Development of Porous Geopolymer Materials from Meta-kaolin and Fly Ash

by

Nurul Fatiehah Binti Abdul Halim

15592

Dissertation submitted in partial fulfilment of

the requirements for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

SEPTEMBER 2015

Universiti Teknologi PETRONAS,

32610, Bandar Seri Iskandar,

Perak Darul Ridzuan.

CERTIFICATION OF APPROVAL

Development of Porous Geopolymer Materials from Meta-kaolin And Fly Ash

by

Nurul Fatiehah Binti Abdul Halim
15592

A project dissertation submitted to the
Chemical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(CHEMICAL ENGINEERING)

Approved by,

_____

(Dr. Muhammad Rashid Bin Shamsuddin)

UNIVERSITI TEKNOLOGI PETRONAS
BANDAR SERI ISKANDAR,PERAK.
September 2015

CERTIFICATION OF ORIGINALITY

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

_____

NURUL FATIEHAH BINTI ABDUL HALIM

ABSTRACT

The present work reports on the development of cost effective porous geopolymer materials from meta-kaolin and fly ash via an addition of three different mechanisms to produce pores. This research focused on synthesizing the geopolymer composites followed by characterization to analyze its physical properties in terms of crystallinity, physical, thermal and mechanical strength. Polymerization process of fly ash and meta-kaolin based geopolymer involved a chemical reaction of alumino-silicate oxides (Si_2O_3, Al_2O_2) with alkaline activators consist of the combination of sodium hydroxide (NaOH) solution and sodium silicate (Na_2O_3Si) solution under high curing temperature conditions. Pore forming agent such as baking soda, calcium metal and corn oil are added to increase the porosity of the geopolymer sample. The geopolymer composites were characterized using Gas Pycnometer test, Scanning Electron Microscopy (SEM), Thermogravimetric Analysis (TGA) and Compression Test. According to SEM test, meta-kaolin based geopolymer containing corn oil exhibit more pores compared to other samples. This sample exhibits higher porosity at 48% porosity, however has the lowest compression strength at 0.44 MPa. While at 16% porosity, sample with baking soda has the highest strength at 1.08 MPa.

ACKNOWLEDGEMENT

First and foremost, I would like to express my gratitude to God for His kind blessings by giving me strong will and determination to complete this progress report for Final Year Project 1 (FYP1) course. Much love and thanks to my mother Mdm Sharifah bt Hashim, my family and friends who has never failed in helping me through the thick and thin of time. It is the unfailing support from them that has enabled me to complete my undergraduate study.

I would like to thank my supervisor, Dr. Mohammad Rashid for his expert guidance, continuous support and encouragement throughout the course of this research project. He has been a very supportive supervisor and willing to share his knowledge, in order to ensure that I could learn and understand every single thing in this project. Besides that, I would also like to take this opportunity to express my deepest thanks to all laboratory technicians who had given guidance in completing this project experiment.

Last but not least, not to forget those who are directly or indirectly involved in the completion of this project.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1.1 Background Study

Since the beginning of the century, adsorption at various interfaces has concerned scientist to perform the researches related to this phenomenon. The technological, environmental and biological importance of adsorption can never be argued. Its practical applications in industry and environmental protection are highly importance (Dąbrowski, 2001). For example, in purification of water, this method is applied to remove any unwanted components or contaminants so that the purified water is safe for other purpose likes drinking.

There are two classes of adsorbent which are based on organic and inorganic materials. Alumina, silica and zeolite are categorized as inorganic materials where as carbon, polymer and biomass fall under organic materials (Knaebel, 2011). On 3750 BC, activated carbon is used as adsorbents in removing the contaminants in wastewater treatment. Activated carbon is highly used because of its low-volume pores that increase the surface area available for adsorption.(Link, 2006).

Nowadays, geopolymers have gained wide attention from many researchers and engineers because it is easy to fabricate, cheap and ecofriendly. Geopolymers represent new material types on the border among glass, ceramics materials and

materials based on classical inorganic bonds (linkage). Geopolymers give the potential possibilities to prepare inorganic bonds and building materials from the waste as slag, fly ash, kaolinitic substances, etc. When geopolymers are prepared, the burning process at high temperatures is not needed as it is during the production of cement or lime. (LOPEZ GUZMAN, 2014). Production of building material production from geopolymers does not in effect encumbrance the environment by CO2 emissions and that is why these materials present the potential possibility of building material production without CO2 emissions. Term geopolymer is coined by Prof Dr. Joseph Davidovits in 1978 (Das, Mohapatra, & Rath). Geopolymer is an inorganic polymer that can be prepared by mixing a reactive aluminosilicate material such as fly ash and meta-kaolin with alkaline activator such as sodium hydroxide (NaOH) solution and sodium silicate (Na_2O_3Si) solution.

1.2 Problem Statement

Commonly used adsorbents in industry application include activated carbon, silica gel and polymers. However, its commercial applications are limited due to high cost. Besides that, such adsorbents are made up from limestone during production which emits large amount of carbon dioxide gas lead to greenhouse effect. Geopolymer is a new class of material derived from alkali-activation of alumino and silicate oxides (Si_2O_3, Al_2O_2). Recent research work (Kadirvelu et al., 2003) showed that this material has characteristics of good absorbents. Being cheap, abundant, eco-friendly and ease of fabrication could make geopolymer a potential material for water treatment membrane.

In order to overcome such problem, in this project, geopolymer is developed into cost effective porous materials from fly-ash and meta-kaolin. Porous structure could be developed using several different methods such as by using calcium metal, by using baking soda and by using corn oil. Later, the materials are characterized for its properties and assessed for quality on waste water treatment. It is intended for the material to be produced into a membrane for future work.

1.3 Objectives

The main objectives of this project are:

- To develop a cost effective porous geopolymer membrane from fly-ash and meta-kaolin by using different mechanisms which are by using calcium metal, using baking soda and by using corn oil.
- To characterize the geopolymer composite properties in terms of crystallinity, physical, thermal and mechanical to assessed quality on waste water treatment.

1.4 Scopes of Study

This project mainly focuses on development of porous geopolymer membrane from fly ash and meta-kaolin. Porous geopolymer membrane produced is then characterized under different techniques. The scope of study for this project is further narrowed into few areas as describe below:

i. Raw Material Preparation
   - Kaolin is cured at 800ºC for 2 hours to form meta-kaolin.
   - Fly ash is taken from combustion by-product in coal power plant and do not require high temperature processing but only require sifting.
   - Meta-kaolin and fly ash reacted with alkaline reagent (mixture of sodium hydroxide solution (NAOH) and sodium silicate solution (Na_2O_3Si)) to produce geopolymer composites.

ii. Synthesis of Porous Geopolymer
   - Porous geopolymer is synthesized by using three different methods which are by using calcium metal, baking soda and corn oil.
     a) By using calcium metal - Hydrogen gas is released during reaction with water. Bubbles produced is

trapped inside the geopolymer composites thus form pores.

b) By using baking soda - Sodium bicarbonate reacted with acetic acid and released carbon dioxide gas. CO_2 gas bubbles are trapped inside the geopolymer composites thus form pores.

c) By using corn oil – The mixture of corn oil and meta-kaolin/fly ash is hardened and form hard solid monolith. Then the monolith is put in THF bath to remove the oil. Pores are produced when the hardened monolith is further dried in oven.

iii. Characterization of Geopolymer Composites

A geopolymer composites were characterized using Archimedes Test/gas pycnometer test, SEM-EDX, TG-DTA and compressive test.

CHAPTER 2

LITERATURE REVIEW

2.1 Adsorption Phenomena

The change in concentration of a given substance at the interface as compared with the neighbouring phases is referred to as an adsorption. In adsorption process, innumerable physical, chemical and biological processes take place at the boundary between two phases, while others are initiated at that interface (Dąbrowski, 2001). Adsorption can occur at various systems likes liquid-gas, liquid-liquid, solid-liquid and solid gas. The process of adsorption arises due to presence of unbalanced or residual forces at the surface of liquid or solid phase. These unbalanced residual forces have tendency to attract and retain the molecular species with which it comes in contact with the surface (Thomas & Crittenden, 1998). Basically, adsorption is a surface phenomenon. This is because, adsorption essentially happens at the surface of the substance only, not distribute uniformly throughout the bulk.

2.2 Types and Applications of Adsorbent

Nowadays, there are many types of adsorbent developed for industrial purposes. The various adsorbent uses is summarized in table below:

Carbon adsorbents	Mineral adsorbents	Other adsorbents
Active carbons	Silica gels	Synthetic polymers
Activated carbon fibres	Activated alumina	Composite adsorbents:
Molecular carbon sieves	Oxides of metals	(complex mineral–carbons,
Mesocarbon microbeads	Hydroxides of metals	X–elutrilithe; X = Zn, Ca)
Fullerenes	Zeolites	Mixed sorbents
Heterofullerenes	Clay minerals	
Carbonaceous	Pillared clays	
nanomaterials	Porous clay hetero-structures (PCHs)	
	Inorganic nanomaterials	

FIGURE 2.1 Type of Industrial Adsorbent (Skouteris, Saroj, Melidis, Hai, & Ouki,

In an ancient Egyptian papyrus, it has been described that active carbon was the first widely used adsorbent as early as 3750 BC. Its application is in the form of charcoal. Activated carbon is used for adsorption of organic substances and non-polar adsorbates and it is also usually used for waste water treatment (Skouteris et al., 2015). It is the most widely used adsorbent since its chemical likes surface groups and physical properties likes pore size distribution and surface area can be manipulated according to what is needed. Its usefulness also derives from its large micropore volume and the resulting high surface area. Activated carbon also commonly used for CO_2 separation in integrated gasification combined cycle (IGCC) processes for energy generation and hydrogen production (Thomas & Crittenden, 1998).

Besides all the adsorbents list above, inorganic polymers, also called geopolymers is categorized as adsorbent. The application only limited in powder form but not yet in membrane. Nowadays, geopolymers are becoming increasingly used as membrane substitutes for Portland cement, in waste remediation applications and as fireproof building materials (MacKenzie, 2015). So, geopolymers has become a new class of materials that has a potential to develop as membrane.

2.3 Background of Geopolymer

Prof Dr. Joseph Davidovits is a French materials scientist that is well known for the invention of geopolymer chemistry. In 1964, Professor David received an annual award from the French Textile Chemical Society (ACIT) for works performed on linear organic polymers. Until 1972, his researches cover the area on binders for foundries, synthetic textile fibers, natural and synthetic leather, collagen and organic membranes (Davidovits).

Geopolymer is an adhesive aluminosilicate forming by alkaline activation of alumina and silica as starting material at high temperature. A reactive aluminosilicate material such as fly ash and meta-kaolin, and alkaline base solution such as sodium hydroxide (NaOH) or potassium hydroxide (KOH) is used to produce geopolymer that consist of Si-O-Al-O bonds.

Many research have been conducted by engineers and academics to develop a sustainable materials (Ferdous, Manalo, Khennane, & Kayali, 2015). Geopolymer cement or concrete has gained wide attention from researchers because of their many advantages such as it provides low carbon emission and thus lead to decrease in global warming potential. Building concrete from geopolymer also can cut production cost. This is because geopolymer is made from recycle industrial waste like fly ash that produced as a by-product in coal fired power plant.

2.4 Method to Produce Pore

Pore in geopolymer membrane can be produced in many ways by using a forming agent or by introducing air into the geopolymer. Nowadays, most popular foaming agent used is aluminium powder. Air can be introduced into the geopolymer solution by using a mixer and after that just let the geopolymer dry by itself. In this project, I will focused on three methods to produce pore membrane such by using calcium metal instead of aluminium, by using baking soda/sodium

bicarbonate and the last one is by using corn oil. Each method is further discussed in table below:

Table 2.1 Method to Produce Pores

	Method 1 : Use calcium metal	Method 2 : Use sodium bicarbonate	Method 3 : Use corn oil
Chemical Equation	$Ca + H_2O \rightarrow H_2 + CaO$	$NaHCO_3 + C_2H_3O_2 \rightarrow NaC_2H_3O_2 + H_2O + CO_2$	-
Pore formation mechanism	In this method, calcium metal reacts with water and mixed with geopolymer paste. During the reaction, hydrogen gas is release and thus produced bubbles inside the geopolymer resin. Pore can be seen on the resin when it dried.	Sodium bicarbonate compound is reacted with acetic acid and then mixed with the geopolymer resin. The reaction released carbon dioxide gas (Deshlahra, Mehra, & Ghosal, 2009). CO2 gas bubbles produced is trapped inside the geopolymer resin as the cement dried and pores are formed.	Geopolymer resin that has been produced is mixed with the corn oil. The mixture is heated to form hard solid monolith and then the monolith is immersed in tetrahydrofuran (THF) bath to remove the oil for 24 hour at 60°C. After that, the monolith is dried and pores can be observed. (Seo, Medpelli, & Seo, 2013)
Previous work done by others	Porous geopolymer synthesized from meta-kaolin, aluminium powder and phosphoric acid at	Baking soda is widely used in the decomposition reaction of sodium bicarbonate to form carbon dioxide	Many inventions have been done regarding the material that can produced foams. The geopolymer resin is

	80 °C for 5 h. The Al powder served as a foaming agent to control porosity, pore size and pore distribution (Le-Ping, Xue-Min, Shu-Heng, Jun-Li, & Lin, 2010). Different mass content of Al powder is added to measure the strength and porosity of the geopolymer.	gas is the basis of its use as a raising agent in baking. Cakes are solid foams. The foam is produced when bubbles of carbon dioxide from the reaction of sodium bicarbonate are trapped in the batter. As the cake bakes, the batter dries, and the trapped bubbles of carbon dioxide form the holes in the cake (Deshlahra et al., 2009).	prepared with same conditions but with different types of vegetable oils to observe which oil can produce more pores. (Seo et al., 2013).

2.5 Characterization Techniques

After the geopolymer composites are developed, six tests will be conducted to observe the properties of the geopolymer. Each technique is summarized as below:

Table 2.2 Characterization Techniques

	Technique	Purpose	Availability in UTP	Method
1	Porosity Test	Determine the powder density of the sample.	Block 4	• Use gas pycnometer Quantachrome Corp Ultrapycnometer 1000 with helium gas.(Chang, 1988)
2	Archimedes Test	Determine the bulk density of	Block 5	• Immersed the geopolymer composites into glass jug

		the sample. (Eddy & Schlessinger, 2003)		half full of water. • Record the weight of sample during submerged and fully submerged. • Calculate density by dividing weight by volume. (Eddy & Schlessinger, 2003)
3	Scanning Electron Microscopy (SEM)	To analyze the pore network microstructure and products formation in sample used. (Eddy & Schlessinger, 2003)	Block 17	• Working tension of 20kV using microanalyzer EDX. (Eddy & Schlessinger, 2003)
4	Thermal Analysis (TGA)	To identify the products presence on the geopolymer sample due to mass loss or gain on heat transformation. (Aguilar, Melo, & Olivares, 2013).	Block 4	• Platinum-rhodium as thermocouples. • Test was performed in N_2 flux (100ml/min). • Alumina as inert reference at 20C/min. (Aguilar et al., 2013).
5	Compression Test	To test the strength of samples from day to day.	Block 13	• Prepare 12 samples and pour to 15cm x 15cm x 15cm mould. • The samples is tested after

		(Aguilar et al., 2013).		7 days curing at 60 ºC. (Aguilar et al., 2013).

CHAPTER 3

METHODOLOGY

3.1 Project Process Flow

Process flow below shows the steps for this research project that must be completed to make sure the objectives can be successfully achieved.

FIGURE 3.1 Project Process Flow

3.2 Gantt Chart

	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Project work continue • Purchasing raw materials. • Perform characterization techniques. • Final geopolymer synthesis.															
2	Submission of Progress Report								9/11/2015							
3	Project work continue • Final geopolymer synthesis. • Perform characterization techniques.															
4	Pre-SEDEX											30/11/2015				
5	Submission of Draft Final Report											1/12/2015				
6	Submission of Dissertation (soft bound)												7/12/2015			
7	Submission of Technical Paper												9/12/2015			
8	Viva													16/12/2015		
9	Submission of Dissertation (hard bound)															12/1/2016

FIGURE 3.2 Gantt Chart for FYP 2

Suggested milestone

Activities

3.3 Project Methodology

3.3.1 Materials and Chemicals

For this project, raw materials used are fly ash and meta-kaolin. Fly ash is obtained from Manjung power plant and meta-kaolin is obtained from RM Chemicals. Baking soda and home vinegar is needed as a pore formation agent. Several chemicals were used which are sodium silicate solution, sodium hydroxide solution, tetrahydrofuran (THF) and calcium metal.

3.3.2 Equipment and Apparatus

Table 3.1 Equipment and Apparatus

No	Apparatus	No	Equipment
1	Beakers	1	Gas pycnometer machine
2	Measuring cylinder	2	Compression test machine
3	Hand mixer	3	SEM machine
4	Dropper	4	Thermal analyzer machine
5	Oven		
6	Plastic container		
7	Electronic balance		
8	10 liter jug container		
9	Spatula		
10	Compression test moulds		

3.3.3 Procedure for Preparation of Sodium Hydroxide (NaOH) Solution

In this research project, a 8M of sodium hydroxide (NaOH) is used. The steps to prepare the solution are described below: (***Das et al.)***

1) 330g of NaOH pellet is pour into 500ml beaker.
2) Distilled water is added slowly into the beaker.
3) The mixture is stir until it is homogeneously mix.
4) Then, the mixture is pour into a 1000ml volumetric flask and water is added until reached the mark.
5) The solution is prepared 24 hours before used.

3.3.4 Procedure for Preparation of Fly Ash Based-Geopolymer

1) Three samples made from fly ash and alkaline activator is respectively prepared.
2) 50g of fly ash is mixed with 27g alkaline activator in the ratio of 25%/75% of sodium hydroxide solution/sodium silate solution respectively. (Seo et al., 2013)
3) The mixture is stir until it become homogeneous.
4) Then, a pore agent is added into the respective mixture and continued to stir for about 5 minutes.
5) The geopolymer paste is poured into a plastic container and was placed in an oven for 24 hours at 60ºC.

3.3.5 Procedure for preparation of Meta Kaolin Based-Geopolymer

1) Three samples made from fly ash and alkaline activator is respectively prepared.
2) 50g of meta-kaolin is mixed with 30.4g alkaline activator in the ratio of 25%/75% of sodium hydroxide solution/sodium silate solution respectively. (Seo et al., 2013)
3) The mixture is stir until it become homogeneous.

4) Then, a pore agent is added into the respective mixture and continued to stir for about 5 minutes.
5) The geopolymer paste is poured into a plastic container and was placed in an oven for 24 hours at 60ºC.

3.3.6 Preparation of Pore Formation Agent

1) Calcium Metal
    - Small amount of calcium metal is mixed with half-tablespoon of water, the reaction between these two substance produced hydrogen gas.
    - The mixture is immediately poured into the geopolymer paste prepared.
    - During heating process, the hydrogen gas produced cause the bubble to form on the surface of the sample.

2) Baking soda
    - 1 tablespoon of baking soda is mixed with 1 tablespoon cooking vinegar, the reaction between sodium bicarbonate and acetic acid produced carbon dioxide gas.
    - The mixture is immediately poured into the geopolymer paste prepared.
    - During heating process, the carbon dioxide gas produced cause the bubble to form on the surface of the sample.

3) Corn oil
    - 2 tablespoon of corn oil is added into the geopolymer paste. Then the sample is dried in the oven.
    - After the sample became hard solid monolith, it is soaked with THF bath to remove the oil inside the sample.
    - Then, the sample will be further dried in the oven for another 24 hours.

3.3.7 Characterization of Fly Ash and Meta-kaolin Based Geopolymer

Characterization of the geopolymer composites is important to determine the physical properties of the samples. There are four recommended analysis that been conducted to analyzed the samples :

1) Porosity Test

   From this test, the porosity of each sample can be determined. First of all, bulk density is calculated by using a Archimedes test, then the sample will be further analyzed with gas pycnometer test to determine the powder density of the sample. Porosity can be calculated by:

$$Porosity\ (\%) = \frac{Bulk\ Density - Powder\ Density}{Bulk\ Density}\ x\ 100 \qquad (1)$$

2) Scanning Electron Microscopy (SEM)

   This analysis was performed to analyze the pore network microstructure on the surface of the sample. From this analysis, we can observe the pore formation on that particular sample.

3) Thermogravimetric Analysis (TGA)

   TGA was conducted to determine mass loss during the heat transformation. In this case, a 10.00 mg of each sample was analyzed from 30ºC to 900 ºC at constant heat rate of 10 ºC/minute..

4) Compression Test

   Compression Test is performed after 7 days the sample is prepared. The purpose of this test is to determine the compressive strength of each sample. From this analysis, the relationship between porosity and compressive strength can be observed.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Result and Discussion

A. Porosity

Table 4.1 Porosity Result

Sample	Bulk Density (g/cm^3)	Powder Density (g/cm^3)	Porosity (%)
Meta-kaolin based geopolymer			
1-Calcium	1.8975	2.4857	31
2-Baking soda	1.8209	2.1061	16
3-Corn oil	1.9373	2.8589	48
Fly Ash based geopolymer			
4-Calcium	2.2381	2.2797	1.8
5-Baking soda	2.1777	2.3827	9.4
6-Corn oil	2.0167	2.3160	15

From the table above, sample made of corn oil for meta-kaolin based geopolymer exhibit higher percentage of porosity compared to other samples which is about 48%. This shows that, this sample contained more void space inside the membrane. The arrangement of molecules inside the sample is less compact which means that the bonds between the particles are less. This result is somewhat similar to SEM result obtained. For fly ash based geopolymer, sample made of corn oil also

exhibit highest porosity compared to others. These two types of geopolymer materials generally share the same properties in terms of porosity.

B. Scanning Electron Microscopy Analysis (SEM)

1) Meta-Kaolin based-geopolymer

i. Method 1: By using calcium metal

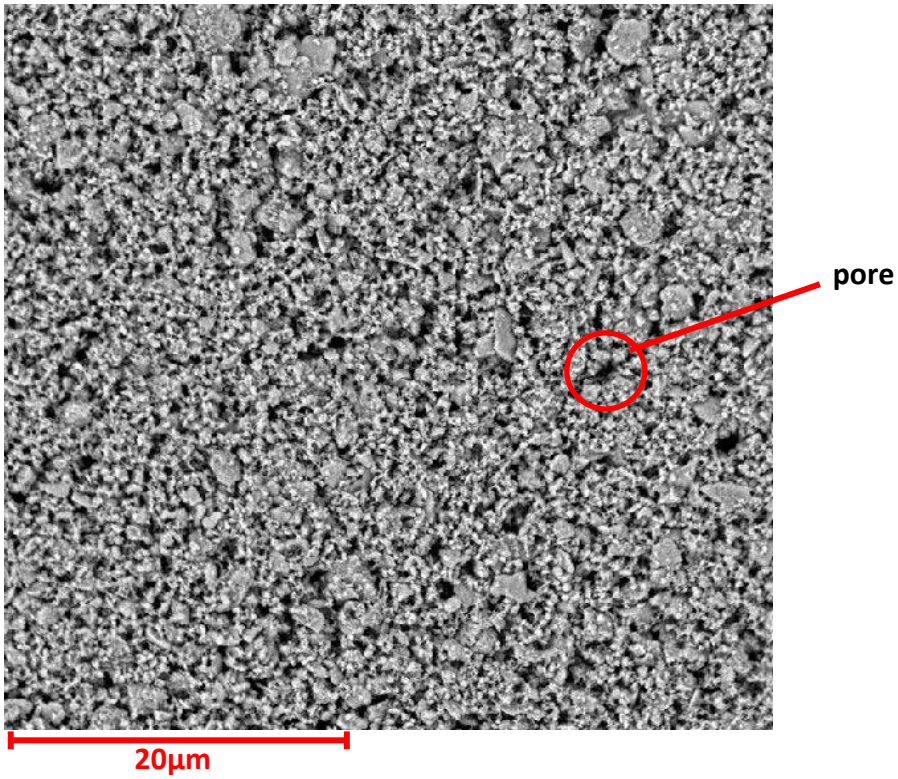


FIGURE 4.1 Meta-kaolin based porous geopolymer (Method 1)

The microstructure geopolymer in Figure 4.1 shows the formation of porous membrane from meta-kaolin by using calcium metal. It clearly shows that the size of the pores is small and almost not visible to see. This is because, more components are attracted to calcium charge since it has +2 free ion. Thus formed a dense network around the calcium molecule. So, the arrangements of the molecules seem more compact.

ii. Method 2: By using baking soda

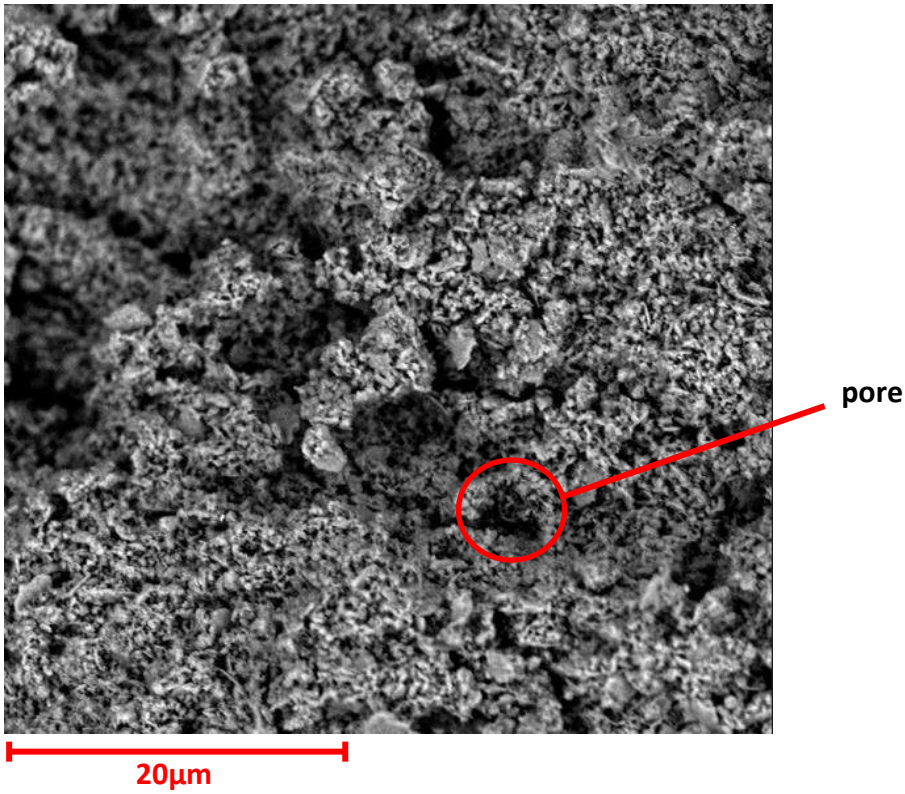


FIGURE 4.2 Meta-kaolin based porous geopolymer (Method 2)

Figure 4.2 shows the formation of porous membrane from meta-kaolin by using method baking soda and acetic acid. It is observed that there are pores produced but the size of the pore is slightly small. Maybe this is because the carbon dioxide gas that has been trapped inside the geopolymer formed a network with another component and formed a large network around the molecules. So, the pores seem to be more bulky.

iii. By using corn oil

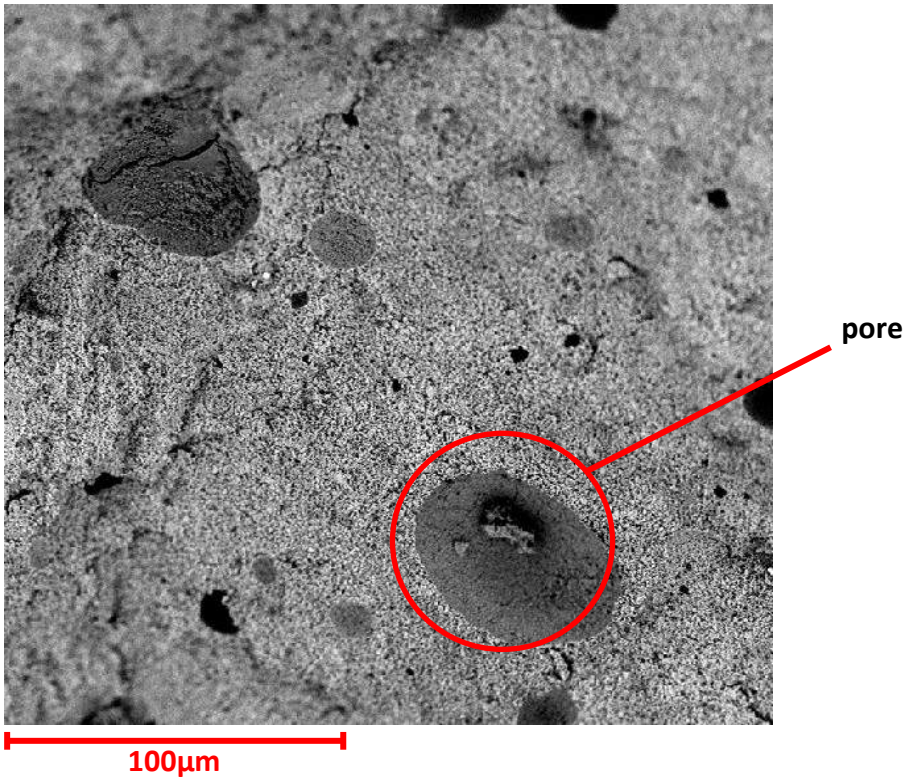


FIGURE 4.3 Meta-kaolin based porous geopolymer (Method 3)

The microstructure geopolymer in Figure 4.3 shows the formation of porous membrane from meta-kaolin by using corn oil. Pores can be clearly seen on the surface of the samples. This is because, during curing process, oil in alkaline emulsion undergoes saponification reaction and decomposed to water soluble material and glycerol molecules which were then extracted with THF to yield porous geopolymer materials.

2) Fly Ash based-geopolymer

i. Method 1: By using calcium metal

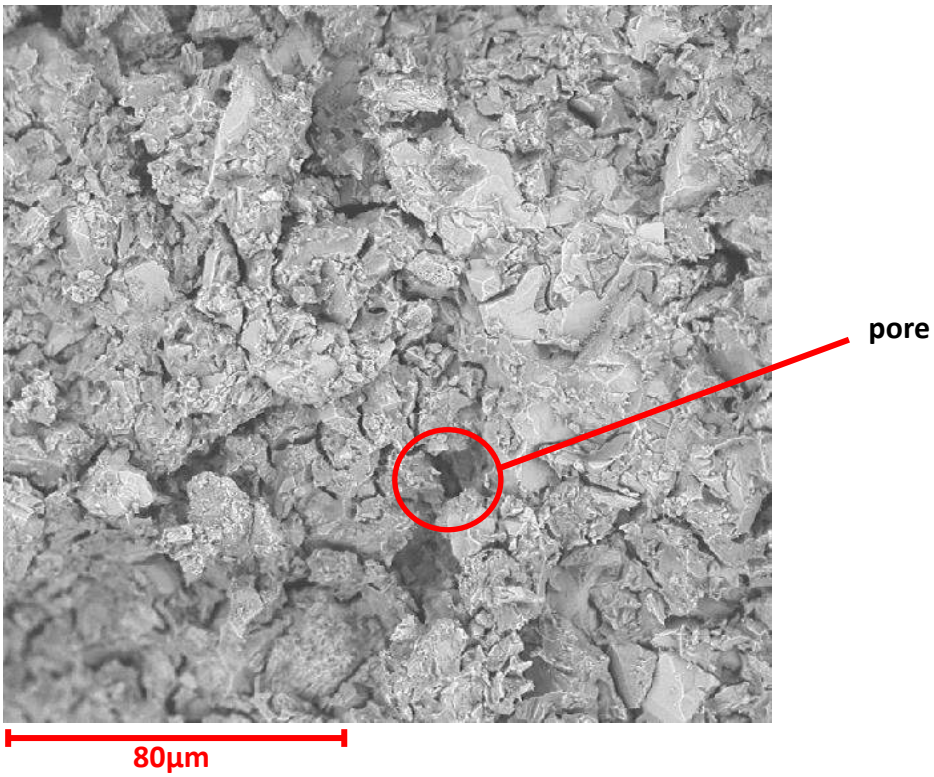


FIGURE 4.4 Fly-ash based porous geopolymer (Method 1)

Figure 4.4 shows the formation of porous membrane from fly-ash by using method 1 (calcium metal). It can be clearly seen that the structure of the sample is like the sample undergo a cracking process. Maybe this is because during the drying process, a high temperature is exposed to the sample thus cause the sample to crack. But, the pore also can be observed at certain area, this is because the amount of calcium metal that has been reacted with water is small thus produced small amount of hydrogen gas.

ii. Method 2: By using baking soda

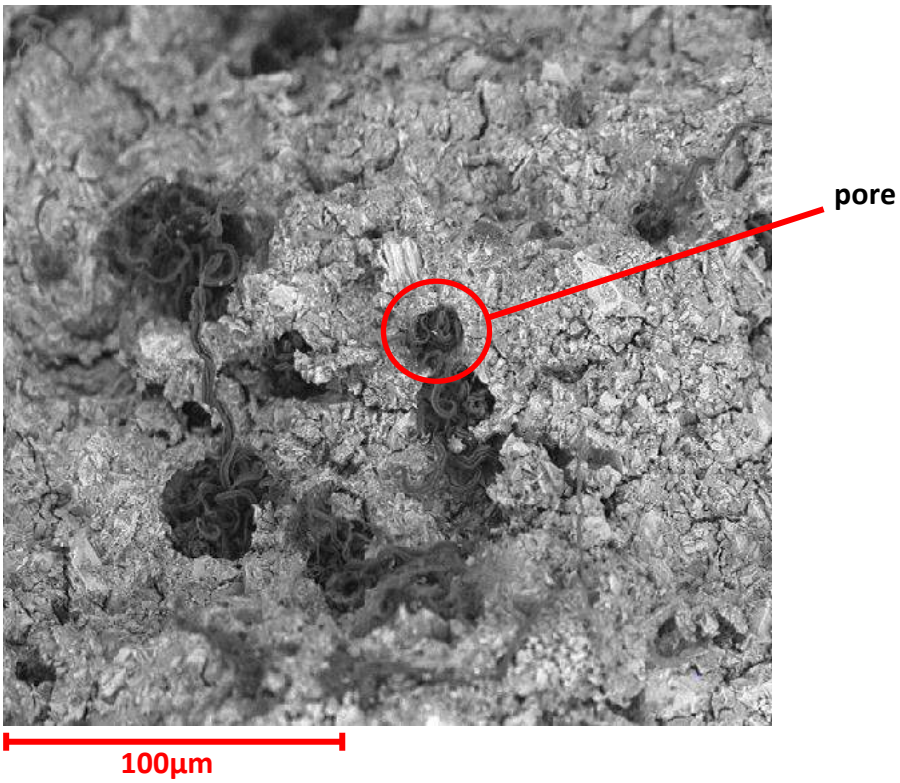


FIGURE 4.5 Fly-ash based porous geopolymer (Method 2)

Figure 4.5 shows the formation of porous membrane from fly-ash by using method 2 (baking soda and acetic acid). It is observed that the pores can be clearly seen but the structure of the samples itself shows that the geopolymer is not homogeneously mixed. Maybe this is because during the formation of alkaline solution, the ratio of sodium hydroxide to sodium silicate solution is not correctly measured. So the structure produced seems to be rough.

iii. Method 3: By using corn oil

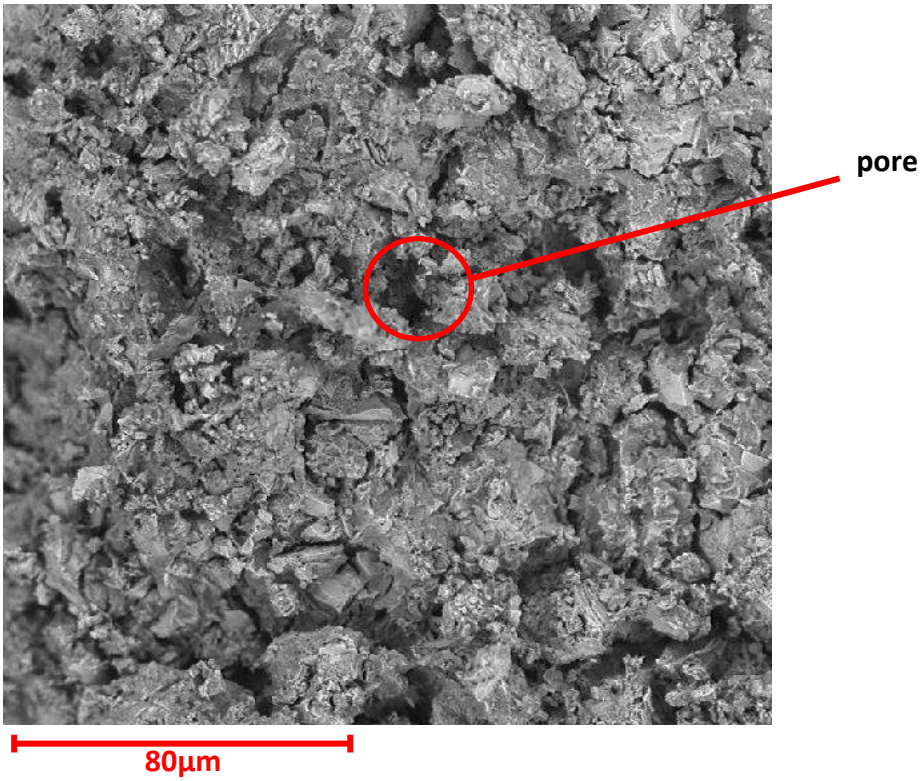


FIGURE 4.6 Fly-ash porous geopolymer (Method 3)

Figure 4.6 shows the formation of porous membrane from fly-ash by using method 3 (corn oil). It can be clearly seen that the structure of the sample have pores but the size of the pores itself is small. This is because during the drying process, the sample is not properly heated and dry. The sample then is soaked with THF solution to remove the oil from the sample. Since a small part of the sample is half-dried, the THF solution cannot removed all the oil that has been trapped inside the sample. So, less void space is produced and cause small distribution of pores is produced.

C. Thermogravimetric Analysis (TGA)

1) Meta-kaolin based-geopolymer

i. Method 1: By using calcium metal

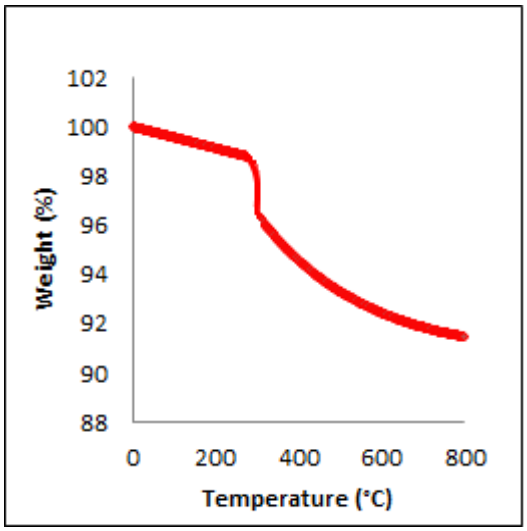


FIGURE 4.7 Meta-kaolin based porous geopolymer (Method 1)

For sample that has been synthesized by using calcium metal, the sample starts to loss its weight at 30 ºC. Between 30 ºC to 100 ºC, water vaporized to atmosphere is at highest rate. After that, the weight of the sample keep decreasing until all the water inside the sample is removed. At the end of the test which is at temperature at around 800 ºC, the weight loss is constant. The percentage of weight reduction is about 10%.

ii. Method 2: By using baking soda

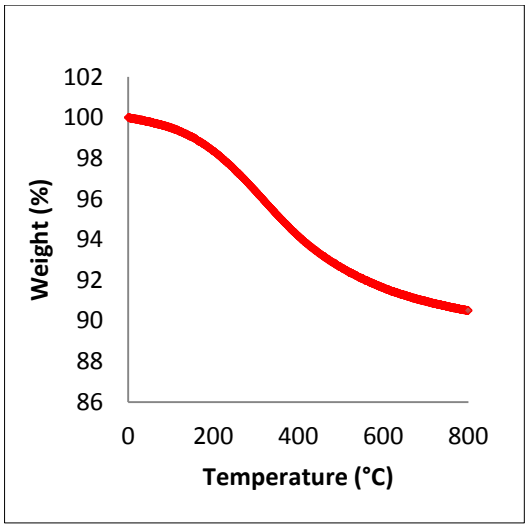


FIGURE 4.8 Meta-kaolin based porous geopolymer (Method 2)

The figure above shows the sample that has been synthesized by using baking soda and acetic acid, the sample starts to loss its weight at 40 ºC. Between 40 ºC to 400 ºC, water vaporized to atmosphere is at highest rate. After that, the weight of the sample keep decreasing until all the water inside the sample is removed. At the end of the test which is at temperature at around 800 ºC, the weight loss is constant. The percentage of weight reduction is about 12%.

iii. Method 3: By using corn oil

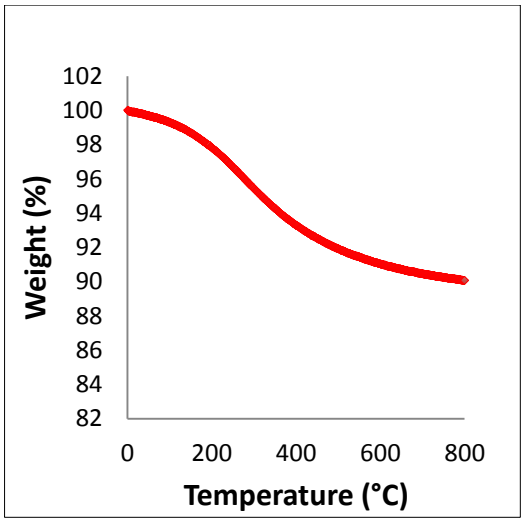


FIGURE 4.9 Meta-kaolin based porous geopolymer (Method 3)

For the sample above, it starts to loss its weight at 30 ºC. Between 30 ºC to 200ºC, most of the water content is vaporized to the atmosphere. After that, the weight of the sample keep decreasing until all the water inside the sample is removed. At the end of the test which is at temperature at around 800 ºC, the weight loss is constant. The percentage of weight reduction is about 17%.

2) Fly Ash based-geopolymer

i) Method 1: By using calcium metal

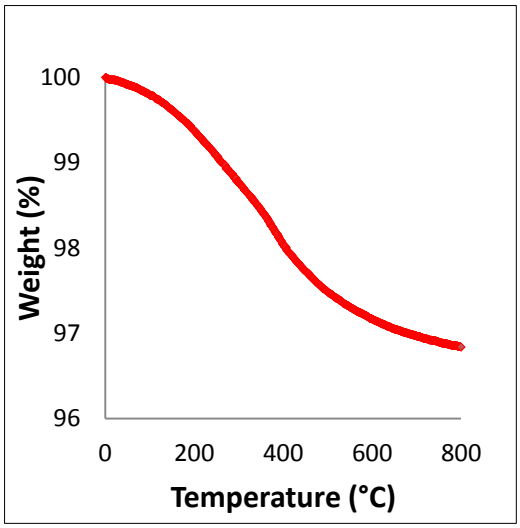


FIGURE 4.10 Fly-ash based porous geopolymer (Method 1)

For sample that has been synthesized by using calcium metal, the sample starts to loss its weight at 20 ºC. Between 30 ºC to 300 ºC, most of the water content inside the geopolymer sample is vaporized to atmosphere. Then, the weight of the sample keep decreasing until all the water inside the sample is removed. At the end of the test which is at temperature at around 8000 ºC, the weight loss is constant. The percentage of weight reduction is small which is about 7% only.

ii) Method 2: By using baking soda

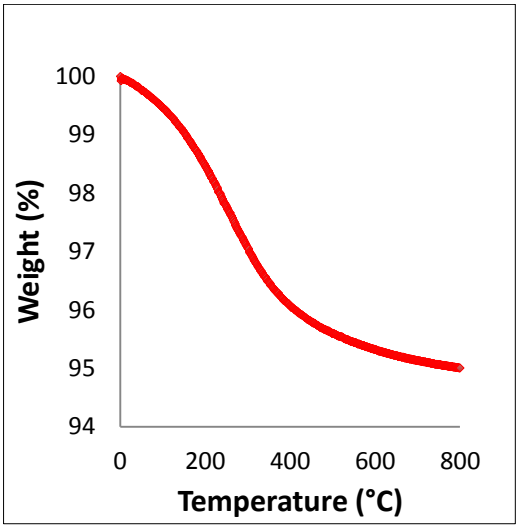


FIGURE 4.11 Fly-ash based porous geopolymer (Method 2)

For sample that has been synthesized by using baking soda, the sample starts to loss its weight at 20 ºC. Between 30 ºC to 300 ºC, water vaporized to atmosphere is at highest rate. After that, the weight of the sample keep decreasing until all the water inside the sample is removed. At the end of the test which is at temperature at around 800 ºC, the weight loss is constant. The percentage of weight reduction is about 10%.

iii) Method 3: By using corn oil

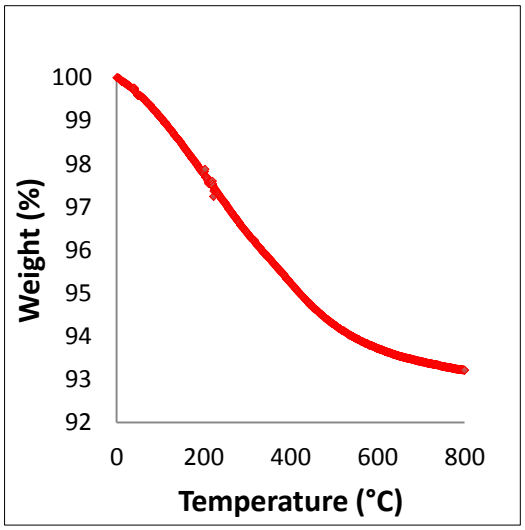


FIGURE 4.12 Fly-ash based porous geopolymer (Method 3)

From the figure above, it shows that the sample starts to loss its weight at 20 ºC. Between 30 ºC to 400ºC, most of the water content is vaporized to the atmosphere. After that, the weight of the sample keep decreasing until all the water inside the sample is removed. At the end of the test which is at temperature at around 800 ºC, the weight loss is constant. The percentage of weight reduction is about 10%.

D. Compression Test

1) Meta-kaolin based-geopolymer

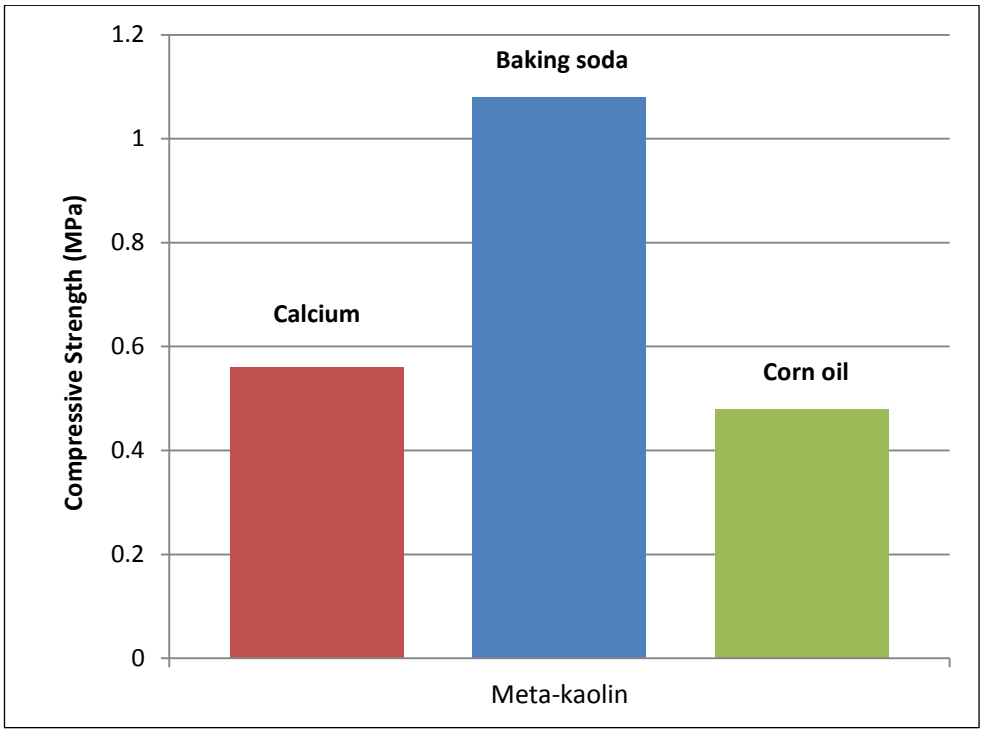


FIGURE 4.13 Compressive Strength for Meta-kaolin based-Geopolymer

From figure above, it shows that sample made of baking soda exhibit highest compressive strength which is about 1.08 MPa compared to calcium metal and corn oil. This is because from the percentage of porosity calculated, baking soda sample produced lowest percentage of porosity. Low porosity decrease the void space between the molecules inside the sample thus provides strong linkage. Stronger linkage between the molecule lead to higher compressive strength.

2) Fly Ash based-geopolymer

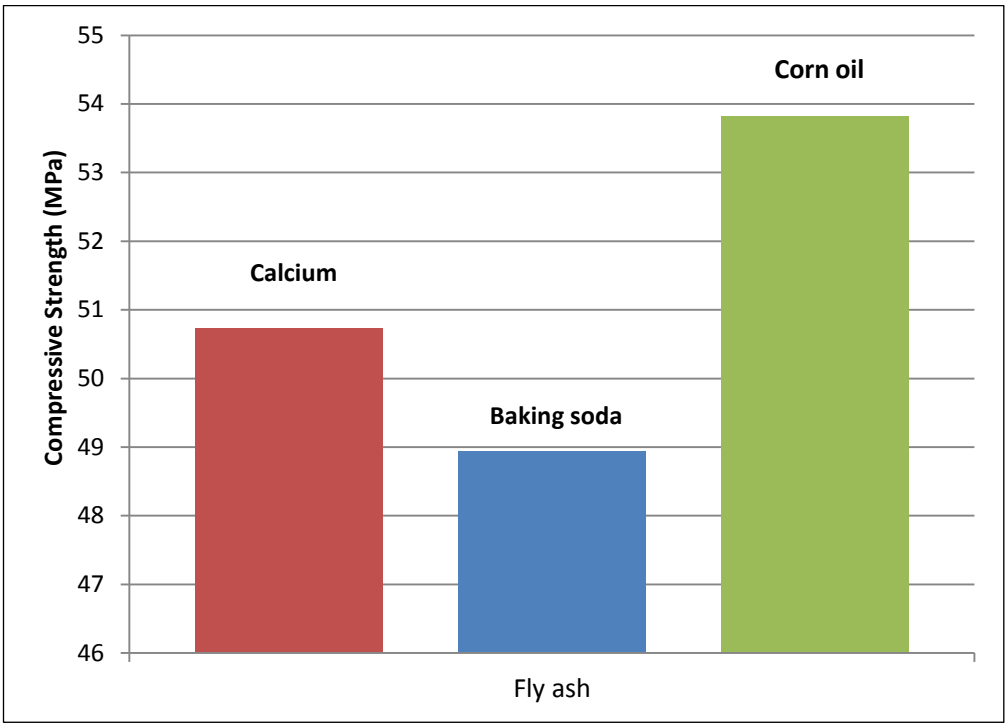


FIGURE 4.14 Compressive Strength for Fly Ash based-Geopolymer

From figure above, it shows that sample made of corn oil exhibit highest compressive strength which is around 54.0 MPa compared to calcium metal and baking soda. This is because from the percentage of porosity calculated, corn oil sample produced lowest percentage of porosity. Low porosity decrease the void space between the molecules inside the sample thus provides strong linkage. Stronger linkage between the molecule lead to higher compressive strength.

For overall comparison, it obviously shows that fly ash based-geopolymer produced higher compressive strength compared to meta-kaolin based geopolymer. This is because during the preparation of sample, sodium silicate used in meta-kaolin based geopolymer is greater than the required amount thus lead to increasing in the chain between the Si molecules. Longer chains have

more inter-chains bonds and cause the sample become more elastic. Higher elasticity of the sample causes the strength to decrease.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This project research presented a brief overall review on geopolymer composites that produced from meta-kaolin and fly-ash. The parameters that vary in this project is the method to produce pores which are by using calcium metal, by using soda and lastly by using corn oil. At the end of this research, a cost-effective geopolymer membrane from both meta-kaolin and fly-ash is successfully developed and all the recommended characterization techniques were analyzed. From the result obtained, it shows that meta-kaolin based geopolymer made of corn oil gives highest percentage of porosity compared to others. He porosity data obtained is supported by the scanning electron microscopy result and compression test. For SEM analysis, meta-kaolin based geopolymer shows a large diameter of void space compared to fly ash based-geopolymer. Higher temperature exposure for TGA test shows that the sample can withstand high temperature and thermally stable, so the materials is suitable to be used in industry For compression test, it also produced lowest compressive strength. This is because higher porosity lead to more void space present inside the sample thus causes the sample to easily broken. In order to assessed the effectiveness of the porous geopolymer membrane in waste water treatment, the percentage of porosity ca be controlled by varying the amount of pore agent used. It is depend on the size of the molecules present inside the wastewater itself.

5.2 Recommendation

In future, the optimum ratio of sodium hydroxide solution to sodium silicate solution can be improved by referring to past research paper to produced most favorable concentration of the geopolymer composites. Optimum ratio ensure the result obtained exhibit a well-mixed geopolymer composites.

Besides that, in order to obtain uniform distribution of pores on the surface of the geopolmer composites, the sample needs to be consistently mixed with automatic mixer during the preparation. Similar mixing time for all sample is important because the longer the time taken during mixing, the higher the amount of bubble produced on the surface of the sample.

Curing time also can affect the geopolymer produced. Higher curing time lead the sample became brittle. So, the optimum curing time should be used in order to get most favorable sample.

REFERENCES

Aguilar, A. S., Melo, J. P., & Olivares, F. H. (2013). Microstructural analysis of aerated cement pastes with fly ash, Metakaolin and Sepiolite additions. *Construction and Building Materials, 47*, 282-292.

Chang, C. (1988). Measuring density and porosity of grain kernels using a gas pycnometer. *Cereal chemistry (USA)*.

Dąbrowski, A. (2001). Adsorption — from theory to practice. *Advances in Colloid and Interface Science, 93*(1–3), 135-224.

Das, S. K., Mohapatra, A. K., & Rath, A. Geo-polymer Concrete–Green Concrete for the Future—A Review.

Davidovits, J. *30 years of successes and failures in geopolymer applications. Market trends and potential breakthroughs*.

Deshlahra, P., Mehra, A., & Ghosal, D. (2009). Evolution of bubble size distribution in baked foods. *Journal of Food Engineering, 93*(2), 192-199.

Drage, T. C., Kozynchenko, O., Pevida, C., Plaza, M. G., Rubiera, F., Pis, J. J., . . . Tennison, S. (2009). Developing activated carbon adsorbents for pre-combustion CO2 capture. *Energy Procedia, 1*(1), 599-605.

Eddy, D. M., & Schlessinger, L. (2003). Validation of the Archimedes diabetes model. *Diabetes Care, 26*(11), 3102-3110.

Ferdous, W., Manalo, A., Khennane, A., & Kayali, O. (2015). Geopolymer concrete-filled pultruded composite beams – Concrete mix design and application. *Cement and Concrete Composites, 58*(0), 1-13.

Kadirvelu, K., Kavipriya, M., Karthika, C., Radhika, M., Vennilamani, N., & Pattabhi, S. (2003). Utilization of various agricultural wastes for activated carbon preparation and application for the removal of dyes and metal ions from aqueous solutions. *Bioresource Technology, 87*(1), 129-132.

Knaebel, K. S. (2011). Adsorbent selection. *Adsorption Research Inc., http//www. adsorption. com/publications/AdsorbentSel1B. pdf (June 2004)*.

Le-Ping, L., Xue-Min, C., Shu-Heng, Q., Jun-Li, Y., & Lin, Z. (2010). Preparation of phosphoric acid-based porous geopolymers. *Applied Clay Science*, *50*(4), 600-603.

Link, C. C. (2006). Properties of activated carbon. *www. activated-carbon. com/carbon. html*.

Lopez Guzman, F. J. (2014). Study of Geopolymer Adsorbents Prepared from Metakaolin and Rice Husk Silica for Targeting to Heavy Metal Capture.

MacKenzie, K. J. D. (2015). 28 - Innovative applications of inorganic polymers (geopolymers). In F. Pacheco-Torgal, J. A. Labrincha, C. Leonelli, A. Palomo & P. Chindaprasirt (Eds.), *Handbook of Alkali-Activated Cements, Mortars and Concretes* (pp. 777-805). Oxford: Woodhead Publishing.

Seo, D., Medpelli, D., & Seo, J. (2013). Porous geopolymer materials: Google Patents.

Skouteris, G., Saroj, D., Melidis, P., Hai, F. I., & Ouki, S. (2015). The effect of activated carbon addition on membrane bioreactor processes for wastewater treatment and reclamation – A critical review. *Bioresource Technology, 185*(0), 399-410.

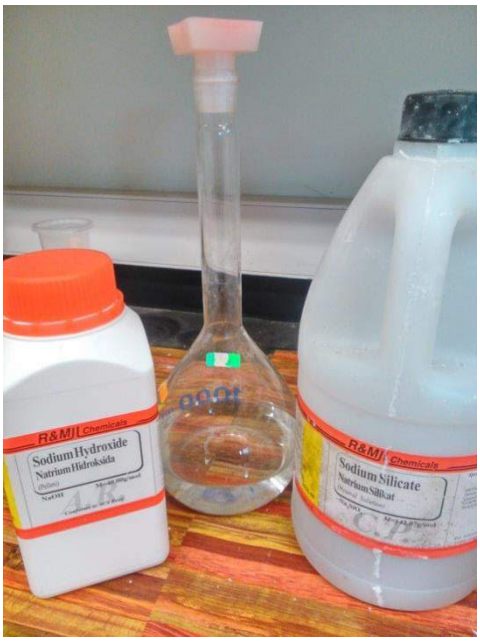
Thomas, W. J., & Crittenden, B. (1998). 3 - Fundamentals of adsorption equilibria. In W. J. Thomas & B. Crittenden (Eds.), *Adsorption Technology & Design* (pp. 31-65). Oxford: Butterworth-Heinemann.

APPENDICES

a. Porosity Equation

$$Porosity\ (\%) = \frac{Bulk\ Density - Powder\ Density}{Bulk\ Density} x\ 100$$

b. Materials and Chemical used



c. Curing process at 60 °C

d. Fly ash based-geopolymer composites

e. Meta-kaolin based-geopolymer

f. Compression Test Machine

g. Gas Pycnometer (Quantachrome Ultrapycnometer 1000) Machine