

**DEVELOPMENT OF THE PORCELAIN TILES BY THE PARTIAL
SUBSTITUTION OF FELDSPAR WITH FLY ASH**

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Special dedicated to my beloved family



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ABSTRACT

Fly ash properties are unusual among engineering materials. Typically used for embankment construction, fly ash is well known in the production of cement, concrete and brick. Fly ash is one of the palm oil waste products from the combustion of palm fiber and shells in the boiler which are collected at the multi-cyclone collectors. In this research, feldspar is gradually replaced by fly ash compositions of 0 – 25 wt. %. The samples were mixed for 12 hours, pressed with a load of 3 tonnes and isostatically pressed by using CIP with a pressure of 100 MPa. The samples were sintered at the temperature range of 1100 – 1300 °C. An investigation on the physical and mechanical properties as well as the crystalline phase and microstructure of porcelain were evaluated in this study. Through XRF analysis, the presence of fluxing component in fly ash significantly proved its potential as fluxing agent in porcelain. The highest strength of the porcelain obtained at the replacement of 5 wt. % of fly ash sintered at 1250 °C concurrently shows it as the best composition of fly ash and the best sintering temperature. Maximum compressive strength of 105.40 MPa and higher Vickers microhardness value obtained through this composition. In fact, the percentage of the total mass loss was increased up to 9.30 %. The bulk density value of the porcelain is reduced to 2.34 g/cm³, which almost 2.5 % loss of density. Lower apparent porosity achieved at this compositions with 0.22 %, indicating almost complete densification takes place. The percentage of mullite content was found to be increased with existence of fly ash in porcelain compositions. SEM studied revealed that intense interlocking and uniform distribution between the primary and secondary mullite in glassy matrix are contributed in achieving higher densification of porcelain. It is concluded that the utilization of fly ash as fluxing component is highly suitable in porcelain and the replacement of 5 wt. % of fly ash successfully improvised the properties and the microstructure of the porcelain with attaining higher mechanical strength.

ABSTRAK

Ciri-ciri abu terbang adalah luar biasa di kalangan bahan-bahan kejuruteraan. Biasanya digunakan untuk pembinaan tambak, abu terbang terkenal dalam pengeluaran simen, konkrit dan batu bata. Abu terbang merupakan salah satu daripada sisa kelapa sawit yang terhasil dari pembakaran serat dan kulit biji sawit di dalam dandang yang dikumpulkan di pengumpul pelbagai siklon. Dalam kajian ini, feldspar digantikan secara berkala pada komposisi 0 – 25 %. Sampel dicampur selama 12 jam, ditekan dengan bebanan sebanyak 3 tan dan ditekan secara isostatik menggunakan CIP di bawah tekanan 100 MPa. Sampel disinter pada jarak suhu 1100 – 1300 °C. Penyiasatan terhadap sifat fizikal dan mekanikal, serta fasa-fasa hablur dan struktur mikro porselin dinilai dalam kajian ini. Melalui analisis XRF, kehadiran komponen fluk dalam abu terbang dengan ketara membuktikan potensinya sebagai agen fluk dalam porselin. Kekuatan maksimum porselin diperoleh pada penggantian 5 % berat abu terbang disinter pada 1250 °C sekaligus menunjukkan ia sebagai komposisi abu terbang dan suhu persinteran yang terbaik. Kekuatan mampatan maksimum iaitu 105.40 MPa dan nilai kekerasan mikro Vickers yang tinggi diperoleh melalui komposisi ini. Malah, peratusan jumlah kehilangan jisim meningkat sehingga 9.30 %. Ketumpatan pukal porselin berkurang sehingga 2.34 g/cm³, dimana hampir 2.5% kehilangan ketumpatan pukal berlaku pada komposisi ini. Keliangan ketara yang lebih rendah diperoleh sebanyak 0.22 %, menunjukkan berlakunya kepadatan yang hampir lengkap. Peratusan kandungan mullite didapati meningkat dengan kehadiran abu terbang dalam komposisi porselin. Kajian SEM membuktikan bahawa pengedaran sengit dan taburan seragam di antara mullit primer dan sekunder dalam matrik berkaca menyumbang dalam mencapai kepadatan porselin yang lebih tinggi. Kesimpulannya, penggunaan abu terbang sebagai komponen fluk sangat sesuai di dalam penghasilan porselin dan penggantian sebanyak 5 % berat abu terbang berjaya menambah baik sifat-sifat dan mikrostruktur porselin dengan pencapaian kekuatan mekanikal yang lebih tinggi.

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LIST OF SYMBOLS AND ABBREVIATIONS

A	-	Area
d	-	Distance
F	-	Force
n	-	Integer
α	-	Alpha
β	-	Beta
θ	-	Angle
λ	-	Wavelength
π	-	Pi
<	-	Less than
d^2	-	Area of the Indentation (mm ²)
d_i	-	Initial Diameter
d_f	-	Final Diameter
h_i	-	Initial Height
h_f	-	Final Height
m_i	-	Initial Mass
m_f	-	Final Mass
W_1	-	Dry Weight
W_2	-	Suspended Weight
W_3	-	Soaked Weight
ρ_w	-	Density of Water (0.99777 g/cm ³)
ASTM	-	American Society for Testing and Materials
AE	-	Auger Electrons
BSE	-	Backscattered Electrons
BPR	-	Ball to Powder Ratio
BS	-	British Standard

CCD	-	Charge-coupled Device
CL	-	Cathodoluminescence
CIP	-	Cold Isostatic Pressing
EDS	-	Energy Dispersive Spectroscopy
FA	-	Fly Ash
FAHM-		Fly-Ash Hollow Microsphere
FP	-	Fly ash Porcelain
HF	-	Hydrofluoric acid
IHM	-	Inorganic Hollow Microsphere
ISO	-	International Standards Organization
ICDD	-	International Centre for Diffraction Data
LFC	-	Lightweight Fly ash Ceramics
LSC	-	Lightweight Sludge Ceramics
NP	-	Normal Porcelain
OHM	-	Organic Hollow Microsphere
OPR	-	Ordinary Portland Cement
POFA	-	Palm Oil Fuel Ash
PVA	-	Polyvinyl Alcohol
PSC	-	Pressure Slip Casting
RHA	-	Rice Husk Ash
SE	-	Secondary Electrons
SEM	-	Scanning Electron Microscopy
XRF	-	X-ray Fluorescence
XRD	-	X-ray Diffraction

LIST OF PUBLICATIONS

Paper

Kutty, N. A. A., & Noh, M. Z (2016). Influence of Phlogopite on the Physico-Mechanical Properties of Porcelain. *Journal of Science and Technology*, 8(1), 9–12.

Kutty, N. A. A., Noh, M. Z., Mayzan, M. Z. H., & Iya, S. G. D. (2018). Influence on the Phase Formation and Strength of Porcelain by Partial Substitution of Fly Ash Compositions. *International Journal of Engineering & Technology*, 7(4.30), 271–275.

Iya, S. G. D, Noh, M. Z., Ab Razak, S. N., Sharip, N., Jasim, M. J., & Kutty, N. A. A. (2020). Developed Formulation Model by the Addition of SiO₂ for Optimum Physicomechanical Properties of Porcelain using Modified Palm Oil Fuel Ash (POFA). *International Journal of Nanoelectronics and Materials*, 13(2), 401 – 410.

Conference

Kutty, N. A. A., Noh, M. Z., Mayzan, M. Z. H., & Iya, S. G. D. (2018). Influence of Different Compositions of Fly Ash as Fluxing Agent in Porcelain. *Advanced Materials Characterization Technique, 16th – 17th August 2017, Genting Highlands, Malaysia* (Poster presentation)

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Ceramic is an inorganic and non-metallic material, and its processing generally involves high temperature. Ceramics materials are grouped according to its mineralogical such as silicate, oxide, non-oxide, nitride and carbide. Besides, ceramic can also be categorized into traditional and advanced ceramics. Traditional ceramics are based on clay and silica, and examples of traditional ceramics are pottery, whiteware, earthenware, stoneware, chinaware and porcelain. In fact, traditional ceramic has been used over 25,000 years, while advanced ceramics were developed within last 100 years (Carter & Norton, 2007). One example of traditional ceramic was invented by the Chinese during T'ang dynasty which namely as white ceramic (Snehesh, 2014).

Porcelain or tri-axial porcelain consists of three different raw materials which are clay or kaolin, feldspar and quartz. Standard compositions of porcelain are approximately 50 % of kaolin, 25 % of quartz and 25 % of feldspar (Koca et al., 2012). Kaolin is the main based raw materials of porcelain where it provides plasticity for forming process. Meanwhile, feldspar helps in enhancing vitrification, and quartz functions as a filler which is able to reduce the shrinkage during firing (Kitouni & Harabi, 2011). Reactivity among these three raw materials provide porcelain with extremely good properties such as less porous, low permeability and elasticity, high resistance to chemical attack and thermal shock, translucence and high hardness rating (Snehesh, 2014).

Regarding its application, porcelain tiles is one of the new products compared to other ceramic tiles, and it shows an increasing rate in its market trends recently. Porcelain tile is a high quality product, and it stands out from other products due to its excellent technical properties. It is a vitrified ceramic product with extremely low porosity ($< 0.5\%$), and a very low percentage of closed porosity which is $< 10\%$ (De Noni et al., 2011; Sánchez et al., 2010). It also has low water absorption, highly creative design, and high abrasion resistance which results in higher mechanical strength (García-Ten et al., 2012). Although porcelain composition can influence the characteristic of the end-product, the sintering temperature is also one of the most important factors in the fabrication of porcelain tiles. The changes in microstructure and vitrification behaviour are dependent on the sintering temperature because it brings out the significant changes in the densification, and it improves the strength of the porcelain tiles (Acchar & Eduardo, 2015).

Over the last few years, modification on the raw materials of porcelain has been executed by utilization of waste materials where it improves the physical and mechanical properties of porcelain. Most of the modifications are focused on the replacement of quartz with waste materials such as fly ash, rice husk ash (RHA), glass waste and slag. Among all waste materials, fly ash had been widely reported on its replacement with quartz in improving the mechanical properties of porcelain (Acchar & Dultra, 2015; Das et al., 2013). However, there are limited studies on the production of lightweight porcelain by the substitution of fly ash. Fly ash is a residue generated from the process of combustion where it is in form of very fine powder and highly content of silica (SiO_2) and alumina (Al_2O_3). In recent years, fly ash is widely used in ceramics due to its potential as a filler and fluxing agent; hence, simultaneously improves its physical and mechanical properties.

1.2 Problem statement

Porcelain tile is a high quality product with excellent technical properties, and it is being commercialized worldwide. Research on the improvise the physical and mechanical properties of porcelain tiles is highly significant, and extremely could expand the uses of this tile for the next few years, since porcelain tiles are being commercialized worldwide which in turn will increase its demand in construction industry.

Over the past 7 years, investigation on the replacement of the raw materials of porcelain with waste materials are mostly including rice husk ash, glass waste, soda lime scrap and palm oil fuel ash. However, the utilization of fly ash in the fabrication of porcelain tiles still remains unclear. The replacement of fly ash on feldspar has not been widely reported compared to quartz. Fly ash is known for its high alumina (Al_2O_3) and silica (SiO_2) content, both of which have potential as a fluxing agent and it is believed could improve the mechanical strength of the porcelain (Das et al., 2013). Fly ash properties are unusual among engineering materials. It has a large uniformity coefficient, and it consists of clay-sized particles (Kim et al., 2014).

Production of porcelain consumes of large amount of kaolin, feldspar and quartz. In the present study, feldspar will be replaced with fly ash in the tri-axial porcelain according to a few compositions that have been highlighted. Utilization of fly ash is expected could improve the properties of the porcelain tiles depending to the compositions of fly ash and other parameters that has been considered in this work. Feldspar is the second most important raw material which acts as a fluxing agent that provides high vitrification of clay bodies by reducing the melting point (Acchar & Eduardo, 2015). The most common fluxes used in clay bodies are potassium oxide and sodium oxide. Basically, two properties which make feldspars useful for industries are their alkali and alumina content (Ogundare et al., 2016).

Therefore, the substitution of fly ash will minimize the cost of the raw materials. Malaysia produces almost 3 million tonnes of fly ash per annum, making fly ash as one of the highest generated waste materials and the disposal of these waste materials contribute to global warming and emission of greenhouses gases (Yap et al., 2013). In fact, the availability of substitution of feldspar with fly ash would encourage the competitiveness of using waste product in ceramic industries. In addition, the environmental pollution issues could also be mitigated through this work.

1.3 Objectives of the study

The study aims to develop the porcelain tiles with improve its properties by substituting feldspar with fly ash in the porcelain. In order to achieve the mentioned aim, a few objectives can be divided as follow:

- i. To determine the best composition of fly ash in order to improve the physical and mechanical properties of the porcelain.
- ii. To determine the suitable sintering temperature of porcelain towards excellent physical and mechanical properties of porcelain.
- iii. To investigate the mineralogy and the microstructure of porcelain due to the substitution of fly ash.

1.4 Scope of the study

The scopes have been designed to make sure that the objectives of the study were implemented successfully. The scopes of the study are as follows:

- i. Standard porcelain compositions (Koca et al., 2012): 50 wt. % of Kaolin, 25 wt. % of quartz and 25 wt. % of feldspar (feldspar are progressively replaced by fly ash from 0 wt. % up to 25 wt. %).
- ii. The sintering temperature of porcelain involved are 1100 °C, 1150 °C, 1200 °C, 1250 °C and 1300 °C.
- iii. Heating and cooling rate in the sintering process are 2 °C/min – 5 °C/min with 2 hours soaking time.
- iv. Pressure exerted in cold isostatic pressing (CIP) is 100 MPa.
- v. Physical properties testing involved in this research are mass loss, volume shrinkage, apparent porosity and bulk density.
- vi. Mechanical properties testing involved in this research are Vickers microhardness and compressive strength.
- vii. Scanning Electron Microscope is used to evaluate the microstructure of the porcelain.
- viii. X-ray diffraction (XRD) analytical technique is used to investigate the mineralogical compositions of the porcelain.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Porcelain tiles is known as highly vitrified ceramic product which has lower water absorption rates (Acchar & Eduardo, 2015). There is two types of tiles commonly being used in tiles industry which are porcelain and ceramic tiles. Both of the tiles can look very similar but there is a distinct difference between them. The most significant identifying factor is the compositions of the raw materials, where porcelain tiles is made of 50 wt. % of kaolin, 25 wt. % of feldspar and 25 wt. % of quartz (Koca et al, 2012). Meanwhile, ceramic tiles is commonly made of by natural clay mixed with various formula with water and sometimes added with organic materials (Mishulovich & Evanko, 2003). However, porcelain tiles is costly due to its remarkable properties, including higher durability, higher thermal shock (Snehesh, 2014).

2.2 Ceramic

Ceramic is a non-metallic materials which is made up from metals or metalloid and non-metals. The word ceramic is originated from the Greek term "*Keramikos*", which means potteries or materials which have been burned out. There are various compositions of ceramics such as oxide, nitrides, carbides, sulphides, fluorides, graphite, etc. Ceramic can be classified into traditional and modern ceramic (Carter & Norton, 2007).

Traditional ceramic is clay-based ceramic such as pottery, whiteware, earthenware, stoneware, chinaware, porcelain, and technical ceramics. Pottery is made up of clay which has been sintered at the sintering temperature of less than 1200 °C. Stoneware and chinaware are typically produced by mixing of clay, feldspar and alumina which has been sintered around 1300 °C. Meanwhile, porcelain and technical ceramic are formed by mixture of clay, feldspar, and quartz which been sintered around 1300 °C and they can probably be sintered over 1300 °C. Traditional ceramics are commonly being applied in industry of tiles, sanitary ware, art ware and table ware (Chiang et al., 1997).

Meanwhile, modern ceramic or generally known as advanced ceramic includes alumina, zirconia, silicon carbide, silicon nitride and other types of nitride and carbide. Advanced ceramics are specifically invented by using ceramic materials that possess good thermal and electrical properties, corrosion or oxidation resistance and superior mechanical properties. The application of advanced ceramics are typically on laser host materials, piezoelectric ceramics, ceramics for dynamic random access memories (DRAMs) and nuclear fusion and fission application (Carter & Norton, 2007).

2.3 Porcelain

Porcelain is a type of ceramic that belongs to the group of silicate ceramics. It is defined as translucent vitreous ware, and it has very fine grained ceramic whiteware. Porcelain is originated from the France and Italy term “*porcella or porcellana*” in 16th century, which means translucent ceramic that uses clay as a major compound (Riley, 2009). Porcelain is being described as a tri-axial ceramic, which is comprised of three different main raw materials, namely clay, feldspar and quartz. Each raw material has its own respective roles. For instance, clay act as plastic materials, feldspar as a fluxing agent, whereas quartz act as a filler (Kitouni & Harabi, 2011).

The primary raw materials of porcelain are nepheline syenite and alumina. Both of the materials are commonly used as feldspar and quartz to improve the mechanical strength of the porcelain. Meanwhile, secondary raw materials include bentonite, bone ash, talc and zircon (Table 2.1), all of which are being commercialized in industry of whiteware ceramics (Carty & Senapati, 1998).

Table 2.1: Secondary raw materials of whiteware ceramics

Raw material	Nominal compositions	Common compositions	References
Bentonite	Montmorillonite: $(M^{2+})(M^{3+})_4(Si,Al)_8O_{20}$ $(OH)_{4-n}H_2O$	Not applicable	(Carty & Senapati, 1998)
	Montmorillonite: $(Al_2, Mg_3) (Si_4O_{10})$ $(OH)_{2-n}H_2O$	Kaolinite: $Al_2Si_2O_5(OH)_4$	(Widodo et al., 2019)
		Illite: $(K,H_3O)(Al,Mg,Fe)_2(Si,Al)_4O_{10}$ $(OH)_2,(H_2O)$	
		Halloysite: $Al_2Si_2O_5(OH)_4$	
		Calcite: $CaCO_3$	
		Pyrite: FeS_2	
		Quartz: SiO_2	
Glass frits	No specific. Generally, frits depends on company specific due to the fusion of variety of minerals in furnace and quenching rapidly of molten materials.	Anorthite–Diopside: $CaO–MgO–Al_2O_3–SiO_2$	(Rasteiro et al., 2007)
		Cordierite–Indialite: $MgO–Al_2O_3–SiO_2$	
		Celsian: $BaO–Al_2O_3–SiO_2$	
		Anorthite: $CaO–Al_2O_3–SiO_2$	
		Nepheline: $Na_2O–Al_2O_3–SiO_2$	
Petalite (lithium feldspar)	$Li_2O \cdot Al_2O_3 \cdot 6SiO_2$	Sodium Oxide: Na_2O	(Carty & Senapati, 1998)
		Potassium Oxide: K_2O	
Bone ash	$Ca_3(PO_4)_2$	Silica: SiO_2	(Zakaria & Haron, 2014)
		Alumina: Al_2O_3	
		Wollastonite: $CaSiO_3$	(Gouvêa et al., 2015)
		Anorthite: $CaAl_2Si_2O_8$	
Talc	$3MgO \cdot 2SiO_2 \cdot 2H_2O$	Chrysotile: $Mg_3(Si_2O_5)(OH)_4$	(Carty & Senapati, 1998)
		Calcium Oxide: CaO	
Whiting	Calcium Carbonate: $CaCO_3$	Magnesium Carbonate: $MgCO_3$	(Carty & Senapati, 1998)
Zircon	Zirconium Silicate: $ZrO_2 \cdot SiO_2 / ZrSiO_4$	Not applicable	(Carty & Senapati, 1998)
	Zirconium Silicate: $ZrSiO_4$	Titanium Dioxide: TiO_2	(Casasola et al., 2012)
		Tin(II) oxide: SnO_2	

Three constituents of the raw materials of porcelain are categorised in phase system [$K_2O \cdot Al_2O_3 \cdot SiO_2$]. Based on the phase diagram in Figure 2.1, the compositions of porcelain is high in silica (SiO_2), and the diagram clearly shows that the major constituents of its raw materials is clay which is comprised of 50 wt. % (Carty & Senapati, 1998). The compositions for tri-axial porcelain are 50 wt. % of clay, 25 wt. % of feldspar and 25 wt. % of quartz. The equilibrium relations between different phase changes due to the vitrification process which requires higher temperature. This leads to the phase formation which determines the microstructure of the porcelain (Iqbal & Lee, 2000). Due to the increased of feldspar, the vitrification can be achieved at lower temperature which leads to a reduction of high energy consumption and cost of sintering process (Kingery et al., 1976).

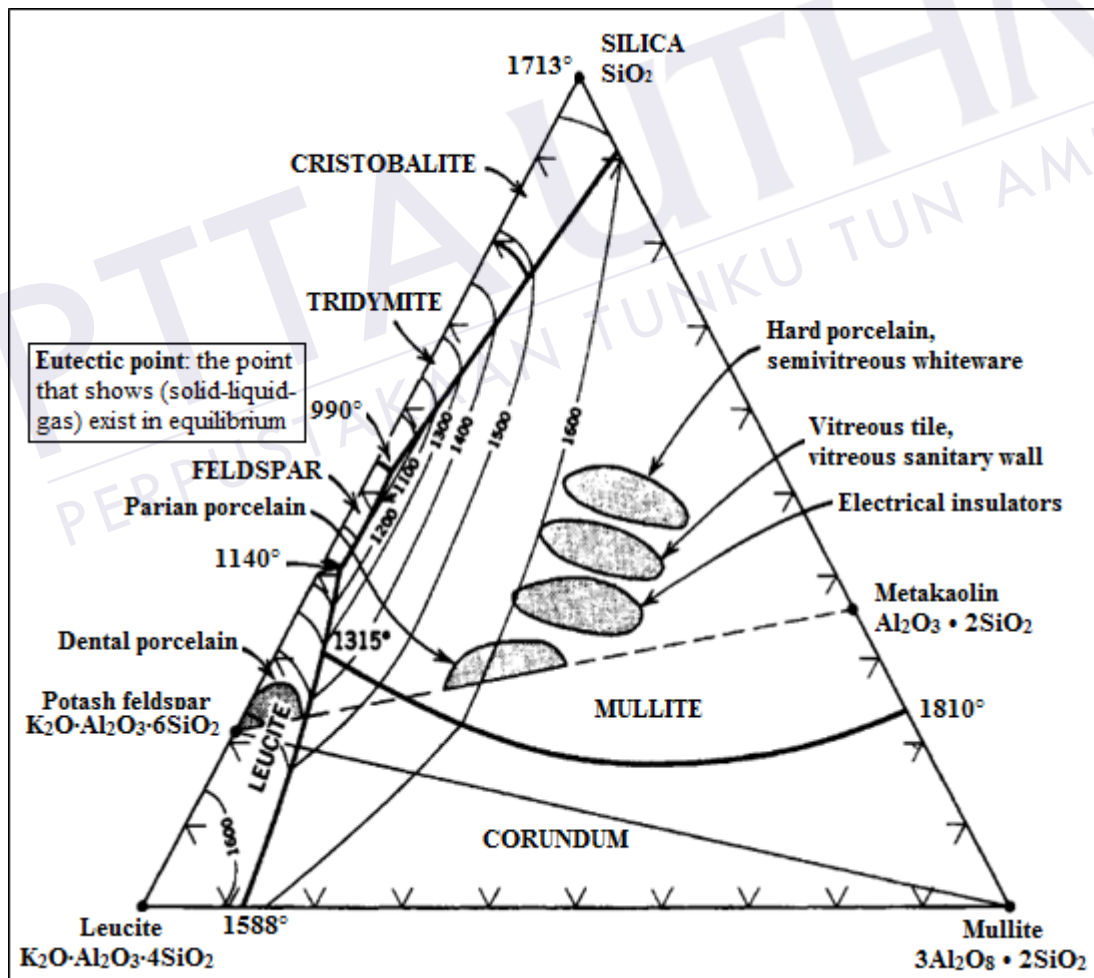
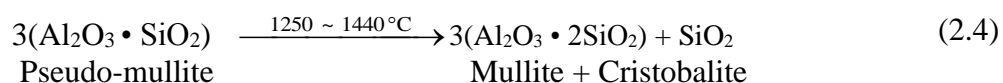
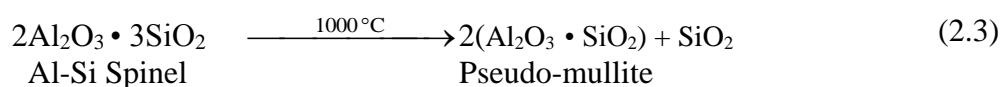
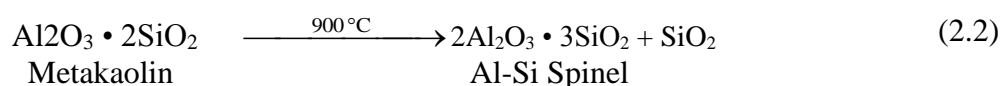
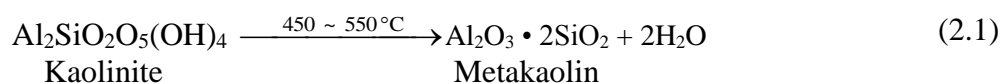


Figure 2.1: Compositions of various types of porcelain in [$K_2O \cdot Al_2O_3 \cdot SiO_2$] phase diagram (Carty & Senapati, 1998)

The ternary-phase of porcelain involves generating volumes of liquid, inducing vitrification and presents the translucency. Generally, the lowest liquid-forming temperature is 695 °C due to melting process of potash feldspar in contact with silicon dioxide. This process is also from the decomposition of the kaolinite. As the temperature increases, the liquid area progressively moves toward Al₂O₃ – SiO₂ axis. Kaolinite dehydroxylated to form metakaolin at the sintering temperature range of 450 – 550 °C, and the chemical equation is presented in Equation 2.1 (Shackelford & Doremus, 2008). Above the sintering temperature of 900 °C, metakaolin starts to form silicon-aluminium spinel phase and free silicon dioxide, resulting in the crystallizations of mullite above 1000 °C. This chemical reactions is presented in Equation 2.2 (Betala, 2014; Jamo, 2015).

However, the melting eutectic point was spotted at 990 °C. The eutectic temperature shows that all the three phases of solid-liquid-gas exists in equilibrium, where the feldspar particles in contact with decomposed kaolinite crystal with the presence of silicon dioxide. Generally, the eutectic temperature depends on the type of the feldspar (Carty & Senapati, 1998). Potash feldspar melts and forms a liquid phase containing silicon dioxide and leucite at 1140 °C, where it can help in the densifications of porcelain (Chiang et al., 1997). Based on Equation 2.3, the primary mullite forms by the decomposition of metakaolin at low temperature (~1000 °C), which at this state, the silicon dioxide is amorphous. Meanwhile, the silicon dioxide crystallizes at higher temperature and turns to cristobalite as presented in Equation 2.4.



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