



**DESIGN, PROCESSING AND PROPERTIES OF
FLY ASH-BASED LIGHTWEIGHT GEOPOLYMER
USING FOAMING AGENT FOR BRICK
APPLICATION**

by

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TABLE OF CONTENTS

	PAGE
DECLARATION OF THESIS	i
DECLARATION OF THESIS	i
ACKNOWLEDGMENT	ii
TABLE OF CONTENTS	iii
LIST OF TABLES	vii
LIST OF FIGURES	ix
LIST OF ABBREVIATIONS	xiii
ABSTRAK	xiv
ABSTRACT	xv
CHAPTER 1 : INTRODUCTION	1
1.1 Research Background	1
1.2 Problem Statement	5
1.3 Research Objective	7
1.4 Scope of Study	7
1.5 Thesis Outline	8
CHAPTER 2 : LITERATURE REVIEW	10
2.1 Introduction	10
2.2 Brick	12
2.2.1 Lightweight Brick	14
2.3 Geopolymer	17
2.4 Geopolymerization Process	21
2.5 Lightweight Geopolymer	24
2.6 Constituents of Lightweight Geopolymer	27
2.6.1 Source Materials (Fly Ash)	28
2.6.2 Alkaline Activator	31
2.6.3 Superplasticizer (Foaming Agents)	34
2.7 Factors Affecting Properties of Lightweight Geopolymer	39
2.7.1 Concentration of Sodium Hydroxide (NaOH) Solution	39
2.7.2 Mix Design	41
2.7.3 Curing Temperature and Curing Time	44

2.8	Properties of Lightweight Geopolymer	49
2.8.1	Compressive strength	49
2.8.2	Water Absorption	51
2.8.3	Density	54
2.8.4	Thermal Properties	57
2.9	Particle Size, Composition and Morphology of Raw Materials	59
2.9.1	Chemical Composition Properties of Raw Materials	59
2.9.2	Microstructure Properties of Raw Materials and Lightweight Geopolymer	60
2.9.3	Particle Size Analysis of Raw Materials	63
2.10	Summary	65
	CHAPTER 3 : METHODOLOGY	66
3.1	Introduction	66
3.2	Research Materials	68
3.2.1	Fly Ash	68
3.2.2	Foaming Agents	68
3.2.3	Alkaline Activator Solution	69
3.3	Preparation of Sodium Hydroxide (NaOH) Solution	71
3.4	Preparation of Alkaline activator Solution	72
3.5	Preparation of Foam from Foaming Agent	72
3.6	Mix Design for Lightweight Geopolymer	74
3.6.1	Concentration of Sodium Hydroxide (NaOH) Solution	74
3.6.2	Foaming Agent/Water Ratio and Foam/Geopolymer Paste Ratio	75
3.6.3	Curing Temperature and Curing Time	77
3.7	Mixing, Moulding and Curing Process of Lightweight Geopolymer	78
3.8	Microstructure for Lightweight Geopolymer and Raw Materials	79
3.8.1	Chemical Composition Characterization	79
3.8.2	Morphology Characterization	79
3.8.3	Particle Size Analysis	80
3.9	Testing for Lightweight Geopolymer	80
3.9.1	Compressive Strength Test	80
3.9.2	Density Analysis	81
3.9.3	Water Absorption Test	82
3.9.4	Thermal Insulation Test	82

CHAPTER 4 : RESULT & DISCUSSION	85
4.1 Introduction	85
4.2 Characterization of Raw Materials	86
4.2.1 Chemical Composition Analysis of Fly Ash and Foaming Agent	86
4.2.2 Particle Size Analysis of Fly Ash	88
4.2.3 Morphology Analysis of Fly Ash	89
4.3 The Effects of NaOH Concentration on Fly Ash-Based Lightweight Geopolymer	90
4.3.1 Compressive Strength of Lightweight Geopolymer	90
4.3.2 Water Absorption of Lightweight Geopolymer	92
4.3.3 Density of lightweight Geopolymer	94
4.3.4 Correlation between Compressive Strength and Water Absorption of Lightweight Geopolymer	96
4.3.5 Correlation between Compressive Strength and Density of Lightweight Geopolymer	97
4.3.6 Microstructure of Lightweight Geopolymer	98
4.4 The Effects of Foaming Agent/Water Ratio and Foam/Geopolymer Paste Ratio of Lightweight Geopolymer	101
4.4.1 Compressive Strength of Lightweight Geopolymer	101
4.4.2 Water Absorption of Lightweight Geopolymer	102
4.4.3 Density of lightweight Geopolymer	104
4.4.4 Microstructure of Lightweight Geopolymer	106
4.5 The Effects of Curing Temperature and Curing Time on Lightweight Geopolymer	108
4.5.1 Compressive Strength of Lightweight Geopolymer	108
4.5.2 Water Absorption of Lightweight Geopolymer	110
4.5.3 Density of Lightweight Geopolymer	111
4.5.4 Microstructure of Lightweight Geopolymer	113
4.6 The Effects of Ageing on Compressive Strength, Density and Thermal Insulation Properties of Lightweight Geopolymer	115
4.6.1 Compressive Strength of Lightweight Geopolymer	115
4.6.2 Density of Lightweight Geopolymer	117
4.6.3 Thermal Conductivity of Lightweight Geopolymer	118
4.6.4 Thermal Diffusivity of Lightweight Geopolymer	120

4.6.5	Specific Heat of Lightweight Geopolymer	121
4.6.6	Correlation between Thermal Conductivity with Compressive Strength and Density of Lightweight Geopolymer	122
4.6.7	Correlation between Thermal Diffusivity with Compressive Strength and Density of Lightweight Geopolymer	125
4.7	Overall Summary	127
CHAPTER 5 : CONCLUSION AND RECOMMENDATIONS		128
5.1	Conclusion	128
5.2	Recommendations	130
REFERENCES		132
APPENDIX A		147
APPENDIX B		149
APPENDIX C		151

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LIST OF TABLES

NO.	PAGE
Table 2.1: Data analysis of different types of bricks/blocks (Mahendran et. al., 2016)	16
Table 2.2: Development of geopolymer application (Davidovits, 2002)	20
Table 2.3: Applications of geopolymer materials depends on Si/Al ratio (Davidovits, 1994)	21
Table 2.4: Chemical requirements of fly ash (ASTM C 618, 2012).	29
Table 2.5: Groups of alkaline activator solution (Pacheco-Torgal et al., 2008a).	32
Table 2.6: Curing conditions for preparation of lightweight geopolymer	48
Table 2.7: Density, water absorption and compressive strength classification requirements for loadbearing concrete masonry units based on ASTM standard (ASTM C 90-11b)	57
Table 2.8: Chemical contents of fly ash	60
Table 3.1: Properties of foaming agent (Polyoxyethylene Alkyether Sulfate)	69
Table 3.2: Properties of sodium hydroxide (NaOH)	70
Table 3.3: Specification of sodium silicate solution (Na ₂ SiO ₃)	71
Table 3.4: Details preparation for one (1) Liter NaOH solution	72
Table 3.5: Mix design for Different NaOH Molarity	75
Table 3.6: Mix design for various ratios of foaming agent/water and geopolymer paste/foam	76
Table 3.7 : Mix design for control sample	76
Table 3.8: Mix design for determination of curing temperature and curing time	77

Table 4.1: Chemical composition of fly ash 87

Table 4.2: Chemical composition of foaming agent (Polyoxyethylene Alkyether Sulfate) 88

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LIST OF FIGURES

NO.	PAGE
Figure 2.1: Projected share of global CO ₂ emissions due to cement production and to all other areas of human activity, in 2012 and 2050 (Provis, 2014).	13
Figure 2.2: The schematic drawings showing the process from fly ash to fly ash-based geopolymer cement/concrete (Zhuang et al., 2016)	18
Figure 2.3: Dependent upon the Si/Al molar ratio, different aluminosilicate chains are formed in the aluminosilicate oligomers which then further to form geopolymer Zhuang et al., (2016).	19
Figure 2.4: Conceptual model for geopolymerization (Duxson et al., 2007).	22
Figure 2.5: Schematic formation of geopolymer (Davidovits, 1994).	23
Figure 2.6: Descriptive model of the alkali activation of fly ash (Fernández-Jiménez, Palomo, & Criado, 2005)	24
Figure 2.7: The possible reaction mechanism of modifier induced foamed geopolymer (Yang et al., 2012)	26
Figure 2.8: Maximum application temperature of some insulation materials (Davidovits, 2008)	27
Figure 2.9: The effects of ageing on compressive strength of geopolymer (Arioz et al., 2013).	46
Figure 2.10: Compressive strength and density of foamed geopolymer after 7 days (Hajimohammadi et al., 2017)	50
Figure 2.11: Relationship between average water absorption and average porosity after day 7, day 14, day 28 until day 90 (Farhana et al., 2014)	53
Figure 2.12: Effects of foam dosage to the water absorption (Zhao et al., 2010)	54

Figure 2.13: Relationship between porosity and density of foamed geopolymer (Yong et al., 2014)	55
Figure 2.14: Microstructure of fly ash (Alehyen et al., 2017)	61
Figure 2.15: Microstructure and distribution of pore of foamed geopolymer (c) and (e): curing at room temperature, (d) and (f) curing at 60 °C (Abdullah et al., 2012)	62
Figure 2.16: SEM images of metakaolin geopolymer foams produced using different amounts of hydrogen peroxide ((a) 0.0; (b) 0.05; (c) 0.10; (d) 0.15) (Bai et al., 2016)	63
Figure 2.17: Particle size distribution pattern of the different ashes (Nazari et al., 2011)	64
Figure 3.1: Flow chart for the preparation of lightweight geopolymer	67
Figure 3.2: Foam generator machine	73
Figure 3.3: A schematic diagram showing the making of foam using foam generato	73
Figure 3.4: Schematic illustration for the preparation of lightweight geopolymer	79
Figure 3.5: Sketch of the Hot Disk TSP 2500 system (Zhang, 2014).	83
Figure 3.6: Schematic diagram of the experimental setup	84
Figure 4.1: Particle size distribution of fly ash	89
Figure 4.2: Microstructure images of fly ash	90
Figure 4.3: The compressive strength of lightweight geopolymer with different molarity of NaOH solution.	91
Figure 4.4: The water absorption of lightweight geopolymer with different molarity of NaOH solution.	93

Figure 4.5: The density of lightweight geopolymer with different molarity of NaOH solution.	95
Figure 4.6: The relationship between compressive strength and water absorption of lightweight geopolymer	97
Figure 4.7: The relationship between compressive strength and density of lightweight geopolymer	98
Figure 4.8: Microstructure of lightweight geopolymer with different molarity of NaOH solution ; (a) 6 M, (b) 8 M, (c) 10 M, (d) 12 M and (e) 14 M	100
Figure 4.9: The compressive strength of lightweight geopolymer with different ratio of foaming agent/water ratio and foam/geopolymer paste ratio	102
Figure 4.10: The water absorption of lightweight geopolymer with different ratio of foaming agent/water ratio and foam/geopolymer paste ratio	103
Figure 4.11: The density of lightweight geopolymer with different ratio of foaming agent/water ratio and foam/geopolymer paste ratio	105
Figure 4.12: The microstructure of lightweight geopolymer with different ratio of foam/geopolymer paste and control sample; (a) control sample, (b) 0.5, (c) 1.0, (d) 1.5 and (e) 2.0, by volume.	107
Figure 4.13: The compressive strength of lightweight geopolymer with different curing conditions	109
Figure 4.14: The water absorption of lightweight geopolymer with different curing conditions	111
Figure 4.15: The density of lightweight geopolymer with different curing conditions	112
Figure 4.16: The microstructure of lightweight geopolymer with different curing time; (a) cured at room temperature, (b) 6 hours, (c) 12 hours, (d) 24 hours and (e) 48 hours.	114

Figure 4.17: The compressive strength of lightweight geopolymer with different ageing time	116
Figure 4.18: The density of lightweight geopolymer with different ageing time	117
Figure 4.19: The thermal conductivity of lightweight geopolymer at different ageing time	119
Figure 4.20: The thermal diffusivity of lightweight geopolymer at different ageing time	120
Figure 4.21: The specific heat of lightweight geopolymer at different ageing time	122
Figure 4.22: The relationship between thermal conductivity and compressive strength of lightweight geopolymer	123
Figure 4.23: The relationship between thermal conductivity and density of lightweight geopolymer	124
Figure 4.24: The relationship between thermal diffusivity and compressive strength of lightweight geopolymer	125
Figure 4.25: The relationship between thermal diffusivity and density of lightweight geopolymer	126

LIST OF ABBREVIATIONS

AAC	Aerated Autoclaved Concrete
Al	Aluminum
Al ₂ O ₃	Aluminum Oxide
ASTM	American Society for Testing and Materials
CaCO ₃	Calcium Carbonate
CLC	Cellular Lightweight Concrete
CO ₂	Carbon Dioxide
EDS	Energy Dispersive Spectroscopy
FeSi	Ferrosilicon
H ₂ O ₂	Hydrogen Peroxide
μm	Micrometer
Na ⁺	Sodium Ion
NaOH	Sodium Hydroxide
OPC	Ordinary Portland Cement
SEM	Scanning Electron Microscope
SiC	Silicon Carbide
XRF	X-ray Fluorescence Spectroscopy
LOI	Loss on Ignition
Wt. %	Weight per cent
Zn	Zinc

Rekabentuk, Pemprosesan Dan Sifat-Sifat Geopolimer Ringan Berasaskan Abu Terbang Menggunakan Agen Berbuih Untuk Aplikasi Bata

ABSTRAK

Konkrit ringan mengurangkan berat keseluruhan struktur yang mengakibatkan pengurangan saiz asas, kos, dan spesifikasi lain. Walaubagaimanapun, penghasilan konkrit ringan konvensional menyebabkan beberapa kesan alam sekitar dan menghasilkan sifat mekanikal yang rendah, jadi terdapat keperluan yang jelas untuk mencari dan menggantikan pilihan yang lebih cekap dan tahan lama bagi mengatasi konkrit ringan konvensional. Geopolymer merupakan peluang yang baik untuk memastikan kemampunan yang lebih besar dalam industri pembinaan terutamanya untuk penggunaan sisa industri seperti abu terbang. Kajian ini memberi tumpuan kepada penyediaan geopolimer ringan berasaskan abu terbang dengan menggunakan superplasticizer sebagai agen berbuih. Superplasticizer (Polyoxyethylene alkyether sulfate) telah disediakan menggunakan kaedah pra-terbentuk dengan gabungan air dan tekanan udara. Kesan parameter bagi sintesis geopolimer seperti kepekatan NaOH (6 M, 8 M, 10 M, 12 M dan 14 M), nisbah agen berbuih kepada air (1/10, 1/20, 1/30 dan 1/40 dengan isipadu, nisbah buih kepada pes geopolimer (0.5, 1.0, 1.5 dan 2.0) mengikut isipadu, suhu pengawetan (40 °C, 60 °C, 80 °C dan 100 °C) dan masa pengawetan (6, 12, 24 dan 48) jam terhadap pes geopolimer ringan yang memberi kesan kepada sifat-sifat mekanikal dan mikrostruktur dikaji secara terperinci. Kekuatan mampatan, penyerapan air, ketumpatan, dikaji untuk menentukan sifat mekanik geopolimer ringan. Ciri-ciri penebat haba disiasat melalui kesan kekonduksian terma, kelesuan haba, dan haba khusus bagi geopolimer ringan pada masa penuaan yang berbeza (3, 7, 28, 60 dan 90) hari. Ciri-ciri mikrostruktur geopolimer ringan telah diuji dengan menggunakan Mikroskop Pengimbasan Elektron. Hasilnya menunjukkan bahawa geopolimer ringan mempunyai kepekatan larutan NaOH yang optimum pada 12 M, dengan kekuatan mampatan maksimum 15.2 MPa pada 7 hari, nisbah optimum agen berbuih kepada air (1/10) dan nisbah buih kepada pes geopolimer (1.0) dengan kekuatan tertinggi 16.6 MPa (7 hari) dan suhu pengawetan optimum (80 °C) dan masa pengawetan (24 jam) menunjukkan kekuatan tertinggi dan ketumpatan terendah sebanyak 15.6 MPa dan 1400 kg / m³. Kekonduksian terma dan kelimpahan terma geopolimer ringan lebih rendah dengan nilai 0.63 W / mK hingga 0.83 W / mk dan 0.26 mm² / s kepada 0.35 mm² / s, masing-masing. Satu bahan pembinaan ringan yang berpotensi boleh dihasilkan dengan menggunakan agen berbuih kos rendah dan mudah diproses bagi penambahan kepada pes geopolimer. Geopolimer ringan berasaskan abu terbang yang dihasilkan dalam kajian ini mempamerkan kekuatan mampatan sesuai dengan standard untuk aplikasi blok ringan pada suhu pengawetan yang lebih rendah (80 °C).

Design, Processing and Properties of Fly Ash-Based Lightweight Geopolymer Using Foaming Agents For Brick Application

ABSTRACT

Lightweight concrete reduces the overall self-weight of the structures resulting in the reduction of the foundation size, cost, and other specification. However, the conventional lightweight concrete production causes several environmental impacts and produce low mechanical properties, so there is a clear need of searching and replacing for more efficient and durable alternatives beyond the limitations of the conventional lightweight concrete. Geopolymer represents a great opportunity to ensure greater sustainability in the construction industry especially for the use of industrial waste such as fly ash. This research focuses on the preparation of fly ash-based lightweight geopolymer using superplasticizer as foaming agent. The superplasticizer (Polyoxyethylene alkyether sulfate) was prepared using pre-formed method by combination with water and air pressure. The effects of geopolymeric synthesis parameters such as the NaOH concentration (6 M, 8 M, 10 M, 12 M and 14 M), ratio of foaming agent to water (1/10, 1/20, 1/30 and 1/40) by volume, ratio of foam to geopolymer paste (0.5, 1.0, 1.5 and 2.0) by volume, curing temperature (40 °C, 60 °C, 80 °C and 100 °C) and curing time (6, 12, 24 and 48) hours on the lightweight geopolymer paste that affect the mechanical and microstructure properties were studied in detailed. The compressive strength, water absorption, density, were studied to determine the mechanical properties of lightweight geopolymer. The thermal insulation properties was investigated through the effects of thermal conductivity, thermal diffusivity, and specific heat of lightweight geopolymer at different ageing time (3, 7, 28, 60 and 90) days. The microstructure properties of lightweight geopolymer were tested by using Scanning Electron Microscope. The results indicated that the lightweight geopolymer have an optimum NaOH concentration of 12 M, with highest compressive strength of 15.2 MPa at 7 days, an optimum ratio of foaming agent to water (1/10) and ratio of foam to geopolymer paste (1.0) with highest strength of 16.6 MPa (7 days), optimum curing temperature (80 °C) and curing time (24 hours) showed the highest strength and lowest density of 15.6 MPa and 1400 kg/m³, respectively. The thermal conductivity and thermal diffusivity of lightweight geopolymer are substantially lower with value of 0.63 W/mK to 0.83 W/mk and 0.26 mm²/s to 0.35 mm²/s, respectively. A potential new lightweight construction material can be produced by using low cost of foaming agent and easy to process for addition to geopolymer paste. The fly ash-based lightweight geopolymer produced in this work exhibit compressive strength in accordance to the standard for masonry lightweight applications at considerably lower curing temperature (80 °C).

CHAPTER 1 : INTRODUCTION

1.1 Research Background

Concrete Masonry Units (CMUs) are one of the most widely used elements in buildings facades and widely used in building construction as loadbearing and non-loadbearing walls (El-Hassan et. al., 2014). However, this type of concrete presents some weaknesses compared to other façade materials, such as high environmental impact, high density, and high thermal conductivity (Madrid et. al., 2017). Due to this problem, some attempt has been made to improve the properties of concrete masonry units by using alternative method and raw materials used which can remedy the weakness of CMUs.

Demand is increasing for affordable and lightweight construction materials with superior mechanical properties. Lightweight concrete can be classed according to its unit weight or density, which normally ranges from 320 kg/m³ to 1920 kg/m³ according to the ACI Committee 213 Guide for Structural Lightweight Aggregate Concrete (ACI 213, 2010). Lightweight concrete masonry produces some advantages such as reduces the dead load of the building, relatively low thermal conductivity, easy to handle and hence reduces the cost of transportation and handling and also comparatively more durable (El-Hassan et. al., 2014). The challenge in making a lightweight concrete is decreasing the density while maintaining the strength of lightweight concrete. Lightweight concrete masonry could be produced either by using lightweight aggregates (natural lightweight aggregates or artificial lightweight aggregates) or

admixtures (air entraining agent or admixtures that develop gases) (Kan and Demirboga, 2009).

The lightweight concrete prepared using admixtures are commonly manufactured by two different methods either by pre-foaming method or mixed foaming method. Pre-foaming method prepared by mixing a pre-formed foam (foam agent with water) mixture into a cement paste or mortar. As the concrete hardens, the bubbles disintegrate leaving air voids of similar sizes (Mustapure, 2014; Thakrele, 2014). In mixed foaming method, known as autoclaved aerated concrete (AAC) consists of a mix of cement, sand, water, lime and an expansion agent (chemical foaming agent). The bubble is made by adding expansion agents (aluminium powder or hydrogen peroxide) to the mix during the mixing process. This creates a chemical reaction that generates gas, either as hydrogen or as oxygen to form a gas-bubble structure within the concrete (Schnitzler, 2006).

There are two types of commercialized lightweight concrete masonry available in markets which are Cellular Lightweight Concrete (CLC) bricks and Aerated Autoclaved Concrete (AAC) bricks. The AAC brick production suffers from some disadvantages such as increased plant precautions due to the explosive nature of hydrogen gas, difficulties in the production control and high energy consumptions (Tsaousi et. al., 2016). Besides, production of these lightweight concrete also involve high pressure steam curing (autoclaved) with high curing temperature (180 °C to 210 °C) to and needs of superplasticizer to improve the compressive strength of lightweight concrete (Wongkeo et. al., 2012; Aminudin et. al., 2015).

The CLC brick was made using cement, water, fly ash and foaming agent and undergoes curing process in water curing procedure for 12 to 14 days and in steam curing procedure 12 hours are required. The performance of CLC brick in term of compressive strength is low for the density of CLC brick range of 400 kg/m³ to 1800 kg/m³ (Awana and Kumar, 2017). CLC also requires pozzolan materials such as fly ash or special additives to improve the properties of CLC bricks and helps in achieving more uniform distribution of air voids (Jitchaiyaphum et. al., 2011; Marunmale, 2014).

Another additional issue is that during production of 1 ton of Portland cement about one ton of carbon dioxide is released. Implementation of geopolymer technology will reduce the production of Portland cement and consequently the amount of carbon dioxide emission in atmosphere (Kargin et. al., 2017). Due to their performance properties, geopolymers have been evaluated as potential replacement for inorganic binders in lightweight concrete (Sanjayan et. al., 2015; Liu et. al., 2014; Wongsa et. al., 2016; Risdanareni et. al., 2017). Geopolymer involves a chemical reaction between aluminosilicate materials with strongly alkaline solution which form a rigid polymer (Davidovits, 1994).

Geopolymeric brick are considered as a new technology in which are eco-sustainable masonry units because they possess good mechanical and thermal properties as well as widen the possibilities to recycle waste material to useful products especially for building material (Petrillo et. al., 2016). Geopolymer lightweight bricks are more sustainable type of the lightweight concrete because of utilization of waste as source materials, manufactured at temperatures below 100 °C, reduces the carbon dioxide

emissions and has better resistance to the chemical and fire (Hajimohammadi et. al., 2017)

One of the most popular source materials for production of geopolymer is fly ash. Fly ash is defined as the ‘finely divided residue causing from the burning of powdered coal or ground, which is conveyed from the fire box through the container by flue gases’ (Senapati, 2011). The main constituents of fly ash are alumina (Al_2O_3), silica (SiO_2), and iron oxides (Fe_2O_3), with varying amounts of calcium, magnesium, sulphur and carbon (Wang and Wu, 2006). The utilization of fly ash as a source material to building and construction materials including cement, concrete, building bricks, and also aggregates is a beneficial approach because it not only transforms the wastes materials into useful materials but it also improves the dumping problems. In the synthesis of geopolymeric materials, utilization of fly ash has been reported by several researchers (Skvara et. al., 2005; Zhuang et. al., 2016; Saravanan et. al., 2013; Van Jaarsveld et. al., 2003).

There is very limited information on the uses of superplasticizer as foaming agent in lightweight geopolymer for concrete masonry applications. Therefore, for this research, the fly ash-based lightweight geopolymer were prepared with pre-form method of foaming agent to study the effect of several mixes of foaming agent and process method to the properties of lightweight geopolymer and the feasibility of using superplasticier only as foaming agent in geopolymer materials. The result is very important for the understanding and future improvement for this lightweight material.

1.2 Problem Statement

Conventional concrete masonry are used in high rise buildings which causes several problems. For examples, causing heavy dead load, greater stresses are generated, and more reinforcement and heavy sections are needed (Marunmale, 2014). This problem could be overcome by reducing the weight of masonry units. Normal weight of concrete masonry units is 2200 kg/m^3 to 2600 kg/m^3 (Aidan et. al., 2009). Lightweight concrete masonry begin in the density of less than 1600 kg/m^3 (Hamad, 2014). Lightweight concrete masonry seems to be a partial solution for general environment problems depending on the raw materials used and method processing. Hence, lightweight geopolymer materials represent decrease load solution and others concrete masonry properties improvement.

Due to increases in construction of multi stories buildings by every passing year, it is necessary to use lightweight material in the building construction but in the same time it does not cause any problems with strength and durability. Most of the published investigations on lightweight concrete masonry used OPC cement, with high curing temperature and longer curing conditions such as Cellular Lightweight Concrete (CLC) and Autoclaved Aerated Concrete (AAC). The manufacturing of AAC and CLC required autoclave curing at $190 \text{ }^\circ\text{C}$ to $210 \text{ }^\circ\text{C}$ and steam curing for 10 hours causes high energy consumptions during production process (Srinivas, 2016; Marunmale, 2014). Due to awareness of these problems, production of lightweight materials with low energy consumption at low temperature (below $100 \text{ }^\circ\text{C}$) and short curing time need to be made to find an alternative environmentally method.

A common issue faced by the conventional lightweight concrete is mainly poor mechanical properties which lead to durability issues (Jitchaiyaphum et. al., 2011). Therefore in this study the need of lightweight geopolymer concrete is important as it promises better mechanical properties such as produce in high compressive strength with low density performance. Existing commercial lightweight concrete also generally possess low thermal insulation properties but has lower strength as compared to lightweight concrete prepared with geopolymer method. In terms of mechanical properties, geopolymer lightweight has enhanced properties without lowering its thermal insulation properties because it is known to have thermal conductivity value within the acceptable market range of 0.4 W/mK to 0.9 W/mK which depends on the density obtained (Zhang et. al., 2015).

Chemical foaming agent used in AAC block proved to have issues in producing lightweight concrete with various size of pores and the chemical reaction occurred rapidly (Hilal et. al., 2015). Due to this problem, utilization of chemical foaming agent required addition of superplasticizer or modifier to slow down the rate of foaming reaction and to form the homogeneous pores in the lightweight concrete (Aini et. al., 2017). In terms of cost evaluation, fly ash-based lightweight geopolymer is proven to be more efficient as it only requires superplasticizer which could be foamed using foam generator because the behavior of this type of superplasticizer function as structure and the degree of polymerization. The effects of strong polar hydrophilic group (SO_3) caused the dispersion of cement particles occurred called as steric hindrance which are better in air entrainment for foaming agent characteristics.

1.3 Research Objective

The aim of this research is to produce fly ash-based lightweight geopolymer for lightweight brick application by using foaming agent through geopolymerization process. The objectives of this study were:

1. To investigate the effects of sodium hydroxide (NaOH) concentration on fly ash-based lightweight geopolymer based on the compressive strength.
2. To obtain the optimum foaming agent/water ratio and foam/geopolymer paste ratio on producing fly ash-based lightweight geopolymer based on the compressive strength performance.
3. To determine the optimum curing temperature and curing time for fly ash-based lightweight geopolymer based on the compressive strength and density analysis.
4. To analyse the effects of ageing on compressive strength, density and thermal insulation properties of fly ash-based lightweight geopolymer.

1.4 Scope of Study

The investigation was conducted using a two phase approach. The first phase of this investigation was to develop a lightweight geopolymer incorporated with foaming agent and to evaluate how these lightweight geopolymer affect the compressive strength and density performances. In order to develop a viable lightweight geopolymer a series of steps must be completed. First, a viable paste mix design must be developed. The geopolymer paste was produced from the reaction of fly ash and alkaline activator

solution (sodium silicate and sodium hydroxide solution). The foaming agent used for this study is come from superplasticizer type. The entire tests conducted are in accordance to the standard which is American Society for Testing and Materials (ASTM). The physical and mechanical properties are focusing on the compressive strength test, water absorption test and density analysis. Different sodium hydroxide concentration, different mixing ratio included foaming agents/water ratio, foam/geopolymer paste ratio, and different curing conditions (curing temperature and curing time) were also studied to investigate their effect on fly ash-based lightweight geopolymer. The thermal insulation including thermal conductivity, thermal diffusivity, and specific heat were analyzed using Transient Plane Source (TPS) method on a Hot Disk Thermal Constants Analyzer at different of ageing time. Microstructure analysis was carried out by using scanning electron microscopy (SEM) and X-Ray Fluorescence (XRF) was performed to investigate the chemical composition of raw materials used and lightweight geopolymer samples.

1.5 Thesis Outline

The thesis is separated into five chapters. Chapter 1 introduces the research background, problem statement, research objective, scope of study, and also the outline of the thesis.

Chapter 2 discusses about the general information of geopolymer, geopolymerization process, literature review based on the lightweight bricks, lightweight geopolymer technology, manufacturing process and properties of lightweight geopolymer. In addition, the lightweight geopolymer performance, types of experiment

carried out by previous researchers and application of lightweight geopolymer materials are also reviewed.

Chapter 3 explains the details of the materials used and the methodology applied in the research to develop the mix design, the mixing process and the curing process of fly ash-based lightweight geopolymer. This chapter also describes the physical, mechanical and microstructural tests of lightweight geopolymer according to the ASTM standards.

Chapter 4 presents the test results and discusses the findings of the experimental program. The materials characterization, the properties and effects of several factors affecting the performance of lightweight geopolymer were also discussed in this chapter.

Chapter 5 states the conclusions of this study and some recommendations for future work. The thesis ends with a Reference List and several Appendices.