



**DEVELOPMENT OF KAOLIN GEOPOLYMER
CERAMIC WITH ADDITION OF ULTRA HIGH
MOLECULAR WEIGHT POLYETHYLENE (UHMWPE)
AS BINDER FOR LIGHTWEIGHT CERAMICS**

by

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TABLE OF CONTENTS

	PAGE
THESIS DECLARATION	i
ACKNOWLEDGEMENT	ii
TABLE OF CONTENTS	iii
LIST OF TABLES	viii
LIST OF FIGURES	ix
LIST OF ABBREVIATIONS	xiii
ABSTRAK	xiv
ABSTRACT	xv
CHAPTER 1 INTRODUCTION	
1.1 Research Background	1
1.2 Problem Statement	4
1.3 Research Objectives	6
1.4 Scope of Study	6
1.5 Thesis Outline	7
CHAPTER 2 LITERATURE REVIEW	
2.1 Introduction	9
2.2 Ceramics	10
2.2.1 Ceramics Fabrication	11
2.2.1.1 Melt Casting	12
2.2.1.2 Sintering of Powder	13
2.2.2 Sintering of Ceramic	14

2.2.3	Ceramic Binder	15
2.2.3.1	Ultra High molecular Weight Polyethylene (UHMWPE)	18
2.3	Lightweight Ceramics	19
2.4	Geopolymers	20
2.4.1	Sources of Materials	23
2.4.1.1	Kaolin	24
2.4.2	Alkaline Activator Solution	27
2.4.2.1	Alkali Hydroxide	29
2.4.2.2	Alkali Silicates	30
2.4.3	Geopolymerization	31
2.4.4	Factor Affecting the Geopolymer Properties	34
2.4.5	High Temperature Exposure Geopolymer	37
2.5	Geopolymer Ceramics	39
2.5.1	Fabrication Method of Geopolymer Ceramics	42
2.5.2	Sintering Temperature of Geopolymer Ceramic	43
2.6	Geopolymer Ceramics Properties	44
2.6.1	Particle Size	44
2.6.2	Flexural Strength	45
2.6.3	Density	46
2.6.4	Shrinkage	48
2.6.5	Phase Properties	49
2.6.6	Thermal Properties	55
2.6.7	Microstructural Properties	57
2.6.8	Functional Group Identification	59
2.7	Summary	62

CHAPTER 3 RESEARCH METHODOLOGY

3.1	Introduction	63
3.2	Research Planning	64
3.3	Materials	66
3.3.1	Kaolin	66
3.3.2	Alkaline Activator	66
3.3.3	Ultra High Molecular Weight Polyethylene (UHMWPE)	67
3.4	Fabrication of Kaolin Geopolymer Ceramics	68
3.4.1	Preparation of Sodium Hydroxide (NaOH) Solution	68
3.4.2	Preparation of Alkaline Activator Solution	68
3.4.3	Fabrication of Kaolin Geopolymer	69
3.4.4	Mixing Process	69
3.4.5	Compaction	70
3.4.6	Sintering	70
3.5	Physical and Mechanical Testing of Sintered Sample	71
3.5.1	Particle Size	72
3.5.2	Flexural Strength Test	72
3.5.3	Density Test	73
3.5.4	Volumetric Shrinkage	73
3.5.5	Water Absorption Test	74
3.6	Materials Characterization Testing	74
3.6.1	Chemical Composition	75
3.6.2	Phase Characterization	75
3.6.3	Thermal Characterization	76
3.6.4	Morphology Characterization	76

3.6.5	Functional Group Identification	76
3.7	Correlation Study	77
CHAPTER 4 RESULTS AND DISCUSSION		
4.1	Introduction	78
4.2	Characterization of Raw Material	79
4.2.1	Particle Size Analysis	79
4.2.2	Chemical Composition Analysis	80
4.2.3	Phase Analysis	81
4.2.4	Thermal Analysis	83
4.2.4	Morphology Analysis	85
4.2.5	Functional Group Analysis	86
4.3	Kaolin Geopolymer Ceramics with Addition Various Weight Content of Ultra High Molecular Weight Polyethylene (UHMWPE) as Binder for Lightweight Application	90
4.3.1	Flexural Strength Analysis	90
4.3.2	Density Analysis	92
4.3.3	Volumetric Shrinkage	94
4.3.4	Water Absorption Analysis	96
4.3.5	Phase Analysis	97
4.3.6	Morphology Analysis	99
4.3.7	Functional Group Analysis	101
4.3.8	Correlation between Various Content of UHMWPE on Kaolin Geopolymer Ceramic	105
4.4	The Effect of Sintering Temperature on Kaolin Geopolymer Ceramics with Addition of UHMWPE as Binder	108
4.4.1	Flexural Strength Analysis	108
4.4.2	Density Analysis	110

4.4.3	Volumetric Shrinkage	112
4.4.4	Water Absorption Analysis	114
4.4.5	Phase Analysis	115
4.4.6	Morphology Analysis	117
4.4.7	Functional Group Analysis	119
4.4.8	Correlation between the Effect of Sintering Temperature on Kaolin Geopolymer Ceramic with addition of UHMWPE as Binder	123
4.5	Effect of Sintering Method on Kaolin Geopolymer Ceramic with Addition of UHMWPE	125
4.5.1	Flexural strength Analysis	125
4.5.2	Density Analysis	127
4.5.3	Volumetric Shrinkage	128
4.5.4	Water Absorption Analysis	130
4.5.5	Phase Analysis	131
4.5.6	Morphology Analysis	133
 CHAPTER 5 CONCLUSION AND RECOMMENDATIONS		
5.1	Conclusion	135
5.2	Recommendations	137
REFERENCES		138
APPENDIX A		151
APPENDIX B		153
APPENDIX C		155

LIST OF TABLES

NO.		PAGE
Table 2.1	Typical methods used to fabricate ceramic materials (Rahaman, 2007)	11
Table 2.2	Possible application of the geopolymers based on Si:Al ratio (Davidovits, 1999)	23
Table 2.3	Flexural strength of the geopolymer and the resulted leucite ceramic (Peigang et al., 2013b)	49
Table 2.4	Summary of main FTIR absorption bands in geopolymer (Liew et al., 2016)	60
Table 3.1	Properties of kaolin	67
Table 3.2	Specification of alkaline activator sodium silicate	68
Table 3.3	The typical properties of UHMWPE	69
Table 3.4	Formulation of kaolin geopolymer ceramic with different UHMWPE content	71
Table 4.1	Chemical composition of raw materials raw materials, kaolin	83
Table 4.2	Main IR spectra absorption peaks of kaolin geopolymer and kaolin geopolymer ceramic	92
Table 4.3	Main IR spectra absorption peaks of kaolin geopolymer ceramic with and without addition of various content UHMWPE sintered at 1200 °C	107
Table 4.4	Main IR spectra absorption peaks of kaolin geopolymer ceramic with addition of 4 wt.% of UHMWPE sintered at different temperatures	127

LIST OF FIGURES

NO.		PAGE
Figure 2.1	Fundamental of geopolymer molecular network (Davidovits, 2002)	22
Figure 2.2	Schematic diagram of a typical mechanism of geopolymerization (Yao et al., 2009)	32
Figure 2.3	Summary of geopolymerization process (Provis et al., 2006)	34
Figure 2.4	Flexural strength and work of fracture of unidirectional carbon fiber reinforced geopolymer composites without and with heat treatment (Peigang et al., 2010)	46
Figure 2.5	Apparent density and specific surface area of Metakaolin-based geopolymer after being heated to the specified temperature (Bell et al., 2009)	47
Figure 2.6	Dilatometry of geopolymer heated at 5 °C/min up to 1200 °C (Peigang et al., 2010b)	49
Figure 2.7	XRD diffractograms of kaolin (K=Kaolinite; Q=Quartz, A=Alunite and D=Dickite) (Heah et al., 2011)	50
Figure 2.8	XRD diffractograms for poly(sialate-siloxo) geopolymers with SiO ₂ /Al ₂ O ₃ molar ratios of (a) 4.02; (b) 3.98; (c) 3.39 and (d) 4.11 (Davidovits & Quentin, 1991)	51
Figure 2.9	XRD diffractograms of pure geopolymer (pure GP) after heating Key: 1 = quartz [SiO ₂], 2 = sodium aluminium silicate [NaAlSi ₂ O ₆], 3 = nepheline [NaAlSiO ₄], 4 = carnegieite [NaAlSiO ₄] (Kuenzel et al., 2013)	52
Figure 2.10	XRD diffractograms for K ₂ O·Al ₂ O ₃ ·4SiO ₂ ·11H ₂ O after being heated to 1000 °C, 1050 °C, 1100 °C, 1150 °C, and 1200 °C (Xie et al., 2010)	53
Figure 2.11	XRD diffractogram for geopolymer samples treated at different temperatures in air with isothermal soak for 120 min (Peigang et al., 2013b)	54
Figure 2.12	TG/DSC curves of the CS-based geopolymer (Peigang & Dechang, 2013a)	55

Figure 2.13	TG/DTA of potassium-based geopolymer at Si/Al ratio of 2.5. Sample was heated at 5 °C/min up to 1200 °C (Peigang, Dechang, Meirong, et al., 2010b)	56
Figure 2.14	SEM micrograph of metakaolin used in producing of leucite ceramics derived from potassium-based geopolymer precursor (Peigang et al., 2013b)	57
Figure 2.15	SEM morphology of geopolymer after being heated to (a) 850 °C, (b) 900 °C and (c) 1200 °C (Kuenzel et al., 2013)	59
Figure 2.16	FTIR spectra of unheated and heated geopolymer for 1 h at the indicated temperature (Barbosa & MacKenzie, 2003)	62
Figure 3.1	Research planning of the kaolin geopolymer ceramic with addition of UHMWPE	66
Figure 3.2	Sintering method used in fabrication of kaolin geopolymer ceramic with addition of UHMWPE (a) conventional method and (b) two-step method	73
Figure 4.1	Particle size distribution of kaolin	82
Figure 4.2	X-Ray diffraction patterns of raw kaolin and kaolin geopolymer (K = kaolinite; Q = quartz; I = illite; D = dickite and Z = zeolite)	84
Figure 4.3	Weight loss of kaolin geopolymer ceramic with addition of (1) 2 wt.% of UHMWPE, (2) 4 wt.% of UHMWPE, (3) 6 wt.% of UHMWPE (4) 8 wt.% of UHMWPE measured by TGA from room temperature to 1000 °C	86
Figure 4.4	SEM micrograph of (a) raw material kaolin and (b) kaolin geopolymer	88
Figure 4.5	IR spectra of (1) kaolin geopolymer and (2) raw kaolin	91
Figure 4.6	Flexural strength of kaolin geopolymer ceramics with various content of UHMWPE sintered at 1200 °C	94
Figure 4.7	Density of kaolin geopolymer ceramics with various content of UHMWPE sintered at 1200 °C	96
Figure 4.8	Volumetric shrinkage of kaolin geopolymer ceramic at various content of UHMWPE sintered at 1200 °C	98
Figure 4.9	Water absorption of kaolin geopolymer ceramic at various content of UHMWPE	100

Figure 4.10	Phase analysis of kaolin geopolymer ceramics with and without addition of various content UHMWPE sintered at 1200 °C (N= Nephaline, C= Carbon)	101
Figure 4.11	SEM micrograph of kaolin geopolymer ceramic at various content of UHMWPE (a) 0 wt.%, (b) 2 wt.%, (c) 4 wt.%, (d) 6 wt.% and (e) 8 wt.%	103
Figure 4.12	IR spectra of kaolin geopolymer ceramic with and without addition of various content UHMWPE sintered at 1200 °C (1)= 0 wt.% of UHMWPE, (2) = 2 wt.% of UHMWPE, (3) = 4 wt.% of UHMWPE, (4) = 6 wt.% of UHMWPE and (5) = 8 wt.% of UHMWPE	106
Figure 4.13	Proposed model of the reaction between UHMWPE and kaolin geopolymer	109
Figure 4.14	The relationship between flexural strength and density	110
Figure 4.15	The relationship between water absorption and density	111
Figure 4.16	The relationship between flexural strength and water absorption	111
Figure 4.17	Visual appearance of kaolin geopolymer ceramic with addition of 4 wt.% of UHMWPE at a) 900 °C, b) 1000 °C, c) 1100 °C, d) 1200 °C, e) 1250 °C	113
Figure 4.18	Flexural Strength of kaolin geopolymer ceramics with and without addition of UHMWPE at different sintering temperatures	114
Figure 4.19	Density of kaolin geopolymer ceramics with and without addition of UHMWPE at different sintering temperatures	116
Figure 4.20	Volumetric shrinkage of kaolin geopolymer ceramic with and without addition of UHMWPE at different sintering temperatures	118
Figure 4.21	Water absorption kaolin geopolymer ceramic with and without addition of UHMWPE at different sintering temperatures	120
Figure 4.22	XRD pattern of kaolin geopolymer and kaolin geopolymer ceramic with addition of 4 wt.% of UHMWPE at different sintering temperatures (N= Nephaline, C= Carbon)	122

Figure 4.23	SEM micrograph of kaolin geopolymer ceramic with addition of 4 wt.% of UHMWPE at different sintering temperature (a) 900 °C, (b) 1000 °C, (c) 1100 °C, and (e) 1200 °C	123
Figure 4.24	IR spectra of (1) unsintered kaolin geopolymer and kaolin geopolymer ceramic with addition of UHMWPE sintered at (2) 900 °C, (3) 1000 °C, (3) 1100 °C, and (5) 1200 °C	126
Figure 4.25	The relationship between flexural strength and density	128
Figure 4.26	The relationship between water absorption and flexural strength	129
Figure 4.27	The relationship between water absorption and density	129
Figure 4.28	Flexural strength of kaolin geopolymer ceramic with and without addition of UHMWPE at different sintering method	131
Figure 4.29	Density of kaolin geopolymer ceramics with and without addition of UHMWPE at different sintering method	133
Figure 4.30	Volumetric shrinkage of kaolin geopolymer ceramics with and without addition of UHMWPE at different sintering method	134
Figure 4.31	Water absorption of kaolin geopolymer ceramic with and without addition of UHMWPE at different sintering method	136
Figure 4.32	XRD pattern for kaolin geopolymer sintered at different sintering method (N= Nepheline, Z= Zeolite)	137
Figure 4.33	XRD pattern for kaolin geopolymer ceramic with addition of 4 wt.% of UHMWPE sintered at different sintering method (N= Nepheline, C= Carbon)	138
Figure 4.34	SEM micrograph of kaolin geopolymer ceramics without addition of UHMWPE sintered using a) conventional method, b) TSS method and with addition of 4 wt.% UHMWPE c) conventional method, d) TSS method	140

LIST OF ABBREVIATIONS

Al	Alumina
Al ₂ O ₃	Aluminum Oxide
ASTM	American Society for Testing and Materials
Ca	Calcium
CaO	Calcium Oxide
CO ₂	Carbon Oxide
K	Potassium
KOH	Potassium Hydroxide
SEM	Scanning Electron Microscopy
TGA	Thermogravimetric Analysis
UHMWPE	Ultra High Molecular Weight Polyethylene
XRD	X-ray Diffraction
XRF	X-ray Fluorescence Spectroscopy
LOI	Loss on Ignition
Wt. %	Weight per cent

Pembentukan Seramik Geopolimer Kaolin dengan Penambahan Polietilena Bermolekul Sangat Tinggi (UHMWPE) sebagai Pengikat di dalam Aplikasi Seramik

ABSTRAK

Bahan seramik ringan yang mempunyai ciri-ciri fizikal, mekanikal dan struktur adalah menjadi bahan terpilih di dalam industri seramik masa kini. Geopolimer teknologi telah digunakan dalam pelbagai aplikasi termasuk penghasilan seramik. Perubahan fasa yang berlaku semasa proses pemanasan geopolimer dari fasa amorfus kepada fasa kristal memerlukan suhu yang lebih rendah berbanding penghasilan seramik konvensional. Bahan utama di dalam perindustrian seramik ringan adalah menggunakan penambah bahan organik semulajadi seperti pengikat, pemplastik, agen aktif permukaan dan pelincir. Kajian ini bertujuan untuk mengkaji penggunaan geopolimer dalam penghasilan bahan seramik di mana kaolin digunakan sebagai sumber bahan utama dan polietilena berjisim molekul yang sangat tinggi ditambah sebagai pengikat. Nisbah pepejal kepada cecair pada 1.0 dan nisbah pengaktif alkali pada 0.24 telah ditetapkan. Selepas kaolin geopolimer dirawat pada suhu 80 °C selama 24 jam, sampel kemudian dihancurkan ke dalam bentuk serbuk. Dengan menggunakan kaedah metalurgi dalam penghasilan kaolin geopolimer seramik, tiga parameter digunakan iaitu suhu pensinteran (900 °C, 1000 °C, 1100 °C, 1200 °C), kandungan pengikat (2 % berat, 4 % berat, 6 % berat, 8 % berat) dan kaedah sinter yang terdiri dari kaedah konvensional dan kaedah sinter dua langkah. Peratus berat optimum bahan pengikat telah dikaji dengan menetapkan suhu pensinteran dan suhu pensinteran optimum dikaji dengan menetapkan peratus berat bahan pengikat berdasarkan kekuatan kelenturan, ketumpatan, penyusutan dan penyerapan air. Seramik geopolimer kaolin dengan penambahan 4 % berat, polietilena berjisim molekul yang sangat tinggi disinter pada 1200 °C menggunakan kaedah sinter dua langkah menunjukkan kekuatan optimum pada 94.32 MPa dan berketumpatan pada 1.71 g/cm³. Permukaan yang rata dan peningkatan pembentukan liang udara juga telah diperhatikan di dalam analisis mikrostruktur. Manakala, analisis fasa menunjukkan puncak fasa nepheline berintensiti tinggi pada sampel dengan penambahan 4 % berat bahan pengikat menyumbang kepada ketumpatan rendah dan kekuatan lenturan tinggi. Kemudian, keupayaan seramik ringan geopolimer kaolin diuji menggunakan ujian sifat mikrostruktur dan sifat mekanikal. Hasil kajian mengesahkan kemungkinan untuk menghasilkan seramik ringan berasaskan geopolimer kaolin yang berpencirian dan sifat-sifat fizikal yang tinggi disamping membuka lebih banyak peluang aplikasi di masa hadapan. Bahan seramik ringan berasaskan geopolimer juga dibuktikan mempunyai potensi disebabkan keupayaan untuk menghasilkan sifat-sifat berprestasi tinggi yang memberi faedah kepada ekonomi dan alam sekitar. Tambahan lagi, penghasilan bahan seramik ringan yang berkualiti tinggi memerlukan rawatan mekanikal dan haba yang membawa kepada kesan positif kepada alam sekitar justeru lebih sesuai dengan kehendak industri mesra alam.

Development of Kaolin Geopolymer Ceramic with Addition of Ultra High Molecular Weight Polyethylene (UHMWPE) as Binder for Lightweight Ceramics

ABSTRACT

A lightweight ceramic material displays physical, mechanical and structural features which is highly preferred in modern ceramic industry. Geopolymer technology has been involved in many applications including in the formation of ceramic. The transformation phase of geopolymer from amorphous to crystalline upon heating require a low processing temperature compared to the conventional ceramics. A major synthetic process for industrialised lightweight ceramics is to use additives which are organic in nature such as binders, plasticizers, surfactants and lubricants. This study investigated the use of geopolymer in producing ceramic materials where kaolin was used as main source material and Ultra High Molecular Weight Polyethylene was added as binder. In this study, the solid-to-liquid ratio of 1.0 and alkaline activator ratio of 0.24 were fixed. Kaolin geopolymer were then cured at 80 °C for 24 hours, the samples were then crushed into powder form. By using powder metallurgy method in producing kaolin geopolymer ceramic, three parameters used are sintering temperature (900 °C, 1000 °C, 1100 °C, 1200 °C), binder content (2 wt.%, 4 wt.%, 6 wt.%, 8 wt.%) and sintering method which are conventional and two-steps sintering method. The optimum weight percent of binder were studied by fixing the sintering temperature, while the optimum of sintering temperature were studied by fixing the weight percent of binder based on testing of flexural strength, density, shrinkage, and water absorption. The results indicated that kaolin geopolymer ceramic with the addition of 4 wt.% of Ultra High Molecular Weight Polyethylene sintered at 1200 °C using two steps sintering method could achieve an optimum strength of 94.32 MPa with a density of 1.71 g/cm³. Also a smooth surface and increasing in formation of pores were observed, which would facilitate the formation of the lightweight and strong structure. Then, the performance of kaolin geopolymer lightweight ceramic was examined by performing microstructural and mechanical properties tests. The outcomes revealed the possibility to produce a lightweight ceramic based kaolin-geopolymer with a considerable characteristics and mechanical properties, which could open the door for many applications in the future. Geopolymer based lightweight ceramic has been claimed as a promising material, due to its ability to produce a high-performance lightweight ceramic and because of its relevant environmental and economic benefits. Furthermore, lower-powered mechanical and thermal treatments are required to ensure the excellent properties and quality to produce the lightweight ceramic materials lead to a positive effect on the environment hence suitable with the desire for eco-friendly industry.

CHAPTER 1: INTRODUCTION

1.1 Research Background

The broad class of materials known as ceramics are inorganic nonmetallic materials consists of metallic and nonmetallic elements bonded together with ionic and/or covalent bonds. Ceramics can be polycrystalline or at least partly polycrystalline structure which are formed by a sintering process (Bell et al., 2009). Besides, ceramics also can be classified as traditional and advanced ceramics. The applications for traditional ceramics and glasses are including structural building materials refractories for furnace linings, tableware and sanitaryware, electrical insulation and transportation vehicles (Mukherjee, 2013). While the diverse application for advanced ceramics have been developed as potential to continue to grow at a reasonable rate where the processing tolerance and economical compare to traditional ceramics (Raman et al., 2012).

The special character of ceramic materials gives rise to a huge application in this modern industry. Fundamentally, ceramics usually have high melting points, therefore there are generally labeled as refractory. Different type of ceramics come with different properties, in general they are also high in modulus, high compression strength, high hardness, low thermal conductivity and chemically inert (Popoola et al., 2014). The ionic and covalent bonds from the crystal structure of ceramic material influence the stability of bonding. Although, the strong bonds increase the fragility of the ceramics, in some way its limits the applications (Iyasara et al., 2014). In addition, an increasing demand for the stiffer, stronger and lightweight ceramic materials recently creates a large interest

to the industry. Thus, the selection materials used, proper fabrication methods and sintering can all affect the texture of the materials to meet the required properties and performance.

Primarily, the suitability properties of ceramic materials for lightweight applications have been determined by specific strength, specific stiffness and density (Bauer et al., 2014). Since the properties of ceramics are highly depend on powder packing and additives, a proper selection of additive is important in striving towards lightweight ceramic materials in order to enhance the mechanical properties during and after manufacturing process (Baklouti et al., 2001; Rajeswari et al., 2015; Taktak et al., 2011). Commonly, types of additives used in ceramic processing consist of binder, plasticizers, surfactants, dispersants, and lubricants. Polymer binder are usually used in ceramics processing which assist the primary function of providing strength to the green ceramics body. Therefore, Ultra High Molecular Weight Polyethylene (UHMWPE) has potential to be used as polymer binder to ceramic, owing to the high mechanical properties such as strength and fire resistance.

Several methods are available for fabricating ceramic materials, and the fabrication method is very important since it can affect the properties of the product. Ceramic fabrication occurs through some sequential steps, which start with raw material, proceed through batch preparation and forming and concludes with firing. Fabrication of conventional ceramic materials require an extremely high temperature treatment up to 1600 °C (Kriven et al., 2013). The use of geopolymer method is an alternative way in producing ceramic materials since the amorphous to semi-crystalline behavior of geopolymer will transforms into crystalline ceramic phases upon heating (Barbosa &

MacKenzie, 2003; Duxson et al., 2006; Peigang et al., 2011). With the help of geopolymerization reactions, high temperature techniques or processes are no longer required to achieve materials of ceramic-like structure and properties (Iwahiro et al., 2001). Moreover, geopolymer also can be directly converted into final structural ceramic part of interest and practically design the chemical compositions of the final product (Peigang et al., 2010a).

Inorganic aluminosilicates produced at low temperatures, also known as geopolymers, have potential applications as matrix in ceramics, coatings, cements and other composite materials (Davidovits, 1991; Davidovits & Davidovics, 1991; Peigang et al., 2010b). Geopolymers are normally made by mixing materials that contain aluminosilicate, such as kaolin, metakaolin and fly ash, into alkaline solution and curing the mixture at a certain temperature (Mustafa Al Bakri et al., 2011b). Highly alkaline solutes, such as sodium hydroxide (NaOH) and potassium hydroxide (KOH), are incorporated into source materials rich in SiO_2 and Al_2O_3 , yielding Si-O-Al-O bonds. Theoretically, any alkali and alkali earth cations can be used as the alkali component in this exothermic process such as geopolymerization. Geopolymerization is a complex multiphase reaction, comprising a series dissolution-reorientation-solidification (Davidovits & Quentin, 1991; Duxson et al., 2005).

The strength of geopolymers influenced by the nature of the source materials. Geopolymers, synthesized using calcined source materials, e.g., fly ash, slag, and metakaolin, have higher compressive strength than those synthesized using non-calcined materials, such as kaolinite, and naturally-occurring minerals (Palomo et al., 1999; Barbosa and MacKenzie, 2003; Xu and Van Deventer, 2000). Many studies on

geopolymer ceramics have concentrated on the use of metakaolin (Bell et al., 2009; Peigang et al., 2011; Peigang et al., 2013b) because of its higher reactivity, which lead to optimal performance, slightly than the less reactive of kaolin (Heah et al., 2011). Despite, the uses of kaolin as a major raw material used in the fabrication of ceramic materials have the advantage to be easily accessible and not expensive.

In this study, geopolymer ceramics were fabricated by using powder metallurgy method. Kaolin was used as source materials and Ultra High Molecular Weight Polyethylene was added as a binder. The kaolin geopolymer were grinded to a powder, followed by mixing with the binder, compressing, and sintering. The mechanical properties, microstructural and phase analysis were investigated.

1.2 Problem Statement

Existing lightweight ceramic materials fabrication routes are often difficult that commonly having complicated chemistry and time consuming. The lengthy heat treatment required can exceed up to 10 hours and mostly having a multi-stage processing steps (Rahaman, 2007). It is worth pointing out that the conventional lightweight ceramics fabrication is a complicated process especially when the manufactured materials need to feature the required properties. In order to meet the growing demand for lightweight and high performance ceramics, the feasibility of using geopolymer technology can be one of the alternative methods in producing ceramic. Common source material in producing geopolymer ceramic is metakaolin which proved to have issues in consuming more energy for calcination thus to produce cost efficient final product making investigation of kaolin geopolymer ceramic very much required.

Most of the published research on lightweight ceramics require high sintering temperature which up to 1800 °C to achieve low density properties (Fu et al., 2016; Zang et al., 2016a). In order to overcome this problem, geopolymer technology can be used in producing high performance lightweight ceramics at a slightly lower temperature with the intention of reducing the energy used towards environmental issues. Despite, to broaden the range of applications of the ceramic materials, these ceramic should not have only high porosity and large interconnections but also dense networks and high in strength. Therefore, the addition of binder in production of geopolymer ceramics can be one of the solutions to achieve all those excellent properties. The purpose of the binder is to plastically deform between ceramic particles thus transmit sufficient strength to the green compact (Yuan and Li, 2012).

Most of the studies on fabrication ceramics from geopolymers performed on directly heat the geopolymer system. Directly heat the geopolymer at elevated temperature will cause cracks and dehydration. Powder metallurgy is one of the methods that have potential for making an almost infinite variety of materials and ceramic products. By compressing and sintering, the powder metallurgy method can provide the desires final shape and dimension of parts (Vogiatzis et al., 2015). In addition, this method is preferred because it produces a uniform distribution of the particles and reduces degradation due to the low processing (Akhlaghi and Pelaseyyed, 2004; Peigang and Dechang, 2013a). Besides, the method selection and material used play important roles towards reducing energy consumption in fabrication of ceramics.

1.3 Research Objectives

The aim of this research is to study the development of kaolin geopolymer ceramics with addition of Ultra High Molecular Weight (UHMWPE) as binder for lightweight application. The details objectives of the study are as follows:

1. To study the addition of different percentage of UHMWPE as binder on properties of the kaolin geopolymer ceramics for lightweight application based on flexural strength.
2. To obtain the optimum sintering temperature on producing kaolin geopolymer ceramics with addition of UHMWPE as binder based on flexural strength.
3. To determine the method of sintering of kaolin geopolymer ceramics with addition of UHMWPE as binder based on flexural strength.

1.4 Scope of Study

The scope of this study is to develop kaolin geopolymer ceramics with addition of UHMWPE as binder that can be used in lightweight application. By using kaolin as source materials in geopolymer production, the performance of this material is covered on characterization study which is morphology analysis, chemical composition analysis, phase analysis and functional group identification analysis. The alkaline solutions used in this research are sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) with ratio of 0.24. The molarity of sodium hydroxide is 12 M.

In order to develop kaolin geopolymer ceramics with addition of UHMWPE as binder, various content of UHMWPE (2 wt.%, 4 wt.%, 6 wt.%, 8 wt.%), sintering temperature (900 °C, 1000 °C, 1100 °C, 1200 °C) and different method of sintering

(conventional and two step sintering method) is carried out. Mechanical study in this research is covered by flexural strength, water absorption, density and shrinkage. The determination of the optimum kaolin geopolymer ceramics with addition of UHMWPE is based on the samples that contribute to high strength with low density and water absorption.

1.5 Thesis Outline

This thesis is divided into five chapters. Chapter 1 describes the background study of this research, problem statement, research objectives, scope of study and also the outline of the thesis.

Chapter 2 discuss on the literature review based on the basic of geopolymer, geopolymer constituent and the process, geopolymer ceramics as another application by using geopolymer technology. There are also reviews on the basic concept, structural, physical, mechanical and morphological properties related to the geopolymer ceramics.

Chapter 3 demonstrates the experiment method that investigates the potential of producing kaolin geopolymer ceramic with addition of UHMWPE as binder. This chapter elaborates more on research materials (kaolin, alkaline activator, and binder used), materials proportion, mixing process, and method used in geopolymer ceramics fabrication. Additionally, this chapter also explained detail on the parameter used, characterization and mechanical testing for geopolymer ceramic as follows the ASTM requirement.

Chapter 4 discussed the experimental results and discussion on the characterization of raw material, the effect of binder content, the effect of sintering temperature and the effect of sintering method used in producing of kaolin geopolymer ceramics. Furthermore, this chapter also explained details on the optimum for geopolymer ceramics with addition of UHMWPE as binder.

Chapter 5 summarizes and concludes the finding from this research and some recommendations for the future work also has been suggested. The end of this thesis is complete by a bibliography and appendices.

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CHAPTER 2 : LITERATURE REVIEW

2.1 Introduction

The polymers based on related aluminosilicate units are a novel family of inorganic polymeric materials, has expanded international interests in the past few years. Their excellent physical properties make them practicable alternatives for many conventional cements and plastics. Geopolymers are also being reflected for a range of applications comprising low CO₂ producing cements (Duxson et al., 2007), refractories (Davidovits, 1991a), fiber-reinforced composites (Davidovits, 2002), and as precursors to ceramic formation. Additionally, their synthesis at low temperature is energy-efficient and more environmentally-friendly than older materials (Barbosa et al., 2000). The formation of geopolymer is a polymerization process similar to the polycondensation of an organic polymer, (Davidovits, 1991a). Therefore, the overall process is termed as geopolymerization. The geopolymerization can transfer Al and Si containing wastes into geopolymers with a high mechanical strength and high durable construction materials. The use of kaolin as source materials in fabrication of geopolymer ceramics can achieve good economic and environmental benefits.

A brief review of some of the relevant literature on fabrication and properties of kaolin based geopolymer ceramics is presented in this chapter covering basic knowledge on ceramics, mechanism of geopolymerization, geopolymer ceramic synthesis with kaolin and properties of geopolymers at elevated temperature. It is observed that there are