash, silica fume, granulated blast furnace slag, rice hush ash and metakaolin are commonly used as source materials. In the production of Geopolymer Concrete, geopolymer act as primary binder and completely replaced OPC. Therefore, Geopolymer Concrete can reduce the dependence on OPC cement. Ultimately, this can reduced the CO_2 emission from the cement industries and about 80% of greenhouse gas emission from this industry can be reduced [6].

The production of Geopolymer Concrete not only reduces the greenhouse effect on environment but it also utilizes the waste material. This is because, most of the source material use in Geopolymer Concrete is waste material. For example, fly ash is a by-product from burning of coal in power station. As the number of world population increase, the demand for power supply is also increasing. Therefore, fly ash is abundantly available. It is estimated that around 600 million tons of fly ash are available world wide, but currently the consumption of fly ash in concrete is only about 10% [7].

1.1 Problem Statement

Concrete is an essential construction material which are composed of binder, aggregate, water and admixtures. The aggregates used in concrete are gravel, limestone and granite as coarse aggregate and sand as fine aggregate. For conventional concrete, Ordinary Portland Cement (OPC) is commonly used as binder. When water is added to OPC a chemical reaction occurs which is known as hydration process. This enables OPC to bind the aggregates together and forms a larger mass.

The production of OPC has caused environmental impacts in all stages of the process. The most significant impact is the emission of carbon dioxide (CO_2) gas. The production of one tonne of OPC emits approximately one tonne of CO_2 into the atmosphere. The other environmental problems associated with OPC production are the production of dust, noise and vibration. The dust can affect human respiration system and thus unhealthy to human health. While the noise and vibration is unpleasant especially to those neighboring residents of OPC plant.

Due to environmental problem, the concern to reduce the utilisation of OPC has arises. This lead to the usage of Cement Replacement Material (CRM), such as fly ash, silica fume, granulated blast furnace, rice-hush ash and etc. The CRM is used as a partial replacement of OPC content and therefore reduces the OPC content in concrete. When CRM is used with OPC in the presence of water and in ambient temperature, it will reacts with calcium hydroxide, $Ca(OH_2)$ which is the unwanted product from hydration process to form the calcium silicate hydrate (C-S-H) gel. This enables the CRM to exhibit the cementitious properties and help to bind the aggregates. This greatly improves the strength and durability of concrete. At the same time the utilization of CRM in concrete will reduces the material cost of concrete.

The advancement of concrete material is continues with the finding of Geopolymer Concrete by Davidovits in 1978. Compared to OPC Concrete, Geopolymer Concrete is more environmental friendly because it replaces cement with geopolymer which act as primary binder. Geopolymer is produced by activated polymerization of alumino-silicate oxide in fine particles with alkali metal solutions. The main material use for geopolymer production is alumino-silicate materials such as fly ash, rice-hush ash and etc, which are waste materials. Therefore, the use of Geopolymer technology not only reduces CO_2 emission, but also utilises the waste materials and thus reducing the environmental impact by reduce the number of waste material to be disposed off at the landfills.

Based on previous research on Geopolymer Concrete, it was found that the compressive strength of Geopolymer Concrete is substantially increase when it is cured in 60°C for 24-hour. This curing method was conducted in oven or steam chamber. Therefore this method is more applicable for pre-cast concrete production. There are limited previous researches that study the potential of Geopolymer Concrete for cast in-situ production. Besides, there are also few published literatures regarding the optimum amount of fly ash needed in Geopolymer Concrete to produce the maximum compressive strength and it only limited to certain curing temperature.

Therefore, this research is devoted to determine the compressive strength of Geopolymer Concrete by utilizing the different amount of low calcium fly ash content and ultimately find the optimum fly ash content in the concrete. At the same time, the concrete will be exposed to different type of curing which are ambient curing, oven curing and external exposure curing to study their effect on the concrete compressive strength.

1.2 Objective

The objectives of this study are:

i. To determine the optimum mix proportion of fly ash in Geopolymer Concrete.
ii. To identify the effect of curing regime on the compressive strength of Geopolymer Concrete.
iii. To ascertain the compressive strength (3, 7, 28 and 56 days) of Geopolymer Concrete.
iv. To identify the microstructure properties of Geopolymer Concrete.

1.3 Scope of Study

Low-calcium fly ash is used as the main material for production of Geopolymer Concrete. The fly ash used in this research is obtained from the same batch that is produced in Manjung Power Station at Perak. The equipment used to manufacture Geopolymer Concrete is the same as use in OPC concrete.

In this study, the content of fly ash used in the concrete are 250, 300, 350, 400 and 450 kg/m^3. For each of Geopolymer Concrete produced with respective fly ash content, there are 3 methods of curing are adopted which are ambient curing, external exposure curing and oven curing.

The tests conducted on Geopolymer Concrete are Compressive Strength Test, Ultrasonic Pulse Velocity (UPV) Test and Field Emission Scanning Electron Microscopy (FESEM) Analysis.

CHAPTER 2
LITERATURE REVIEW

2. LITERATURE REVIEW

This chapter presents the background that lead to the development of alternative binders to manufacture concrete. The available published literature on low calcium fly ash and Geopolymer Concrete is also briefly reviewed.

2.1 Concrete And Environment

Since the past few decades, concrete had became the most widely used construction material. As the world continues to growth, the demand for this material also increases. In 2002, Mc Caffrey estimated that demand of cement increasing by 3% annually [2]. On the other hand, huge demand for concrete has become serious concern among the countries in the world due to its negative impacts to the global environment.

In the concrete production, cement is mostly use as primary binder to hold all the aggregate together, thus form a larger and stronger material. Due to its high capability to hold the aggregates, it becomes the most popular binder among the concrete manufacturer. However, it is well known that cement production is contributing considerable amount of carbon dioxide (CO_2) gases to the atmosphere. Many researches have been done in the past to study the impact of cement production to the environment. From the researches, it was found that approximately 1 tonne of CO_2 gases is emitted for every tonne of cement produced [2, 3]. Roy, 1999 states that CO_2 is produced during the carbonation process of limestone in the kiln during the manufacturing of cement and the combustion of fossil fuels [8].

Currently, Ordinary Portland Cement (OPC) is the most popular type of cement uses in the concrete production. During the manufacturing process of OPC cement, approximately about 1.35 billion tonne of CO_2 gases is emit annually in the worldwide which is comprises of 7% of total greenhouse gas emissions to the earth's atmosphere [1].

As the time passed by, cement industry has become one of the major contributors of greenhouse effect by consistently emit CO_2 gas into the atmosphere. The most adverse impact of greenhouse effect is the global warming which causes increment in global temperature. Aside emitting CO_2 gases, it also emits dusts and other particles into the environment. Therefore, it also contributes to global dimming phenomena. Global dimming is a phenomenon which occurs due to the reduction of the amount of sunlight reach earth. This phenomenon is causes by the particles in air blocking the sunlight. Even though there are many efforts have been execute to reduce the effect of greenhouse gases. It was found that the global dimming phenomena can be reduced; however it will increase the effect of global warming [9].

Due to greenhouse effect, the concern to reduce the greenhouse gas emission is increased. Therefore in 1997, Kyoto Protocol to the United Nations Framework Convention on Climate Change had been established which aimed at combating global warming. The Kyoto Protocol is a legal agreement under which industrialised countries will reduce their collective emissions of greenhouses gases by 5.2% compared to the year 1990. The main goal of this treaty is to reduce the overall emissions from six greenhouse gases which are carbon dioxide, methane, nitrous oxide, sulphur hexafluoride, HFCs and PFCs. National targets range from 8% reductions for the European Union and some others to 7% for the US, 6% for Japan, 0% for Russia, and permitted increases of 8% for Australia and 10% for Iceland. This target must be achieved by period 2008-2012 [10].

2.2 Fly Ash

American Concrete Institute (ACI) Committee 116R define fly ash as 'the finely divided residue that results from the combustion of ground or powdered coal and that is transported by flue gasses from the combustion zone to the particle removed system' [11]. Fly ash is a by-product from burning pulverized coal in electrical power generating plants. Fly ash is forms during the burning process of coal where the mineral impurities in the coal fuse and float in the combustion chamber. Then it cool down and solidifies into glassy spherical particles which are known as fly ash [12].

There are two types of fly ash commonly used in concrete which are Class C and Class F. Class C fly ash is known as high-calcium fly ash which produced from burning of sub-bituminous coal such as lignite. While the fly ash produced from burning of bituminous coal is known as low-calcium fly ash (Class F fly ash). Class C fly ash contained about 20% of calcium oxide (CaO) which is higher compared to Class F fly ash which contain less than 10% of CaO [13, 14].

Fly ash powder does resemble OPC cement but its particles size is finer than OPC and lime which ranging between 1μm to 150μm. The chemical composition of fly ash is also different from OPC cement. Generally, fly ash mainly composes of silicon oxide (SiO), aluminium (Al_2O_3), iron (Fe_2O_3) and calcium (CaO). Magnesium, potassium, sodium, titanium, and sulfur are also present in fly ash but only in small amount. The combustion of sub-bituminous coal contains more calcium and iron than fly ash from bituminous coal. There are different types of coal with different chemical composition. Therefore, the chemical composition of fly ash is varies depended on the type and amount of incombustible matter in the coal [13].

Fly ash has been produced in all around the world. Therefore, the availability of fly ash is abundant. In 2007, U.S. produced about 70 million tonnes of fly ash. Nearly 45 percent was recycled and the rest was disposed to landfill. There are about 40% of recycled fly ash was used in concrete industry. Fly ash also is reused in other fields such as in highway, embankment and etc [15]. While in Australia, the total fly ash production is approximately 12.5 million tonnes in 2007. From that amount, only 2.1 million tonnes is reused regardless whether the ash is sold or used internally by the ash producer [16].

By incorporating fly ash in concrete production, the environmental impact can significantly be reduced by decreasing the amount of cement. Therefore, this will reduced the greenhouse gas production from concrete industry and also reduced the energy consumption. Other than that, it also can preserve the natural resources since cement production consumes many natural resources such as limestone. The usage of fly ash in concrete also can reduce the amount of fly ash that must be disposed in landfills.

2.3 Fly Ash as Cement Replacement Material in Concrete

Due to concern of greenhouse gases effect, the demands to reduce the CO_2 emission in the concrete industry has arise. It is impossible to eliminate concrete in construction field because it is the main material. Therefore, many efforts had been done in the past in order to produce more environmental friendly concrete. One of the efforts taken is to reduce the amount of cement in concrete and replaced with other material which is known as Cement Replacement Material (CRM).

Fly ash is one of the most popular CRM incorporated in concrete production. Fly ash is suitable to be use as CRM because it is a pozzolan. Pozzolan is a material which exhibits cementitous properties when combine with calcium hydroxide. In concrete production, cement will react with water during the hydration process and produce calcium-silicate-hydrate (C-S-H) gel and calcium hydroxide. Then, fly ash will react with calcium hydroxide and produced more C-S-H gel. The calcium hydroxide is undesired in concrete because it can cause many problems associated with concrete durability. Since fly ash consumes calcium hydroxide in concrete, it improves the durability of the concrete. Besides improved concrete durability, it also can improve concrete workability. The spherical shape of fly ash helps to improved workability. While the small particle size of fly ash plays as filler of voids in concrete, hence produce dense and durable concrete.

Malhotra and Mehta define high volume fly ash (HVFA) concrete as concrete that contain 50% or more cement replacement material. The concrete exhibit 50% or higher CRM is possible to produce sustainable and high performance concrete that have high workability, high ultimate strength and high durability [7]. In 2002, Malhotra had successful to develop HVFA concrete that utilized up to 60 % of fly ash, and yet possessed excellent mechanical properties with enhanced durability performance. The test results showed that HVFA concrete is more durable than Portland cement concrete [17].

2.4 Geopolymer

Davidovits introduced the term 'Geopolymer' in 1978 to represent the inorganic polymers resulted from polymeric reaction of alumina-silicate material with alkaline solution. Geopolymer is synthesized from predominantly silicon (Si) and aluminium (Al) material of geological origin or by-product materials. The chemical composition of geopolymer materials is similar to zeolite, but they revealed an amorphous microstructure [5]. During the synthesized process, silicon and aluminium atoms are combined to form building of blocks that are chemically and structurally comparable to the conventional binder in concrete.

The research done by Davidovits showed that, the polymerization process occurred in Geopolymer Concrete produced three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds, as follow [5]:

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

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 polymerization; 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) [5, 18]:

$$\underset{\text{(Si-Al materials)}}{n(Si_2O_5.Al_2O_2)}+2nSiO_2+4nH_2O+NaOH \text{ or } KOH \rightarrow Na^+,K^+ + \underset{\text{(Geopolymer precursor)}}{n(OH)_3\text{-}Si\text{-}O\text{-}\underset{\displaystyle (OH)_2}{\underset{|}{Al^-}}\text{-}O\text{-}Si\text{-}(OH)_3} \quad (2\text{-}2)$$

$$n(OH)_3\text{-}Si\text{-}O\text{-}\underset{\displaystyle (OH)_2}{\underset{|}{Al^-}}\text{-}O\text{-}Si\text{-}(OH)_3 + NaOH \text{ or } KOH \rightarrow \underset{\text{(Geopolymer backbone)}}{(Na+,K+)\text{-}(\text{-}\underset{\underset{|}{O}}{\underset{|}{\overset{|}{Si}}}\text{-}O\text{-}\underset{\underset{|}{O}}{\underset{|}{\overset{|}{Al^-}}}\text{-}O\text{-}\underset{\underset{|}{O}}{\underset{|}{\overset{|}{Si}}}\text{-}O\text{-})} + 4nH_2O \quad (2\text{-}3)$$

Davidovits stated that there are 3 processes involved in formation of geopolymer concrete [5, 19]:

- Dissolution of Si and Al atoms from the source material through the action of hydroxide ions
- Condensation of precursor ions into monomers
- Polymerization of monomer into polymer structures

However, it is difficult to evaluate each process individually because they can overlap with each other and occurred almost instantaneously [20].

In the Geopolymer Concrete, water played no role in chemical reaction. This is in contrast with conventional concrete where water played an important role in hydration process and also influenced the concrete strength. Even thought water do not contributed in chemical reaction in Geopolymer Concrete, it provided workability to the mixture during the handling process. Water will be expelled from the concrete during the curing period (refer to equation 2-3).

The Geopolymer can be use in various applications depending on the molar ratio of Si to Al. Table 2.1 shows the possible application of Geopolymer as proposed by Davidovits[5].

Table 2.1: Application of Geopolymers

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

2.4.1 Fly Ash as Source Materials

Fly ash is one of the by-product materials that contain high silicon (Si) and aluminium (Al). There are many researches have been conducted to investigate the suitability of fly ash as binder in Geopolymer Concrete.

In order to determine which type of fly ash produced higher compressive strength, van Jaarsveld et al, 1999 had carried out an experiment by utilizing both type of fly ash. From the research, it was found that Class C fly ash produced higher compressive strength than Class F fly ash. The researchers claimed that high content of CaO helped to reduce the porosity of concrete and improved compressive strength [21].

On the other hand, Gourley found that calcium content in concrete can significantly affect polymerization process and also can altered the microstructure. Therefore, the researcher claimed that low calcium fly ash is more preferred to be used as source material in Geopolymer Concrete [22].

Besides, Fernández-Jimenez and Palomo (2003) also found that low calcium fly ash is more suitable than high calcium fly ash. These researchers claimed that to produce optimal binding properties, the low-calcium fly ash should have the percentage of unburned material (LOI) less than 5%, Fe_2O_3 content should not exceed 10%, and low CaO content, reactive silica content should be between 40-50%, and 80-90% of particles should be smaller than 45 μm [23].

The calcined source materials such as fly ash, slag and calcined kaolin demonstrated high compressive strength compared with using non-calcined material such as kaolin clay, mine tailings and geological origin material [24]. However, the combination of calcined and non-calcined material produced higher compressive strength compared to the usage of single source material [25].

The other characteristics that influenced the suitability of fly ash to be source material for geopolymers are the particle size, amorphous content and the origin of fly ash.

2.4.2 Alkaline Liquids

In the past researches, the combination of hydroxide solution and silicate solution were commonly used as alkaline liquid in Geopolymer Concrete production. The hydroxide solution used either sodium hydroxide (NaOH) or potassium hydroxide (KOH). While the silicate solution used were either sodium silicate ($NaSiO_2$) or potassium silicate. Among the research that have been conducted using those alkaline solutions are Davidovits 1999 [5]; Palomo et al. 1999 [20]; Barbosa et al. 2000 [24]; Xu and van Deventer 2000 [19]; Swanepoel and Strydom 2002 [26]; Xu and van Deventer 2002[25].

There were some researches that use single alkaline solution such as reported by Palomo et al. and Teixeira-Pinto et al [20, 27]. Based on the research, Palomo et al concluded that the type of alkaline liquid played an important role in the polymerization process. High reaction rate occurred when using combination of hydroxide solution and silicate solution compared to the use of only alkaline hydroxides. Xu and van Deventer (2000) confirmed that the addition of sodium silicate solution to the sodium hydroxide solution as the alkaline liquid enhanced the reaction between the source material and the solution. Furthermore, after a study of the polymerization of sixteen natural Al-Si minerals, researchers found that generally the NaOH solution caused a higher extent of dissolution of minerals than the KOH solution [19].

2.4.3 Mixture Proportions

The mix proportions for Geopolymer Concrete had been studied by many researchers in order to find the optimum mix proportion corresponding to the compressive strength. The compressive strength of Geopolymer Concrete depends on it constituent. Therefore, variation of mixture proportion of Geopolymer Concrete had been design and tested previously.

In 2005, Hardjito and Rangan studied the variation of mix proportion on compressive strength. From the research, the researches claim that the following factors contributed to high compressive strength [28]:

- Usage of high concentration of sodium hydroxide solution
- High ratio of sodium silicate-to-sodium hydroxide ratio (by mass)
- Low ratio of H_2O-to-Na_2O
- Low ratio of water-to-geopolymer solids (by mass)

In 1999, Palomo et al studied the geopolymerisation of low-calcium ASTM Class F fly ash (molar Si/Al=1.81) using four different solutions with the solution-to-fly ash ratio by mass of 0.25 to 0.30. The molar SiO_2/Na_2O of the solutions was in the range of 0.63 to 1.23. The best compressive strength obtained was more than 60 MPa for mixtures that used combination of sodium hydroxide and sodium silicate solution, after curing the specimens for 24 hours at 65°C [20].

Van Jaarsveld et al (1998) studied the ratio of alkaline solution to alumino- silicate powder of fly ash with kaolin or calcined kaolin as source material. The liquid component used by mass are 3.5% of sodium silicate, 20% of water and 4% of sodium hydroxide or potassium hydroxide. From the study, the researchers found that the optimum ratio of solution to alumino-silicate powder was 0.39 [29].

The same experiment was conducted by Xu and van Deventer (2000). But instead of using fly ash based material, the researchers used stilbite. From the experiment, the researchers claims that in order to allow polymeric reaction to occur, the proportion of alkaline liquid to alumino-silicate powder by mass is approximately 0.33 [19].

Based on previous work done by Davidovits (1982), Barbosa et al (2000) prepared seven mixture compositions of geopolymer paste by using kaolin as source material, for the following range of molar oxide ratios: $0.2<Na_2O/SiO_2<0.48$; $3.3<SiO_2/Al_2O_3<4.5$ and $10<H_2O/Na_2O<25$. From the tests performed on the paste specimens, they found that the optimum composition occurred when the ratio of Na_2O/SiO_2 was 0.25, the ratio of H_2O/Na_2O was 10.0, and the ratio of SiO_2/Al_2O_3 was 3.3. Mixtures with high water content, i.e. $H_2O/Na_2O = 25$, developed very low compressive strengths, and thus underlying the importance of water content in the mixture. There was no information regarding the size of the specimens, while the moulds used were of a thin polyethylene film [24].

2.4.4 Manufacturing Process

Most of the manufacturing process of making geopolymer paste involved the dry mixing of the source materials, followed by adding the alkaline solution and then further mixing for another specified period of time [26, 27 & 29].

2.4.5 Curing

Hardjito and Rangan (2005) found that, as the curing temperature is increase from 30°C to 90°C, the compressive strength also increase [28].

Palomo et al (1999) stated that curing temperature and curing time have been reported to play important roles in determining the properties of the geopolymer materials made from by-product material such as fly ash. The increase in curing temperature resulted in higher compressive strength [20].

CHAPTER 3
METHODOLOGY

3. METHODOLOGY

This chapter describes details on manufacturing process of Geopolymer Concrete that is adopted in this research. The material used in Geopolymer Concrete includes fly ash, alkaline liquids and aggregates are discussed. Besides, the detail of mix proportion used is also presented. The mixing process and curing process of Geopolymer Concrete is also included in this chapter.

In this research, the harden Geopolymer Concrete is tested for compressive strength, porosity and microstructure. The detail of the tests is briefly discussed in this chapter.

3.1 Materials

The materials used to produce Geopolymer Concrete are low calcium fly ash, sodium hydroxide solution and sodium silicate solution as alkaline liquid, coarse aggregate, fine aggregate and water.

3.1.1 Fly Ash

Low calcium fly ash (ASTM Class F) is incorporated in this research as primary binder. This binder is obtained from Manjung Power Station, Perak, Malaysia. The different batch of fly ash produced in this plant will have different distribution of chemical content which is depends on many factors such as combustion rates, type of coal and etc.

Therefore, the same batch of fly ash is utilized in all mix proportion to ensure same chemical distribution of chemical content in each concrete specimen.

The chemical distribution of fly ash is determined from X-Ray Fluorescence (XRF) Analysis, as given in Table 3.1. From the analysis, the results shows that the Fe_2O_3 content is 6.6%, SiO_2 content is 51.19% and loss on ignition (LOI) value is 6.1%. While, the calcium oxide (CaO) content is 5.57% that is lower than 10%. Malhotra and Ramezanianpour (1994) claimed that the low calcium fly ash must contain CaO lower than 10% [13].

Fernández-Jimenez and Palomo (2003) stated that, the binding property of Geopolymer Concrete is influenced by the chemical composition of low calcium fly ash. Therefore, in order to produce the optimum binding properties, the low calcium fly ash should have Fe_2O_3 lower than 10%, low CaO content, 40-50% of reactive silica content; and 5% or lower LOI value [23]. Even though the SiO_2 content and LOI value is slightly higher, it is still in acceptable limit. Thus, the fly ash used in this research will produce optimum binding properties of Geopolymer Concrete.

The fly ash also contain molar ratio of Si/Al about 2 which is same as proposed by Davidovit (1999) [5]. The colour of fly ash is dark brown.

Table 3.1: Composition of Fly Ash as Determine by XRF Analysis

Compounds	Percentages (%)
SiO_2	51.19
Al_2O_3	24
Fe_2O_3	6.6
CaO	5.57
MgO	2.4
SO_3	0.88
K_2O	1.14
Na_2O	2.12
LOI	6.1

3.1.2 Alkaline Liquid

The combination of sodium hydroxide (NaOH) solution and sodium silicate (Na_2SiO_3) solution were used as the alkaline liquid. In this research, sodium hydroxide was chosen instead of potassium hydroxide because it promotes higher dissolution of fly ash mineral. Besides, it is cheaper and easily available. The concentration of sodium hydroxide solution used is 8M. In order to produce 8M sodium hydroxide solution, 294 grams of NaOH pellet is diluted with 706 grams of water to produce 1 kilogram of NaOH solution. On the other hand, the chemical composition of sodium silicate solution is composed of 14.73% of Na_2O, 29.75% of SiO_2 and 55.52% of water. This solution is obtained from Malay-Sino Chemical Industries Sdn Bhd. The ratio of Na_2SiO_3 to NaOH used is 2.5 as proposed by Hardjito and Rangan (2005) [28].

3.1.3 Aggregate

The aggregates used in this research are similar to the aggregates used in conventional concrete. The coarse aggregate used is granite rock in crushed form, while fine aggregate is sand in uncrushed form. Both coarse and fine aggregates were prepared in saturated-surface-dry (SSD) condition before mixing process. This is to ensure that the aggregates will not absorb water that being added to the mixture. The particles size distribution of aggregates is determined by Sieve Analysis. The Sieve Analysis results of coarse aggregate and fine aggregate are shown in Table 3.2 and Table 3.3 respectively.

Table 3.2: Sieve Analysis Results of Coarse Aggregate

Sieve Size (mm)	Mass Retained (g)	% Mass Retained	Σ % 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

Table 3.3: Sieve Analysis Results of Fine Aggregate

Sieve Size (mm)	Mass Retained (g)	% Mass Retained	Σ % 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

3.1.4 Water

The purpose of adding water in Geopolymer Concrete is to enhance the workability of fresh concrete so that it will be easy to handle. Compared to OPC concrete, the presence of water in Geopolymer Concrete is not affecting its compressive strength. Eventually, water inside the concrete will be evaporated during the curing process. Therefore, this study utilized 15% of water from fly ash (by mass) to improve the concrete workability.

3.2 Mixture Proportion

The mixture proportion used in this research was developed based on the past research on geopolymer paste as discussed in Chapter 2. Therefore, the following proportions were selected for the Geopolymer Concrete mixtures.

- Low calcium (ASTM Class F) fly ash
  - The content of fly ash is varied: 250 kg/m^3, 300 kg/m^3, 350 kg/m^3, 400 kg/m^3 and 450 kg/m^3.
- Alkaline liquid
  - Combination of sodium hydroxide solution and sodium silicate solution.
  - Molarity of sodium hydroxide (NaOH) solution is 8M.
  - Ratio of sodium silicate solution-to-sodium hydroxide solution (by mass) is 2.5.

- Aggregate
  - Coarse and fine aggregates are approximately 74% to 81% of the entire mixture by mass. This value is almost similar to that used in OPC concrete (75% to 80% by mass).
- Extra water
  - Extra water was taken as 15% of fly ash (by mass)
- Curing method
  - 3 type of curing: Ambient curing, oven curing and external exposure curing

The detail of mix proportion used is given in Table 3.4.

Table 3.4: Mix Proportion of Geopolymer Concrete

Fly Ash Content (kg/m^3)	Coarse Aggregate (kg/m^3)	Fine Aggregate (kg/m^3)	NaOH solution (kg/m^3)	Na_2SiO_3 solution (kg/m^3)	Extra water (kg/m^3)	Curing
250	1200	645	41	103	37.5	Ambient
250	1200	645	41	103	37.5	External exposure
250	1200	645	41	103	37.5	Oven
300	1200	645	41	103	45.0	Ambient
300	1200	645	41	103	45.0	External exposure
300	1200	645	41	103	45.0	Oven
350	1200	645	41	103	52.5	Ambient
350	1200	645	41	103	52.5	External exposure
350	1200	645	41	103	52.5	Oven
400	1200	645	41	103	60.0	Ambient
400	1200	645	41	103	60.0	External exposure
400	1200	645	41	103	60.0	Oven
450	1200	645	41	103	67.5	Ambient
450	1200	645	41	103	67.5	External exposure
450	1200	645	41	103	67.5	Oven

3.3 Mixing, Casting and Curing

3.3.1 Mixing Process

Geopolymer Concrete can be manufactured by adopting the same equipments and techniques as in the manufacture of OPC concrete. For mixing process, a rotating pan mixer with fixed blades was used.

Before mixing process started, the solid materials and liquid materials was prepared. The solid components are consists of coarse aggregates, fine aggregates and fly ash. All the aggregates were prepared in saturated-surface dry (SSD) condition. While for liquid components, all the liquid (sodium hydroxide, sodium silicate and water) were mixed together before mixing process.

During the mixing process, all the solid components were mixed together for two and half minutes. This process is called dry mixed. Dry mixed is performed to ensure the aggregates are homogenously mixed with fly ash. After that, all the liquid components were added to the solid components and the mixing continued for another one and half minutes.

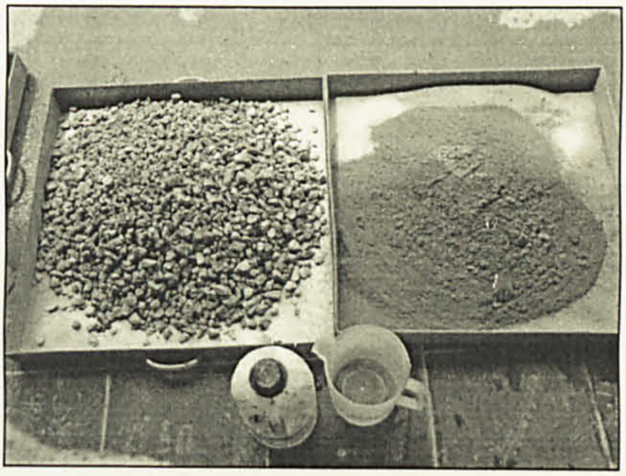


Figure 3.1: Materials for Geopolymer Concrete

3.3.2 Casting

After mixing process, the fresh Geopolymer Concrete was directly cast into 100x100x100 mm size moulds. 12 specimens were prepared for each mixed proportions. All the specimens were compacted by using the vibrating machine in order to eliminate air bubbles in the specimens. This is because the presence of air bubbles may reduce the strength of Geopolymer Concrete. At the same time, slump test was performed to measure the workability of Geopolymer Concrete. In the slump test, the Geopolymer Concrete was filled in three layers in which each layer was tamped 25 times using 16mm diameter steel rod.

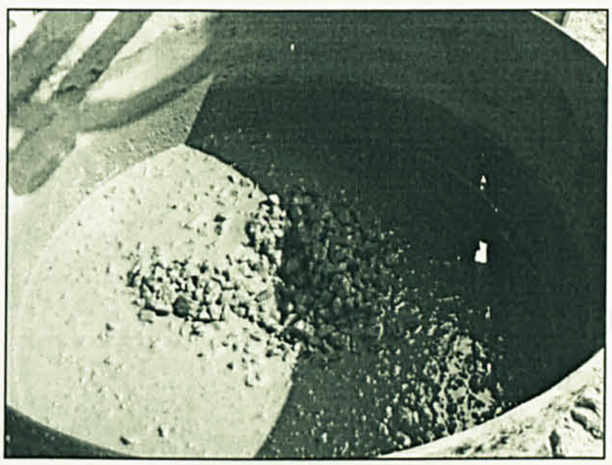


Figure 3.2: Fresh Geopolymer Concrete

Figure 3.3: Casting and Compaction process of Geopolymer Concrete

3.3.3 Curing

As mention previously, curing method is one of the important parameters in this research. There are three types of curing were adopted which are ambient curing, oven curing and external exposure curing.

For ambient curing, the concrete specimens were cured at ambient condition where the temperature is between 27°C to 32°C. While for external exposure curing, the specimens were put inside a chamber that was placed under direct sunlight. The temperature inside the chamber is varied between 33°C to 40°C. In this curing method, concrete was undergoing cyclic heating.

For oven curing, the specimens were left at ambient condition for 1 hour after casting. This is term as delay time. Then, the specimens were placed inside oven at 65°C for 24 hours. After 24 hours, the specimens were removed from oven and placed at ambient condition.

All the hardened concrete specimens were removed from moulds at 24 hours after casting. Then the specimens were placed depending on respective type of curing until the testing day.

Figure 3.4: Concrete specimens at ambient curing

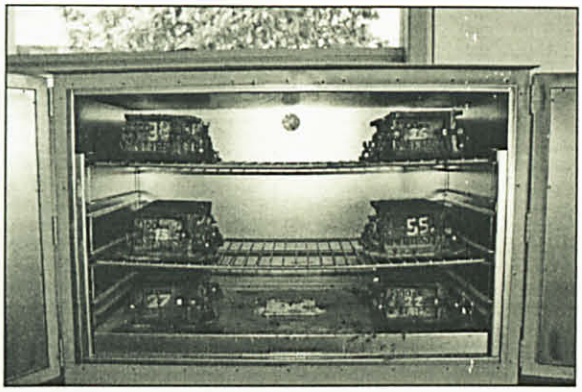


Figure 3.5: Concrete specimens at oven curing

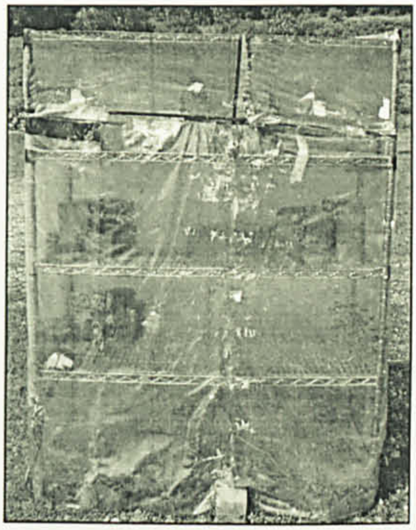


Figure 3.6: Concrete specimens at external exposure curing

3.4 Geopolymer Concrete Tests

Testing of concrete samples was conducted for fresh and hardened concrete. For fresh concrete, slump test was used to analyze the workability characteristic. While for hardened concrete, 2 methods of testing which are non-destructive and destructive test were carried out. Non-destructive method adopted is Ultrasonic Pulse Velocity (UPV) and destructive method used is Compressive Strength Test. The destructive test is conducted after non-destructive test was performed. The microstructure properties of Geopolymer Concrete was determined by Field Emission Scanning Electron Microscopy (FESEM) Analysis.

3.4.1 Compressive Strength Test

This test was conducted to determine the compressive strength of Geopolymer Concrete. The machine used to conduct this test is ELE ADR 3000 testing machine. The specimens were tested at 3-day, 7-day, 28-day and 56-day for ambient and external exposure curing. While for oven curing, the specimens where tested at 1-day, 3-day, 7-day and 28-day. 3 concrete specimens were tested for every compressive strength test conducted.

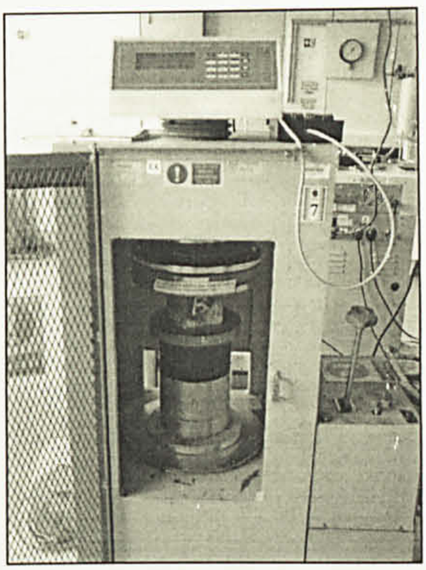


Figure 3.7: ELE ADR 3000 Testing Machine

3.4.2 Ultrasonic Pulse Velocity Test

Ultrasonic Pulse Velocity (UPV) test was conducted using Portable Ultrasonic Non Destructive Digital Indicative Tester (PUNDIT). In this test, the electro acoustical transmitter was placed on the opposite surface of longitudinal pulse receiver. The time taken for the pulse to travel from the transmitter to receiver was measured on three opposite surfaces for each cube. Pulse velocity that transmitted through the concrete will be used to analyze the development of concrete inner properties.

Figure 3.8: UPV PUNDIT Tester

3.4.3 Field Emission Scanning Electron Microscopy (FESEM) Analysis

The FESEM Analysis was carried out to study the relation between compressive strength with microstructure properties of Geopolymer Concrete. The microstructure of Geopolymer Concrete was analyzed at age 56-day. The machine used to perform this analysis is Supra 55VP Inca x-act Oxford FESEM. In this test, the concrete was placed in the vacuum chamber inside the FESEM machine. The FESEM is operated at specific pressure in order to facilitate the operation of filament and electron inside FESEM.

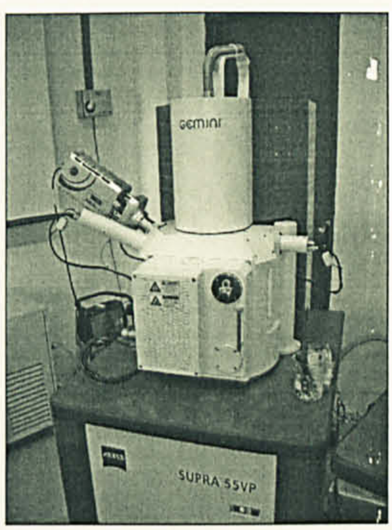


Figure 3.9: Supra 55VP Inca x-act Oxford FESEM Instrument

CHAPTER 4
RESULT AND DISCUSSION

4. RESULT AND DISCUSSION

In this chapter, the experimental results on fresh and hardened Geopolymer Concrete are presented and analyzed. The analyses involved are the properties of fresh Geopolymer Concrete, Ultrasonic Pulse Velocity (UPV) Test, Compressive Strength Test and Field Emission Scanning Electron Microscopy (FESEM) Analysis. Beside, the density of concrete also presented.

4.1 Properties of Fresh Geopolymer Concrete

The properties of fresh Geopolymer Concrete was measured based on its workability characteristics. In this experiment, slump test was used to measure the workability. The slump test was conducted for each fly ash content (250, 300, 350, 400 and 450 kg/m^3) of fresh concrete. Table 4.1 shows the workability characteristic for fresh concrete with different fly ash content.

Table 4.1: Workability Characteristic of Geopolymer Concrete

Fly Ash Content (kg/m^3)	Slump (mm)	Workability Characteristic
250	21.5	Workable but stiff
300	21.2	Workable but stiff
350	22.8	Workable
400	22.7	Workable
450	24.7	Highly Workable

From Table 4.1, the characteristic of fresh concrete with 250 and 300 kg/m^3 fly ash content was workable but a little bit stiff while for fly ash content of 350 and 400 kg/m^3, the concrete was workable. But, fresh concrete with 450 kg/m^3 fly ash content was highly workable. Therefore it shows that, the larger amount of fly ash incorporated in Geopolymer Concrete produce higher concrete workability. This is because, the amount of fly ash in this concrete is larger compared to other samples, so it contributes more fine particles in concrete. Therefore, its improved the concrete workability.

Figure 4.1: Slump test conducted during the experiment

4.2 Ultrasonic Pulse Velocity (UPV) Test

UPV Test was conducted to analyze the presence of voids in Geopolymer Concrete samples. Pulse was transmitted from transmitter and travelled through inner part of concrete to the receiver. The time (micro second) taken for the pulse to travel is recorded by PUNDIT machine and the distance for transmitter to receiver is 100mm which is the size of concrete sample. Therefore, the pulse velocity (V) can be calculated. Table 4.2 shows the entire UPV Test result.

Table 4.2: Ultrasonic Pulse Velocity Test Results of Geopolymer Concrete Samples

Curing	Code	Fly Ash Content (kg/m^3)	Velocity (km/s)			
			3 day	7 day	28 day	56 day
Ambient	D1	250	2.85	2.96	3.18	3.30
	C1	300	2.99	3.09	3.52	3.65
	E1	350	2.97	3.05	3.37	3.51
	A1	400	2.89	3.00	3.31	3.37
	B1	450	2.50	2.75	3.07	3.23
External Exposure	D2	250	2.96	3.05	3.26	3.35
	C2	300	3.07	3.25	3.63	3.72
	E2	350	3.15	3.27	3.71	3.82
	A2	400	3.00	3.17	3.45	3.54
	B2	450	2.81	2.93	3.15	3.31
			1 day	3 day	7 day	28 day
Oven	D3	250	3.09	3.30	3.53	3.73
	C3	300	3.34	3.45	3.58	3.83
	E3	350	3.51	3.57	3.67	3.97
	A3	400	4.00	4.07	4.15	4.25
	B3	450	3.02	3.24	3.46	3.68

Figure 4.2, 4.3, 4.4, 4.5 and 4.6 show the trend of UPV results for each fly ash content. Pulse velocity indicates the time taken for pulse to travel from transmitter to receiver. If pulse travels through many voids inside the concrete, the time taken is longer for the pulse to reach receiver. Therefore, it indicates that the pulse is travelling with slow velocity. The presence of voids can affect the concrete strength. This is because the voids can reduce the bonding between geopolymer pastes with aggregates. Hence, the ability of concrete to sustain heavy load is reduced.

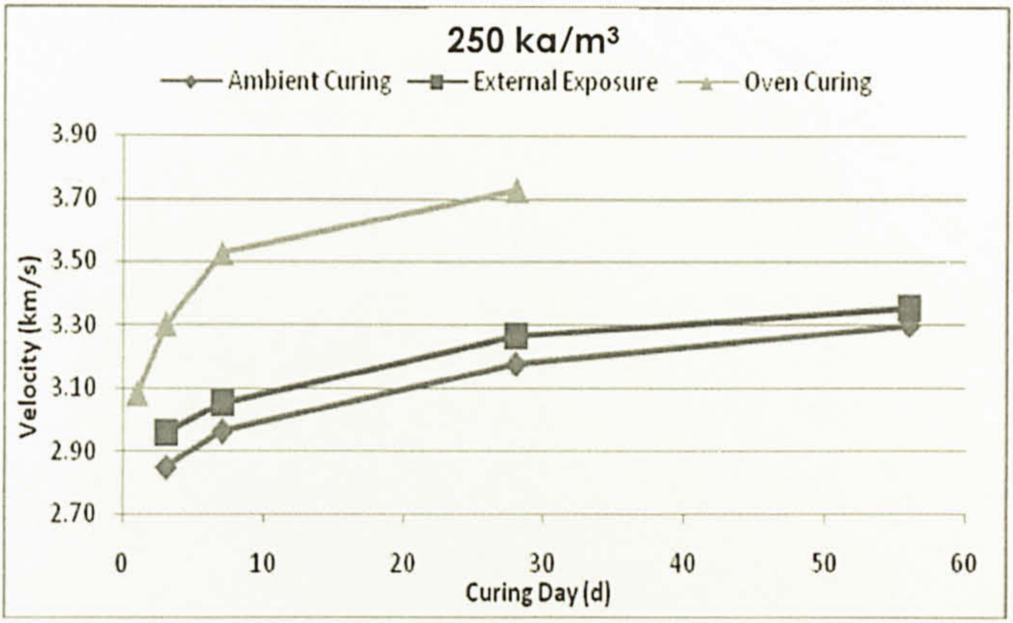


Figure 4.2: Pulse Velocity of Fly Ash Based Geopolymer Concrete with 250 kg/m^3 of Fly Ash Content.

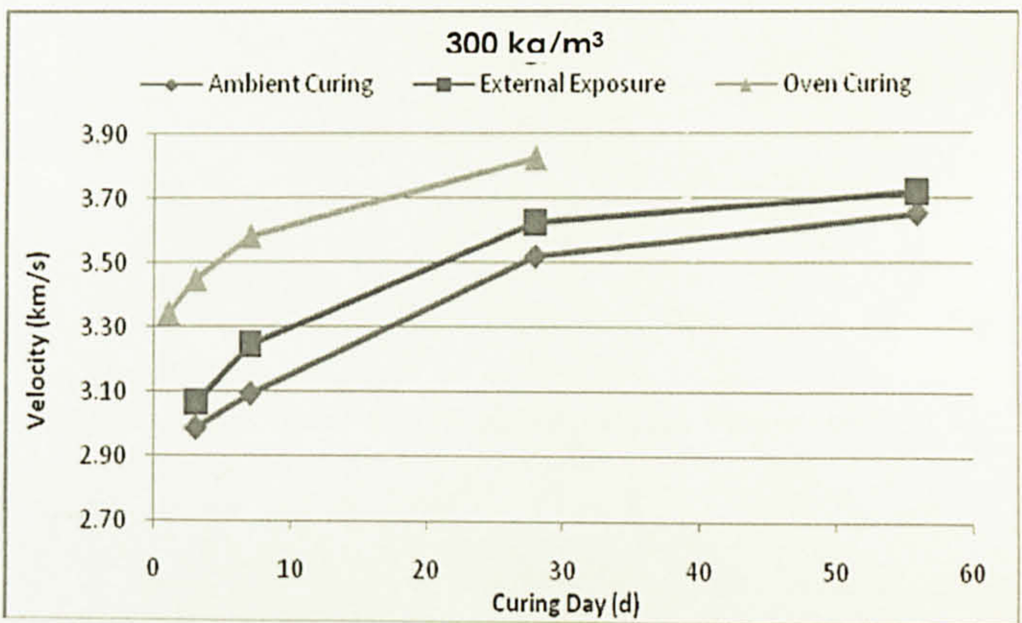


Figure 4.3: Pulse Velocity of Fly Ash Based Geopolymer Concrete with 300 kg/m^3 of Fly Ash Content.

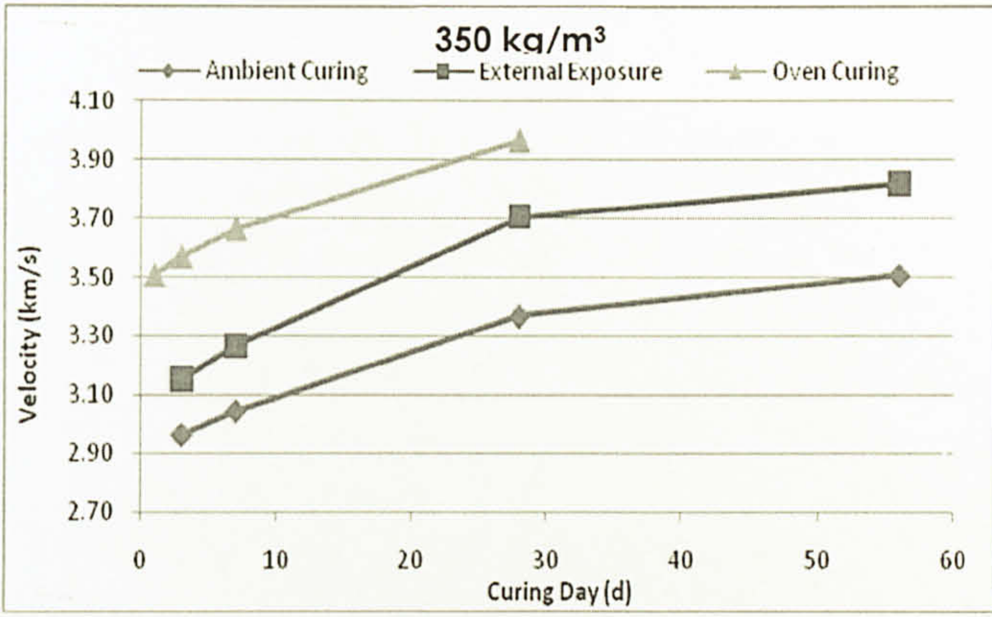


Figure 4.4: Pulse Velocity of Fly Ash Based Geopolymer Concrete with 350 kg/m^3 of Fly Ash Content.

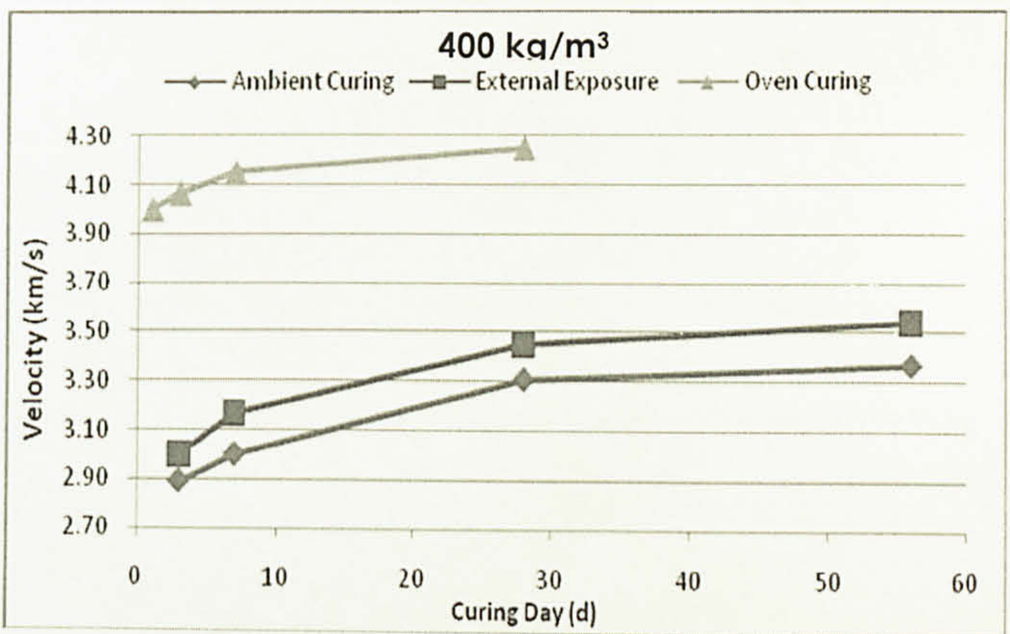


Figure 4.5: Pulse Velocity of Fly Ash Based Geopolymer Concrete with 400 kg/m^3 of Fly Ash Content.

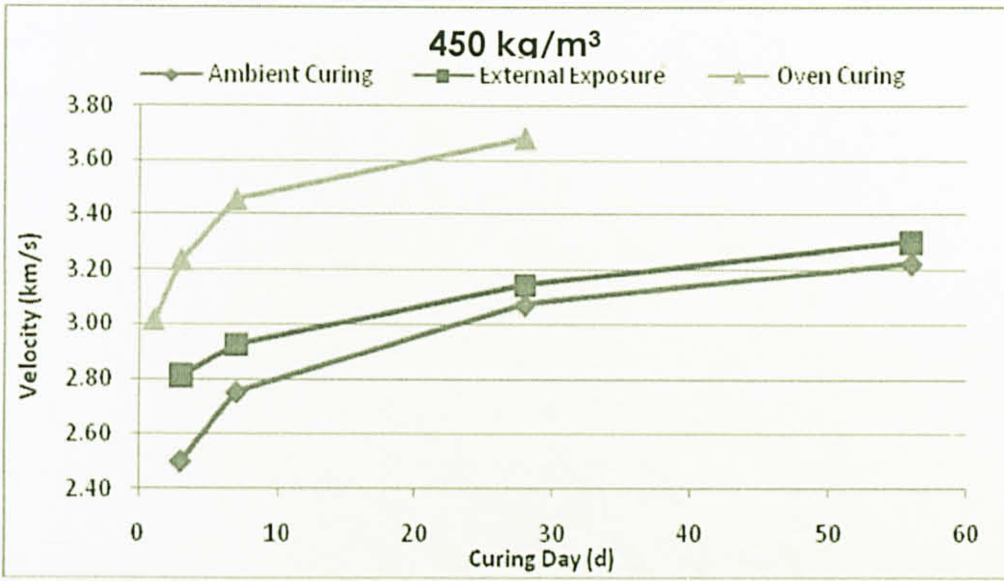


Figure 4.6: Pulse Velocity of Fly Ash Based Geopolymer Concrete with 450 kg/m^3 of Fly Ash Content.

Figure 4.2 shows pulse velocity for Geopolymer Concrete with 250 kg/m^3 fly ash content. From the result, the oven cured concrete showed the highest pulse velocity. Since the curing temperature was high, the water inside Geopolymer Concrete was easily evaporated. After water was evaporated, it leaves small pores inside concrete. Compared this with ambient curing concrete and external exposure curing concrete where temperature is not as high as oven curing concrete, water was not easily evaporated and thus water was still trapped inside the pores. The pulse can travelled faster in empty pores compared to water-trapped pores. That explains why oven curing concrete exhibits the highest pulse velocity. This is followed by external exposure curing because its curing temperature was higher compared to ambient curing which possessed lowest pulse velocity. The other experiments conducted by using different amount of fly ash also produced the similar result (refer to Figure 4.11 to Figure 4.14).

For oven curing concrete, the pulse velocity was rapidly increased from 1 day to 7 days. This is because residue heat was still presence inside the concrete after being exposed to high temperature. Thus, the residue heat will promote the un-evaporated water to be evaporated. After 7 days, the pulse velocity was still increased but in slower rate. This indicates that water is almost completely evaporated from the concrete sample and leave small pores inside concrete.

For external exposure curing and ambient curing, the pattern of pulse velocity at 3, 7, 28 and 56 days are quite similar. From 3 to 28 curing days, the pulse velocity was rapidly increased but after 28 days, the pulse velocity was increased in a very slow rate and shows the tendency of becoming constant. This shows that water inside concrete evaporated in faster rate before 28 days and become slower after that.

Even though the temperature of external exposure curing was higher than ambient curing, the results not showed much difference from each other. This is due to the condition of weather during the curing process. The temperature of external exposure curing was changing due to rainy season and sometimes the temperature is approximately the same as ambient curing.

These results can be used in analyzing the compressive strength of Geopolymer Concrete, since UPV data have direct relation to concrete strength development.

4.3 Compressive Strength Test

Compressive Strength Test was conducted to analyze the impact of fly ash content on Geopolymer Concrete, in order to obtain the optimum mix proportion of fly ash. Besides, it is also conducted to determine the impact of curing regimes to the concrete strength development.

The concrete strength development was measured at 3, 7, 28 and 56 days of age for ambient curing concrete and external exposure curing concrete. While for oven curing concrete, the strength development was measured at 1, 3, 7 and 28 days of age. Table 4.3 shows the overall compressive strength development result of Geopolymer Concrete.

Table 4.3: Compressive Strength Development of Geopolymer Concrete

Curing	Code	Fly Ash Content (kg/m^3)	Compressive Strength (MPa)			
			3 day	7 day	28 day	56 day
Ambient	D1	250	7.3	11.9	24.1	26.3
	C1	300	9.0	16 5	27.8	33.6
	E1	350	8.6	14.4	27.0	31.6
	A1	400	8.3	13.9	25.1	30.6
	B1	450	5.7	8.7	16.5	19.1
External Exposure	D2	250	11.9	19.7	25.7	30.0
	C2	300	12.1	20.3	28.6	34.4
	E2	350	12.9	20.5	30.4	35.8
	A2	400	12.8	20.0	25.7	30.9
	B2	450	7.5	11.3	17.6	20.5
Oven			1 day	3 day	7 day	28 day
	D3	250	25.7	27.2	28.3	31.5
	C3	300	36.5	37.6	37.6	39.6
	E3	350	41.4	42.6	44.6	46.4
	A3	400	41.8	44.0	46.1	48.7
	B3	450	22.1	22.7	23.7	26.3

Figure 4.7, 4.8, 4.9, 4.10 and 4.11 shows the compressive strength results for each fly ash content with different curing regime.

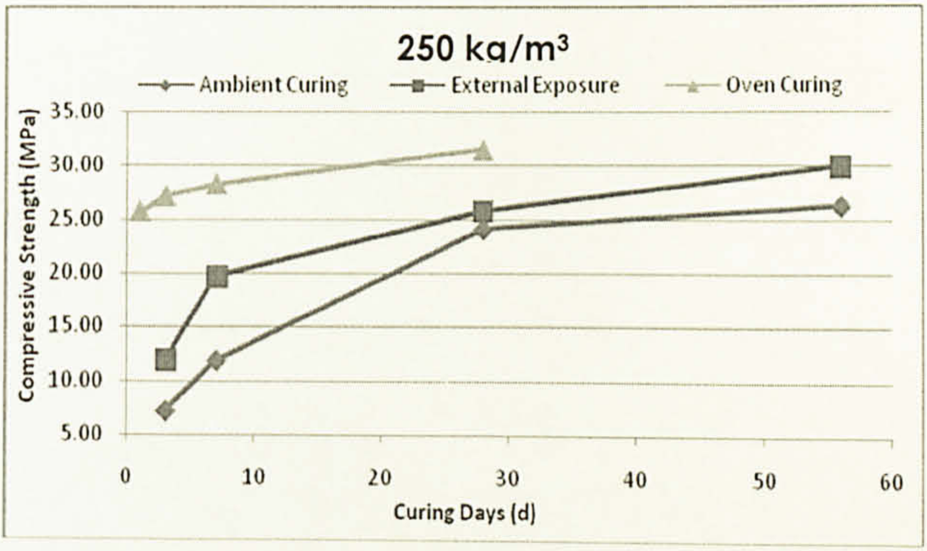


Figure 4.7: Compressive Strength of Fly Ash Based Geopolymer Concrete with 250 kg/m^3 of Fly Ash Content.

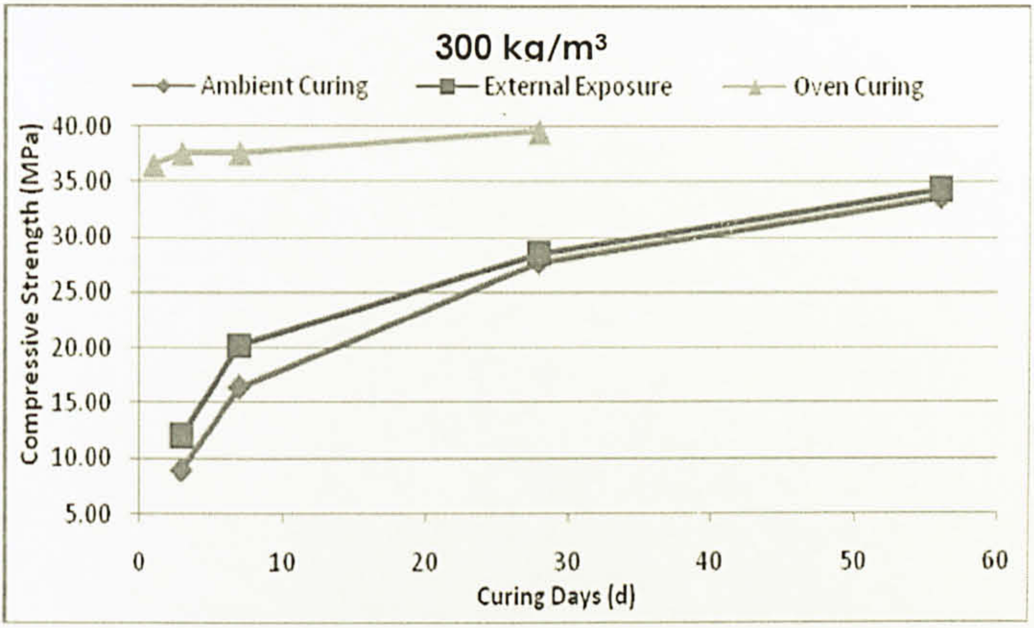


Figure 4.8: Compressive Strength of Fly Ash Based Geopolymer Concrete with 300 kg/m^3 of Fly Ash Content.

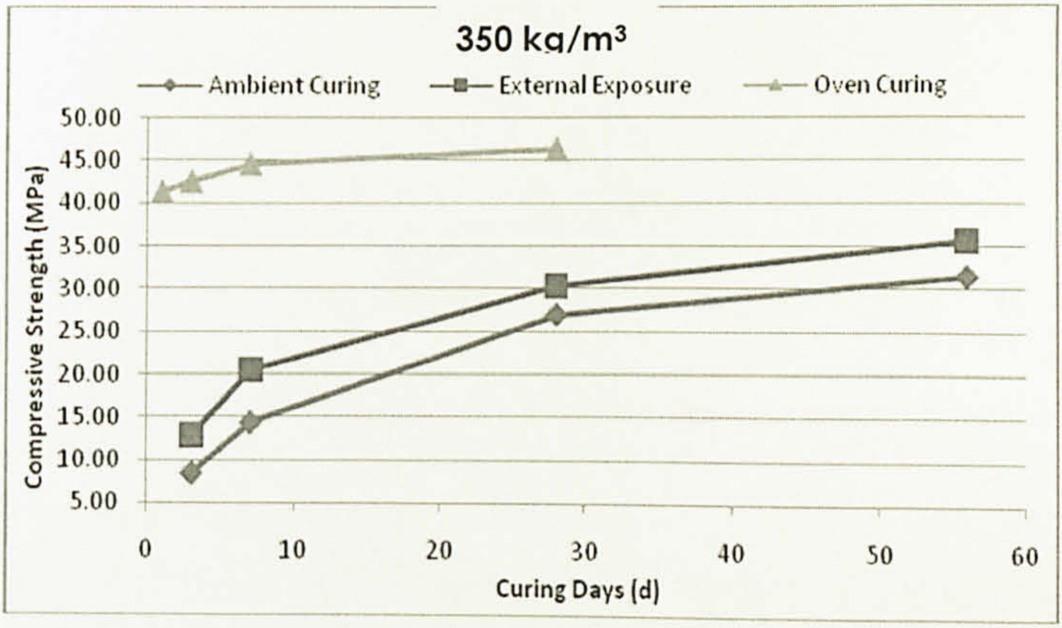


Figure 4.9: Compressive Strength of Fly Ash Based Geopolymer Concrete with 350 kg/m^3 of Fly Ash Content.

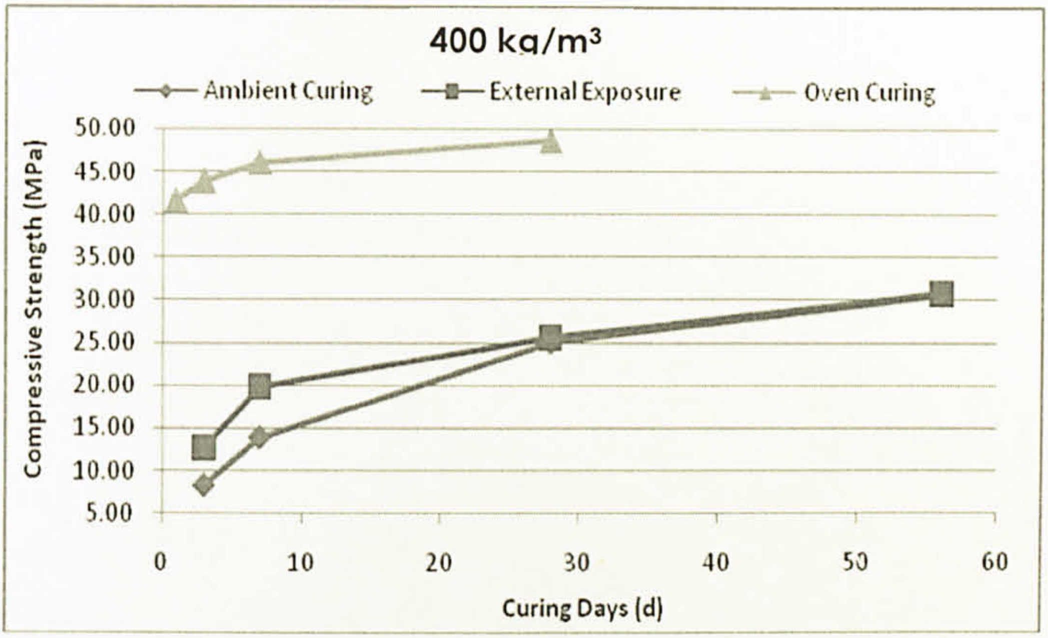


Figure 4.10: Compressive Strength of Fly Ash Based Geopolymer Concrete with 400 kg/m^3 of Fly Ash Content.

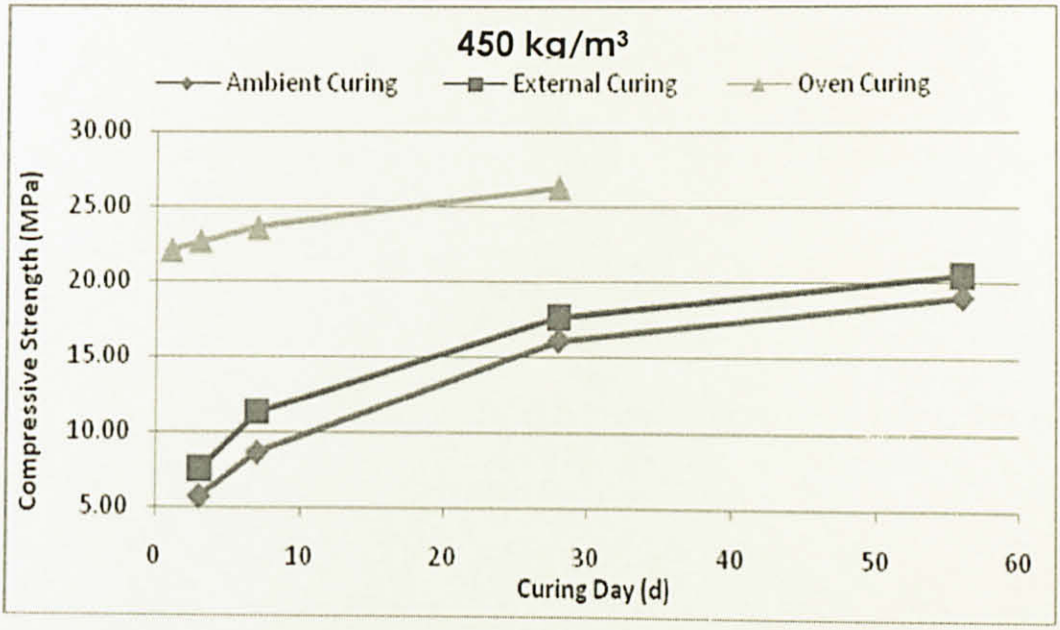


Figure 4.11: Compressive Strength of Fly Ash Based Geopolymer Concrete with 450 kg/m^3 of Fly Ash Content.

Figure 4.7 shows the compressive strength of Geopolymer Concrete that utilized 250 kg/m^3 fly ash content. From the graph, oven curing shows the highest compressive strength and followed by external exposure curing. While ambient curing demonstrated the lowest compressive strength. This is because the strength of Geopolymer Concrete was influenced by curing temperature. High curing temperature will accelerate the polymerization process between fly ash and alkaline solutions. Thus, enhance the bonding between geopolymers and bonding between geopolymer and aggregates in the concrete. High compressive strength can be achieved if bonding inside concrete is strong because heavy load is needed to break the bonding.

Water was added in Geopolymer Concrete mixture to improve the workability of fresh concrete. But, water do not involved in polymerization process and it will be expel from concrete during the curing process. Therefore, the presences of water molecules in Geopolymer Concrete will interrupt the polymerization process and thus affecting the concrete strength. Higher temperature will promote higher rate of water evaporation. Therefore, water in concrete can be reduced or drive out faster compared to lower temperature. Since oven curing method involved highest curing temperature which is 65°C, compared to external exposure (55°C) and ambient curing (30°C) method, thus its exhibited highest compressive strength. Meanwhile, the ambient curing involved lowest curing temperature, hence the concrete strength is low. This trend is similar for other fly ash content (refer to Figure 4.8 to Figure 4.11).

From Figure 4.7, 4.8, 4.9, 4.10 and 4.11, the compressive strength of oven curing concrete was significantly increased after 24 hour. After that, its compressive strength still increased but in slower rate. This shown that, the oven curing method can accelerated the polymerization process at a short time and thus enhanced concrete strength. After 7 days, the compressive strength started to become constant.

Ambient curing and external exposure curing showed quite similar trend. It can be seen that compressive strength development was constantly increased from 3 to 28 days. But after 28 days, the compressive strength increased in slowed rate and also showed the tendency of became constant.

Figure 4.12, 4.13 and 4.14 show the compressive strength of Geopolymer Concrete with different amount of fly ash at 28 days for ambient curing, external exposure curing and oven curing respectively.

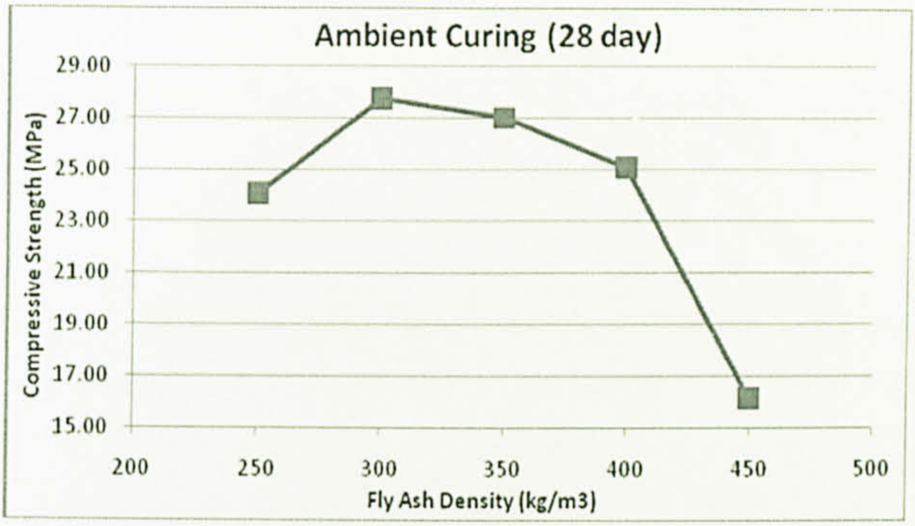


Figure 4.12: Compressive Strength of Fly Ash Based Geopolymer Concrete with Different Amount of Fly Ash Content in Ambient Curing at 28-day.

In ambient curing (refer Figure 4.12), the compressive strength increased from 24.1 MPa to 27.8 MPa when 250 and 300 kg/m^3 fly ash content was utilized in the concrete. But when higher fly ash content was utilized, the compressive strength decreased from 27.8 MPa for 300 kg/m^3 fly ash content to 16.5 MPa for 450 kg/m^3 fly ash content. Therefore, the optimum fly ash content for ambient curing concrete achieved when 300 kg/m^3 fly ash content was utilized. The addition of fly ash content which is higher than optimum level will increase the number of unreacted material inside concrete and thus disturbed the polymerization process. Therefore, it explains the reason of lower compressive strength when 350, 400 and 450 kg/m^3 fly ash content was utilized in concrete.

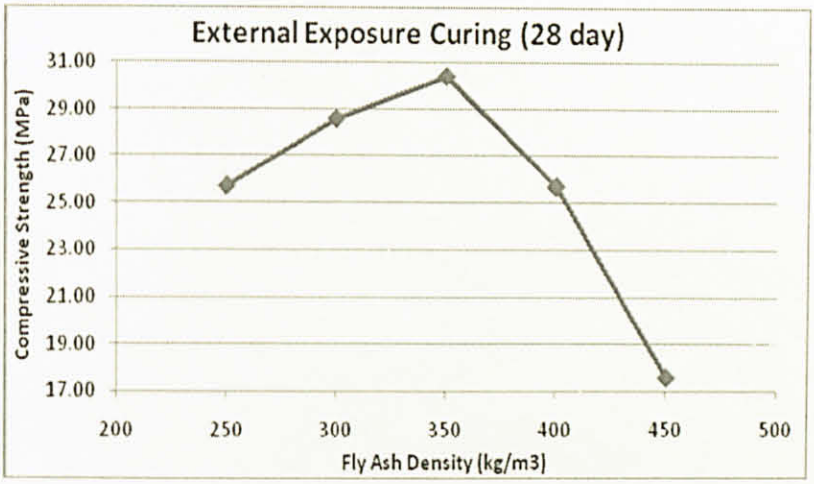


Figure 4.13: Compressive Strength of Fly Ash Based Geopolymer Concrete with Different Amount of Fly Ash Content in External Exposure Curing at 28-day.

Figure 4.13 shows the compressive strength of Geopolymer Concrete that undergone external exposure curing. The figure shows that the compressive strength increased from 25.7 MPa to 30.4 MPa when content of fly ash increased from 250 to 350 kg/m^3 respectively. After that, further addition of higher fly ash content will reduce the compressive strength. Therefore, the optimum amount of fly ash content for external exposure curing is 350 kg/m^3.

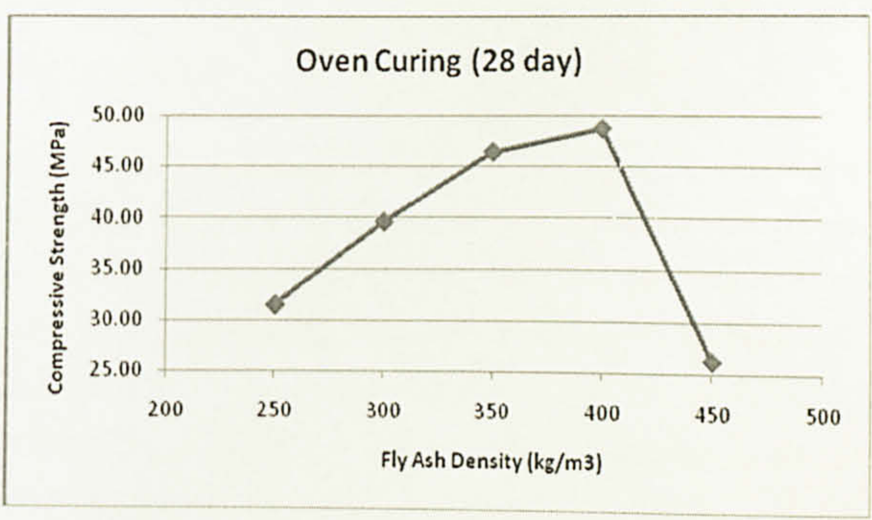


Figure 4.14: Compressive Strength of Fly Ash Based Geopolymer Concrete with Different Amount of Fly Ash Content in Oven Curing at 28-day.

The compressive strength of Geopolymer Concrete that undergone oven curing is shown in Figure 4.14. From Figure 4.14, it shows that the highest compressive strength recorded when 400 kg/m^3 fly ash content was utilized in the concrete. The compressive strength increased from 31.5 MPa to 48.7 MPa when content of fly ash is increased from 250 to 400 kg/m^3 respectively. But, when 450 kg/m^3 fly ash was utilized, the compressive strength is only 26.3 MPa. This proved that the usage of higher fly ash content which exceeded the optimum level will disturb the polymerization process and hence resulted in low compressive strength.

The optimum amount of fly ash content for ambient curing is 300 kg/m^3 and for external exposure curing is 350 kg/m^3, while for oven curing is 400 kg/m^3. As stated before, at higher curing temperature, the rate of polymerization process is higher. Therefore, it can utilize more fly ash to react with alkaline solution and produced greater bonding between the geopolymer itself and bonding between geopolymer with aggregates.

4.4 Field Emission Scanning Electron Microscopy (FESEM) Analysis

FESEM Analysis was carried out to observe the microstructure of Geopolymer Concrete. The microstructure properties was analyzed to determine the relation between the Interfacial Transition Zone (ITZ) with the compressive strength performance. FESEM Analysis was performed on hardened concrete samples with 56 days of age. Concrete samples for FESEM Analysis were taken from:

1. 250 kg/m^3 fly ash with External Exposure Curing
2. 400 kg/m^3 fly ash with External Exposure Curing
3. 250 kg/m^3 fly ash with Oven Curing
4. 400 kg/m^3 fly ash with Oven Curing

ITZ can be identified as the presence of gap between aggregate and Geopolymer paste. Its indicates low bonding strength of Geopolymer paste caused by low amount of heats absorbed during the polymerization process during maturing period. When concrete loaded with certain load, ITZ connected with micro pores and micro cracks and caused premature failure of concrete sample.

4.4.1 FESEM Analysis of Geopolymer Concrete with External Exposure Curing

Figure 4.15 and 4.16 show the inner part of Geopolymer Concrete sample with 450 and 250 kg/m^3 fly ash content respectively.

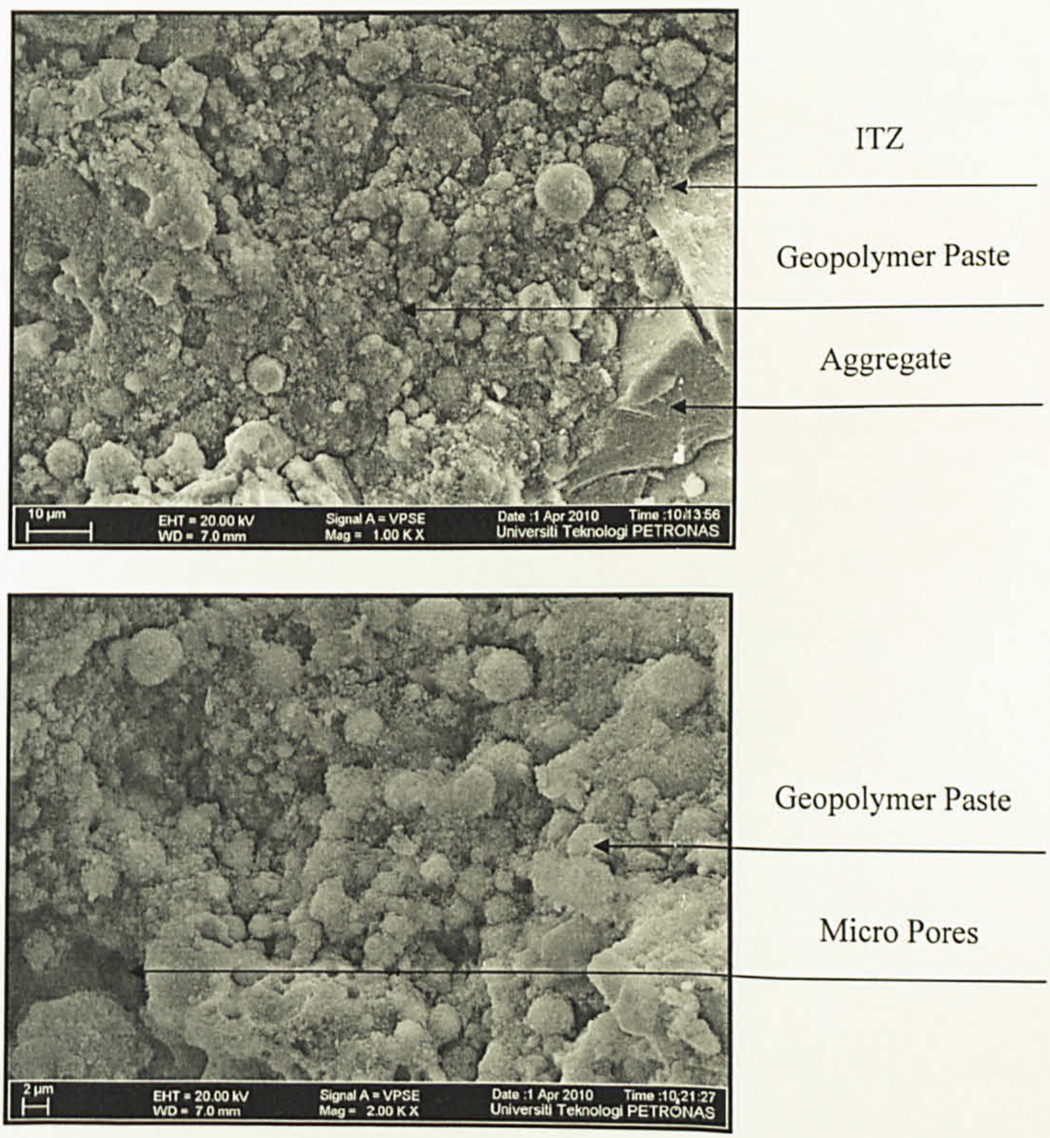


Figure 4.15: FESEM Images of External Exposure Curing Concrete with 450 kg/m^3 fly ash Content

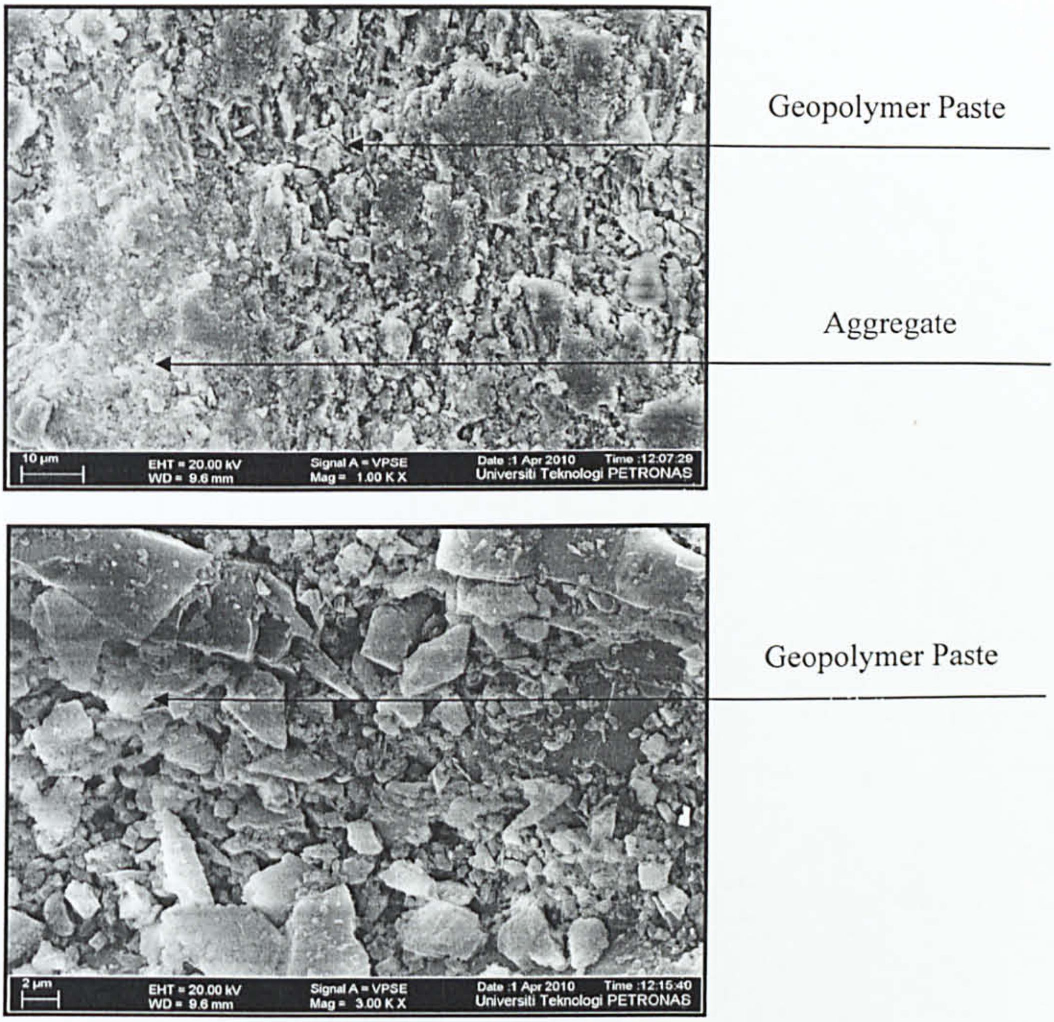


Figure 4.16: FESEM Images of External Exposure Curing Concrete with 250 kg/m^3 fly ash Content

As seen in Figure 4.15 and 4.16, Geopolymer paste was completely hardened and bonded with aggregate. In Figure 4.15, ITZ appeared between aggregate and Geopolymer paste, and but in Figure 4.16, ITZ was not significant. Besides, the micro pores was smaller in Figure 4.16 and hardly been seen compared to Figure 4.15 where the structure contained more pores. This shows that, the bonding between aggregate and Geopolymer paste is stronger in concrete 250 kg/m^3 fly ash content than 450 kg/m^3. This can be verified with compressive strength where the compressive strength of 450 kg/m^3 fly ash content was only 20.5 MPa, whereby for 250 kg/m^3 was 26.3 MPa.

4.4.2 FESEM Analysis of Geopolymer Concrete with Oven Curing

Figure 4.17 and 4.18 show the inner part of Geopolymer Concrete sample with 450 and 250 kg/m^3 fly ash content respectively. Based on compressive strength test, concrete with 250 kg/m^3 fly ash content exhibit higher compressive strength which is 31.5 MPa compared to 26.3 MPa for 450 kg/m^3 fly ash content.

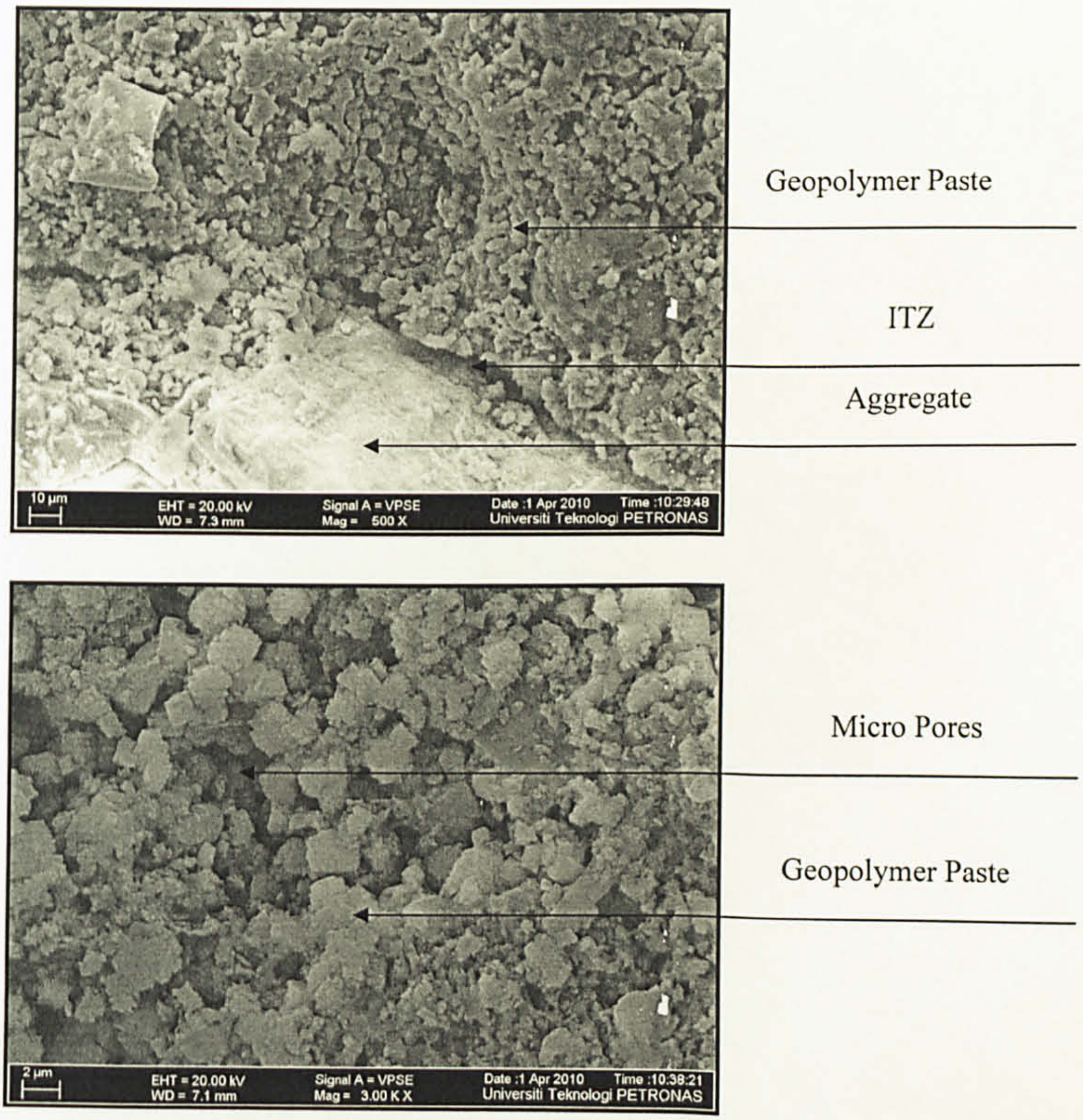
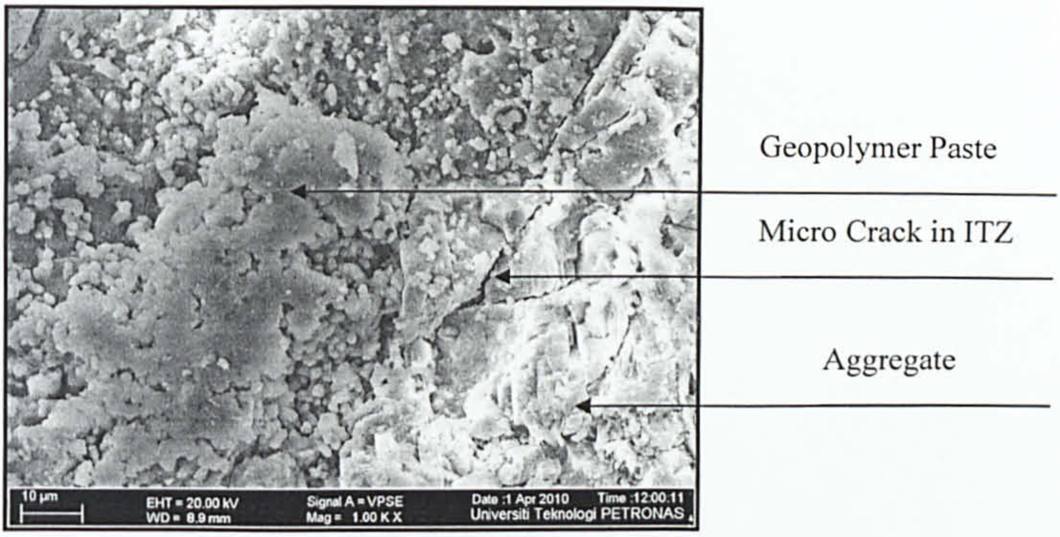


Figure 4.17: FESEM Images of Oven Curing Concrete with 450 kg/m^3 Fly Ash Content



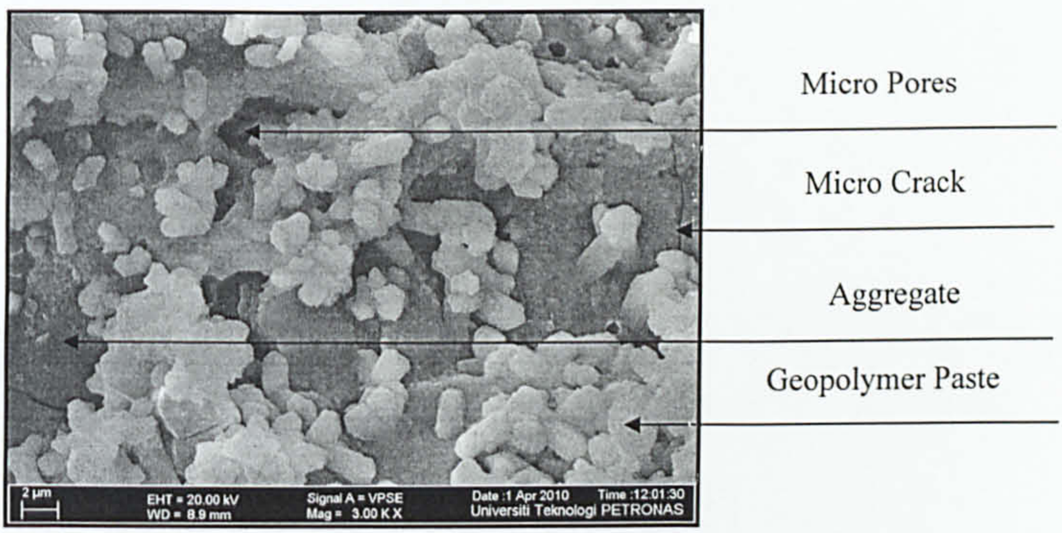


Figure 4.18: FESEM Images of Oven Curing Concrete with 250 kg/m^3 Fly Ash Content

The clear appearance of ITZ and porous structure of Geopolymer paste can be seen in Figure 4.17. This explains the lower compressive strength of concrete with 450 kg/m^3 fly ash content. In Figure 4.18, it can be seen that the Geopolymer paste was firmly bonded with the aggregate. The presence of micro crack can be seen in this sample. This is resulted from creep and shrinkage of concrete due to high curing temperature. Despites the presence of micro crack, the compressive strength of this concrete was higher because there is less number of micro pores and also the bonding between Geopolymer paste and aggregates was stronger.

4.5 Geopolymer Concrete Density

The density of normal concrete is 2400 kN/m^3. Since the density of fly ash used in the Geopolymer Concrete is varied, therefore it is essential to determine the Geopolymer Concrete density. Table 4.4 shows the concrete density for all the concrete samples.

Table 4.4: Geopolymer Concrete Density

Curing	Code	Fly Ash Content (kg/m^3)	Average Weight (kg)	Concrete Volume (m^3)	Density (kN/m^3)
Ambient	D1	250	2.412	0.001	2412
	C1	300	2.436	0.001	2436
	E1	350	2.445	0.001	2445
	A1	400	2.464	0.001	2464
	B1	450	2.416	0.001	2416
External Exposure	D2	250	2.371	0.001	2371
	C2	300	2.375	0.001	2375
	E2	350	2.389	0.001	2389
	A2	400	2.388	0.001	2388
	B2	450	2.335	0.001	2335
Oven	D3	250	2.381	0.001	2381
	C3	300	2.405	0.001	2405
	E3	350	2.403	0.001	2403
	A3	400	2.459	0.001	2459
	B3	450	2.374	0.001	2374

For concrete with fly ash content of 250 kg/m^3, the concrete density is ranging from 2371 to 2412 kN/m^3, while for fly ash content of 300 kg/m^3, the concrete density is in range of 2375 to 2436 kN/m^3. The concrete density is ranging from 2389 kN/m^3 to 2445 kN/m^3 for concrete with 350 of fly ash kg/m^3. Whereas for fly ash content of 400 kg/m^3 and 450 kg/m^3, the concrete density is ranging from 2388 kN/m^3 to 2464 kN/m^3 and from 2335 kN/m^3 to 2416 kN/m^3 respectively. Overall, the minimum concrete density is 2335 kN/m^3 and the maximum density is 2464 kN/m^3. This value is quite similar with normal concrete density.

CHAPTER 5
CONCLUSION AND RECOMMENDATION

5. CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE RESEARCH

5.1 Conclusions

This research was conducted to identify the properties of Geopolymer Concrete which utilized fly ash as binder. The content of fly ash used in this research were varies from 250, 300, 350, 400 and 450 kg/m^3 of Geopolymer Concrete, in order to determine the optimum amount of fly ash in concrete. The Geopolymer Concrete was undergone 3 types of curing which are ambient curing, external exposure curing and oven curing. This is to determine the compressive strength development of concrete with respective curing type. The following conclusions can be drawn from this study:

1. The optimum proportion of fly ash in Geopolymer Concrete depended on the curing type. For ambient curing, the optimum proportion of fly ash obtained was 300 kg/m^3 of Geopolymer Concrete which exhibit compressive strength of 27.8 MPa at 28 days. While for external exposure curing, 350 kg/m^3 fly ash content was identified as the optimum fly ash proportion with compressive strength of 30.4 MPa at 28 days. The optimum amount of fly ash for oven curing concrete was 400 kg/m^3 with compressive strength of 46.1 MPa at 28 days.

2. Oven curing shows the highest compressive strength development compared to ambient curing and external exposure curing. While, ambient curing recorded the lowest compressive strength development. This is because at higher curing temperature, the polymerization process was accelerated. Hence, it can promote

stronger bonding between geopolymer pastes and bonding between geopolymer pastes with aggregates.

3. The compressive strength development of Geopolymer Concrete was recorded at 3, 7, 28 and 56 days for ambient curing and external exposure curing and at 1, 3, 7 and 28 days for oven curing. It was found that, the compressive strength development of oven curing concrete is dramatically increased after 24 hours of curing and thus exhibit high early strength performance. For ambient and external exposure curing, the compressive strength of concrete is rapidly increased from 3 to 7 days and started to become constant after reached 28 days.

4. FESEM Analysis was conducted to observe microstructure of Geopolymer Concrete. This is to determine the relation between ITZ with concrete compressive strength performance. From this analysis, it was found that the presence of ITZ in concrete can reduced concrete strength. Besides, the presence of micro pores and micro cracks can also contributed to low compressive strength of Geopolymer Concrete.

5.2 Recommendations for Future Research

Future expansion of this research can be made to explore the potential of Geopolymer Concrete application in construction industry especially for cast in-situ production. The Geopolymer Concrete with external exposure curing had shown the potential of replacing conventional OPC concrete for cast in-situ production. So, extensive studies should be concentrating on external exposure curing of Geopolymer Concrete, to improve the compressive strength. Besides, other concrete properties such as tensile strength, creep and drying shrinkage, durability and etceteras should also being look into. Currently, the Geopolymer Concrete is composed of solid (fly ash) and liquid part (alkaline solution). The preference of developing Geopolymer Cement in dry form is an advantage for in-situ production. Therefore, the study on developing Geopolymer Cement is a great need to the industry.

CHAPTER 6
ECONOMIC BENEFITS

6. ECONOMIC BENEFITS

This chapter describes the cost of Geopolymer Concrete production based on the experiment conducted during the research work. The total costs were contributed from cost of fly ash, coarse and fine aggregates, sodium hydroxide pallet and sodium silicate solution. The detail of calculation is presented in this chapter.

6.1. Cost of Project

Table 6.1: Total content of material used in Geopolymer Concrete

Mix Code	Fly Ash Content (kg/m^3)	Coarse Aggregate (kg/m^3)	Fine Aggregate (kg/m^3)	NaOH solution (kg/m^3)	Na_2SiO_3 solution (kg/m^3)	Extra water (kg/m^3)	Curing
A1	400	1200	645	41	103	60.0	Ambient
A2	400	1200	645	41	103	60.0	External Exposure
A3	400	1200	645	41	103	60.0	Oven
B1	450	1200	645	41	103	67.5	Ambient
B2	450	1200	645	41	103	67.5	External Exposure
B3	450	1200	645	41	103	67.5	Oven
C1	300	1200	645	41	103	45.0	Ambient
C2	300	1200	645	41	103	45.0	External Exposure
C3	300	1200	645	41	103	45.0	Oven
D1	250	1200	645	41	103	37.5	Ambient
D2	250	1200	645	41	103	37.5	External Exposure
D3	250	1200	645	41	103	37.5	Oven

Mix Code	Fly Ash Content (kg/m^3)	Coarse Aggregate (kg/m^3)	Fine Aggregate (kg/m^3)	NaOH solution (kg/m^3)	Na_2SiO_3 solution (kg/m^3)	Extra water (kg/m^3)	Curing
E1	350	1200	645	41	103	52.5	Ambient
E2	350	1200	645	41	103	52.5	External Exposure
E3	350	1200	645	41	103	52.5	Oven
Total	5250	18000	9675	615	1545	787.5	

No. of cube = 12

Cube size = 100x100x100mm

Volume/ cube = 0.1x0.1x0.1

= $0.001\ m^3$

Total volume = $12 \times 0.001\ m^3$

= $0.012\ m^3$

Table 6.2: Cost of Geopolymer Concrete

Material	Quantity (kg/m3)	Volume (m3)	Quantity (kg)	Unit Price	Total Price (RM)
Sodium Hydroxide (NaOH) Pallet (*)	615 (NaOH in solution)	-	2.17	RM 0.75/kg	1.63
Sodium Silicate (Na_2SiO_3) Solution	1545	0.012	18.54	RM 1.20/kg	22.25
Fly Ash	5250	0.012	63	-	-
Coarse Aggregate	18000	0.012	216	RM 180/tonne (RM 0.18/kg)	38.88
Fine Aggregate	9675	0.012	116.10	RM 40/tonne (RM 0.04/kg)	4.64
				Total Cost	RM 67.40

(*) Sodium hydroxide (NaOH) solution = 615 kg/m3

= 615 x 0.012

= 7.38 kg of NaOH solution

1 kg of NaOH solution = 0.294 kg of NaOH pallet

7.38 kg of NaOH solution = 2.17 kg of NaOH pallet

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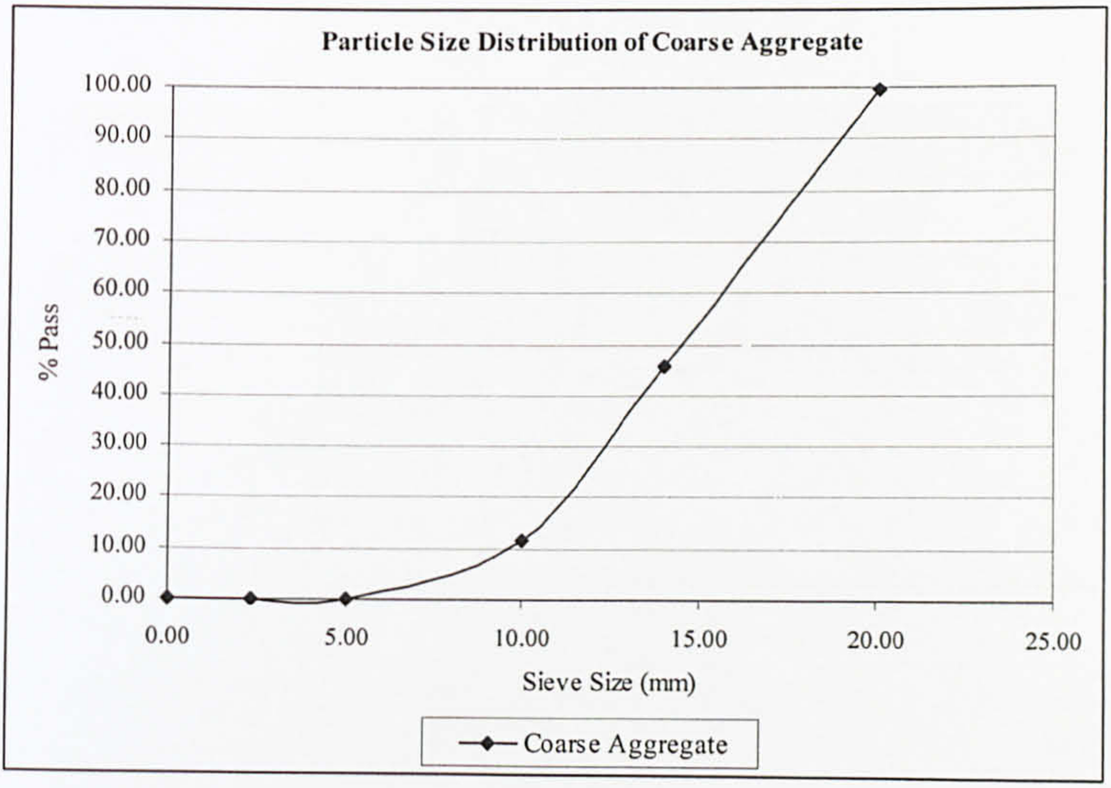
APPENDIX A

SIEVE ANALYSIS OF COARSE AND FINE AGGREGATES

Sieve Analysis Results of Coarse Aggregate

Sieve Size (mm)	Mass Retained (g)	% Mass Retained	Σ % 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	Σ % 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