EFFECT DIFFERENT RATIO OF SODA FELDSPAR AND POTASH FELDSPAR ON THE PROPERTIES OF PORCELAIN TILES

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DECLARATION

I hereby declare that I have conducted completed the research work and written the dissertation entitled "**Effect Different Ratio of Soda Feldspar and Potash Feldspar on the Properties of Porcelain Tiles**". I also declared that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or university.

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LIST OF SYMBOLS

α	Alpha
β	Beta
°C	Degree Celsius
°C/min	Degree Celsius per Minute
g	Gram
g/cm3	Gram per Centimeter Cube
h	hour
µm	Micron Meter
MPa	Mega Pascal
mm	Millimeter
Min	Minute
%	Percentage
wt%	Weight Percent

LIST OF ABBREVIATIONS

Fe_3O_4	Magnetite
$3Al2O_3 \cdot 2SiO_2$	Mullite
SiO_2	Silica
$Al_2Si_2O_5(OH)_4$	Kaolinite
$AlNaO_8Si_3$	Soda Feldspar
$KAlSi_3O_8$	Potash Feldspar
ICDD	International Center for Diffraction Data
MS ISO	Malaysian Standard International Organization for Standardization
USM	Universiti Sains Malaysia
SEM	Scanning Electron Microscope
MOR	Modulus of Rupture
PSA	Particle Size Analyzer
WA	Water Absorption
XRD	X-ray Diffraction

KESAN PERBEZAAN NISBAH SODA FELDSPAR DAN KALIUM FELDSPAR TERHADAP SIFAT JUBIN PORSELIN

ABSTRAK

Objektif kajian ini untuk mengkaji kesan komposisi berbeza kalium feldspar dan soda feldspar serta suhu pembakaran berbeza terhadap sifat jubin porselin. Dalam kajian ini, bahan mentah untuk jubin porselin ialah tanah liat Trong, silika, kalium feldspar dan soda feldspar. Parameter - parameter yang dikaji adalah komposisi soda feldspar terhadap kalium feldspar [0:40 (T1, T7), 5:35 (T2, T8), 10:30 (T3, T9), 20:20 (T4, T10), 30:10 (T5, T11), 40:0 (T6, T12)] pada dua suhu pembakaran yang berbeza (1150°C and 1200°C). Serbuk dicampur dengan kaedah pencampuran kering dan diikuti dengan proses pengisaran selama 8 jam bagi mencapai keseragaman campuran. Seterusnya, serbuk ditekan bagi membentuk segi empat dengan menggunakan tekanan 35MPa. Sampel dikeringkan di dalam ketuhar untuk menyingkirkan kelembapan berlebihan sebelum dibakar pada dua suhu berbeza (1150°C and 1200°C) dalam tempoh rendaman selama 2 jam dan kadar pembakaran 5°C/min. Sampel yang dibakar dicirikan dengan sifat fizikal dan sifat mekanikal (modulus kepecahan). Mikroskop elektron imbasan (SEM) dan Pembelauan Sinar-X (XRD) digunakan untuk menganalisis mikrostruktur dan fasa di dalam jubin porselin. Komposisi 40% soda feldspar ($T12_F$) yang dibakar pada suhu 1200°C mempunyai serapan air paling rendah (0.12%) dan modulus kepecahan tertinggi (65.2 N/mm^2) disebabkan oleh pembentukan keliangan terendah (0.2%). Analisis fasa XRD menunjukan pembentukan fasa mulit pada suhu bakar 1200°C menjadi faktor penting bagi kekuatan jubin porselin. Kesimpulannya, komposisi 40% soda feldspar menghasilkan komposisi optimum yang boleh diganti kedalam jubin porselin pada suhu pembakaran 1200°C.

EFFECT DIFFERENT RATIO OF SODA FELDSPAR AND POTASH FELDSPAR ON THE PROPERTIES OF PORCELAIN TILES

ABSTRACT

The objective of this work is to study the effect of different composition of potash feldspar and soda feldspar at different firing temperatures to the properties of the porcelain tiles. The raw materials of porcelain tiles in this work were Trong clay, silica, potash feldspar and soda feldspar. Parameters studied in this work were ratio composition of soda feldspar to potash feldspar [0:40 ($T1_A$, $T7_A$), 5:35 ($T2_B$, $T8_B$), 10:30 ($T3_C$, $T9_C$), 20:20 ($T4_D$, $T10_D$), 30:10 ($T5_E$, $T11_E$), 40:0 ($T6_F$, $T12_F$)] with two different firing temperature (1150°C and 1200°C). The powder was mixed using a dry mixing method and then followed by a milling process for 8 hours to achieve homogeneous mixture. Then, the powder was pressed to the rectangular shape using 35MPa of pressure. The sample was dried overnight in the oven in order to remove the excess moisture before being fired at two different temperatures (1150°C and 1200°C) with 2 hours soaking time and 5°C/min of heating rate. Fired samples were characterized by physical properties and mechanical properties (modulus of rupture, MOR). Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD) were used to observe the microstructural analysis of fracture surfaces and phases present in the fired bodies of porcelain tiles. Composition 40% of soda feldspar ($T12_F$) fired at temperature 1200°C has lowest water absorption (0.12%) and highest MOR (65.2 N/mm^2) due to lowest porosity (0.2%) formed. The XRD phase analysis shows the formation of the mullite phase which is a significant factor for the strength of porcelain tiles. In conclusion, 40% soda feldspar provided the optimum composition that can be substituted in the porcelain tiles at firing temperature 1200°C.

CHAPTER 1

INTRODUCTION

1.1 Background Research

Porcelain is a ceramic product that is created from a mixture of clay, silica, and feldspar. It is a type of ceramic that requires it to be fired at a high temperature to achieve vitreous, or glassy phase, including translucence and low porosity (Advameg, 2021). The primary constituents of porcelain are clay, silica, and feldspar. Clays are utilized in ceramics for shaping, providing plasticity and strength throughout the fabrication process. Silica is used to promote thermal and dimensional stability due to its high melting point. Feldspar acts as a fluxing agent in ceramic bodies, where it is used to lower the melting temperature of the ceramic body during firing and forms a glassy phase in the body. It melts at an early stage during the firing process and then develops the glass phase, which binds other components together (Potter, 1998).

Production of porcelain tiles has become the most significant product in the ceramic industry due to its excellent physical and mechanical characteristics, which include low water absorption, high fracture toughness, high density, and high strength. Porcelain usually has mixtures of ~50wt% of kaolin and clay, ~30wt% of flux (sodium or potassium feldspar) and ~20wt% of quartz (Bouzidi et al., 2013). In addition, by varying the quantities of the three primary constituents which are clay, feldspar and silica, the physical and mechanical properties of ceramic products also can be improved. It was addressed in the previous study that a better understanding of the porcelain properties is required in the starting composition of raw materials and their processing methods (De Noni et al., 2010; Ochen et al., 2021). Therefore, it is necessary to use higher purity raw materials in the starting composition to fabricate the porcelain tiles with desired properties.

1.2 Problem statements

Nowadays, porcelain tiles are the most technologically advanced ceramic tile material, and it stands out for being denser, fully glazed, and very low porosity. Because of these properties, porcelain has excellent mechanical and chemical properties that make it very resistant to chemicals, cleaning agents, abrasion, and fracture (Piccolo et al., 2022). Porcelain tile is a product with high technical properties and aesthetic value. Porcelain is a ceramic material with a very compact structure and has extremely low porosity with water absorption less than 0.5% which make it more suitable for their applications (Zanelli et al., 2004).

In ceramic industries, porcelain commonly has mixtures of 40-50 wt% of kaolin and clays, 35-45wt% of flux (sodium or potassium feldspar) and 10-15wt% of quartz (Bouzidi et al., 2013; J. Martín-Márquez et al., 2008). Porcelain production usually required a high firing temperature as high as 1200°C to achieve densification of tile (Fatile, 2014; Sawadogo et al., 2020). The higher purity of clay, silica and feldspar in the starting composition is necessary to exhibit desired properties of the porcelain tiles. Alkali feldspars are most commonly used as a raw material in ceramic and glass making industries (Alimon & Ahmad, 2011), which is used to lower the melting temperature of the ceramic body during firing and forms a glassy phase in the body (Potter, 1998).

Ozturk and Yildiz, (2015) mentioned that the high alkaline contents (10-15%) are suitable for fluxing agents as it promotes the formation of viscous liquid phases that helps to enhance the densification process of porcelain tiles. According to Ajayi, (2014), feldspar with high potassium content has been discovered to be better for body formulation, while feldspar with high sodium content is more useful in glaze formulation because of its low melting point. Therefore, the flux that is widely used in porcelain composition is potassium feldspar. However, several authors noted that mixed feldspars are ideal for fluxing agents for porcelain manufacture since this feldspar develop a very viscous liquid phase that embeds the new forming crystal, resulting in the densification process (Kamseu et al., 2007; Sarkar, 2019). This densification process will help to indicate the properties of the porcelain tiles produced.

According to the Anak Ginung and Abdullah (2015), in Malaysia, sodium-rich feldspar deposits have been discovered in Gua Musang, Kelantan, and Merapoh, Pahang, while high potassium feldspar volcanic rocks deposits have been discovered in Grik, Perak. Anak Ginung and Abdullah (2015) also noted that the ceramic and glass-making industry in Malaysia rely on imported feldspar from Netherlands, Thailand, Turkey, China, and India due to shortage of feldspar deposits. If local soda feldspar can be applied to make porcelain tiles, it will increase the use of local soda feldspar since deposits of sodium-rich feldspar in Malaysia have high production compared to deposits of potassium-rich feldspar. This will help to increase the use of local soda feldspar and reduce dependency on imported feldspars.

In this research work, we propose a substitution of potash feldspar with soda feldspar for fabrication of porcelain tiles. The ratio composition of soda feldspar to potash feldspar that used are [0:40 ($T1_A$, $T7_A$), 5:35 ($T2_B$, $T8_B$), 10:30 ($T3_C$, $T9_C$), 20:20 ($T4_D$, $T10_D$), 30:10 ($T5_E$, $T11_E$), 40:0 ($T6_F$, $T12_F$)] with two firing temperature (1150°C and 1200°C). The suitability and performance substitution of potash feldspar with soda feldspar for producing high quality porcelain tiles were evaluated in terms of physical and mechanical properties. It expected that the porcelain tiles produced by using soda feldspar give higher strength and low water absorption compared to potash feldspar, thus helping to increase the use of local soda feldspar and reduce the dependency on imported feldspar.

1.3 Objectives

The objectives of these studies are as follows:

i. To evaluate the substitution of potash feldspar with soda feldspar at different compositions and temperatures.
ii. To identify the composition of soda-potash feldspar combination to the properties of porcelain tiles.

1.4 1.4 Scope of Research

This research work was conducted to investigate the potential of substitution of potash feldspar with soda feldspar at different compositions and temperatures for production of porcelain tiles. This research work is divided into three stages as shown in Figure 1.1.

Stage I is the characterization of the raw materials to verify and validate the raw materials. Several characterizations were conducted such as X-ray Diffraction (XRD) for phase analysis, Particle Size Analysis and Scanning Electron Microscope (SEM) for morphology of raw material.

Stage II is the fabrication of porcelain tiles which began with preparation of raw materials. The samples were prepared for six different compositions. The mixture of raw materials was dry mixed and milled for 8 hours to achieve homogeneity. The dry powder weighed about 35grams and then pressed at 34 MPa to create rectangular tiles shapes with dimensions of length (103 mm), width (39mm), and thickness (5.2mm) The tiles were drying in the oven for 24 hours at 80°C to remove any moisture before being fired at two different temperatures (1150°C and 1200°C) with 2 hours soaking time and 5°C/min of heating rate.

Stage III is the characterization of fired bodies of porcelain tiles in terms of physical properties (appearance observation, firing shrinkage, water absorption, bulk density, apparent porosity) and mechanical properties via Modulus of Rupture (MOR) testing. Lastly, X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM) were conducted to analyze the phase and microstructure of optimum fired porcelain tiles that were selected based on the maximum and minimum water absorption at temperatures 1150°C and 1200°C.

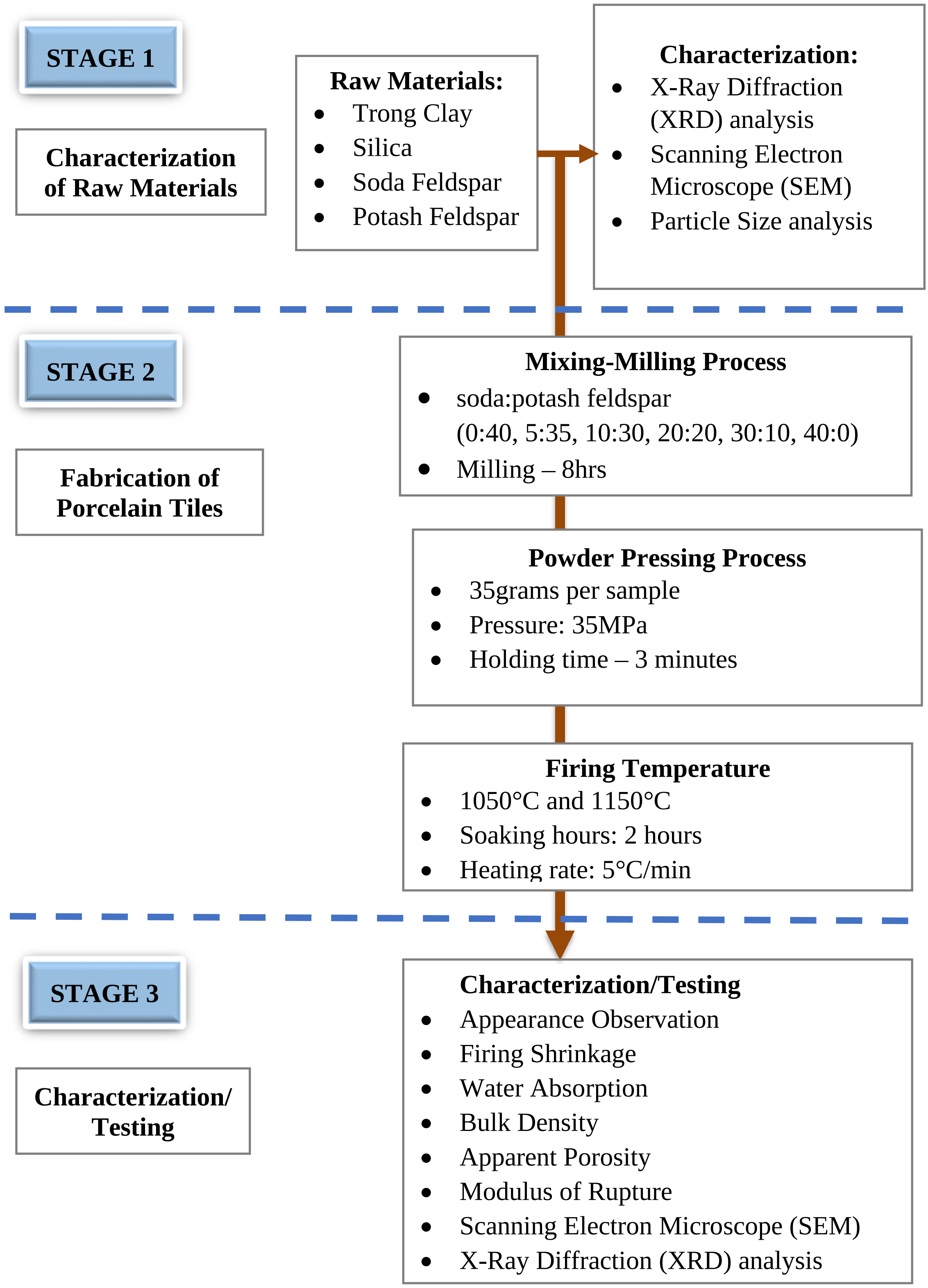

Figure 1. 1: shows the process flowchart of fabrication of porcelain tiles.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction to Ceramic Tiles

Ceramic tiles are typically used for flooring, wall decorating and roof covering. A thin slab with a full cross-section made of clays and other inorganic raw materials are generally formed by extruding or dry pressing at room temperature, but which may also be formed using other processes. The slab is then dried and fired at high temperatures to develop the necessary properties. Majority ceramic tiles have either a white or red body color under the glazed finish.

Nowadays, there are various types of ceramic tiles on the market that vary based on the design, and characteristics of the final product. Among ceramic tiles, porcelain is the most technologically advanced ceramic tile material, and it stands out for being denser, fully glazed, and very low porosity. Because of these properties, porcelain has excellent mechanical and chemical properties that make it very resistant to chemicals, cleaning agents, abrasion, and fracture (Piccolo et al., 2022).

2.2 Body Formulation of Ceramic Tiles

The performance of ceramic tiles is optimized by starting materials which are clay, feldspar and silica. Clay provides plasticity that facilitates the shape of the ceramic body, feldspar is used as fluxing agent to lower the firing temperature by forming a glassy phase and silica is acts as a filler and stabilizer material (Baccour et al., 2009; Kamseu et al., 2007).

2.2.1 Clay

Clay primarily composed of fine-grained minerals that typically plastic at appropriate water contents and hardens when dried or fired (Mejia, 2015; Reeves et al., 2006). The ceramics industry is interested in clay's plasticity, which helps with body shape, chemical and mineral composition, thermal properties, color properties, densification and mechanical strength after firing (Baccour et al., 2009; Burst, 1991; Nwankwojike et al., 2020). Alkaline oxides, primarily K_2O and Na_2O, allow the glassy phase and liquid phase to form during the firing of clay when the temperature is above 900^{o}C. These alkaline oxides encourage the formation of a liquid phase that contributes densification to occur (Baccour et al., 2009).

According to Anak Ginung and Abdullah (2015), the ceramics industry is the largest consumer of clays. Clays are primarily used in making of ceramics, concrete, bricks, and cement. Clays can be classified as common clay, ball clay, and fire clay. Common clay is used to make heavy clay products such as building bricks, and structural tiles. Meanwhile, ball clay is usually used to produce products such as pottery, floor and wall tiles, and sanitaryware. Fire clay is used to produce refractory products including fire bricks and blocks and high alumina bricks (Anak Ginung & Abdullah, 2015).

2.2.1(a) Chemical Structure of Clay

The clay minerals are a group class of hydrous aluminosilicates that are often present in sediments and soils. Each clay mineral consists of a unique arrangement of layers that are made up of sheet formed of either tetrahedral or octahedral structural units (Mejia, 2015; Reeves et al., 2006). Si and Al are linked together in a planar arrangement of sheets that connect their tetrahedral and octahedral layers. Two planes of oxygen atoms are formed in tetrahedral coordination around Si atoms in a single sheet, while around Al atoms in the centre of the other sheet, OH groups are arranged in octahedral coordination. The clays can be formed by two layers, three layers, etc and can be classified according to the number of layers as shown in Table 2.1 (Mejia, 2015).

Table 2. 1: Classification of clays minerals. The layer type refers to the tetrahedral: octahedral sheet ratio (Mejia, 2015)

Layer Type	**Group**	**Mineral Species**
1:1	Kaolin-Serpentine	kaolinite, dickite, nacrite and halloysite.
2:1	Mica-Illite	muscovite, illite, glauconite, celadonite, paragonite.
	Smectite	montmorillonite, beidellite, nontronite
	Chlorite	clinochlore, chamosite, pennantite.
	Vermiculite	vermiculite.

According to Mejia (2015), kaolin and serpentine are the most fundamental arrangements. Kaolin is an important raw material for the ceramic industry that is mostly used for the production of porcelain and refractory products. The primary component of kaolin is Kaolinite ($Al_2Si_2O_5(OH)_4$). In addition, Mejia (2015) also noted that mullite ($3Al_2O_3 \cdot 2SiO_2$) is the primary product phase after firing of kaolin at high temperatures. When the stability limits of the crystalline clay structures are reached during firing, they partially decompose while additional phases are simultaneously created.

2.2.2 Silica

Silica (SiO_2) is an important raw material in the ceramic industry and a major source of silica is sand. The most frequent stable forms of silica are quartz, cristobalite, and tridymite. At room temperature, quartz is stable form, when temperature between 870°C and 1470°C, tridymite is stable, while temperature 1470°C to the melting point 1713°C, cristobalite is stable (Heaney & Veblen, 1991). Quartz is the most common mineral about 12% of the Earth's crust. It is constituent of igneous rocks and found in most metamorphic rocks, comprising a major part of the sandstone. Quartz is composed of silica tetrahedral grouped in such a way to form spirals with all the tetrahedral oxygen bonded to silicon (Silberberg, 2012).

A quartz content in typical porcelain tile composition consists of 5 to 25%. Quartz must be present in order to reduce drying and firing shrinkage (Santos et al., 2021). Nwankwojike et al., (2020) noted that silica helps to reduce the shrinkage and increase the whiteness of the fired body. This is also supported by Das and Dana, (2003) that silica helps to increase the whiteness and reduce the shrinkage due to the presence of viscous glass.

2.2.2(a) Structure of Silica

There are two forms of quartz which are α and β quartz. Up to 573 °C, α-quartz is stable, and β-quartz is stable above this temperature. The quartz changed from α-Quartz to β-Quartz as the temperature approached 573°C during firing. Heaney and Veblen (1991) stated that α-quartz with rhombohedral structure transforms to β-quartz with hexagonal structure. This transformation of α↔β quartz inversion is reversible. During the cooling stage, the inversion from β-quartz to α-quartz is accompanied with shrinkage, which can lead to enough deformation to fracture in porcelain stoneware tile bodies.

2.2.3 Feldspar

Feldspar is the most abundant mineral roughly 60% of the earth's crust, which is found in practically all igneous and metamorphic rocks, as well as several sedimentary deposits. Feldspar raw materials consist of alumina (Al_2O_3), alkali (Na_2O and K_2O), and silica (SiO_2). Feldspars are considered as fluxing agents because they provide alkaline oxides such as Li_2O, Na_2O and K_2O that help to produce low-viscosity liquid phases at high temperatures during firing process (Locks et al., 2021). Feldspar minerals with high alkali content (10 - 15 %) are often used as fluxing agents in tile compositions (Fatile, 2014). It is generally known that the proportion of alkaline oxides is an essential component that determines the viscosity, amount of glassy phase and crystal phases of ceramic bodies (Ozturk & Yildiz, 2015). In terms of composition, feldspars belong to a ternary system made up of three groups of potassium, sodium and calcium feldspars (Fuertes et al., 2022).

In the ceramic industry, there are several factors that need to be considered about the use of feldspar. Firstly, their granulometry must be smaller than 74 μm since fluxing power is inversely proportional to grain size (Fuertes et al., 2022). Secondly, their purity which is influenced by the type of final product. Iron is one of the impurities as it will give the final ceramic product a dark coloring (Fuertes et al., 2022). This will lower the quality of final products.

Feldspar is an essential raw material in batches for both ceramic industries about 35% and glass making about 60% (Fuertes et al., 2022). In the ceramic industry, feldspar is used as fluxing agent to help reduce vitrifying temperature of ceramic body and form a viscous liquid phase in the body during firing process, while in glass industry, alumina (Al_2O_3) from the feldspar help to improve hardness, durability and resistance to chemical corrosion of the product. Locks et al., (2021) noted the feldspar form a viscous liquid phase that fills the pores of the ceramic composition by a capillary effect at temperatures above 1100°C, bringing the particles of silica and clay closer together and promote densification of the ceramic bodies in firing process. The liquid phase that formed in the ceramic body will interact with other elements (clay and silica) and bind the particles together, resulting in the densification of the body (Bakr et al., 2009).

There are only potassium and sodium feldspar that are commercially valuable and are utilized in the glass and ceramic industries (Anak Ginung & Abdullah, 2015). These feldspars produce the liquid phases during the firing process, which contributes to increase the densification and reduce the porosity of the product. During the firing process, large amounts of liquid phases are formed, resulting in low porosity. According to Sarkar (2019), in practice, both potash and soda feldspar are needed to manufacture whiteware where potash gives a high viscous liquid and a wide fire temperature range, while soda feldspar provides low viscosity and significant fluxing agent in glazes. High alkaline feldspars (10-15 %) are suitable fluxing agents for porcelain production, according to (Fatile, 2014), because it promotes the formation of a very viscous liquid phase that allows the porcelain tile to densify. However, in a previous study it noted that potassium feldspar is widely used as fluxes in porcelain composition due to high firing temperature (1200°C) of porcelain (Cam & Senapati, 1997).

Feldspar is used to improve the performance of the final ceramic product such as durability, whiteness, strength, toughness, and chemical resistance. These properties allow the feldspar to be utilized in a variety of applications with varying feldspar content, such as dinnerware (17- 20%), sanitary wares, floor tiles (55-60%), wall tile

(0-11%), high-tension electrical porcelains (25-35%), dental porcelains (60-80%), kitchen and ovenware (10%) (Fuertes et al., 2022). One of applications of feldspars is ceramic tiles such as wall tile, floor tile, vitrified tile, and industrial tile. According to IMARC Group (2022), the global ceramic tiles market has been reached US$ 75.4 billion in 2021. This market is expected to reach US$ 107.3 Billion by 2027, exhibiting a (Compound Annual Growth Rate (CAGR) of 5.9% during 2022-2027 (IMARC Group, 2022).

2.2.3(a) Chemical Structure of Feldspar

The chemical composition of feldspar is important when considering their properties. The most common criterion for assessing the use of a specific feldspar in a whiteware body and behaviour of material under various conditions is chemical analysis.

Feldspars are minerals that belong to the group of silicates where the basic unit of silicates is the silicon tetrahedron $(SiO_4)^{4-}$ (Fuertes et al., 2022). The structure of feldspars is made up of four-tetrahedra rings which are joined by two tetrahedra and has oxygen-filled corners (Kyonka & Cook, 1954). Different subgroups will be distinguished depending on how these tetrahedra are joined. Chemically, feldspar is an aluminosilicate mineral of which the formation occurs when Al^{3+} replaces Si^{4+} in some silicon tetrahedra. This substitution results in a positive charge defect that is balanced out by a variety of alkali and alkaline-earth cations (Fuertes et al., 2022). Common feldspars have a general formula of $XAl_{(1-2)}\,Si_{(3-2)}\,O_8$, where X can be either sodium (Na) or potassium (K) or calcium (Ca).

2.2.3(b) Types of Feldspar

There are three types of feldspar that are commonly used in the ceramic and glass industry are potash feldspar, soda feldspar and calcium feldspar.

2.2.1(b)(i) Potash Feldspar

Potash feldspar or known as potassium feldspars ($KAlSi_3O_8$), crystallizes as orthoclase in monoclinic structure and as microcline in a triclinic structure. Despite having slightly different crystal structures, orthoclase and microcline share the same element and have similar physical properties. Orthoclase is a common component of many igneous rocks and is frequently present in large masses. Microcline is a potassium-rich alkali feldspar that typically contains minor amounts of sodium. Microcline was formed during slow cooling of orthoclase, which is more stable than orthoclase at lower temperatures. Microclines are totally dissolved in melt above 1200°C (Jorge Martín-Márquez et al., 2009).

2.2.1(b)(ii) Soda Feldspar

Soda feldspars or sodium feldspars ($NaAlSi_3O_8$) crystallize as albite in a triclinic form. Albite is a relatively common and significant rock-making mineral. Albite is totally dissolved at temperature 1100°C (Jorge Martín-Márquez et al., 2009). Many ceramic and glaze manufacturers prefer soda feldspar because it has a stronger fluxing property than potash feldspar, which regulates the degree of vitrification (Kyonka & Cook, 1954). It has been discovered that soda feldspar melts uniformly to a very viscous liquid at a temperature of 1118 ± 3°C (Kyonka & Cook, 1954). Soda feldspar exhibits a lower viscosity than potassium feldspar when melted at the same temperature (Sarkar, 2019). This allows vitrification at lower temperatures, but it also increases the risk of deformation.

2.2.1(b)(iii) Calcium Feldspar

Calcium feldspars ($CaAl_2Si_2O_8$) or also known as lime feldspar occurs a white or grayish, brittle phase which is also used in the manufacture of ceramic and glass. It is crystallized as anorthite in a triclinic form. Anorthite is plagioclase feldspar. Plagioclase is a framework silicate mineral which is commonly defined based on the content of anorthite (An) in the solid solution (Fuertes et al., 2022).

Table 2. 2: Classification of feldspar group attending to their chemical composition, Si/Al distribution, structure (Fuertes et al., 2022).

Chemical composition	Specie	Si/Al distribution	Structure
K-feldspars	orthoclase	Fully disordered	Monoclinic
	Low Microcline	Fully ordered	Triclinic
	Intermediate Microcline	Partly disordered	Triclinic
Na-feldspars	Analbite	Fully disordered	Triclinic
	High Albite	Fully disordered	Triclinic
	Intermediate Albite	Partly disordered	Triclinic
	Low Albite	Fully ordered	Triclinic
	Monoalbite	Fully disordered	Monoclinic
Ca-feldspar	Anorthite	Disordered	Triclinic
	anorthite	Mostly ordered	Triclinic

2.2.3(c) Feldspar Deposit in Malaysia

Deposits of feldspar in Malaysia are in Merapoh (Kuala Lipis, Pahang), Bukit Mor (Muar, Johor), Perak (Simpang Pulai, Menglembu and Grik), Tanah Putih (Gua Musang, Kelantan), and Gemencheh (Negeri Sembilan). Based on the Minerals and Geoscience Department, they have identified sodium rich feldspar deposits in Gua

Musang, Kelantan and in Merapoh, Pahang, meanwhile high potassium feldspar volcanic rocks deposits in Gerik, Perak. However, feldspar deposits in Malaysia produce less workable feldspar due to low quality of feldspar, which prevents use in high-end applications (Alimon & Ahmad, 2011). The feldspar was produced from three feldspar-rich rock mines at Tanah Putih, Gua Musang, Kelantan and one in Gemencheh, Negeri Sembilan. According to Figure 2.1, feldspar production increased by around 282.5% from 314399 tonnes in 2012 to 1202727 tonnes in 2014 (Anak Ginung & Abdullah, 2015).

Table 2. 3: Production of Feldspar in Malaysia (Anak Ginung & Abdullah, 2015).

State	**2011**		**2012**		**2013**		**2014**	
	tonnes	**mines**	**tonnes**	**mines**	**tonnes**	**mines**	**tonnes**	**mines**
Kelantan	322029	3	456746	3	277061	3	1189947	3
N. Sembilan	57600	6	26160	6	37338	2	12780	2
Total	**379629**	**9**		**9**	**314399**	**5**	**1202727**	**5**

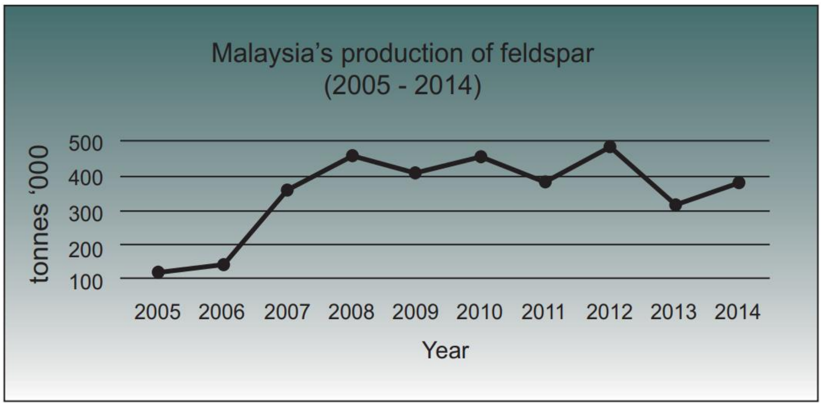

Figure 2. 1**:** Production of Feldspar in Malaysia from 2005 to 2014 (Anak Ginung & Abdullah, 2015).

Many industries used imported feldspars. The imported feldspar came from Netherlands, Thailand, China, India, and Turkey. Feldspar imported a total 124,947 Tonnes worth RM53.53 million in 2014. (Minerals and Geoscience Department, 2014). The importation of the feldspar in Malaysia in 2012 to 2014 is shown in Table 2.4.

Table 2. 4: Malaysian Imported Feldspar from 2012 to 2014 (Anak Ginung & Abdullah, 2015)

Country	2012		2013		2014	
	Quantity (Tonnes)	**Value (RM)**	**Quantity (Tonnes)**	**Value (RM)**	**Quantity (Tonnes)**	**Value (RM)**
Netherlands	96	466536	550	2640713	3079	15744751
Thailand	37302	7906732	10111	8125795	49504	12183575
China	20426	14863865	42319	13819657	42110	10547390
India	23011	7126199	15746	4810808	22106	6916418
Turkey	8926	4476971	5142	2717586	6069	3611969
Others	75053	33420616	3439	12251237	2079	4529885
Total	164814	68260919	77308	44365796	124947	53533988

2.3 Fabrication of Porcelain Tiles

2.3.1 Raw Material

There are three major ingredients to be considered in ceramic mixtures during processing which are clay, feldspar, and silica. Clays are the major component in the ceramic industry, which provides the aluminosilicates that will form the crystalline phase of the material and exhibit plastic behavior in ceramic bodies, giving the pieces the ability to be molded and strength needed for handling (Frizzo et al., 2020). During the firing process, clay will develop a mullite and glassy phase. Feldspars form a glassy phase at low temperatures and enable the achievement of almost zero (less than 0.5%) open porosity and a low level of closed porosity (less than 10%). Silica contributes to reducing the shrinkage and increasing the whiteness of the fired body (Sánchez et al., 2010). The common of porcelain has mixtures of 40-50 wt% of kaolin and clays, 35-45wt% of flux (sodium or potassium feldspar) and 10-15wt% of quartz (Bouzidi et al., 2013; J. Martín-Márquez et al., 2008).

2.3.2 Mixing Process

Mixing is necessary to combine the components of a ceramic powder to create a more chemically and physically uniform material during the forming process. Ceramic materials are frequently mixed in pug mills. To mix the ceramic components, all raw materials are added to pug mills after being weighed in accordance with their body composition. The properties of the mix and the final product are greatly influenced by the mixing time, intensity, and sequence of mixing (Ceramics, 2005). To obtain required physicochemical uniformity, the prepared raw materials must be mixed and homogenized.

2.3.3 Forming Process

The method used to shape the ceramic can have a significant impact on the final properties of ceramic products. Mostly, the ceramic tiles are formed using a dry pressing method. In dry forming, dry ceramic powders are simultaneously compacted and shaped in a hard die or flexible mold (Ceramics, 2005). Hydraulic pressing is widely employed for the shaping of tiles which can give high compaction force, high production, uniformity, and simple adjustment during the forming process (Ceramics, 2005).

2.3.4 Drying Process

Drying process is carried out to eliminate excess water and harden the unfired ceramic tile body before the firing process. The excess water is eliminated to prevent water from being entrapped in the ceramic tile body, enhancing the strength of ceramic bodies. Drying must be properly managed in order to reduce drying time while preventing differential shrinkage, warping, and distortion. Ceramics products are mostly dried via convection, which involves circulating warm air around the ceramics (Ceramics, 2005).

2.3.5 Firing Process

Firing is an important process in the production of ceramic products as it influences many properties of the final products, including mechanical strength, abrasion resistance, dimensional stability, resistance to water and chemicals, and fire resistance. Furthermore, the ceramic's strength, porosity, size, and color depend on the firing temperature and environment.

Sintering of the material will happen during the firing stage. The pores between the particles will be eliminated as a result of solid-state mechanisms that operate at lower temperatures, which causes the ceramic material to shrink. Sintering is also driven by viscous flow mechanisms that possess a higher effect on the densification of the green body at temperatures when liquid phase begins to form (German et al., 2009). As the firing temperature increases, the density increases until it reaches a maximum where there is enough liquid phase to completely fill off the open porosity (Wattanasiriwech et al., 2009).

2.4 Type of Ceramic Tiles

According to the ISO 13006 standard, ceramic tiles are categorized into three groups based on the water absorption which are Group I (<3% of water absorption), Group II (3–10% of water absorption) and Group III (>10% of water absorption (Dondi et al., 2014). Group I and Group II are divided into two subgroups each at the water absorption of 0.5% and 6%, respectively (Dondi et al., 2014).

2.4.1 Wall Tiles

Wall tile is suited for interior usage or that is commonly non-vitreous for enhanced adhesion to vertical facades. The general classification of general wall tiles includes products produced by both double and single firing. Ceramic tiles are classified into several groups by ISO 13006 according to their forming method and water absorption of the finished product. Based on ISO 13006 standard, porous wall tiles are included in the group III, in which the water absorption is higher than 10% (Dondi et al., 2014).

2.4.2 Floor Tiles

One of the important components of the building is the floor tiles, thus, it must be capable of withstanding heavy wear and mechanical stress. Typically, concrete floor surfaces are often used in the construction industry due to the strength, durability and affordability of concrete floors. The International Standard classified floor tiles as either having water absorption between 6% to 10% (J. Martín-Márquez et al., 2008).

2.4.3 Porcelain Tiles

Porcelain tile is a product with high technical properties and aesthetic value. Porcelain is a ceramic material with a very compact structure and has extremely low porosity with water absorption less than 0.5% and group BIa of ISO 13006 (Cavalcante et al., 2004; Zanelli et al., 2004). Ceramic material must have low porosity as it provides high mechanical and chemical properties to the material (Fatile, 2014). This makes them ideal for outdoor flooring and wall cladding in extreme temperatures.

Usually, porcelain tile is a white, thin, and translucent that possesses a metal-like ringing sound when tapped. It is made from kaolin, quartz, and feldspar and followed by heat treated to form a mixture of glass and crystalline phases (Leonelli et al., 2001). Porcelain commonly has mixtures of 40-50 wt% of kaolin and clays, 35-45wt% of flux (sodium or potassium feldspar) and 10-15wt% of quartz (Bouzidi et al., 2013; J. Martín-Márquez et al., 2008).

2.5 Properties of Porcelain Tiles

2.5.1 Firing Shrinkage

According to Sawadogo et al., (2020), firing shrinkage increases with temperature where the shrinkage increases rapidly from 1200°C to 1250°C, and slightly increases up to 1300°C. In general, increasing the firing temperature causes the shrinkage to increase (Kummoonin et al., 2014). This shrinkage shows the material is consolidated with volume reduction. According to J. Martín-Márquez et al., (2008), open porosity is closely related to shrinkage. Materials start to shrink, and the consolidation of the structure takes place once the transition temperature is reached, resulting in the reduction of the porosity of the materials. Shrinkage properties play a significant role in the ceramic industry in determining the size of the final product since dimensional control of the manufactured pieces is necessary for industrial ceramic products, such as floor and wall tile to have better quality of final products (Correia et al., 2004).

2.5.2 Bulk Density

Bulk density is an indicator for compaction of raw material in the ceramic body. The density is increased as the firing temperature increases. This is due to the formation of the liquid phases during firing above 1050℃, thus, enhancing the densification process (Sawadogo et al., 2020). The densification is driven by the gradual melting of feldspars. This indicates that the density of the material increases with temperature as a result of agglomeration or a reaction between the raw material's particles (De Noni et al., 2010; Sawadogo et al., 2020). De Noni et al., (2010) stated that maximum densification can be obtained rather than zero porosity under firing conditions by rearrangement of particle and viscous flow. Particle size distribution and the presence of flux (such K_2O and Na_2O), which encourage the formation of glassy phase, have an impact on the densification behavior. An essential engineering quality for building materials is the use of denser products (Baccour et al., 2009).

2.5.3 Water Absorption

The ceramic tiles are categorized using the water absorption property, which is a standardized characteristic based on the amount of water that a finished product can absorb under controlled conditions (Frizzo et al., 2020). The water absorption for porcelain tile is commonly less than 0.5% (De Noni et al., 2010). Water absorption is also related to densification and open porosity of ceramic bodies as a function of the firing temperature. When temperature increases, the water absorption will decrease. Das and Dana (2003) also noted that water absorption decreases with increase in firing temperature due to elimination of pores through formation of liquid phase. The liquid phase formed during firing, thereby increasing consolidation of the ceramic material at high temperatures (Kamseu et al., 2007). In addition, according to Baccour et al., (2009), above 1050°C, the values of water absorption decrease, as it is associated to a more significant liquid phase formation. Under the surface energy forces created by the fine pores contained in the ceramic body, the liquid phase tends to approach the pores, closing them and isolating nearby pores, resulting open porosity decreases (Kingery et al., 1976; Kummoonin et al., 2014; J. Martín-Márquez et al., 2008; Mathur et al., 2015).

2.5.4 Flexural Strength

Temperature enhances the strength of porcelain, especially above 1200°C (Sawadogo et al., 2020). Porcelain tiles are known to have a high flexural strength, which is greater than 35 ± 2 MPa, which makes them suited for applications (Ochen et al., 2021). Theoretically, when apparent porosity reaches zero, the flexural strength developed in a porcelain body reaches its maximum (Das & Dana, 2003). Usually, in ceramic compositions that contain clays and feldspars, higher temperatures cause more mullite phase to form, thus, improves the mechanical properties of ceramic (Kamseu et al., 2007).

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter presents the materials and methodologies for the research work. The research was carried out in order to evaluate the effect of substitution of potash feldspar with soda feldspar and firing temperature on the properties of porcelain tiles. Trong clay, soda feldspar, potash feldspar and silica were used as raw materials in this work. Two parameters carried out in this study are different ratio composition of soda feldspar to potash feldspar (0:40, 5:35, 10:30, 20:20, 30:10, 40:0) and firing temperature (1150°C and 1200°C).

This chapter can be divided into three stages where the first stage is characterization of raw materials, the second stage is fabrication of porcelain body, followed by the characterization of fired tiles as third stage. The process flow of substitution of potash feldspar with soda feldspar to fabricate porcelain tiles is shown in Figure 3.1.

For the first stage, the raw materials used in this work will be characterized to verify and validate the chemical content, morphology, and phase of the materials. The raw materials were characterized by X-Ray Diffraction (XRD) for phase analysis, X-Ray Fluorescence (XRF) for chemical composition of raw materials, Particle Size Analysis (PSA) for determine particle size and Scanning Electron Microscope (SEM) for morphology of the raw material.

In the second stage, the fabrication of porcelain tiles began with preparation of raw materials. The samples were prepared for six different compositions. The sample was batched and mixed using a dry mixing method, then followed by a milling process