EXPERIMENTAL STUDY ON THE KINETICS OF GEOPOLYMER'S SOLIDIFICATION

By

Ahmad Hafizi bin Pauzi

Dissertation submitted in partial fulfilment of

The requirements for the

BACHELOR OF CHEMICAL ENGINEERING (Hons)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the

Chemical Engineering Programme

Universiti Teknologi PETRONAS

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Approved by,

………………………..
(Dr. Lukman bin Ismail)

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CERTIFICATION OF ORIGINALITY

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

(AHMAD HAFIZI BIN PAUZI)

“It doesn’t matter how beautiful your theory is, it doesn’t matter how smart you are. If it doesn’t agree with the experiment, it’s wrong.” – Richard Feynman

ABSTRACT

Geopolymer material is formed through the reaction of silica and alumina oxides with alkali activator and has appeared as a viable alternative to the Ordinary Portland Cement (OPC) in the construction field due to good properties such as more resistance to corrosion and fire, high compressive and tensile strength, low permeability and good acid resistance. Furthermore, the production of geopolymer concrete offers a solution to the environmental issue as it utilized the industrial waste that consist of Si and Al such as fly ash and wastewater from chemical industry. However, there are limited research were conducted to investigate the effect of parameters before setting time especially at the solidification phase in geopolymerization process and explained it through Avrami's Kinetic Theory's perspective that closely related to the nucleation of new particles and growth rate of particles into spherical shaped. This research is to study the solidification of fly ash geopolymer by determining setting time by varying curing temperature, alkaline activator and concentrations of alkaline. However, prior to the study of the effects, the feasibility of equipment used between LFRA Texture Analyzer and Vicat Needle was determined. Further characterization on the phase exists during the solidification process of geopolymer was done using XRD analysis. Besides that, this research also focuses on the transformation of crystal in geopolymerization through Avrami's Kinetic Theory. From the experimental results, the alkaline activation with the addition of sodium silicate (Na_2O_3Si) to sodium hydroxide (NaOH) produced shortest time for solidification geopolymer followed by potassium hydroxide (KOH) solution and sodium hydroxide (NaOH) solution. Moreover, the increase in concentration of alkaline solution and curing temperature produce shorter setting time for geopolymer's solidification. From the Avrami Kinetic Theory's perspective, the growth form of crystal in the geopolymerization process exhibits two and three dimensional structure while certain samples of geopolymer shows there are presence of secondary nucleation in geopolymer's growth.

ACKNOWLEDGEMENT

First and foremost, my deepest gratitude to Dr. Lukman bin Ismail as my supervisor for giving me the great opportunity to do this research, letting me take his ideas for what he wanted me to do for a project, twist them into all sorts of different shapes, and produce something that in the end is quite different from what we had initially planned. I was fortunate to get exactly the supervisor I needed for this project - providing me with support, advice and freedom to go chasing crazy ideas to complete this project successfully.

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Thanks to my family – none of this would have been possible without all of you, and the support you've given me throughout this project has been absolutely amazing and thanks to everyone in the Geopolymer Laboratory for lending the helps and places in finishing this project. On a slightly more serious note, all sorts of funding bodies contributed money towards the successful completion of this project, and all are worthy of acknowledgement. The Ministry of Higher Education (MOHE) has supported me throughout this work by funding under the Long Term Research Grant Scheme (LRGS) and Universiti Teknologi PETRONAS (UTP) for their dedication and continuous support especially in term of welfare and personal matters.

And last but definitely not least, I have to thank Allah. He created everything and I am just trying to understand my little corner of it.

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CHAPTER 1

INTRODUCTION

1.1 Project Background

Geopolymer is one of the types of concrete that available and widely used in structural material in the world due to its simplicity in operation, easy availability, low cost of ingredients as well as strong in structure (Islam, Rahman and Ahmed, 2011). Venderly (2003) states that probably concrete made from Ordinary Portland Cement (OPC) is the most important material in the world. The reason of this statement is due to the presence of gypsum in OPC that improves workability of cement. However, Portland cement also has its drawback where it has risks to induce climate change due to the emission of carbon dioxide (CO_2) into atmosphere during combustion of fossil fuels and decarbonisation of limestone (Worrell *et al*., 2001). In addition, Madeleine Rubenstein (2012) from The Earth Institute of Columbia University states that the CO_2 released to environment by cement industry was reportedly about 5% - 7% in all over the world.

Consequently, a new kind of material was found, known as 'geopolymer' which is emerging as viable alternative to conventional cements especially in construction filed. According to Davidovits (2011), geopolymer cement represents a broad range of material characterized by chain of inorganic molecule. The main constituent in geopolymer is fly ash, rich with the silica and alumina that reacts with alkaline solution like sodium silicate oxide (geopolymerization process) to form gel which binds the fine and coarse aggregates (Aleem and Arumairaj, 2012). This fact had been supported by Yen *et al*. (2006), where the study shows that geopolymer is the most stable material and best alternative for cement the earth can offer. Moreover, the main advantage of geopolymer concrete is it helps to reduce OPC contribution to CO_2 emission during concrete production while in the same time produce stronger material that have high sustainability towards the corrosion especially from acid and water penetrability which provides good characteristic for the construction materials.

However, in order to produce geopolymer concrete, several factors need to be considered especially in the solidification phase during geopolymerization process. The study of solidification phase or setting time of geopolymer is very important in order to produce geopolymer with good quality with regard to its final properties. According to De Silva and Sagoe-Crenstil (2009), there are number of factors that might affect the final properties of solidification of geopolymer likes curing temperature, ageing time, water content as well as types and concentration of alkaline solution. The main purpose of this research is to study the effect of different types of alkaline activator, the effect of different concentrations of alkaline solution and the effect of curing temperature in the solidification of geopolymer. The main material used in the geopolymerization process is fly ash while types of alkaline activator used are potassium hydroxide (KOH), sodium silicate solution (Na_2O_3Si) and sodium hydroxide solution (NaOH). The solidification phase will be tested using LFRA Texture Analyser, Vicat Needle and X-Ray Diffraction. The result will be presented and explained based on the Avrami's Kinetic Theory.

1.2 Problem Statement

Studies about the ability to achieve an excellent compressive strength of geopolymeric materials by proper mix design are well-documented (Duxson *et al.*, 2006). Conversely, the underlying mechanisms controlling geopolymer formation and alkali activation in general are not well understood. Most studies related to geopolymer are based on its chemical and physical properties after setting time such as compressive strength, acid resistance, water penetrability and stability of geopolymer (Nurhanie *et al.*, 2012). However, there are only a few researches were conducted to investigate the effect of parameters before setting time especially at the solidification phase in geopolymerization process and explained it from the Avrami's Kinetic Theory's perspective. Therefore, this research focusing more to the effect of different types of alkaline activator, the effect of different concentrations of alkaline solution as well as effect of different curing temperature on the solidification of geopolymer. The research also studies the feasibility of LFRA Texture Analyzer and Vicat Needle in determining the solidification of geopolymer. In respect to Avrami's Kinetic Theory, the transformation of crystal from nucleation phase until its growth of crystal will be explained and justified based on this theory.

1.3 Aim and Objectives

Throughout this research, the main aim is to study the formation of solidification based on three main parameters which are different types of alkaline solution, different concentrations of alkaline solution and temperature curing. All the details about the phase transition will be explained through Avrami's Kinetic Theory.

The objectives that have been identified for this research are:

I. To investigate the feasibility of using LFRA Texture Analyzer and Vicat Needle in determining the solidification of geopolymer.
II. To determine the effect of different types of alkaline activator (NaOH, KOH and Na_2O_3Si) on the solidification of geopolymer;
III. To determine the effect of different concentration of alkaline solution on the solidification of geopolymer;
IV. To determine the effect of different curing temperature on the solidification of geopolymer.

1.4 Scope of Study

The main scope of this study is to analyse the formation of crystal based on a few parameters which are alkaline solution and temperature. The common types of alkaline solution used in the geopolymerization process are sodium silicate solution (Na_2O_3Si) and sodium hydroxide solution (NaOH). Materials with high content of alumina and silica are needed in order to complete geopolymerization process. Material used in this research is known as fly ash, a waste product from industry which is very reactive in alkaline solution such as sodium silicate solution and potassium silicate solution to form gel before moulding process. Next, for the effect of difference concentration of alkaline solution, it will be varies from 6M to 14M each of alkaline solution. Then, the effect of temperature will be determined at set-up temperature range from 60^oC to 90^oC. Lastly, LFRA Texture Analyzer and Vicat Needle will be used to study the feasibility of both equipments in determination of geopolymer crystallization. X-Ray Diffraction will be used to identify the characterization of geopolymer after the final setting time has achieved. Above all, Avrami's Kinetic Theory is used to describe and explain the formation of crystal in geopolymerization process.

1.5 Relevancy and Feasibility of the Project

Solidification phase plays an important role in production of good quality of geopolymer in term of physical and chemical properties. The compressive strength, low permeability, good acid resistance and more resistance to corrosion of geopolymer only can be produced through the good establishment of synthesis condition during solidification phase (De Silva *et al*., 2009). Furthermore, geopolymer concrete would be very useful material in constructional field as a replacement to Portland cement and study of crystallization kinetic would very helpful to engineers and designers to produce new material in future. Moreover, this research can be considered as feasible as all the equipment including materials and apparatus are available in Chemical Engineering Laboratory, Universiti Teknologi PETRONAS. Time constraint also has been considered and this project shall be completed within the time specified in the project Gantt chart.

CHAPTER 2

LITERATURE REVIEW

This chapter covers about the concept of geopolymer and geopolymerization process. This part also explained the disadvantages of Ordinary Portland Cement (OPC) and importance of geopolymer in industrial field. Next, it reviews the concept solidification of geopolymer based on three (3) parameters including factors of different types of alkaline activator, the different concentration of alkaline solution as well as curing temperature. Then, this chapter also explained about Avrami's Kinetic Theory that being used to analyse all of these parameters. Lastly, this chapter covers the LFRA Texture Analyzer, Vicat Needle and X-Ray Diffraction that have been used to study the solidification physical characteristics.

2.1 Geopolymer

The term 'geopolymer' can be described as a group of inorganic polymers obtained by low-temperature alkali activation of alumina (Al_2O_3) and silica (SiO_2) containing materials (De Silva *et al*., 2009). It is a new materials that have been widely used for coatings and adhesive, new binders for fibre composites, waste encapsulation as well new cement for concrete in construction field (Davidovits *et al*., 2011). Geopolymer can be divided into two types; pure inorganic geopolymer and organic containing geopolymer, synthetic analogues of naturally occurring macromolecules (Yen *et al*., 2006). Basically, there are two main constituents in the process of geopolymerization which are source materials and alkaline solution.

The main sources of material used in making of geopolymer are alumina-silicate; rich with alumina (Al) and silica (Si) content and for alkaline solution while liquid used is alkali silicates or alkali hydroxides (Bakharev *et al*., 2004). The microstructure of geopolymer is essentially based on temperature dependent where it is exist as X-rays amorphous structure at room temperature but it evolved into a crystalline matrix structure like leucite or pollucite when the temperatures higher than 900 °C (He, Jia, Wang and Zhou, 2010). De-guang and Da-gen (2005) proves the theory of microstructure by conducting an experiment between metakaolin with the sodium silicate solution. The result of the experiment showed under SEM Micrograph, the microstructure of geopolymer is X-ray amorphous at room temperature as shown in Figure 2.1.

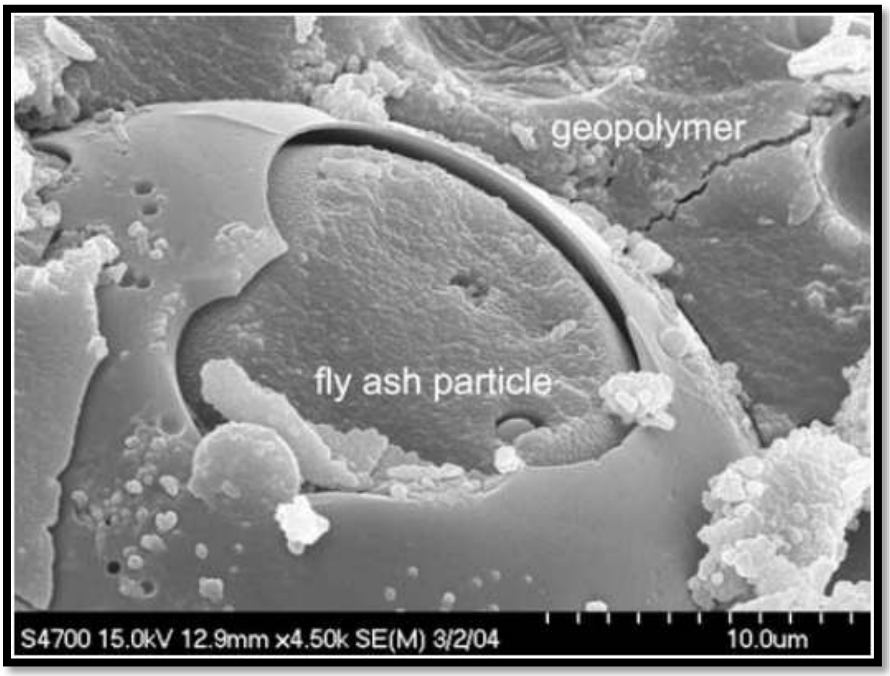


Figure 2.1: The microstructure of geopolymer under SEM Micrograph.

(Source: Frantisek *et al.*, 2005)

2.1.1 Drawback of Ordinary Portland Cement (OPC)

The Ordinary Portland Cement (OPC) concrete is widely used in constructional field and it will continue to grow as the result of continuous urban development all over the world. Global cement demand keep increasing as times move on from 2005 to 2010 as developing country likes Malaysia requires large quantity of cement because of the needs for infrastructure to cope with high growth phase. However, the production of OPC concrete inherent some disadvantages to the environment in term of carbon dioxide emission that led to global climate changes.

There are two major drawbacks in production of OPC which are about 1.5 tonnes of raw material needed to produce every tonne of OPC while at the same time; one tonne of carbon dioxide (CO_2) will be released during the production (Anuar *et al.*, 2011). According to Aleem and Arumairaj (2012), the remaining of natural limestone in all over the world will deplete in 25-50 years. The contribution of OPC towards CO_2 emission is approximately 5%-7% all over the world. The major factors that contribute to the emission of CO_2 are calcination of limestone ($CaCO_3$) into the calcium oxide (CaO) and the combustion of fossil fuel (Sreevidyaet *et al*., 2012). Figure 2.2 shows the increasing of CO_2 emission in all over the world started from year 1930 until year 2000.

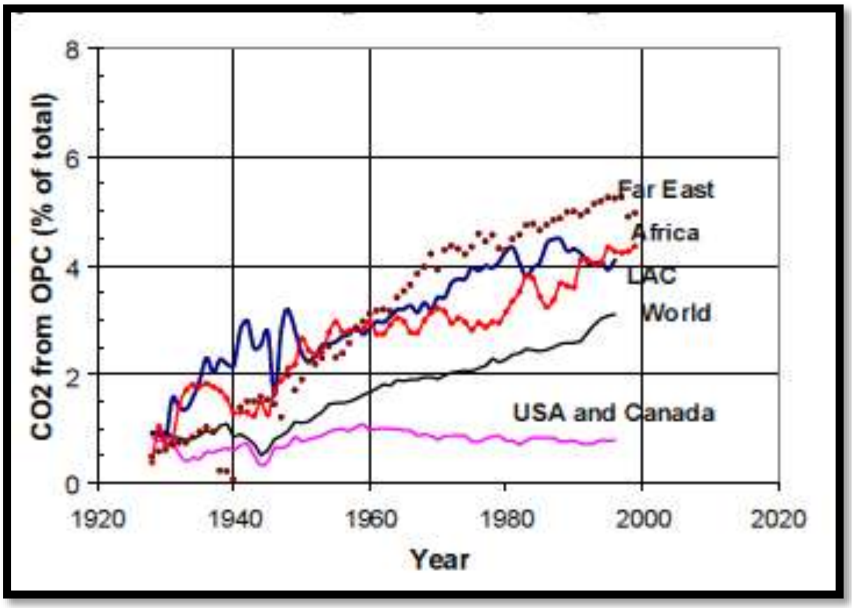


Figure 2.2: CO_2 emission from 1930 until 2000 (Source: Venderly, 2003)

CO_2 is released as a byproduct in the calcination process in a rotary kiln at temperature of 600-900^{o}C to induce a series of complex chemical reaction. Besides that, it also requires extremely resource and huge energy for combustion process. The **Equation 1** shows the calcination of $CaCO_3$ into the CaO and CO_2.

$$\boxed{CaCO_3\,(s) + \text{heat} \longrightarrow CaO\,(s) + CO_2\,(g)}$$

The microstructure of OPC is coarse stacking of grains matter which resulted possibility of crack and low compressive strength of OPC. The presence of water is very harmful in OPC production due to its ability to cause leaching compound in cement paste. Water will increase the possibility of hydration reaction in OPC, producing calcium silicate hydrate and calcium hydroxide which resulted low resistance to heat and chemical attack (Aleem and Arumairaj, 2012). Water carries harmful dissolve species like acid (chloride or sulphate) into the concrete that may cause deterioration of concrete structure by provoking expansion and cracking.

Nurhanie *et al.* (2012), states that the ability of water to form large pores in the cement paste may cause leaching of compound from concrete. Figure 2.3 shows the microstructure of Portland cement.

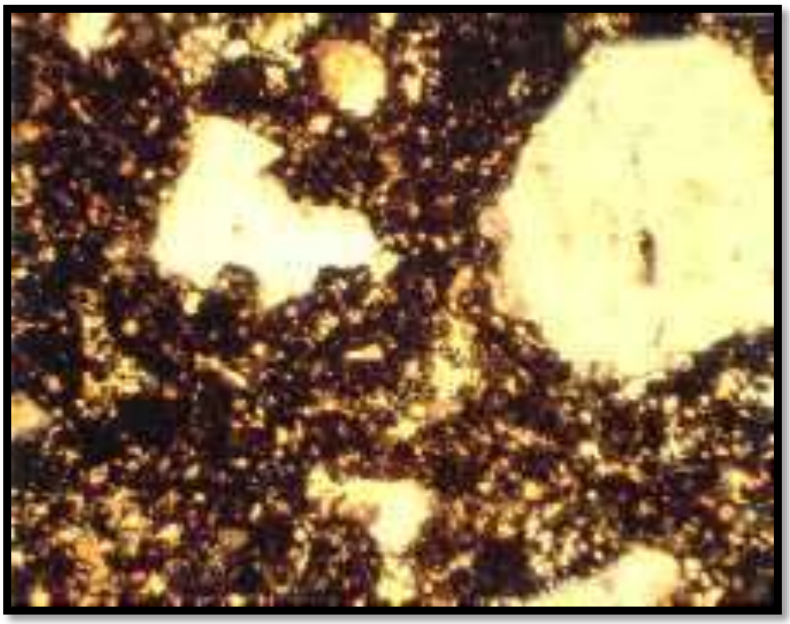


Figure 2.3: The structure of Ordinary Portland Cement (Source: Geopolymer's Institute, from: http://www.geopolymer.org/)

Thus, the introduction of geopolymer concrete in the constructional field is very important to reduce the impact of OPC towards the environment.

2.1.2 Necessity of Geopolymer

The geopolymer technique was first developed in 1978 by French Professor Davidovits to be applied for preparation of monolithic materials from reaction of metakaolin with reactive silica with strong alkaline like potassium hydroxide (He, Jia, Wang and Zhou, 2010). Then it was further developed into geopolymeric binder due to its ability as inorganic molecule to transform and undergo polymerization at low temperature (Davidovits *et al.*, 2002). Since last decade, geopolymer are very important in industrial area especially in civil engineering after several researches has been done regarding to its chemical and physical properties. Furthermore, it becomes well-known due to their low cost, excellent mechanical and physical properties, low energy consumption as well as reducing "greenhouse emission" during elaboration process (Muniz-Villareal *et al.*, 2010). However, the ultimate structure of the geopolymer is largely dependent on the ratio of Si to Al (Si: Al). The Table 2.1 shows the application of geometric material based on Si:Al ratio.

Table 2.1: Application of Geometric Material Based on Si:Al Ratio (Source: Davidovits, 1999)

Si:Al ratio	Applications
1	Bricks, Ceramics, Fire protection
2	Low CO_2 cements and concretes Radioactive and toxic waste encapsulation
3	Fire protection fibre glass composite, Foundry equipments, Heat resistance composite from 200°C to 1000°C, Tooling for aeronautics titanium process.
>3	Sealant for industry, 200°C to 600°C, Tooling for aeronautics SPF aluminium.
20-35	Fire resistant and heat resistance fibre composites.

The main impact of geopolymer as green materials can be seen in the construction field by replacing the Portland cement. The conventional production of cement in large scale will generate carbon dioxide (CO_2) which can lead to the global climate. Geopolymer development also offers an incredible solution to the environmental issue posed by the industries by utilizing the thermal industrial waste in the form of fly ash and also uses the waste water from chemical industry in geopolymer concrete production. Considering the fact that geopolymer concrete does not need any cement, reduction in cement consumption is expected thus solving the potential issue of limestone depletion in the future (Aleem and Arumairaj, 2012).

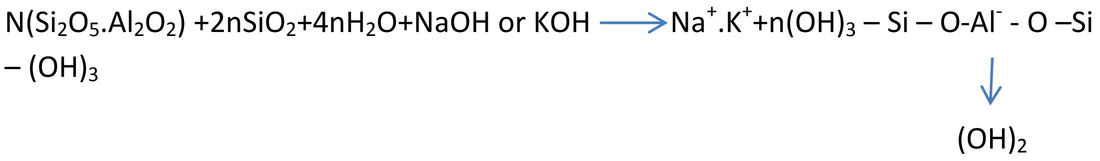
Figure 2.4: Snapshot of geopolymer concrete (Source: Geopolymer House, from: http://geopolymerhouses.wordpress.com/page/2/)

2.1.3 Geopolymerization Process

Geopolymerization is the process of combining many of small molecules (oligomers) into large covalent bond network. Professor B. Vijaya Rangan (2008) from Curtin University, Australia stated that, “the polymerization process involves a substantially fast chemical reaction under alkaline conditions on silicon-aluminium minerals that results in a three-dimensional polymeric chain and ring structure...”. The empirical formula of geopolymer is $M_n\,[-(SiO_2)_z\text{-}AlO_2]_n.wH_2O$ where ***z*** is 1, 2 or 3; *M* is an alkali cation and *n* is degree of polymerization (Bakharev *et al*., 2004).

Aleem and Arumairaj (2012) presented the schematic formation of geopolymer material through the **Equation 2** and **Equation 3**.

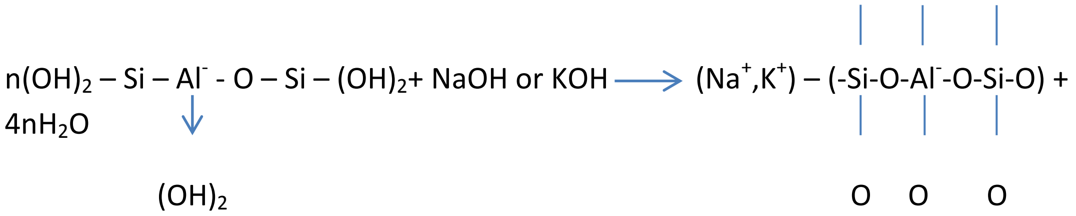
Equation 2



$$N(Si_2O_5.Al_2O_2) + 2nSiO_2 + 4nH_2O + NaOH \text{ or } KOH \longrightarrow Na^+.K^+ + n(OH)_3 - Si - O\text{-}\underset{\substack{\downarrow\\(OH)_2}}{Al^-} - O - Si - (OH)_3$$

(Geopolymer Precursor)

Equation 3



$$n(OH)_2 - Si - \underset{\substack{\downarrow\\(OH)_2}}{Al^-} - O - Si - (OH)_2 + NaOH \text{ or } KOH \longrightarrow (Na^+,K^+) - (\text{-}\overset{|}{\underset{\substack{|\\O}}{Si}}\text{-}O\text{-}\overset{|}{\underset{\substack{|\\O}}{Al^-}}\text{-}O\text{-}\overset{|}{\underset{\substack{|\\O}}{Si}}\text{-}O) + 4nH_2O$$

(Geopolymer Backbone)

Fundamentally, the geopolymerization process can be divided into three phase starting from deconstruction (dissolution of material in alkaline solution), then it continued with polymerization of alumina/silica-hydroxy species and oligomers and the final phase is stabilization where small gels are formed and transform into large networks through reorganization (Muniz-Villareal *et al*., 2012). Figure 2.5 shows the phases of geopolymerization process.

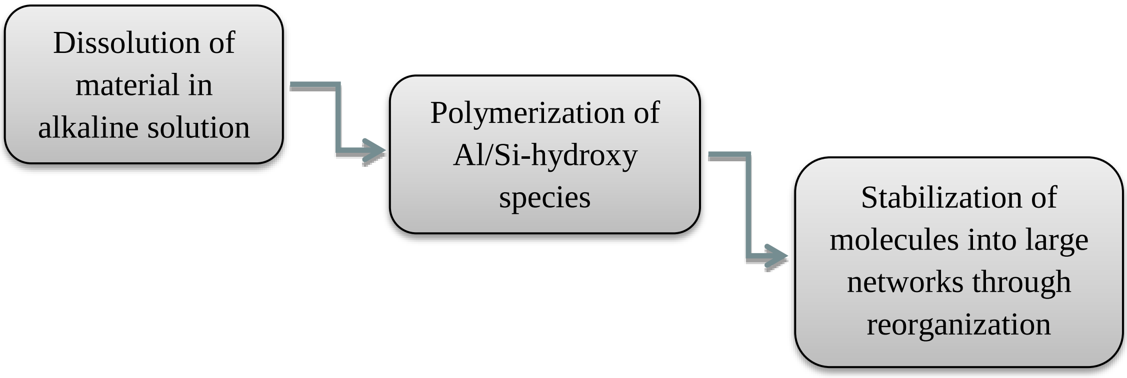


Figure 2.5: Step mechanism of geopolymerization process (Yao, 2009)

A geopolymer can be presented through one of these three basic forms (Chanh *et al.*, 2008) as shown in Figure 2.6 while Figure 2.7 show the basic polymerization of monomers in geopolymerization process.

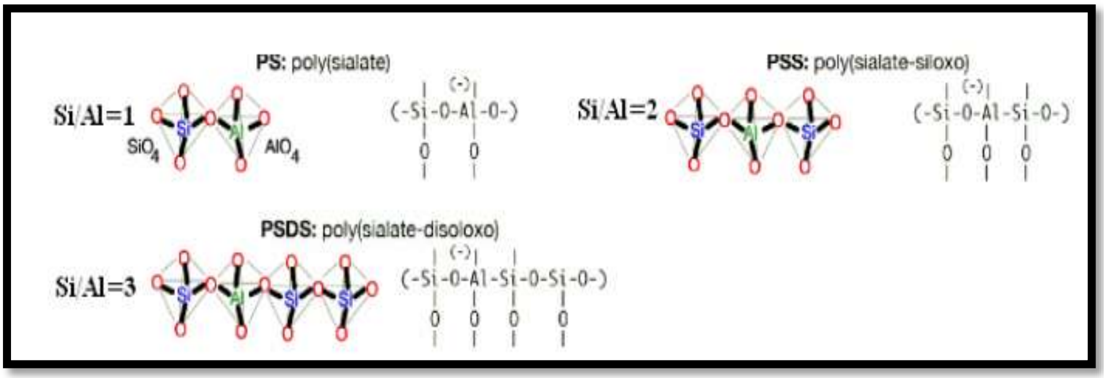


Figure 2.6: Three (3) basic forms of geopolymer (Chanh *et al.*, 2008)

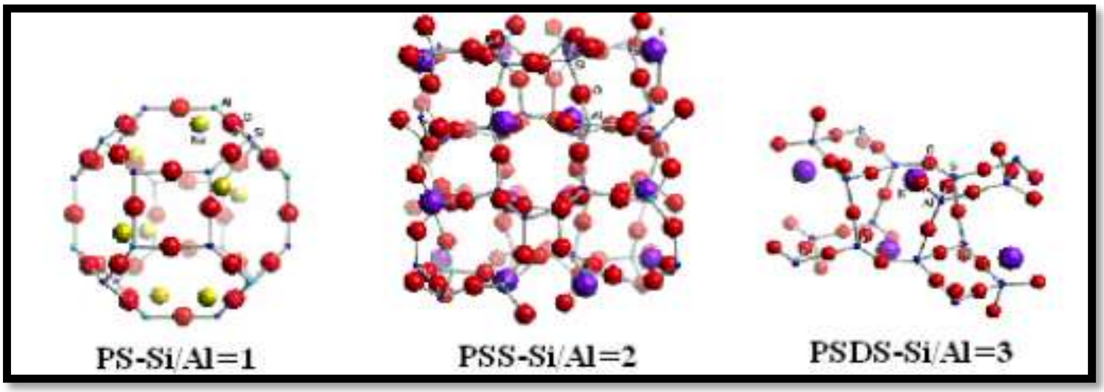


Figure 2.7: Polymerization of monomers (Chanh *et al.*, 2008)

The polymerization of monomers as shown in Figure 2.7 is called polysialates. According to Barbosa and MacKenzie (2002), polysialates are based on alumino silicate units such as sialate siloxo (-Si-O-Al-O-Si-O-) and sialate disiloxo (-Si-O-

Al-O-Si-O-Si-O-). The term of sialate (-Si-O-Al-O-) stands for silicon-oxo-aluminate building unit where it consists of SiO_4 and AlO_4 tetrahedra linked by sharing all oxygen atoms. However, the presence of positive ion (K+, Na+, Ca+…) is important to balance the negative charge of Al in 4-fold coordination. Formation of chain and ring polymers will appear in cross-linked coordination through a sialate -Si-O-Al- bridge (Mustafa Al Bakri *et al.*, 2011). The microstructure for polysialate (geopolymer) is X-ray amorphous in three dimensional silico-aluminate structures.

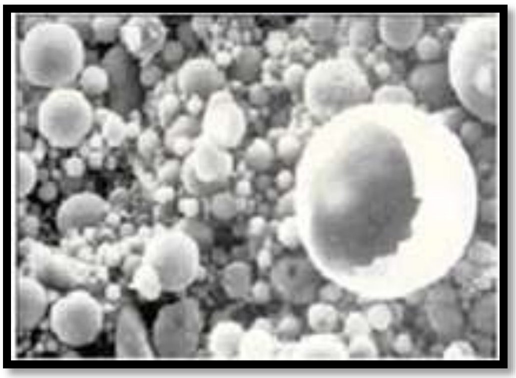


Figure 2.8: Fly ash

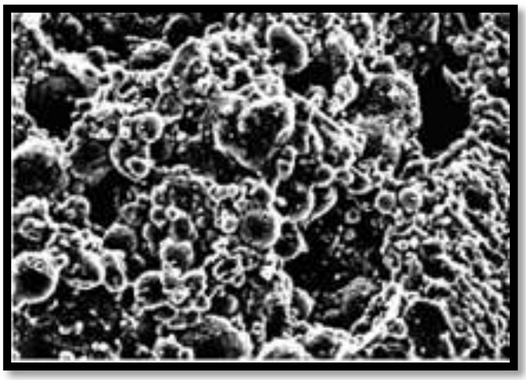


Figure 2.9: Fly ash with NaOH

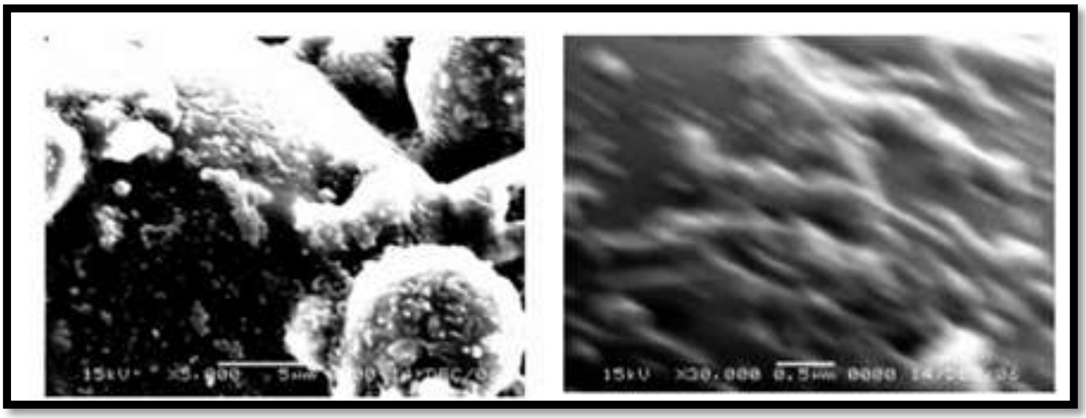


Figure 2.10: Fly ash after reaction with NaOH (x5000 and x30000)

(Source: Chanh *et al.*, 2008)

Figure 2.8, Figure 2.9 and Figure 2.10 show the microscopic reaction of fly ash-based geopolymer. Figure 2.8 shows the particles of fly ash before the reaction of NaOH while Figure 2.9 and Figure 2.10 display the particles of fly ash reacting with NaOH. These figures show that the particles of fly ash are really reactive with particles of alkaline solution. As a result of this geopolymerization process along with good ratio of Si:Al ratio, strong geopolymer can be produced.

2.2 Solidification of Geopolymer

Solidification phase in the geopolymerization process plays an important role in the determination of geopolymer's chemical and physical properties. Since the synthesis of geopolymerization processes almost the same with zeolite especially regarding to its chemical composition, the key different difference is the microstructure of geopolymer is X-ray amorphous in room temperature while zeolite are well-developed crystalline structure (De Silva *et al.*, 2009). However, this type structure (X-ray amorphous) makes the synthesis of geopolymer are quite challenging and the stability of geopolymer phase in respect to solidification is an interesting field to be explored. The main focus of this research is the solidification of geopolymer based on several parameters:

I. Effect of different types of alkaline activator on the solidification of geopolymer;
II. Effect of different concentrations of alkaline solution on the solidification of geopolymer;
III. Effect of different curing temperature on the solidification of geopolymer.

2.2.1 Effect of Different Types of Alkaline Activator

Geopolymerization process requires two main components which are materials rich with alumina-silica contents and the alkaline solution (Yudthana *et al.*, 2009). The basic alkaline solutions used in the production of geopolymer are sodium hydroxide (NaOH) and potassium hydroxide (KOH) while alkali silicates used are potassium silicate (K_2O_3Si) and sodium silicate (Na_2O_3Si). The alkaline solution acting as activator to highly reactive materials (MK or fly ash) and subsequently polymerizes in molecular chain and become the binder (Aleem and Arumairaj, 2012).

The main function of alkaline activator in the geopolymerization process is a tool to dissolute of materials (MK or fly ash) at the early stage of process. The presence of OH^- anions in the alkaline solution will attack the Si-O and Al-O bonds during dissolution phase (Yao *et al.*, 2009). Furthermore, the types of alkaline activator are very important in the reaction rate of geopolymer. The rate of geopolymerization in the potassium hydroxide solution is slightly higher than sodium hydroxide due to slight different in chemical characteristic of both caustic bases. The reaction of KOH and NaOH with water is strongly exothermic that will produce hydrogen and heat but KOH solution is slightly less exothermic. Shelly Morgan (2011) reported that potassium hydroxide solution is more soluble in water compared to sodium hydroxide as 121g of KOH will dissolve in 100ml of water while 100g of NaOH is needed to dissolve same amount of water. This different of characteristic might be decisive factors that affected the result of setting in geopolymer.

The reaction rate of alkaline activator that contained soluble silicate is higher than reaction rate of hydroxides alkaline (Hardjito *et al*., 2005). This fact was proven five (5) years later by Yao (2009) through the experiment; where the study states that the potassium silicate solutions have better activation efficiency compared to potassium hydroxide solution even with higher alkali content. Figure 2.11 and Figure 2.12 presented perfect examples regarding the effect of two different alkaline solutions on the geopolymerization process. Since the reaction in dissolution phase is exothermic, the higher heat evolution means the higher rate of reaction of geopolymerization process. From both figures, it clearly shows that heat evolution in the reaction of potassium silicate solution is higher than heat evolution in reaction of potassium hydroxide solution (Yao *et al*., 2009).

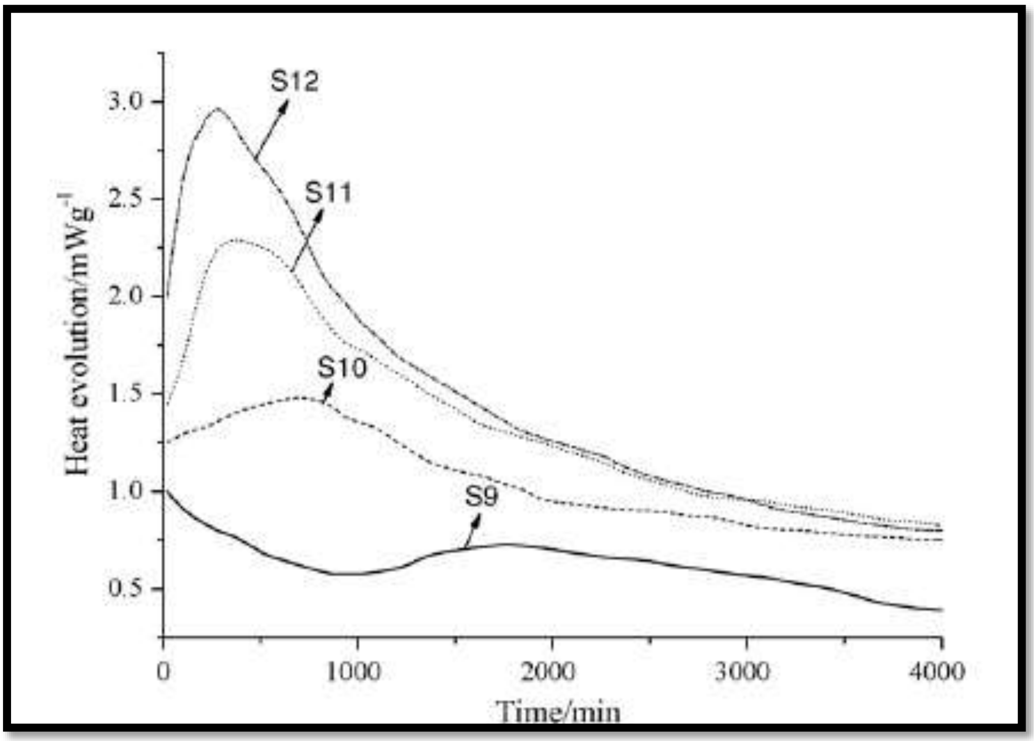


Figure 2.11: Effect of KOH solution in MK-based geopolymerization

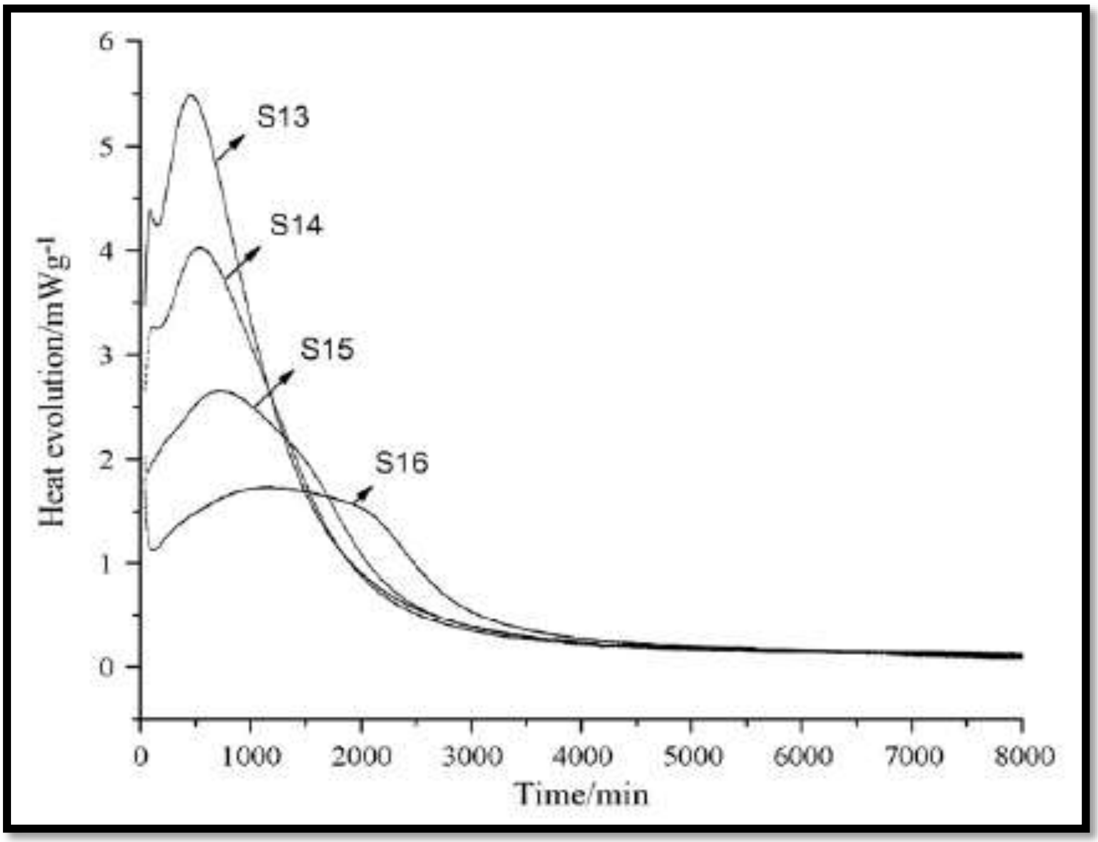


Figure 2.12: Effect of modulus K-water glass on MK-based geopolymerization (Source: Yao *et al*., 2009)

The compressive strength of geopolymer produced largely dependent on the presence of alkaline solution in the geopolymerization process. More dissolution of Si-O and Al-O bonds by alkaline activator in geopolymerization process will result in faster setting time for geopolymer to solidify. According to Mustafa Al Bakri *et al*. (2011), increasing the waterglass content in the alkaline activator significantly increased the geopolymerization rate and subsequently provides better compressive strength compared to NaOH activator. The increasing of waterglass or silicate solution in NaOH concentration helps in providing extra SiO_2 species in the solution which leading to an increase in the ratio of SiO_2/Al_2O_3, thus more formation of Si-O-Si bonds are formed. As the result of this matter, stronger geopolymer will be produced (Mustafa Al Bakri *et al*., 2011). Normally, the faster setting time for geopolymer to solidify will successively produce a higher compressive strength of geopolymer if all the critical factors are met.

2.2.2 Effect of Different Concentrations of Alkaline Solution

The effect of concentration in the alkaline solution to the mechanical properties of geopolymer is well-documented (Yao *et al*., 2009). Different concentration of alkaline solution will produce huge different in mechanical properties of geopolymer's produced especially in term of compressive strength. Compressive strength measurement is used as a tool to determine the quality of geopolymer produced. With respect to this fact, several studies and researches have been done for past decade. Hardjito and Rangan (2005) observed that the higher concentration (molar) of sodium hydroxide (NaOH) solution will produced higher compressive strength of geopolymer. Table 2.2 below was presented by Hardjito and Rangan to demonstrate the compressive strength of geopolymer produced after seven (7) days.

Table 2.2: Effect of concentration to the compressive strength of geopolymer

Mixture	Concentration of NaOH solution (M)	Sodium Silicate/ NaOH ratio by mass	7-day compressive strength after curing at 60°C for 24h (MPa)
1	8	0.4	17.3
2	8	2.5	56.8
3	14	0.4	47.9
4	14	2.5	67.6

From data presented in the Table 2.2, it clearly shows that the strength of geopolymer is largely dependent on the concentration of alkaline solution. Alkaline concentration plays a significant factor in controlling the leaching of alumina and silica from materials during dissolution phase; subsequently affect the mechanical properties of geopolymer and setting time of geopolymer. Ubolluk and Prinya (2009) supported the idea effect of concentration alkaline activator by running the experiment to study the influent of concentration of NaOH on the synthesis of fly ash based geopolymer. The experiment was conducted by varies different concentrations of NaOH at 5M, 10M and 15M towards fly ash as material that rich with alumina-silica content.

The result of the experiment is demonstrated in the Figure 2.13 and Figure 2.14. From both figures, the concentration of NaOH at 10M is the maximum due to high base of OH^- for Al^{3+} and Si^{4+} leaching process and at 5M, the dissolution was low due to low base condition. However, even the concentration of 15M shows dissolution is below than 10M, it is due to the coagulation of silica during the process (Bergna and Roberts, 2006).

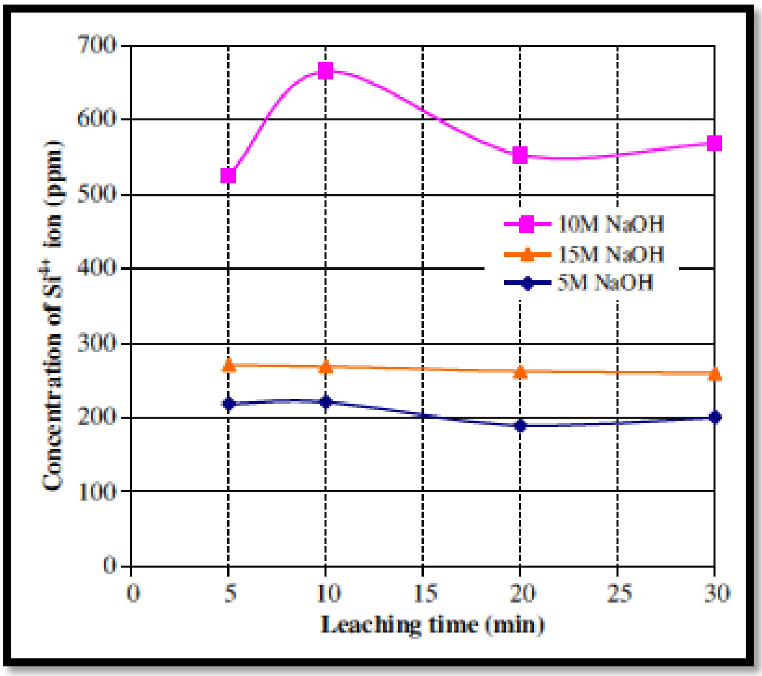


Figure 2.13: Si^{4+} ion concentration with fly ash/NaOH 3:1 in 5M, 10M and 15M

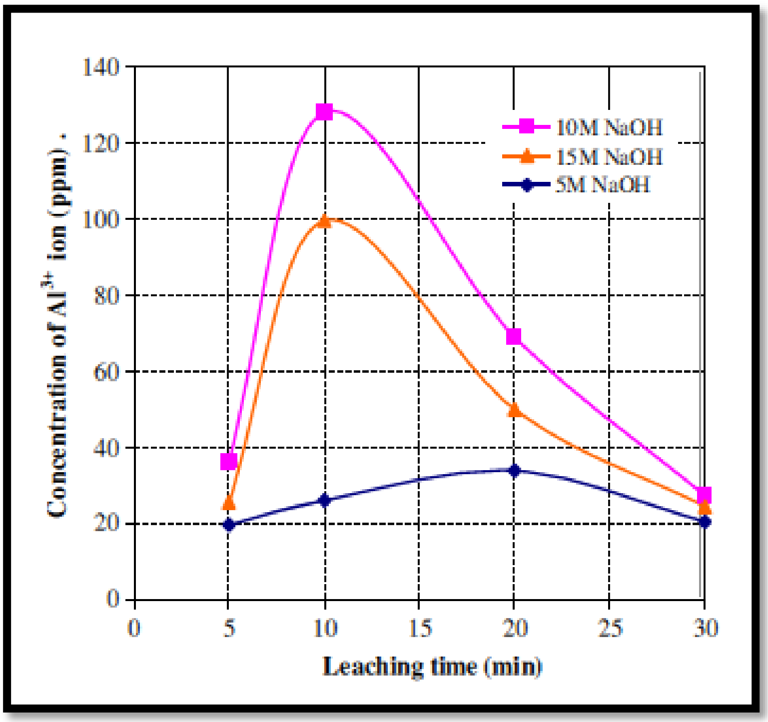


Figure 2.14: Al^{3+} ion concentration with FA/ NaOH 3:1 in 5M, 10M and 15M

This experiment also shows the formation of crystal during ‘solid’ gel phase under the Scanning Electron Microscope (SEM) as shown in Figure 2.15.

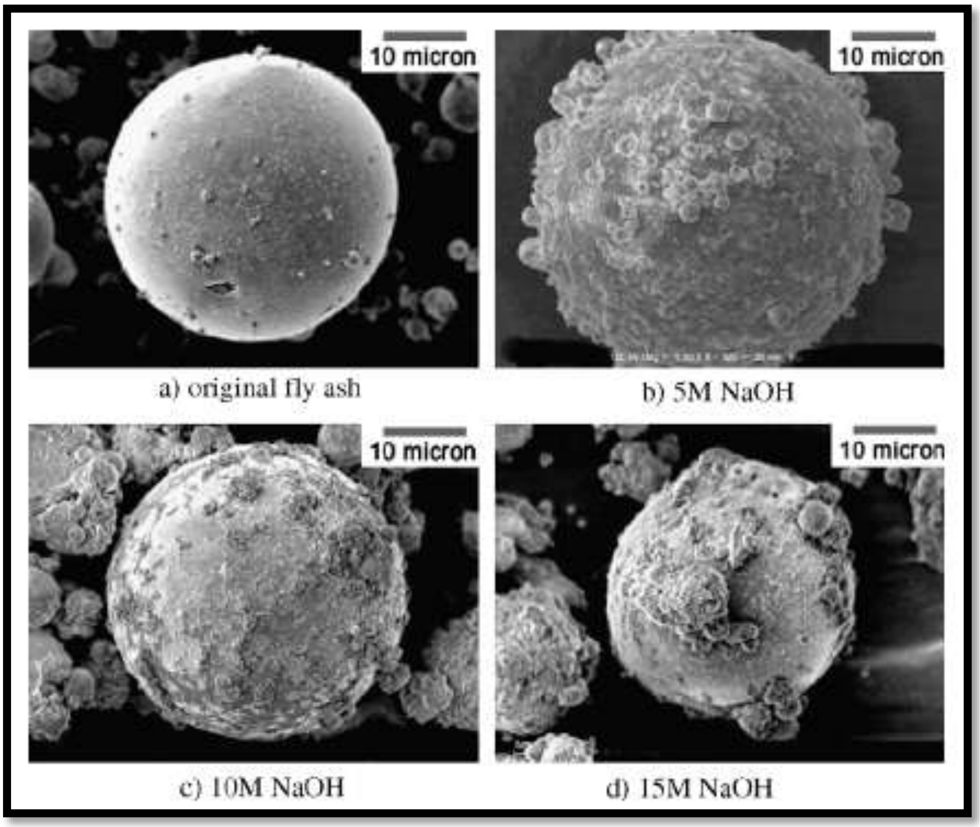


Figure 2.15: Geopolymer’s crystal (Source: Ubolluk and Prinya, 2009)

Figure 2.15 displays a perfect example of solidification of geopolymer affecting by concentration of alkaline solution. SEM shows clearly the difference formation of crystal caused by different concentrations of alkaline solution. The difference of concentration can be seen in the figure of 5M, the formation of spherical units of gel are formed at fly ash particles whereas for 10M of concentration of NaOH, small spherical units of gel merged and formed larger mass of new gel.

Ubolluk and Prinya (2009) also explained the formation of new gel had two opposing effects. First, the depletion of ions will increase further leaching ion from fly ash particle and secondly, it will thickening the solution resulted to the lower immobility of solution. Kamarudin (2011) continuing the study of influence of alkaline solution to the solidification of metakaolin based geopolymer. The result of study can be seen in Figure 2.16. The result and explanation of this experiment are almost similar as presented by Obolluk and Prinya but different between in concentration of NaOH tested.

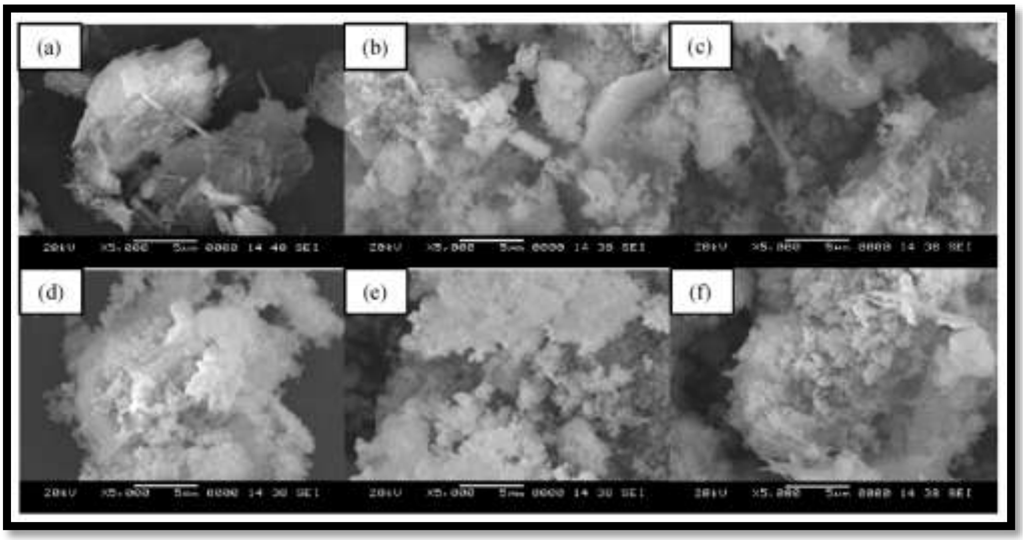


Figure 2.16: SEM micrograph of kaolin (a), geopolymer synthesis using different NaOH concentration of 6M (b), 8M (c), 10M (d), 12M (e), 14M (f).

2.2.3 Effect of Different Curing Temperature

Despite the effect of alkaline solution, the effect of curing temperature also is one of the major factors that will determine the setting time and subsequently the compressive strength of geopolymer (Chanh *et al*., 2008). However, there are only few reports studied about the relationship between synthesis of temperature and kinetics of geopolymerization and its influence to the physical and mechanical properties (Muniz-Villarreal *et al*., 2010). According to Wang and Cheng (2003), the increasing of curing temperature will increase the rate of geopolymerization process.

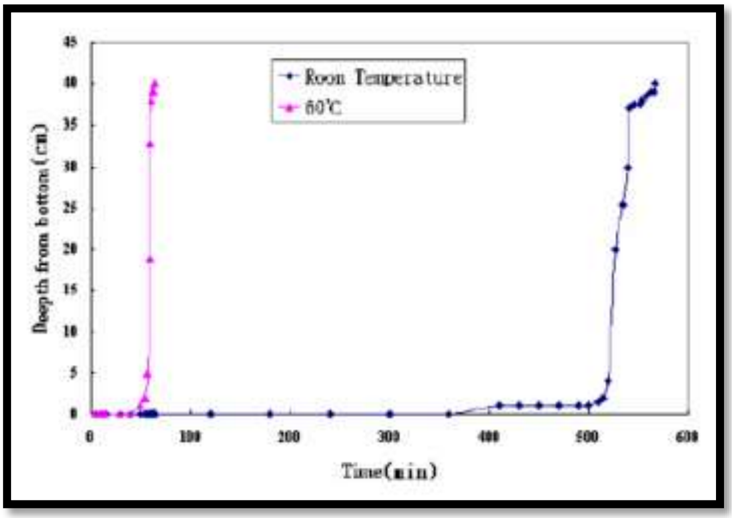


Figure 2.17: Effect of temperature on setting time (Source: Wang and Cheng, 2003)

Figure 2.17 shows the effect of curing temperature (at room temperature and 60^{o}C) on setting time of geopolymerization process. The difference between two temperatures is quite obvious that geopolymer's setting time at 60^{o}C is faster than setting time at room temperature (26^{o}C). The setting time for the geopolymer to solidify at the temperature of 60^{o}C is about 60 minutes while at the room temperature (26^{o}C) is 9.5 hours. The reason behind this difference is water loss at 60^{o}C increasing the setting time for geopolymer to solidify. Muniz-Villarreal *et al.* (2009) proved this fact in their study of influence of temperature on metakaolin-based geopolymer; it is observed that the best geopolymerization process occurred at the optimum curing temperature of 60^{o}C. Figure 2.18 shows the effect of curing temperature (at room temperature and 60^{o}C) on the compressive strength where the compressive strength of geopolymer at 60^{o}C is higher than at room temperature.

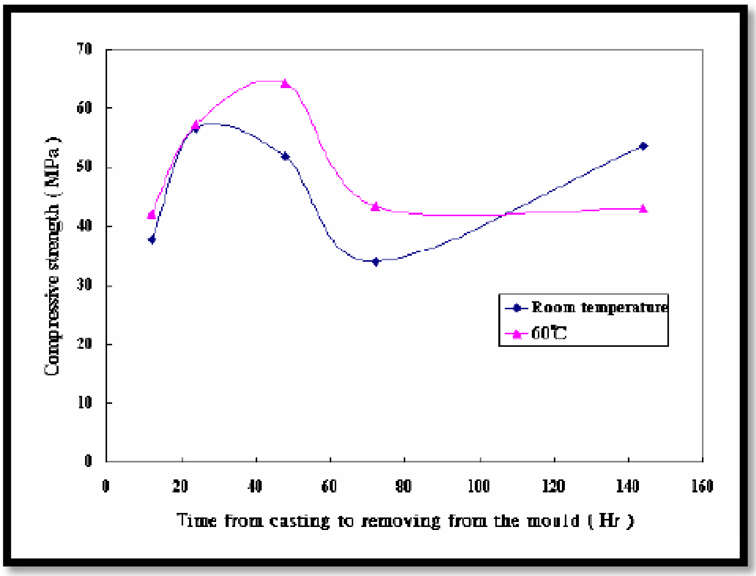


Figure 2.18: Effect of temperature on geopolymer strength

(Source: Wang and Cheng, 2003)

The difference in compressive strength of geopolymer due to the effect of temperature curing can be explained through the factor of porosity distribution. Muniz-Villarreal states that the compressive strength of geopolymer is strongly dependent on the size of porosity during geopolymerization process. Based on their experiment at the optimum temperature of 60^{o}C, the percentage of porosity is low compared to low temperature (30^{o}C) and high temperature (90^{o}C) and resulting to highest compressive strength of geopolymer.

2.3 Avrami's Kinetic Theory

The theory of kinetic transformation commonly described based on Johnson-Mehl-Avrami-Kolgmogoroff (JMAK) phenomenological model (Fanfoni *et al.*, 1996). This theory also known as Avrami's Kinetic Theory functioning to demonstrate the isothermal solid transformation from one phase to another phase by nucleation. Basically, the kinetic transformation of solid is closely related to the nucleation of new particles and growth rate of particles into spherical shaped. This theory can specifically describe the kinetics of solidification and the rate of transformation usually follows the characteristic of s-shaped or sigmoidal.

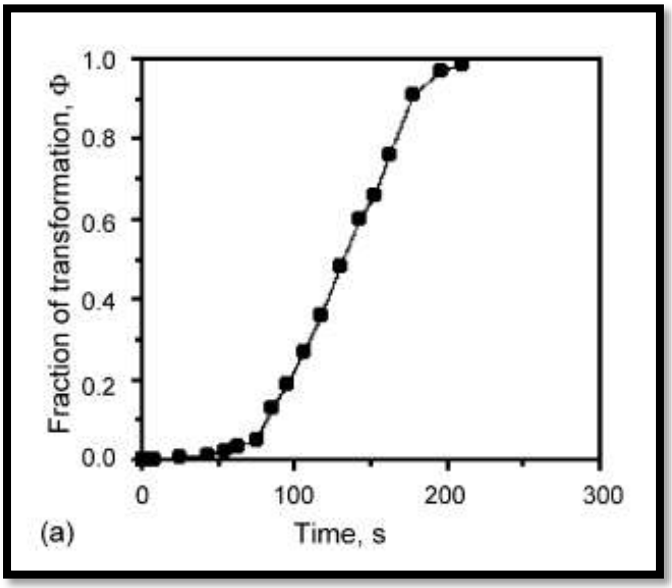


Figure 2.19: The rate of solidification of NiTi thin film at 495°C.

Based on Figure 2.19, the curves represent the solidification process of NiTi thin film at 495°C presented by Ramirez *et al.* (2006). It is found that the transformation rate of solid is slow at the beginning, thus the initial curve has low tangential gradient while the same observation also can be made at the final curve. Those rates are slow because the time needed for number of nuclei of the new phase to form and begin growing is considerably long. However, the transformation of the crystal is rapid at the intermediate period, as shown by the steep curve in the middle region of the graph. The nuclei growth into particles and consumption of the old phase while nuclei continue to form the remaining parent phase are the reasons for the significant rise in the solidification rate. Moving on to the final phase, the process is slow and takes relatively longer time due to these two reasons; the untransformed material where the nuclei could form in is less and the existing particles are in contact with each other, creating a boundary which inhibit the growth of the crystal particles.

Referring to Jiao Yang and Benjamin J. McCoy (2005), the equation for bulk solidification of polymers can be described as

$$\boxed{1\text{-}X = e^{-Vt}}$$

Equation 4

Where X is the degree of solidification and V_t is the volume of solidification material. Conversely, for the case of sporadic nucleation, the Avrami's equation can be modified and presented as

$$\boxed{1\text{-}X = \mathrm{e}^{-\mathrm{Ktn}}}$$

Equation 5

Where K is the growth rate and n is Avrami's exponent. The Avrami's exponent (n) is sensitive to the dimensionality of growth as well as time-dependence of nucleation and growth (Ramirez *et al*., 2006). However, Lukman *et al*. (2008) states that the degree of crystallinity is first measured by geopolymer deposition (δ_r). Deposition of geopolymer is the mass fractions of the crystal deposited on the wall during the cooling process. This statement can be expressed as

$$\boxed{\partial r = \frac{\partial t - \partial \mathrm{o}}{\partial\infty - \partial o}}$$

Equation 6

δt - deposition at time (min)

δ∞ - maximum or asymptotic deposition from deposition curve

δ0 - initial mass of geopolymer content in liquid (g)

Replacing the X in the Equation 5 with δ_r in Equation 6, and taking log twice in Equation 5 can be written as

$$\boxed{\mathbf{\log\,[-\ln(1-\delta_r)] = \log \mathit{K} + n \log (\mathit{t})}}$$

Equation 7

Equation 7 represents the equation of straight line of Y= mX+c if the graph of log [-ln(1- δ_r)] vs. log (t) is plotted. The gradient of the straight line in the graph represents the value of Avrami's exponent (n) while y-intercept of the graph symbolizes growth value (K) of geopolymer's particle. The graph of log [-ln(1- δ_r)] vs log (t) can be represented in Figure 2.20.

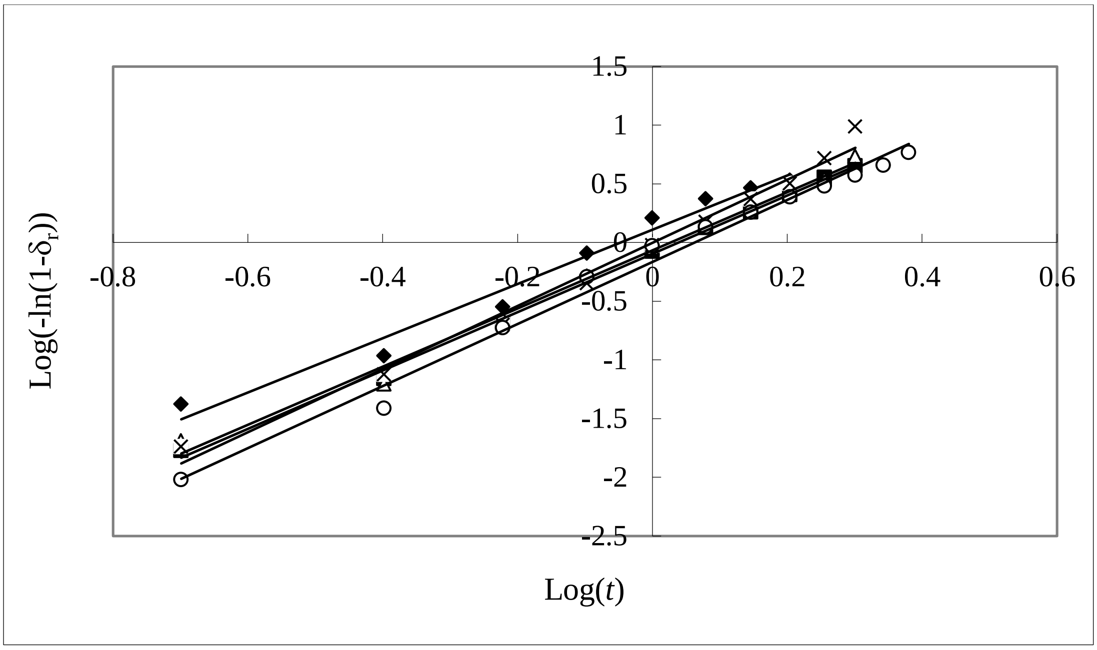
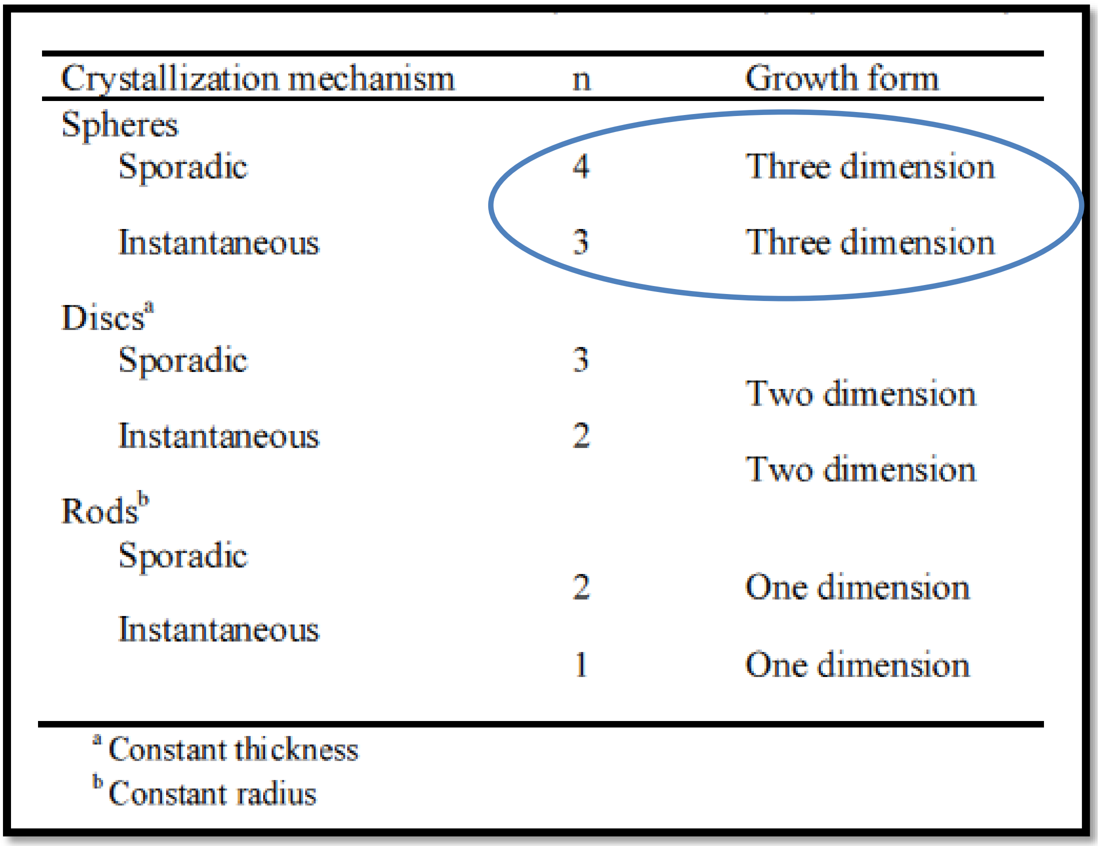


Figure 2.20: log [-ln(1- δr)] vs log (t) (Source: Ismail *et al.,* 2008)

Modified Avrami's Kinetic Theory specifically chosen in order to determine the microstructure of geopolymer where it can be decided through the value of Avrami's exponent (*n*) in the equation. Figure 2.21 shows the Avrami's parameter for solidification of polymer by Ismail *et al.* (2006). According to van Deventer (2007), the microstructure's type of geopolymer is ranging from X-ray amorphous until semi-crystalline phase in three dimensional solico-aluminate structures. Thus, in order to be feasible to this theory, the range of value of Avrami's exponent (*n*) should be in between three (3) and four (4). This modified equation also used to show the nucleation's growth of crystal during geopolymerization process. The growth of particles is described through the value of *K*, which is the y-intercept in the graph of log [-ln(1- δr)] vs. log (t). It specifically described the dimensional growth of the crystal formation during geopolymerization process.



Crystallization mechanism	n	Growth form
Spheres		
Sporadic	4	Three dimension
Instantaneous	3	Three dimension
Discs[a]		
Sporadic	3	Two dimension
Instantaneous	2	Two dimension
Rods[b]		
Sporadic	2	One dimension
Instantaneous	1	One dimension

[a] Constant thickness

[b] Constant radius

Figure 2.21: Avrami's parameter for crystallization of polymer

(Source: Ismail *et al.*, 2008)

2.4 LFRA Texture Analyzer

The LFRA Texture Analyzer is the industry standard instrument for gelatine Bloom assessment (AOAC Standard Reference). This instrument was developed by the Leatherhead Food Research Association (LFRA) in England and widely well known as a part of Universal Testing Machine (UTM) (Bourne *et al*., 1982). LFRA Texture Analyzer uses a load cell that is fitted to a travelling beam, where various kinds of test probes and accessories are mounted. It applies mechanical energy to the sample via the controlled movement. Then, based on the geometry of the probes, the resultant forces generated will vary into compression, puncture, extrusion, snapping, shear and many others. This analysis will yield either one of these two; correlation between the condition imposed with the sample response which can be obtained using sensory panel or the key functional properties which include break point, hardness, spreadability and ripeness (Hellyer *et al*., 2004).

One of the examples of the test is extrusion test. This test applying mechanical force to the sample until it flows throughout the holes, slots and annular spaces. The mechanism of extrusion test can be represented in the Figure 2.22.

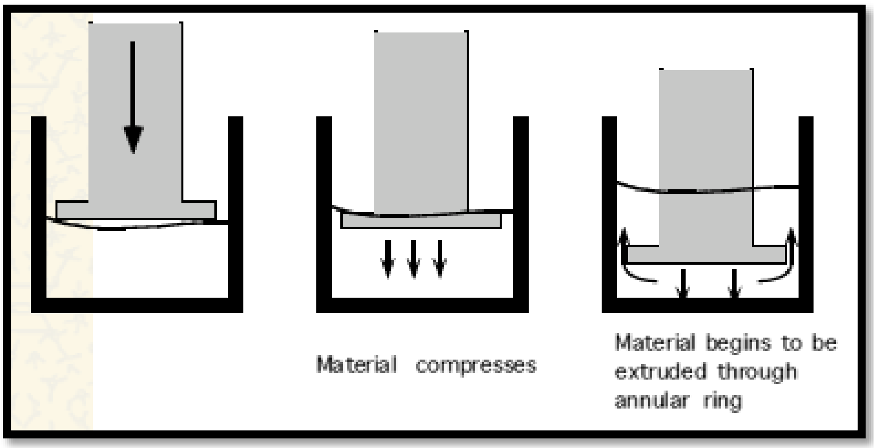


Figure 2.22: Simple cell extrusion test (Source: Hellyer *et al*., 2004)

First figure shows the plunger goes down and began to contact the surface of test sample. Next, a mechanical force is applied to the test sample resulting some liquid might be squeezed out from material compressed. Lastly, third figure displays the material is extruded through annular ring where the strength of material can be determined. In the extrusion test, maximum force that has been applied to extrude the material is measured as firmness or tenderness or material.

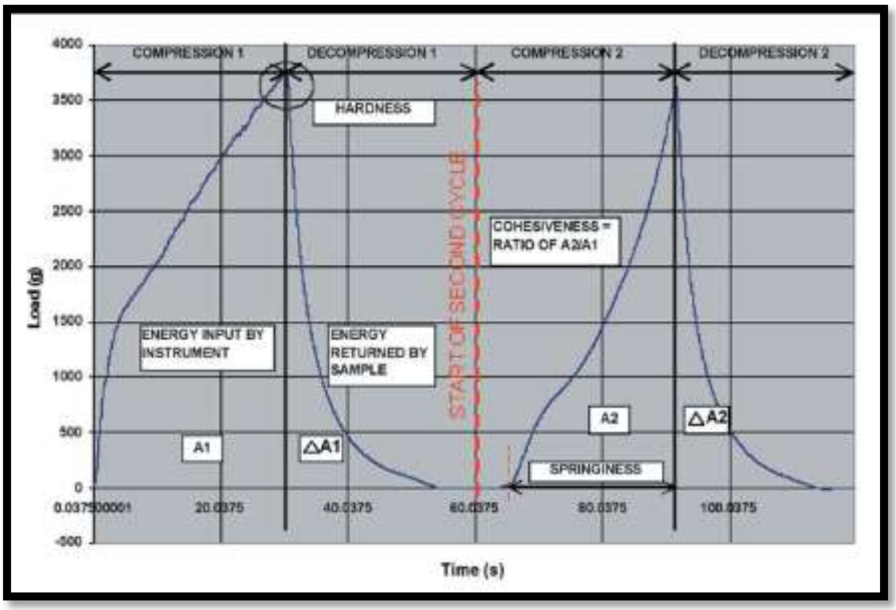


Figure 2.23: Typical Texture Profile Analysis (TPA) trace from large scale deformation of bread crumb (Source: Hellyer *et al*., 2004)

Figure 2.23 illustrates texture profile of white bread as indicator of freshness and ingredient functionality in the finished loaf. Figure shows the hardness of sample tested can be identified on the force peak on the first compression while the cohesiveness can be acknowledged from the ratio of positive force area under the first and second compression (A2/A1). On the other hand, the springiness of sample is shown by the force area of second compression (A2). Pathiraj *et al*. (2012) states that LFRA Texture Analyzer is a decent choice of testing machine to determine the compression test using cylinder to produce reproducible results at room temperature.

2.5 Vicat Needle

The most common apparatus used in determination of cement or plastic solidification is by using Vicat needle. The uses of Vicat needle also can be used in determining the setting time for geopolymer to harden from liquid into solid form. Normally, two test methods are given depending on material tested. First, Method A known as Reference Test Method whereby using manually operated standard Vicat apparatus while Method B permit the use of an automatic Vicat machine (International Standard Worldwide). The values stated in the Vicat measurement is in millimetre (mm) that shows the depth of penetration on the sample tested in order to determine the material's hardness as shown in Figure 2.24.

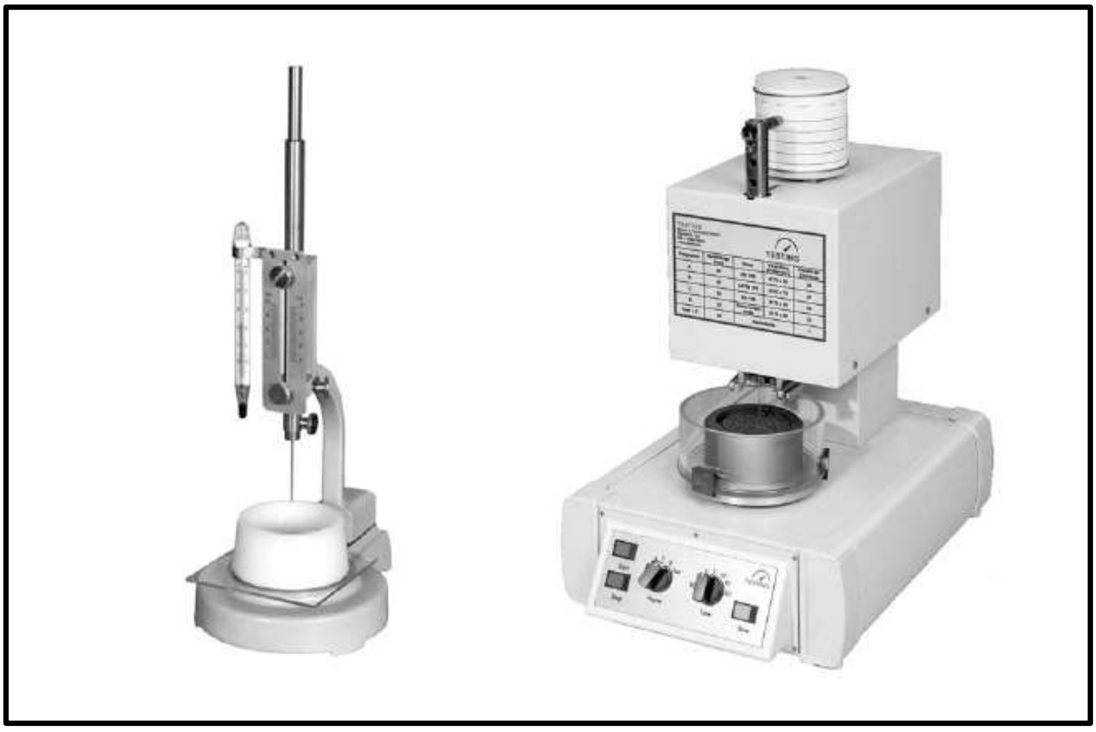


Figure 2.24: Manual (left) and automatic (right) Vicat apparatus

Vicat needle apparatus is used in geopolymer field in order to measure the setting time for solidification process from liquid to solid. Normally, the Vicat Needle ASTM C191-04 is used in testing the geopolymer's hardening with the needle's diameter of 1.00±0.05mm and 10.00±0.05mm. However, the best needle's diameter to be used in measuring the setting time of geopolymer's hardening is 1.00±0.05mm (Nath and Saker, 2012)

The working principle of Vicat needle is quite simple starting with material tested with the depth of 40 mm will be placed at the centre of the specimen under the 10mm end of Vicat needle and the movable rod is lowered until the needle end makes contact with the material phase. Next, the indicator of measurement is set at zero and movable rod will be allowed to free fall for penetration of needle. The depth of penetration will be recorded and repeated every five minutes until the material solidified. The solidification of material can be identified once the Vicat needle unable to penetrate the material (Hardjito, Cheak and Lee Ing, 2008).

2.6 X-Ray Diffraction (XRD)

X-Ray Diffraction or XRD is a non-destructive analytical method which is used to determine the atomic and molecular structure of a crystal where crystalline atoms causing a diffraction into many specific directions. Crystal structure can be considered as built of layers or planes, each acting as a semi-transparent mirror. When x-rays that have similar wavelength with the distances between those planes hit them, reflection of the rays will occur in such a manner that the angle of incidence is equal to angle of reflection. This phenomenon is called "diffraction and can be described by Bragg's Law.

However, in this research, XRD is used for phase analysis of the starting materials and to investigate the crystallinity of the resulting geopolymer. For the determining the phase analysis of the starting material (fly ash), the random powder sample of fly ash are prepared by lightly pressing powder samples into aluminium holders. The geopolymer characteristic can be investigated by taking resulting geopolymer in powder form or by cutting 0.5 mm thick slices of specimen and mounting in aluminium holder (Yudthana *et al*., 2009).

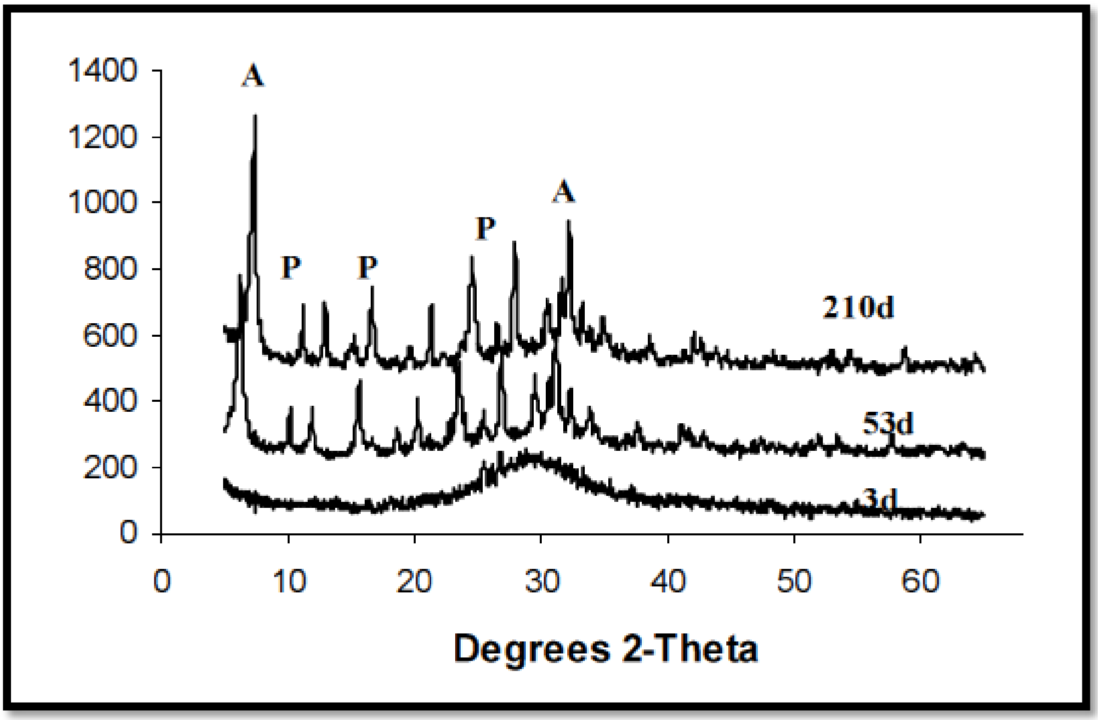


Figure 2.25: XRD pattern of geopolymer (De Silva and Sagoe-Crenstil, 2009)

A diffraction pattern records the X-ray intensity as a function of 2-theta angle. Figure 2.25 shows the sample of XRD pattern of geopolymer mixtures at different curing intervals presented by De-Silva and Sagoe-Crenstil (2009). The letter of *A* represents the microstructure of zeolite A while letter of *P* represent the microstructure of zeolite P. From this figure, XRD clearly shows that the different curing interval affecting the strength of geopolymer where longer curing of time will produce stronger geopolymer.

The analysis of diffraction pattern gives huge advantage of XRD compared to other method in determining chemical analysis of the samples. XRD helps in quantify each phase present, the crystallinity of sample, the crystal structure of the sample and its lattice parameters as well as size of crystal itself which is vital in determining material characterization and quality control.

Summary of the research works done in geopolymer field are as listed as follow:

Year	Reference	Title	Findings
2002	Davidovits et al.	30 years of Success in Geopolymer Application	• The origin of geopolymer founder in 1978. • The application of geopolymer in the construction field.
2003	Wang and Cheng	Geopolymer production materials by fly ash	• Increased in temperature lead to increase the setting time and the compressive strength of geopolymer. • At room temperature, time taken to harden is 9.5 hours while at 60°C is 1 hours
2004	Hadjito and Rangan	Development of FA-Based Geopolymer Concrete.	• Higher concentration of NaOH solution produced higher compressive strength. • Longer curing time, higher compressive strength.
2007	Provis and Devanter	Geopolymerization kinetic. 2. Reaction kinetic modelling	• Development of mathematical model that can be applied in the geopolymerization field.
2008	Chanh et al.	Recent research geopolymer concrete	• Geopolymer has excellent strength against acid and salt compared to Portland cement. • The microstructure of geopolymer is amorphous in standard condition.
2009	Yudthana et al.	The Role of NaOH concentration in FA Geopolymer.	• Class F FA has higher content amount of silica alumina. • The best ratio for FA: NaOH solution is 3:1. • Increasing in sodium silicate solution will increase the geopolymerization reaction.
2009	Yao and Zhang	Geopolymerization process of alkali-metakaolinite characterized by isothermal calorimetry.	• Potassium silicate solution has better activation efficiency than KOH solution even with higher concentration. • The presence of silicate solution added the silica content in geopolymer that resulting in higher strength.
2009	De Silva and Sagoe-Crenstil	Role of Al2O3, SiO2 and Na2O on the amorphous to solidification phase	• The structure of geopolymer in amorphous in standard condition but turn to crystalline in the presence of higher temperature.

2009	Ubollluk and Prinya	Influence of NaOH solution on the synthesis of fly ash geopolymer	• Leaching process depending on NaOH concentration and time. • Increase in concentration of NaOH solution will increase the geopolymerization process.
2011	Mustafa Al Bakri et al.	Mechanism and Chemical Reaction of FA Geopolymer	• The geopolymerization process occurs in 3 stages: Dissolution, polymerization and reorganization.
2011	Muniz-Vilarreal et al.	Effect of temperature on geopolymerization process of MK geopolymer	• The increase in temperature will increase geopolymerization process. • The best geopolymerization process occurred at $T=60^{o}C$.
2011	Anuar et al.	Strength characteristic of Geopolymer concrete	• Different concentration of NaOH s solution influences the strength of geopolymer produced.
2011	Mustafa Al Bakri et al.	The effect of alkaline activator on fly ash based geopolymer.	• Higher silica alumina ratio will increase the compressive strength of geopolymer. • The best silica-alumina ratio is 1.00.
2012	Aleem and Arumairaj	Optimum mix for the Geopolymer concrete	• Compressive strength of geopolymer increased with the increased of aggregates during reorganization phase. • This is due to the high bonding between aggregates and alkaline solution.
2012	Aleem and Arumairaj	Geopolymer concrete- review	• The curing temperature is the most important factor in determination of strength. • Increased in concentration of alkaline solution, curing temperature and time will increase the compressive strength of geopolymer.
2012	Nurhanie et al.	Crystallization of geopolymer	• The KOH solution produced faster setting time compared to the NaOH solution. • Increased in temperature and concentration will shortening the setting time. • Avrami’s theory can be used to describe the transformation of crystal from liquid to solid.

CHAPTER 3

METHODOLOGY

This section explains in detail all the process routes as well as related procedures during conducting the experiment. Besides that, this section also describes all the equipment, materials and apparatus needed with its function. Apart from that, the key milestone and Gantt chart also included.

3.1 Research Methodology

The methodology used in conducting this research project is based on the discovery and experiment. First, gather all the important information about geopolymer from the previous journal, paper work and engineering book and compiled it as literature review. The research continues with conducting the pre-experiment to determine the feasibility of equipments used in solidification of geopolymer followed by other experiments to determine effects of parameters on the solidification of geopolymer. Parameters selected in this research are different types of alkaline activator, different concentrations of alkaline solution and different temperature curing. Lastly, all the results from the experiment will be demonstrated and analysed based on the Avrami's Kinetic Theory.

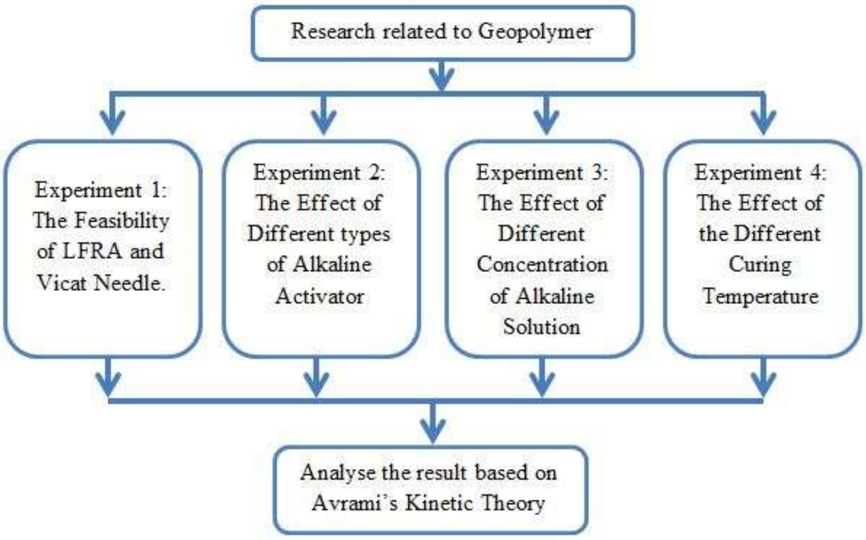


Figure 3.1: The overall process flow of the research

3.2 Project Activities

The details of procedure are very important before conducting the experiment in laboratory. The chemicals needed in the experiment must be prepared carefully especially in dealing with high reactive chemical likes high concentration of sodium hydroxide and sodium silicate solution. Furthermore, the equipment and apparatus needed in the experiment must be verified clearly in procedure to avoid any problems appeared. Lastly, safety in handling the equipment must be followed throughout the experiment to avoid any unexpected accident.

There are four sets of different experiments are needed in order to fulfil all the objectives of this research. Every parameters stated in the objectives will be tested using different experimental procedures to acquire accurate result. However, the procedure for all three experiments almost the same but differs in term of types of alkaline activator, concentrations of alkaline solution and temperature curing during moulding process. Below is the step by step procedure in the performing the experiment in geopolymerization process.

1. Sodium hydroxide (NaOH) pellet is mixed with distilled water in a volumetric flask based on selected concentrations (6M, 10M and 14M).
2. The hydroxide and silicate solution are kept in store at room temperature for 24 hours to remove heat as geopolymerization is exothermic reaction. (sodium silicate solution is already prepared in laboratory)
3. Alkali silicate (waterglass) is added into the solution depending on ratio required. (most suitable NaOH/Silicate ratio is 1:2.5 (Nath and Sarker, 2012)
4. Fly ash powder is mixed well with alkali hydroxide/alkali silicate prepared depending on ratio required. (most suitable fly ash/alkaline ratio is 3:1)
5. The fresh mixture of fly ash and alkaline solution is rapidly poured into steel mould for moulding process and exposed it into desired temperature (26^oC, 60^oC and 90^oC).
6. Time is set and the mould is observed and tested every 5 minutes using LFRA Texture Analyzer and Vicat needles until it solidified. Time and data tested from both equipments will be recorded.
7. The result achieved in this experiment (solidification of geopolymers) will be analyse and discuss based on Avrami's Kinetic Theory.

3.2.1 Experiment 1: The Feasibility of LFRA Texture Analyzer and Vicat Needle as Testing Equipment.

The main objective in this experiment is to study the feasibility of the LFRA Texture Analyzer and Vicat Needle apparatus in determining the solidification of geopolymer. In this experiment, two samples were prepared in order to observe which equipment will produce more reliable result in respect to study the setting time for geopolymer to solidify. Both of the samples will be prepared by mixing the fly ash with 10M of sodium hydroxide (NaOH) solution and being cured at temperature of 60^{o}C. Both of the samples will be tested every 15 minutes by using both LFRA Texture Analyzer and Vicat Needle to determine the degree of geopolymer's solidification. The average of 15 minutes was chosen due to the location of LFRA is quite far from the oven where the sample being cured at specified temperature. In order to produce more reliable of result, the Vicat needle apparatus also being tested every fifteen minutes. The result of geopolymer's solidification from both experiments will be recorded and compared to see the accuracy of the result. Figure 3.2 shows the samples of geopolymer being tested by both LFRA Texture Analyzer (a) and Vicat Needle (b).

(a) **(b)**

Figure 3.2: a) LFRA Texture Analyzer b) Vicat Needle

3.2.2 Experiment 2: The Effect of Different Types of Alkaline Activator

For this experiment, the three types of alkaline activator used are sodium hydroxide solution (NaOH), potassium hydroxide solution (KOH) and sodium silicate solution (Na_2O_3Si). First step in conducting this experiment is the preparation of two (2) sets of samples of NaOH solution and one set of KOH solution with desired concentration of 10M. Sodium silicate solution is added into one (1) of the NaOH solution 24 hours before mixing it with solid constituent for the leaching process. The fly ash powder is molded in the mould with these three different alkaline activators with 3:1 ratio for each of the alkaline activator. Next, the mixture is exposed it to the setting temperature (60^{o}C). Every five minutes, the mould will be tested using Vicat Needle and data is recorded. Lastly, the result of geopolymer's solidification will be explained based on the Avrami's Kinetic Theory.

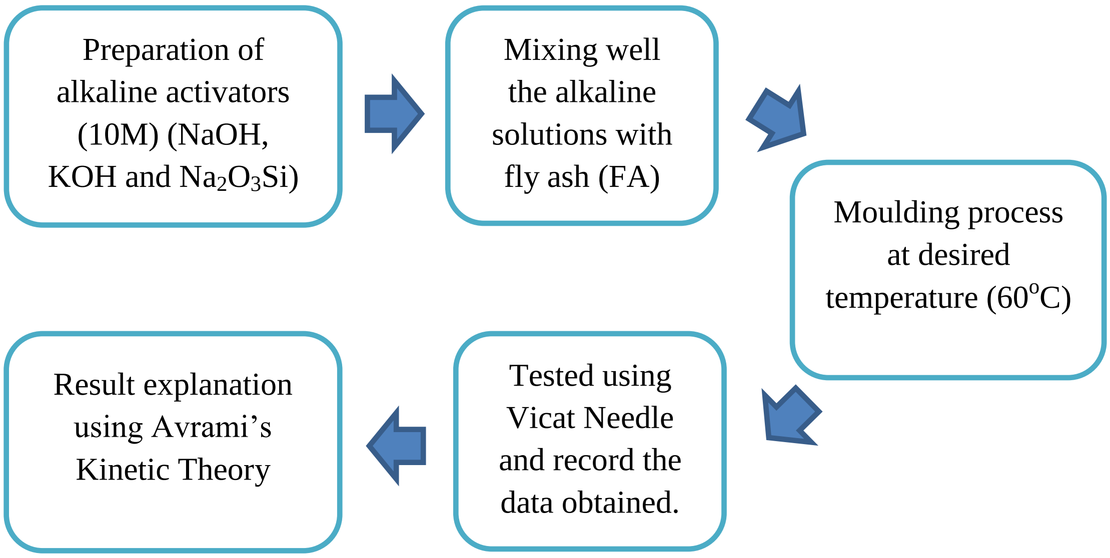


Figure 3.3: Summary of procedure for Experiment 2

(a) (b) (c)

Figure 3.4: a) Sodium Hydroxide b) Potassium Hydroxide c) Sodium Silicate

3.2.3 Experiment 3: The Effect of Different Concentrations of Alkaline Solution

The parameter tested in this experiment is different concentrations of alkaline solution where the concentration of NaOH solution at 6M, 10M and 14M are chosen since it is common alkaline activator used in the geopolymerization process. In order to achieve desired NaOH concentration, the amount of NaOH pellet and distilled water required is different for all three sample tested. The preparation of alkaline concentration should be done 24 hours prior to mixing with solid constituent. Next, the samples of NaOH were mixed with fly ash with the ratio 3:1 in the mould and exposed it to the setting temperature (60^{o}C). Every five minutes, the samples will be tested using Vicat Needle and XRD analysis was done to the samples for determination of geopolymer's microstructure. The result of geopolymer's solidification was recorded and explained based on the Avrami's Kinetic Theory. The simplified procedure in this experiment is presented in the Figure 3.5.

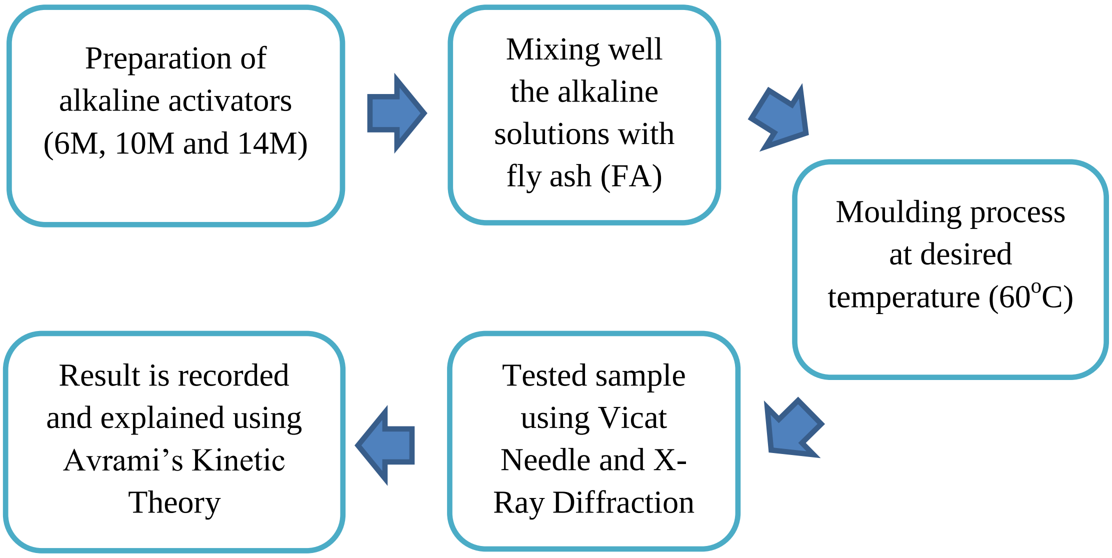


Figure 3.5: Summary of procedure for Experiment 3

Figure 3.6: Alkaline solution with different concentration

3.2.4 Experiment 4: The Effect of Different Curing Temperature.

The curing temperature during moulding process is a crucial phase for formation of crystal in the geopolymerization process. Thus, in the Experiment 3 for studying the effect of temperature curing on the solidification of geopolymer, the temperature setting in the moulding process will varies from 60^{o}C, 75^{o}C and 90^{o}C. These three (3) temperatures will be tested to determine the optimum temperature in the synthesis of geopolymer. First, three samples of alkaline solution is prepared and blended with fly ash with ratio of 1:3 until well mixed. Then, the mixture is molded in mould and it is exposed to the desired temperature of 60^{o}C, 75^{o}C and 90^{o}C. After that, the mould is observed and tested every five minutes using Vicat Needle. Then, all the data was recorded and result of solidification was explained based on the Avrami's Kinetic Theory. Figure 3.7 illustrates the step by step procedure of conducting this experiment.

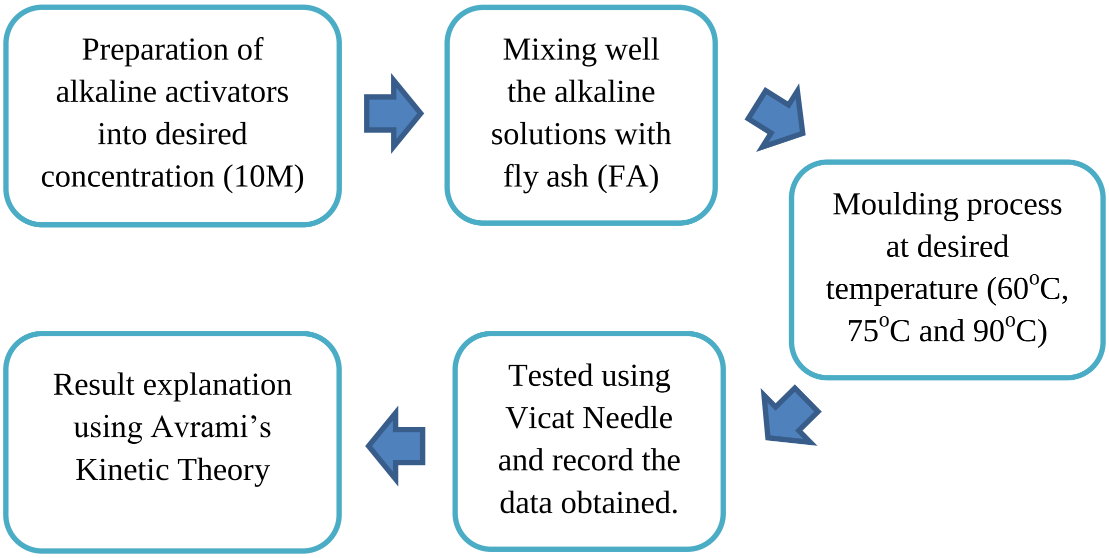
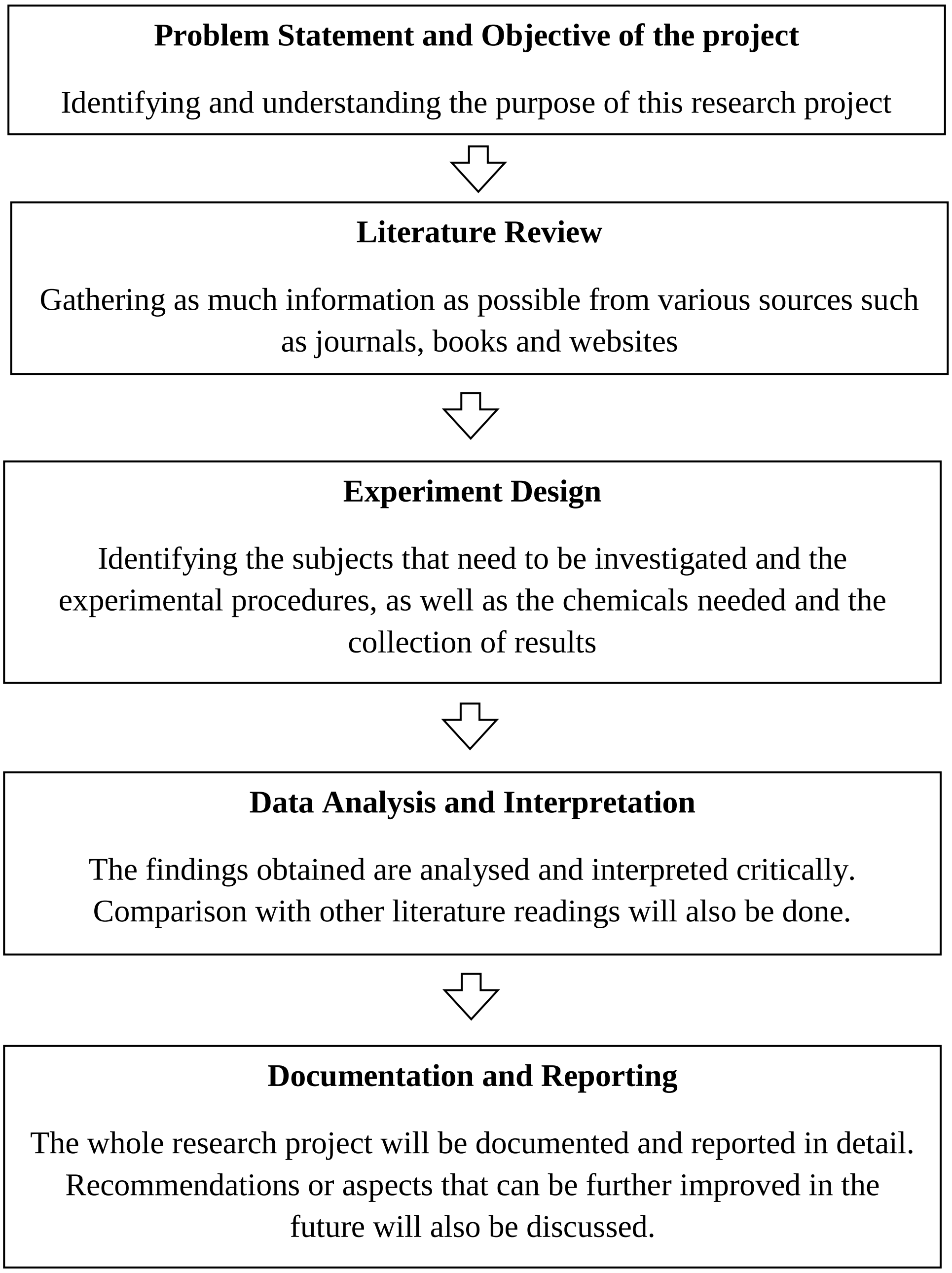


Figure 3.7: Summary of procedure for Experiment 4

Figure 3.8: Sample being cured at 60^{o}C

3.3 Key Milestones

Several key milestones for this research project must be achieved in order to meet all the objectives of this project:



Problem Statement and Objective of the project

Identifying and understanding the purpose of this research project

Literature Review

Gathering as much information as possible from various sources such as journals, books and websites

Experiment Design

Identifying the subjects that need to be investigated and the experimental procedures, as well as the chemicals needed and the collection of results

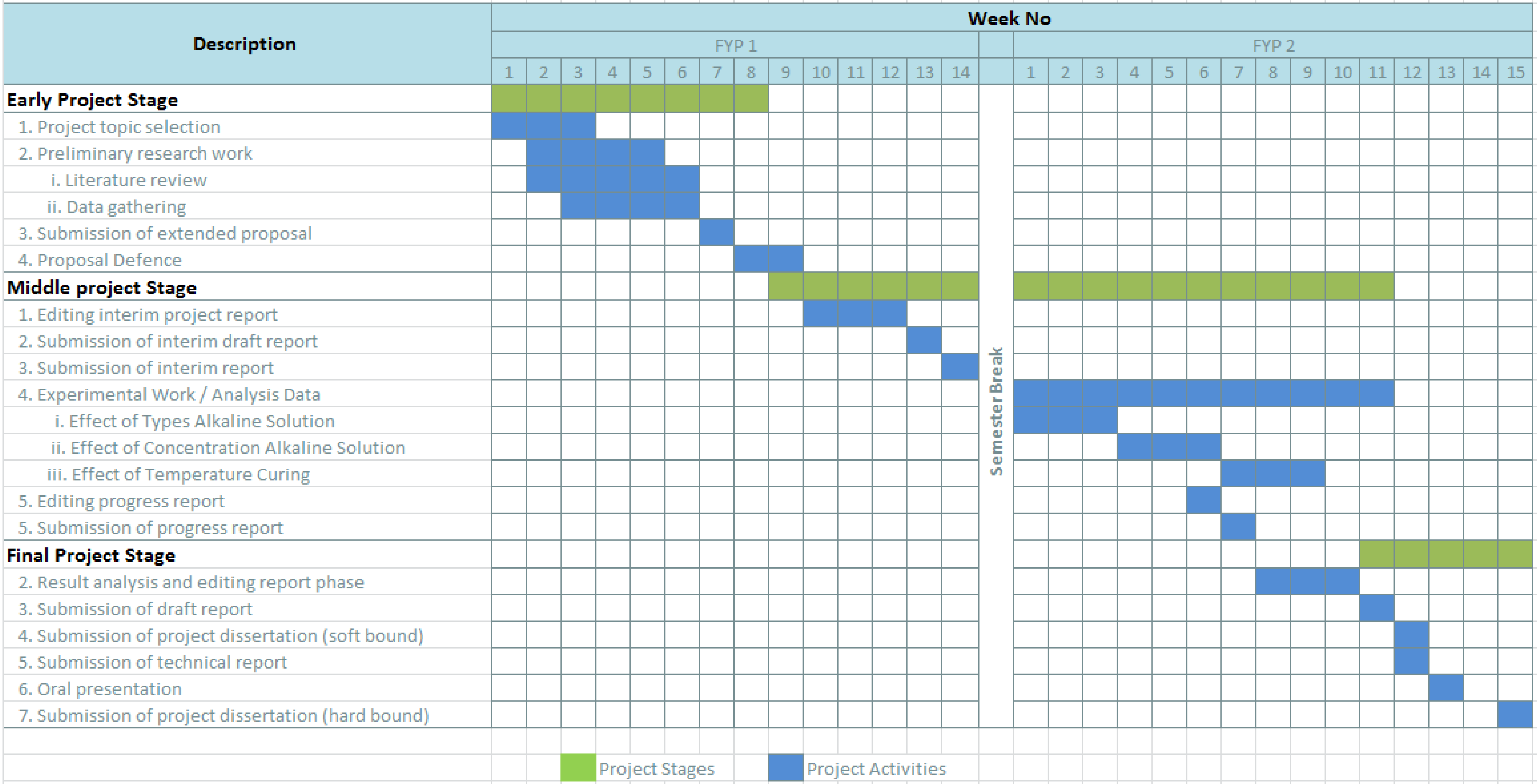
Data Analysis and Interpretation

The findings obtained are analysed and interpreted critically. Comparison with other literature readings will also be done.

Documentation and Reporting

The whole research project will be documented and reported in detail. Recommendations or aspects that can be further improved in the future will also be discussed.

3.4 Gantt Chart



Description	Week No																													
	FYP 1															FYP 2														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Early Project Stage	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩							Semester Break															
1. Project topic selection	🟦	🟦	🟦																											
2. Preliminary research work		🟦	🟦	🟦	🟦																									
i. Literature review		🟦	🟦	🟦	🟦	🟦																								
ii. Data gathering			🟦	🟦	🟦	🟦																								
3. Submission of extended proposal							🟦																							
4. Proposal Defence								🟦	🟦																					
Middle project Stage									🟩	🟩	🟩	🟩	🟩	🟩		🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩				
1. Editing interim project report										🟦	🟦	🟦																		
2. Submission of interim draft report													🟦																	
3. Submission of interim report														🟦																
4. Experimental Work / Analysis Data																🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦				
i. Effect of Types Alkaline Solution																🟦	🟦	🟦												
ii. Effect of Concentration Alkaline Solution																			🟦	🟦	🟦									
iii. Effect of Temperature Curing																						🟦	🟦	🟦						
5. Editing progress report																					🟦									
5. Submission of progress report																						🟦								
Final Project Stage																										🟩	🟩	🟩	🟩	🟩
2. Result analysis and editing report phase																							🟦	🟦	🟦					
3. Submission of draft report																										🟦				
4. Submission of project dissertation (soft bound)																											🟦			
5. Submission of technical report																											🟦			
6. Oral presentation																												🟦		
7. Submission of project dissertation (hard bound)																														🟦

🟩 Project Stages 🟦 Project Activities

3.5 Equipment, Apparatus and Material Required

The basic tools required in this experiment are available in the Chemical Engineering Laboratory in Universiti Teknologi PETRONAS (UTP). The tools can be divided into the equipment, apparatus as well as material. The basics equipment used in this experiment are Vicat Needle, LFRA Texture Analyzer and XRD Diffraction. Equipment in this experiment will be used depending on their specific purposed. For example, Vicat Needle and LFRA Texture Analyzer will be used to determine the hardness of geopolymer produced while XRD is used to identify the solidification characteristic of geopolymer. LFRA Texture Analyzer and Vicat Needle are available in Block 5, UTP Chemical Engineering Laboratory while XRD Diffraction can be found in Block 17, UTP Mechanical Engineering Laboratory.

The apparatus is the main important part in conducting the experiment. In these three (3) experiments, the apparatus needed are beaker, volumetric flask, spatula, glass rod, measuring cylinder and weighing scale. Every apparatus have their specific purpose in these experiments. The beaker and glass rod will be used in the preparation on NaOH solution to dissolve NaOH pellet in the distilled water. After that, volumetric flask will be used to dilute the NaOH into desired concentration. Next, measuring cylinder is required in determining amount of alkaline solution before mixing it with fly ash powder while weighing scale is needed to weight the fly ash’s powder as it needs to follow the ratio of 3:1. Then, mould will be used to mould the mixture.

These experiments cannot be completed without the presence of materials such as fly ash, sodium hydroxide, potassium hydroxide, sodium silicate as well as distilled water. The fly ash will act as material rich with alumina silica content while both sodium hydroxide and sodium silicate acting as alkaline solutions to provide the OH^- ion in the synthesis of geopolymer. The fly ash used in this experiment is from Class F type which the characterization and composition can be referred in the **Appendix 1**. Lastly, the distilled water will be used to dissolve NaOH and KOH pellets in the preparation of alkaline solution. NaOH, KOH and fly ash already available in UTP Chemical Engineering Laboratory while sodium silicate solution is obtained from Avantis Laboratory Supply, Perak.

CHAPTER 4

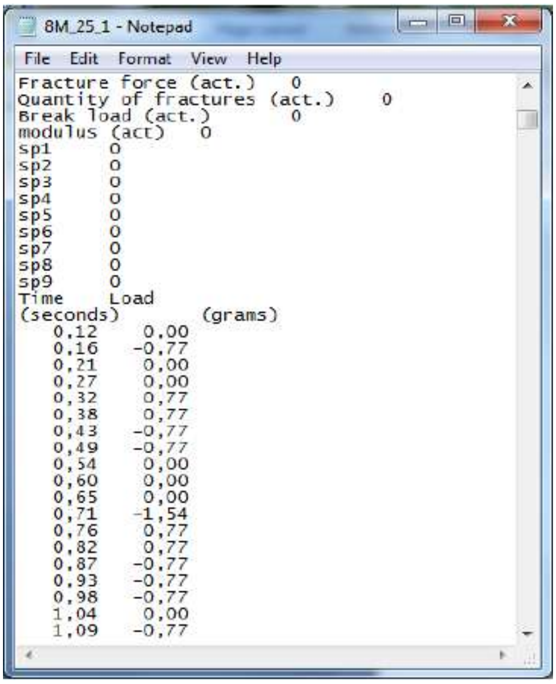
RESULT AND DISCUSSION

In this section, the experimental results are presented and discussed where all the test data points plotted in the figures correspondence to the mean value of each setting time for all types of parameters investigated. The standard deviations are plotted on the test data points as the as the error bar. The totals of twelve samples were prepared in order to study various parameters on setting time of geopolymer's solidification. This section will be divided into three subsections including 4.1; feasibility of equipment between LFRA Texture Analyzer and Vicat Needle apparatus, 4.2; the experimental result for all parameters and 4.3; the reaction kinetic analysis from Avrami's Kinetic Theory.

4.1 LFRA Texture Analyzer vs. Vicat Needle Apparatus

One of the main objectives in this research is to study the feasibility of LFRA Texture Analyzer and Vicat Needle apparatus in determining the solidification point of geopolymer. Both of the equipments will be used to test the transformation of geopolymer from liquid after the mixing until final setting time of geopolymer fully hardened in Experiment 1. This part discussed more on the difference in measurement for both equipments as well as provides the justification which equipment will produce more reliable result. The reliability of the result for both of the equipments is presented on the graph through the value of R^2 which shows the accuracy of equipment in determination of solidification point of geopolymer.

The LFRA Texture Analyzer test was performed in this experiment is to study the strength of geopolymer during the solid transformation. Normally, the data of sample tested will be obtained from the LFRA using the software in the form of notepad. The type of probe used in this experiment was known as TA 10 with the diameter of 10 mm. The probe also was set at constant speed of 2 mm/s with the penetration depth of 4 mm inside the samples. There are a few parameters that can be extracted from the LFRA Texture Analyzer such as the hardness, adhesiveness and springiness of the sample. Figure 4.1 shows the snapshot of the notepad from the computer that connected to the LFRA for 6M of NaOH concentration presented by Nurhanie *et al*. 2012. Based on this data, the graph of hardness (g) versus time (min) can be plotted to show the solidification point of geopolymer as shown in Figure 4.2.



```
Fracture force (act.)    0
Quantity of fractures (act.)     0
Break load (act.)        0
modulus (act)    0
sp1      0
sp2      0
sp3      0
sp4      0
sp5      0
sp6      0
sp7      0
sp8      0
sp9      0
Time     Load
(seconds)         (grams)
    0,12     0,00
    0,16    -0,77
    0,21     0,00
    0,27     0,00
    0,32     0,77
    0,38     0,77
    0,43    -0,77
    0,49    -0,77
    0,54     0,00
    0,60     0,00
    0,65     0,00
    0,71    -1,54
    0,76     0,77
    0,82     0,77
    0,87    -0,77
    0,93    -0,77
    0,98    -0,77
    1,04     0,00
    1,09    -0,77
```

Figure 4.1: Snapshot of LFRA Texture Analyzer (Nurhanie *et al*., 2012)

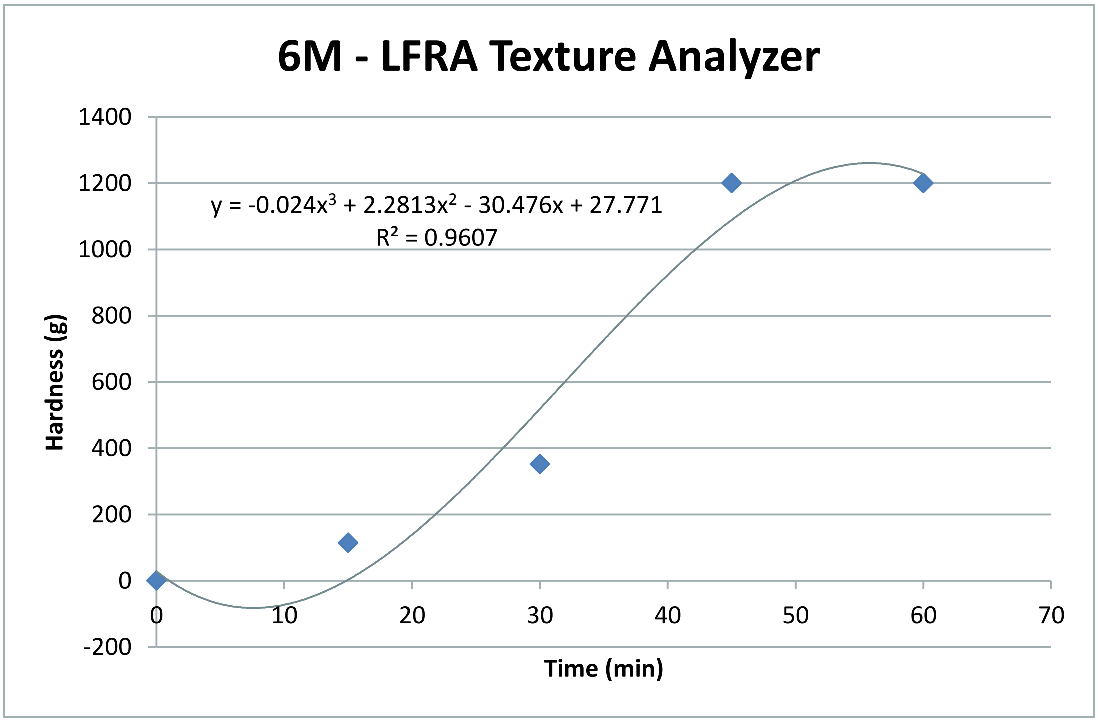


Figure 4.2: The graph hardness vs. time for 6M NaOH concentration

The most common equipment used in determining the setting time of geopolymer is Vicat needle apparatus where it is widely used in the cement and plastic industry. The measurement of setting time is based on the depth of penetration of Vicat needle into the sample whereby the initial and final setting time will be recorded. The maximum depth of penetration was set at 40 mm that shows the geopolymer is fully hardened. The penetration of Vicat needle will be tested three times in order to increase the accuracy of the result as shown in Table 4.1.

Table 4.1: Sample of data taken from Vicat Needle testing (6M)

Time (min)	**Depth from bottom (mm)**			
	1st	**2nd**	**3rd**	**Average**
0	0.0	0.0	0.0	0.00
15	5.5	5.0	5.0	5.17
30	13.0	12.5	12.0	12.50
45	36.0	37.0	36.0	36.33
60	39.5	40.0	40.5	40.00
75	40.0	40.0	41.0	40.33

Based on the result in the Table 4.1, the graph of depth of penetration (mm) vs. time (s) shall be constructed as shown in Figure 4.3.

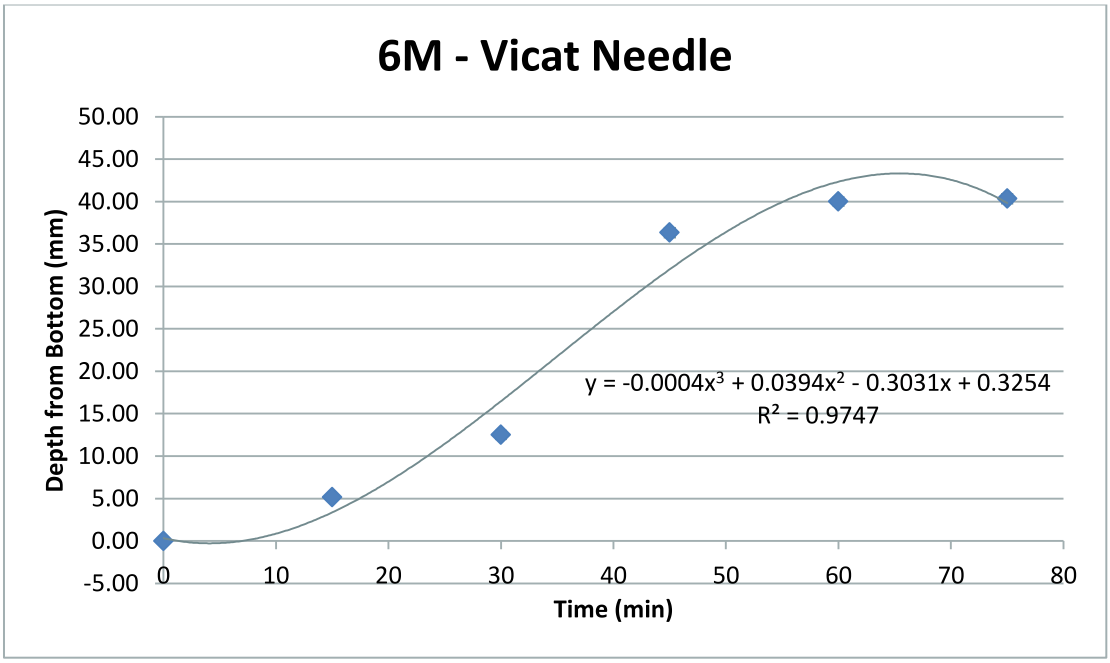


Figure 4.3: The graph depth from bottom vs. time for 6M NaOH

There are a few advantages of using Vicat Needle apparatus compared to LFRA Texture Analyzer in determination of the setting time of geopolymer. The LFRA possesses a few limitations in determination of hardness of geopolymer which make it less suitable to be used as testing equipment. First, the main function of LFRA is used to analyze the food texture with the maximum strength recorded is 1200g. The nature of food texture itself can be considered soft to be compared with the hardness of geopolymer. Thus, the maximum hardness of LFRA with the strength of 1200g can be considered as soft and far from the real hardness for geopolymer even when the geopolymer still not fully harden. In short, the geopolymer strength at the final setting time is harder compared to the penetration capability of the LFRA.

Next, the smallest probe's type (TA 10) that has being used in the experiment is 10mm in diameter which can be considered as quite big for the penetration of geopolymer and it would slightly affect the result of geopolymer tested especially in term of actual time for geopolymer to start solidifying. The larger surface area of probe makes the penetration of probe into the sample harder. Apart from that, the LFRA takes almost two minutes for the probe to penetrate the sample tested. These two limitations will ultimately affect the accuracy of the result. It should be noticed that the solidification of geopolymer are very sensitive to the change in temperature. It can be seen that from the both graphs of LFRA and Vicat Needle where the samples of same composition and being cured at same temperature, but differs in final setting time of solidification. Furthermore, the distance between LFRA and the oven are quite far thus the geopolymer is exposed to the changes in temperature for a significant period of time which will result in the less reliable data.

The used of Vicat Needle offers a better option as testing equipment to determine the solidification of geopolymer. First, the smallest Vicat Needle's diameter that can be used for sample penetration is 1mm. This is a highly desirable so that the penetration of the sample can be done in an easier manner. Moreover, time taken for the Vicat Needle to penetrate is considerably shorter than the LFRA. The fact that geopolymer solidification is time sensitive makes Vicat Needle the better option for the testing. Lastly, the Vicat Needle possesses the mobility which enables the sample testing to be conducted near the oven where the geopolymer sample is being cured at the temperature of 60° Celcius. Based on the facts mentioned, it can be concluded that Vicat Needle is clearly the better option as the testing equipment for the sample.

4.2 The Experimental Results (Experiment 2, 3 and 4)

The total of three experiments were conducted in this research in order to study the effect of different types and concentrations of alkaline activator as well as the effect of different curing temperature on the solidification of geopolymer. This subsection shows the analysis of experimental result for all three experiments that have been tested using Vicat Needle apparatus. The analysis of data is based on the theory stated in the journals and books from previous studies.

4.2.1 Experiment 2: The Effect of Different Types of Alkaline Activator

The main objective for this experiment is to investigate the effect of different type of alkaline activator on the solidification of geopolymer. In this experiment, three different types of alkaline activator that will be used to dissolute the fly ash in geopolymerization process. The alkaline activators used in this experiment are sodium hydroxide (NaOH), potassium hydroxide (KOH) and sodium hydroxide with the addition of sodium silicate (Na_2O_3Si). Figure 4.4 demonstrates the overall combination of result for the different types of alkaline activator of NaOH, KOH and modulus alkali of Na_2O_3Si. As shown in previous discussion, the result showed in this experiment is the graph of Vicat Needle penetration's depth vs. time for different types of alkaline concentration.

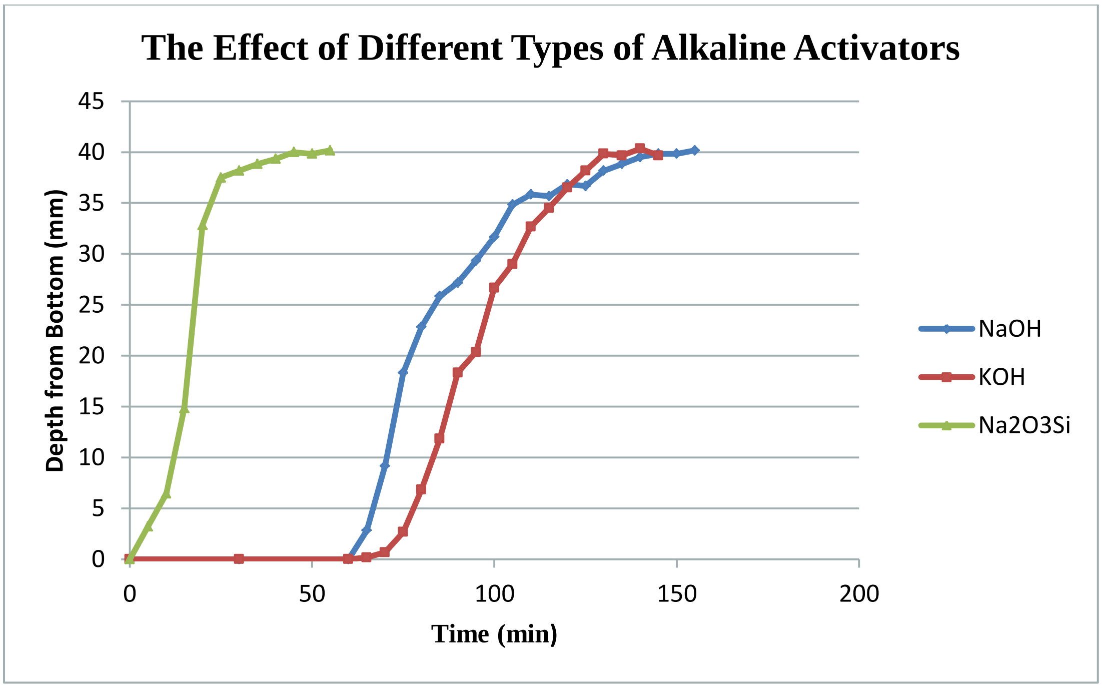


Figure 4.4: The Effect of Different Types of Alkaline Activators

The figure displays the result from Experiment 2 where it clearly showed that the different types of alkaline activator plays significant role in setting time for solidification of geopolymer. The role of alkaline activator in the geopolymerization process is a tool to dissolute of fly ash at the early stage of process where the presence of OH- anions in the alkaline solution will attack the Si-O and Al-O bonds during dissolution phase (Yao *et al.*, 2009). From this graph, it shows that the time needed for geopolymer to harden for NaOH is 155 minutes, KOH is 140 minutes and NaOH with addition of modulus alkali (Na_2O_3Si) is 45 minutes. This result shows that the geopolymerization process occurs rapidly in the reaction of fly ash with NaOH in the presence modulus alkali silicate, followed by reaction fly ash with KOH solution and lastly the reaction with NaOH solution.

This result can be explained through two difference approaches including the addition of modulus alkali silicate to the NaOH solution and different in results of KOH and NaOH. First, the addition of sodium silicate in the NaOH solution causes the increasing in number of Si^{4+} ion in the solution which is crucial for leaching process to occur. The addition of silica ion will possess higher dissolution as compared to alumina as alumina content in fly ash is half of silica (Xu and Van Devanter, 2000). Thus, the addition of modulus alkali silicate content will accelerate the geopolymerization process thus subsequently will shorter the setting time for the hardness of geopolymer compared to other activators.

Next, the comparison in setting time for geopolymer to solidify can be seen in the different results of sodium hydroxide solution and potassium hydroxide solution. NaOH and KOH are caustic base but slightly different in chemistry and practical application (Nurhanie *et al.*, 2012). The reaction of water with both base solutions are strongly exothermic with KOH solution is slightly exothermic. The water solubility of KOH solution is higher to be compared with NaOH solution in the same amount of water (Shelly Morgan, 2011). All of this alkalinity factors might plays a role in the explanation of result for this experiment. It is observed that the setting time for the geopolymer to solidify is slightly faster in KOH solution compared to NaOH solution might be due to the higher alkalinity of KOH solution (Yao *et al.*, 2009). It seems that aluminosilicate is more dissolvable in KOH solution which is consistent to the research conducted by van Jarsveld and van Devanter in 1997 has shown that K^+ is positive to the strength of geopolymeric materials.

4.2.2 Experiment 3: The Effect of Different Concentrations of Alkaline Solution (NaOH)

The objective of Experiment 3 is to study the effect of different concentrations of alkaline activator on the solidification behaviour of geopolymer. The alkaline activator used in this experiment is sodium hydroxide (NaOH) solution where its concentrations are varying from 6M, 10M and 14M. Figure 4.5 shows the overall combination of result for the concentration of 6M, 10M and 14M. The result shows the graph of Vicat needle penetration's depth vs. time for each concentration tested. In this experiment, the samples tested was send to XRD analysis where the result shows the structure of geopolymer in all concentrations tested are amorphous structure as shown in **Appendix 2**.

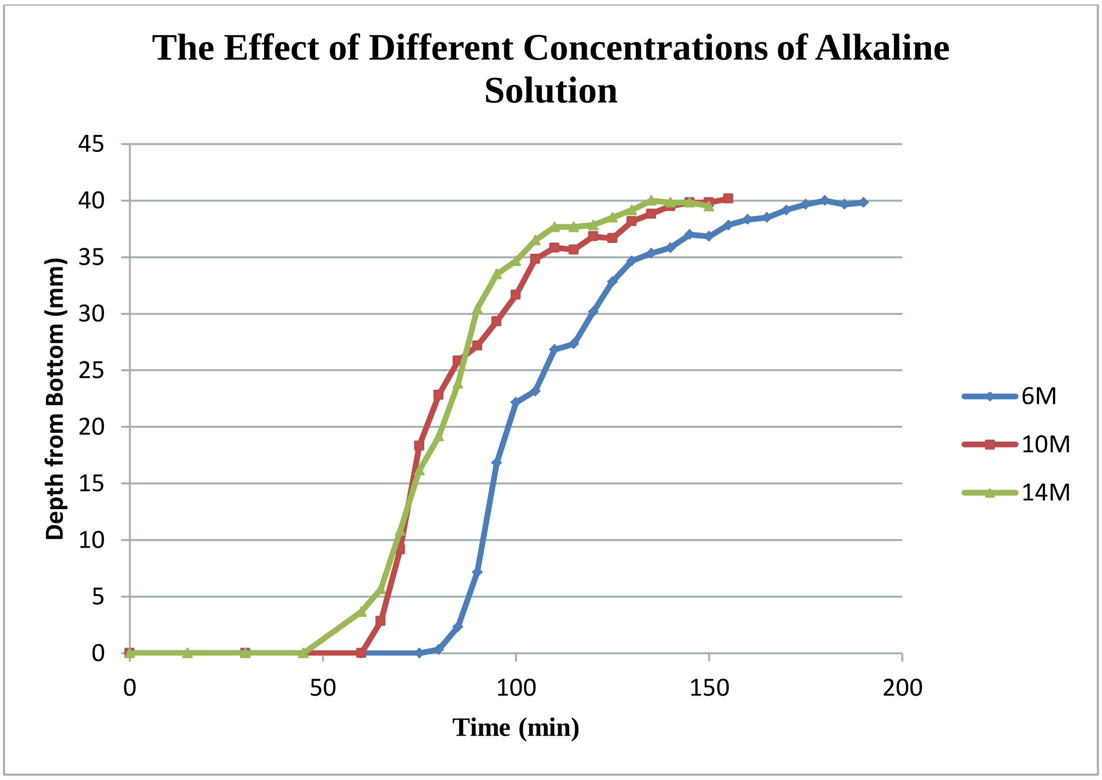


Figure 4.5: The Effect of Different Concentrations of Alkaline Solution

The figure shows the result for the different concentration of alkaline solution on the solidification of geopolymer. The sample of 14M and 10M of NaOH concentration achieved maximum hardening faster compared to the sample of 6M of NaOH solution. Furthermore, it should be noticed that the concentration of 14M achieved maximum hardening slightly faster than 10M which means the rate of solidification process for 14M is higher compared to concentration of 10M. The time taken for the sample of 14M to harden is 135 min followed by sample of 10M (155 min) and sample 6M took relatively longer period of time with 180 min.

When the fly ash material begins coming to contact with NaOH solution, leaching of Si, Al and other minor ions started. The amount of dissolution is largely depending on the concentration of NaOH and curing time. Thus, increase in the number of concentrations will increase the number of OH^- ions in the solution for leaching process that lead to acceleration of the geopolymerization process. The presence of high molarity also adding the number Si^{4+} and Al^{3+} in the solution which can lead to higher compressive strength of geopolymer. However, based on the literature review, when the concentration of NaOH solution is higher than 15M, the dissolution supposed to be decreasing due to increase in coagulation of silica (Bergna and Roberts, 2006). In short, the higher molarity of NaOH concentration leads to higher geopolymerization process which reducing the setting time of geopolymer.

4.2.3 Experiment 4: The Effect of Different Curing Temperature

The presence of higher temperature plays an important role in the geopolymerization process. Thus, the objective of this experiment is to study the effect of curing temperature on the setting time for the geopolymer to solidify. In this experiment, it is observed that the 10M of fresh fly ash-based geopolymer being cured in room temperature did not harden for at least one (1) day. The result of curing temperature at room temperature sharing the similar result was obtained by previous research conducted by Hardjito *et al.* (2004). Thus, in order to produce more reliable result, the different curing temperatures tested were at 60^oC, 75^oC and 90^oC.

Figure 4.6 shows the graph for this experiment where it can be seen that the time taken for geopolymer to solidify is shorter at temperature of 90^oC compared to the curing temperature at 75^oC and 60^oC. The time taken for the geopolymer’s sample to fully solidify at 90^oC is 55 minutes while at curing temperature of 75^oC is 90 minutes and 60^oC is 155 minutes. This is due to the rate of water loss that decreased the setting rate (Wang and Cheng, 2008). At the 90^oC, rate of water loss is higher than other temperatures due to the high reaction kinetic at high temperature. However, despite the samples being cured at 90^oC and 75^oC showing the faster setting time, both of the samples produce crack which subsequently affecting the compressing strength of geopolymer as shown in Figure 4.7. Based on the literature review, the best geopolymerization process occurred at the optimum curing temperature of 60°C (Muniz-Villarreal *et al*., 2009). In short, the increase of curing temperature will shorter the setting time for solidification of geopolymer.

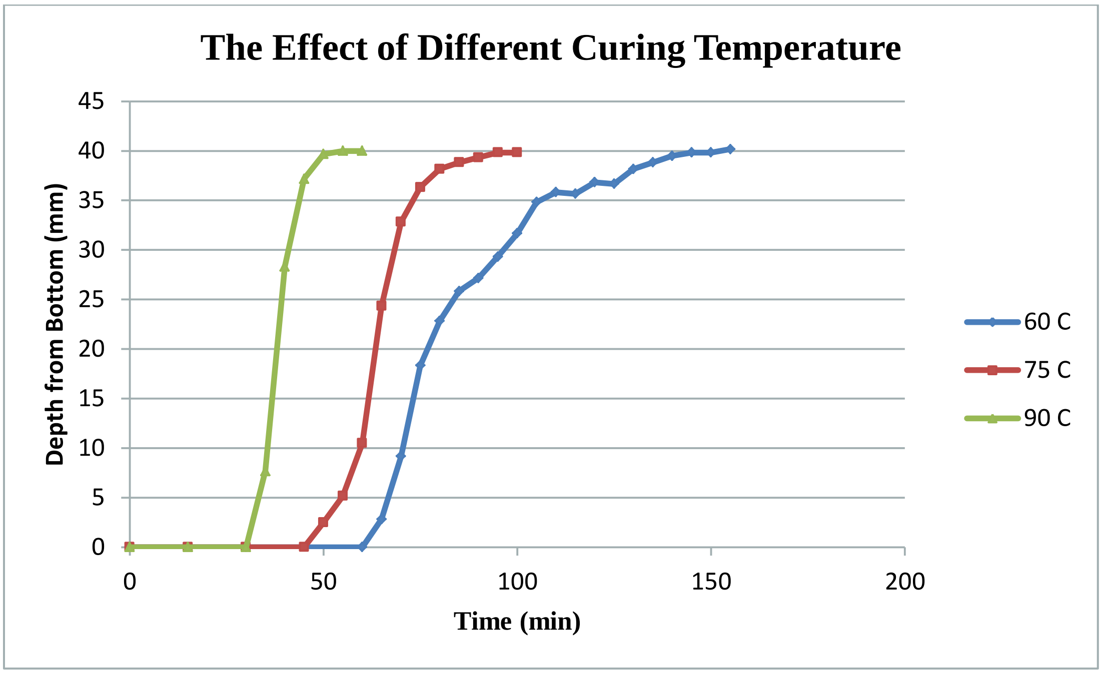


Figure 4.6: The Effect of Curing Temperature

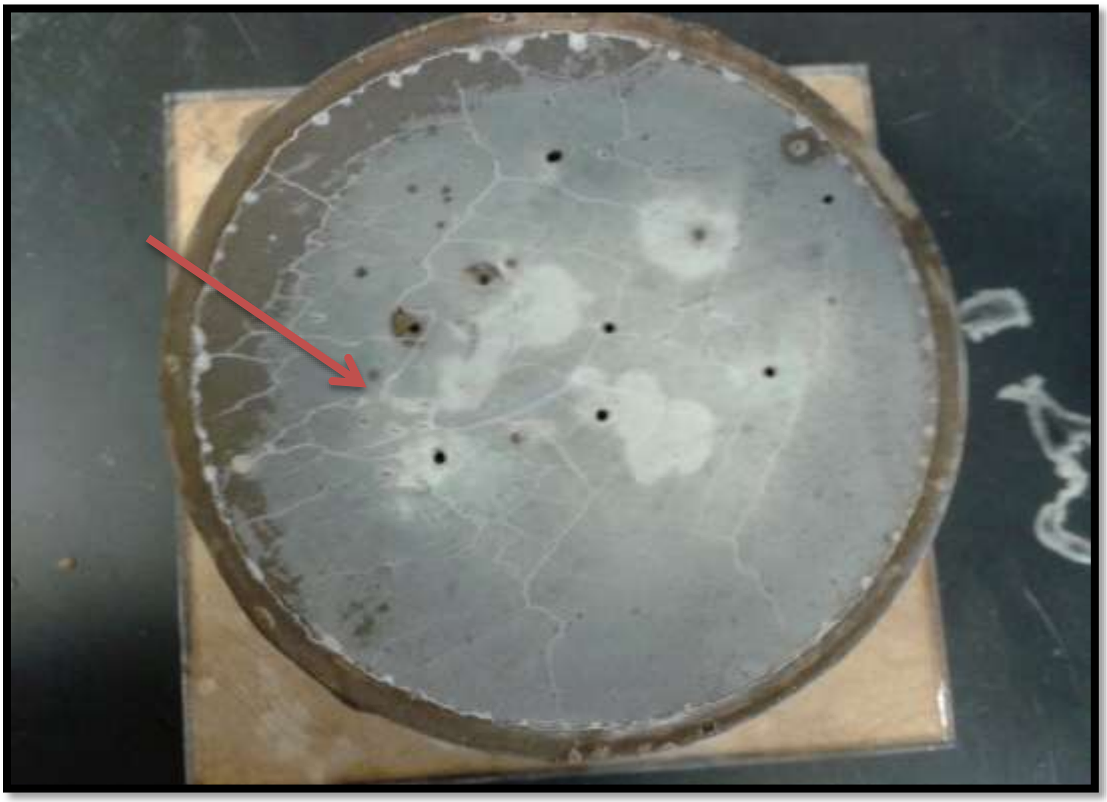


Figure 4.7: The crack of sample being cured at 90°C.

4.3 Reaction Kinetic Analysis from Avrami's Theory

Based on the graph of depth of Vicat Needle penetration versus time for the Experiment 2, Experiment 3 and Experiment 4, the curve of the graph shall be analyzed using Avrami's Theory in order to extract the kinetic of crystallization of geopolymer. The sigmoidal graph from the all three experimental results shall be converted into the linear graph of log [-ln (1-X)] versus log T. Throughout the transformation of graph from sigmoidal graph to linear graph, the significance different of data might deviates the actual values of Avrami's exponent (*n*) and growth rate (*K*). Thus, the average data from the needle's penetration plays an important role in order to get more accurate results.

Figure 4.8 shows the graph of Avrami's plot for the effect different types of alkaline activator while Figure 4.9 displays a result for different concentration of alkaline solution and Figure 4.10 shows the result for effect of different curing temperature on the solidification of geopolymer. From the Avrami's plot, the value of Avrami's exponent (*n*) and growth rate constant (*K*) is extracted and tabulated on Table 4.

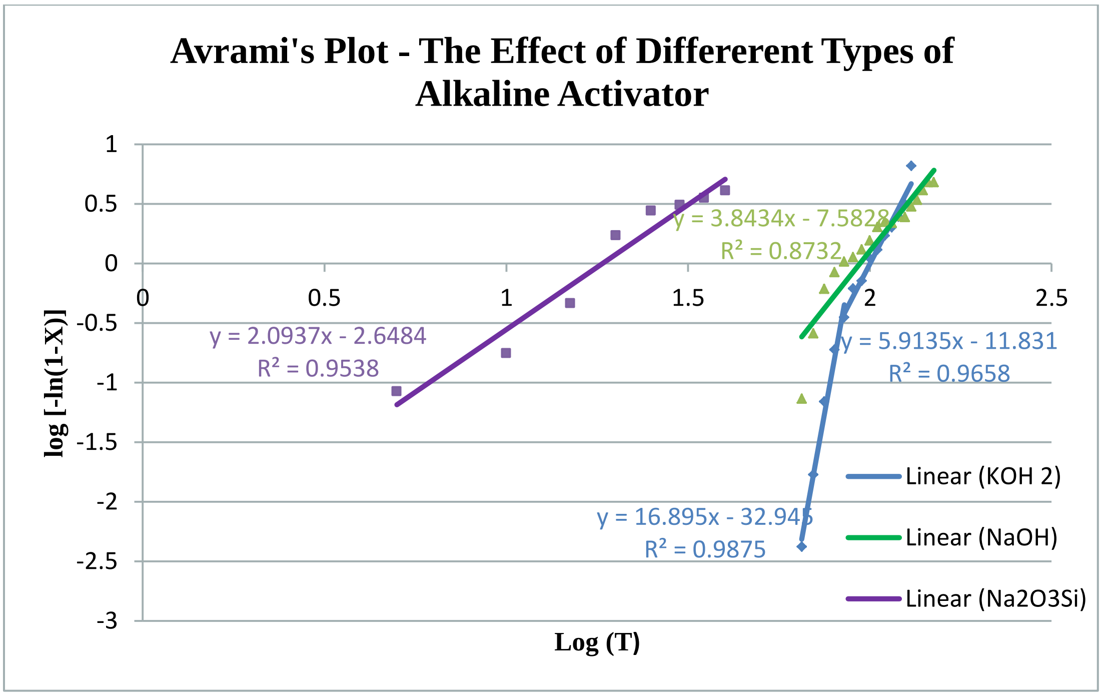


Figure 4.8: Avrami's plot for Experiment 2

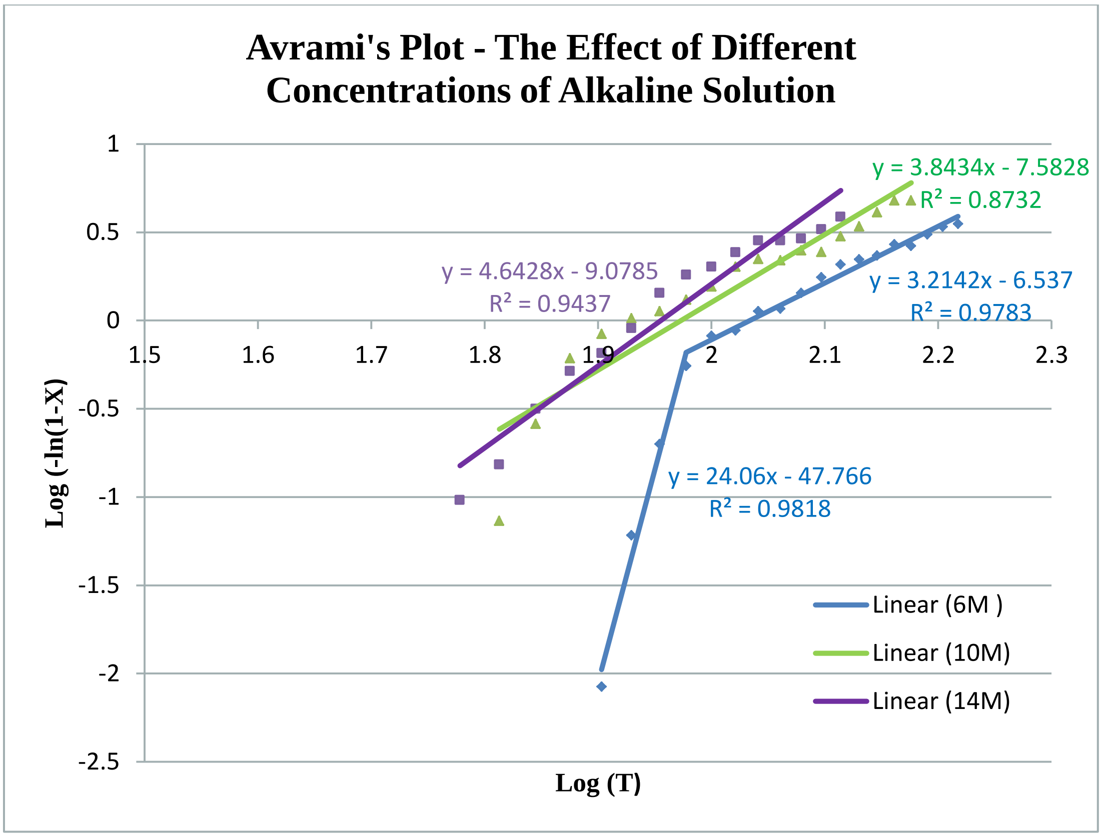


Figure 4.9: Avrami's plot for Experiment 3

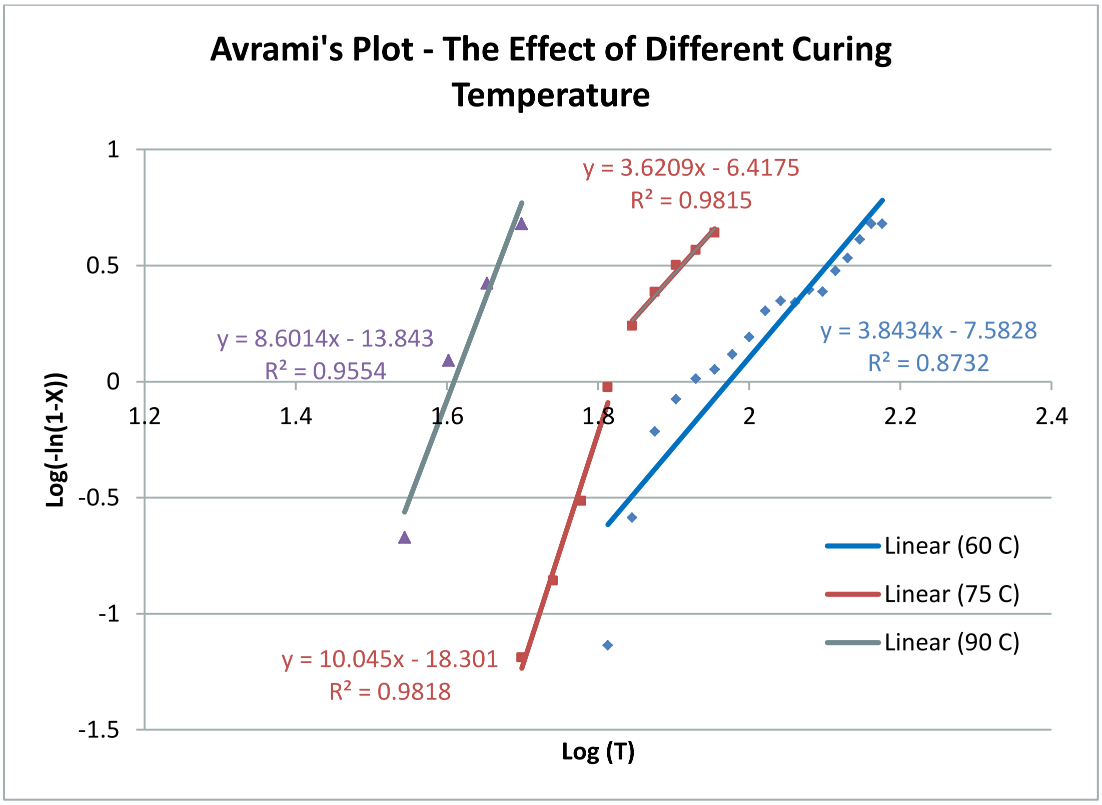


Figure 4.10: Avrami's plot for Experiment 4

From all the figures, the extraction of data from the Avrami's plot can be tabulated in the Table 4.2 for further analysis. There are some of the samples show two values of Avrami's exponent (*n*) and growth rate (*K*) in a single graph.

Table 4.2 (a): Avrami's exponent for Experiment 2

Types of Alkali Activator	Avrami's Exponent (n)	Growth Rate (K),(min^{-1})
NaOH	3.8434	2.61×10^{-8}
KOH (1)	5.9135	1.48×10^{-12}
KOH (2)	16.895	1.13×10^{-33}
NaOH + Na_2O_3Si	2.0937	2.25×10^{-3}

Table 4.2 (b): Avrami's exponent for Experiment 3

Concentration (M)	Avrami's Exponent (n)	Growth Rate (K), (min^{-1})
6 (1)	3.2142	2.90×10^{-7}
6 (2)	24.06	9.99×10^{-48}
10	3.8434	2.61×10^{-8}
14	4.6428	8.35×10^{-10}

Table 4.2 (c): Avrami's exponent for Experiment 4

Temperature (^oC)	Avrami's Exponent (n)	Growth Rate (K),(min^{-1})
60	3.8434	2.61×10^{-8}
75 (1)	3.6209	3.82×10^{-7}
75 (2)	10.045	4.27×10^{-19}
90	8.6014	1.43×10^{-14}

Based on the tables presented, the obvious trend shall be seen that the value of Avrami's exponents are varies in all the parameters tested. The value of *n* is ranging from two until twenty-four where the smallest *n* value is calculated for the sample of 10M of NaOH solution with the presence of Na_2O_3Si while the biggest value lies on the 6M concentration of NaOH solution at the 60^oC. The values of Avrami's exponent are very important for determining the growth form of geopolymer. Additionally, there are certain results shows the two values of the *n* in a single graph due to the pattern of the Avrami's plot as shown in the sample of KOH solution, sample of 6M of NaOH solution and the sample of NaOH solution that being cured at 75°C which resulted into very large of value of *n* in these samples. These results suggested the presence of secondary crystallization in the formation of geopolymer (Toro-Vazquez *et al.*, 2001).

According to Lukman *et al.*, (2008), when the value of *n*=1, it shows the growth of from instantaneous nuclei whereas the *n*=3 and *n*=4 refers to spherulitic growth from either sporadic or instantaneous nucleation. The value of *n* between 2 and 3 indicates growth form in two- or three- dimensional nucleation of the crystal growth. However, for the geopolymer growth, the expected value of exponent is ranging from 3-4 in three dimensional forms. This result shows that the growth of geopolymer form of geopolymer can be said that as two and three dimensional as some of the value of Avrami's calculated are varies from two up to above four. In addition, the presence of secondary crystallization in some of the sample, it can be said that the growth form of geopolymer is not fixed to one formation. In short, the longer setting time of geopolymer's solidification, the higher value of Avrami's exponent that led to geopolymer is formed in 3-dimensional structure.

On the other hand, the value of growth rate (*K*) also plays an important role in determine the speed of geopolymerization process. The values of Avrami's exponent (*n*) is inversely proportional to the growth rate (*K*) value where the as the value of *n* increases, the value of *K* will decreases (Nurhanie *et al.*, 2012). For Experiment 2, the value of growth rate of NaOH solution with the presence of Na_2O_3Si is higher than sample with NaOH solution. This result is consistent to the theory as the addition of modulus alkali silicate will accelerate the geopolymerization process. The expected value of growth is increases as the NaOH concentration increases that accelerate the geopolymerization process as reported by Hardjito *et al.*, (2004). However, the values of *K* in this experiment is fluctuated might be due to the parallax error during taking the measurement which subsequently affecting the calculation of growth rate in Avrami's equation. Lastly, the Experiment 4 shows that the growth rate of geopolymerization at curing temperature of 60°C is higher than at the curing temperature of 75°C and 90°C. It is consistent to the result by Muniz-Villarreal in 2009 as the best geopolymerization process occurred at 60°C.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Based on the result and discussion in Chapter 4 proves that the effect of different types of alkaline activator, different concentrations of alkaline solution and different curing temperature play an important roles on the solidification of geopolymer. In short, it can be concluded that:

- The Vicat needle offers better option than LFRA Texture Analyzer in term of determining the solidification of geopolymer.
- The best alkaline activator for the geopolymerization process is NaOH with the addition of modulus alkali silicate.
- Increase in alkaline concentration until 14M will increase the rate of geopolymerization process that subsequently shorter time for the geopolymer to solidify.
- The increase in curing temperature until 60^{o}C will increase the rate of geopolymerization process that afterward shorter time for solidification of geopolymer.

Based on Avrami’s Kinetic Theory:

- The growth rate of particle is faster as the curing temperature increases as well as with the addition of modulus alkali silicate into solution.
- The range value of Avrami’s exponent for all the samples are ranging from the value of 2 up to 24. Thus, it shall be concluded that the geopolymer’s growth form as two and three dimensional structure.
- There are several samples of geopolymer as presented in the sample of KOH solution, sample of NaOH at 6M and sample’s cured at 75^{o}C suggested the presence of secondary crystallization in the geopolymerization process.

5.2 Recommendations

In future work plan, there are a few recommendations are suggested in order to expand and improve this project.

- The ratio of alkaline solution and material solid shall be considered in order to get the best mixing time for geopolymer to solidify.
- The effect of mixing time also can be considered in this project since it plays a definite role in determining the setting time of geopolymer solidification.
- The average data taken from the Vicat Needle must be close enough between the points to get more accurate result of Avrami's plot.
- The position of eyes during the taking the needle's measurement must be perpendicular to the scale in order to avoid parallax error that subsequently affecting the calculation of Avrami's exponent.

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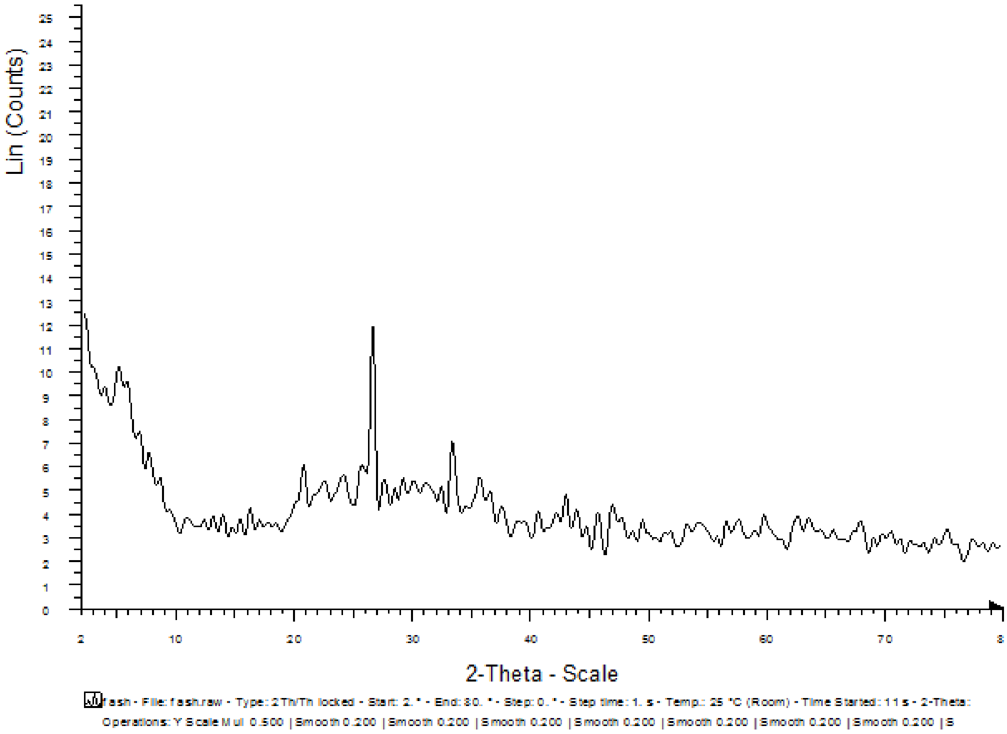
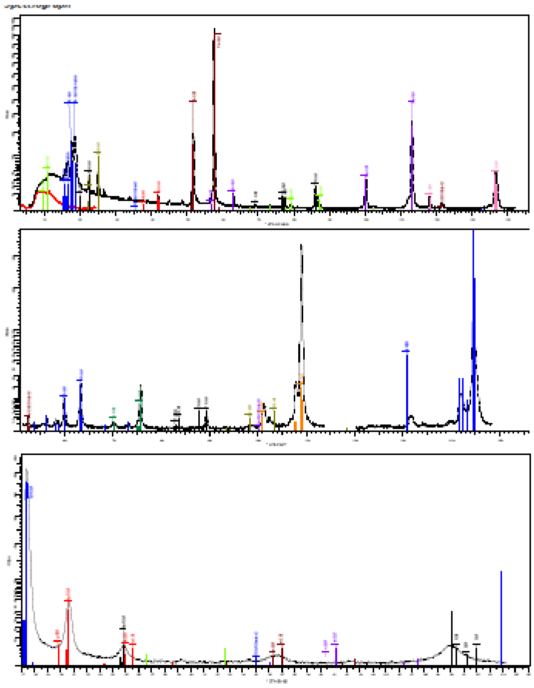
APPENDICES

Appendix 1: Fly Ash Characterization (Class F)

1) X-Ray Diffraction (XRD)



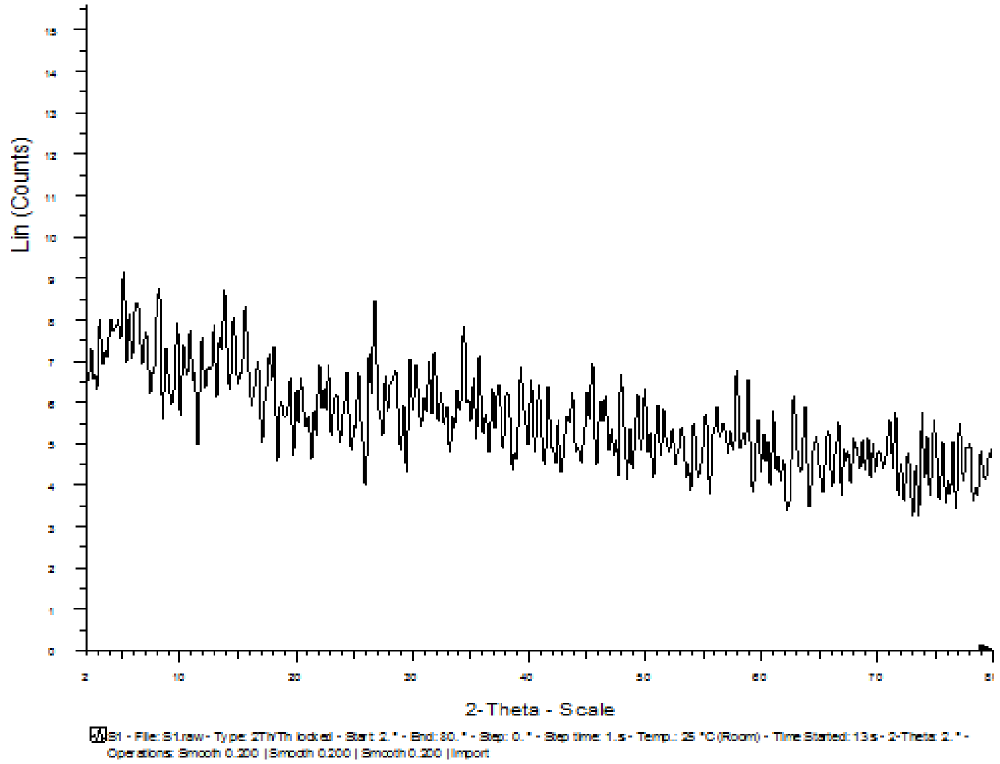
2) X-Ray Fluorescence Spectrometry Scanning (XRF)



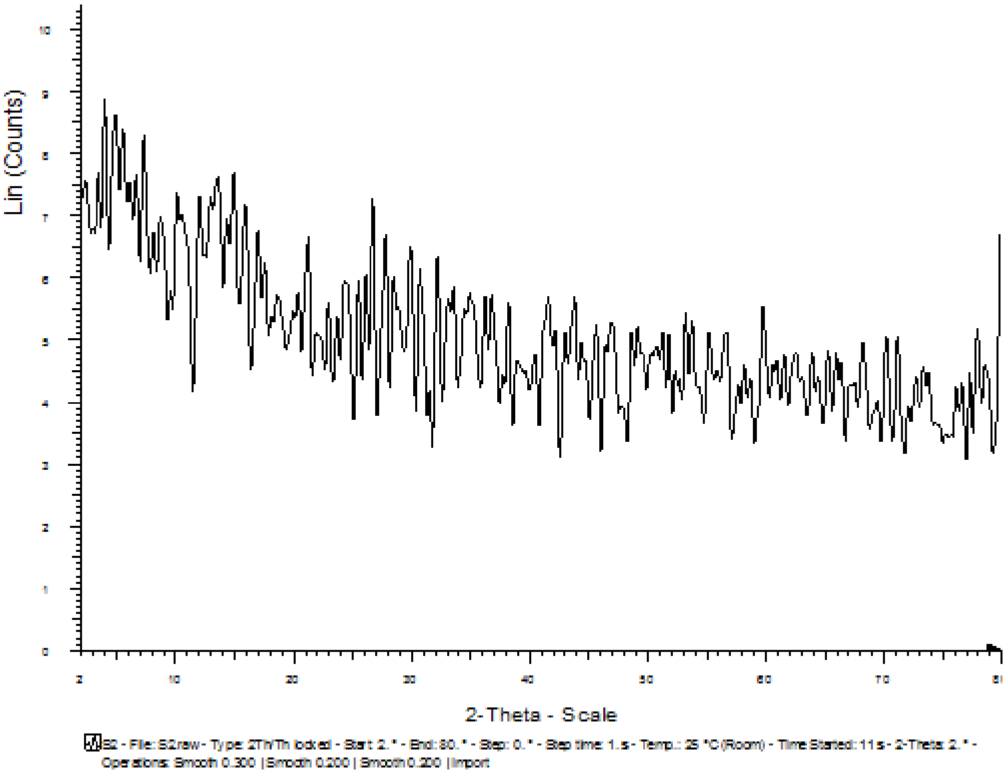
Formula	Concentration
SiO2	43.34%
Al2O3	20.77%
Fe2O3	12.41%
CaO	11.13%
MgO	3.75%
K2O	1.98%
SO3	1.45%
Na2O	0.95%
TiO2	0.88%
P2O5	0.32%
BaO	0.17%
SrO	0.12%
MnO	0.11%
V2O5	0.04%
ZrO2	0.04%
ZnO	0.02%
CuO	0.01%
Cr2O3	0.01%
Rb2O	95 PPM
As2O3	58 PPM
CoO	58 PPM

Appendix 2: The XRD results for 6M, 10M and 14M of NaOH concentration

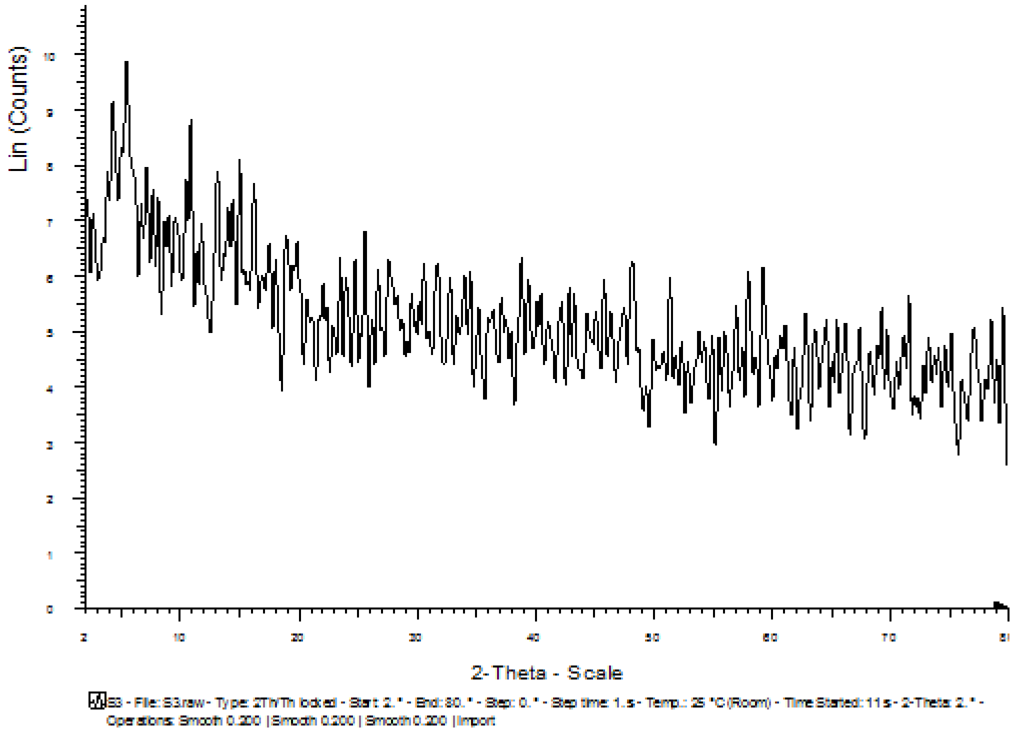
I. 6M



II. 10M



III. 14M


Lin (Counts)
0
1
2
3
4
5
6
7
8
9
10
2
10
20
30
40
50
60
70
80
2-Theta - Scale
S3 - File: S3.raw - Type: 2Th/Th locked - Start: 2. ° - End: 80. ° - Step: 0. ° - Step time: 1. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 2. ° -
Operations: Smooth 0.200 | Smooth 0.200 | Smooth 0.200 | Import