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FABRICATION AND CHARACTERIZATION OF TRI-LAYER LIGHTWEIGHT CERAMIC TILE

By

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled "**Fabrication and Characterization of Tri-layer Lightweight Ceramic Tile**". I also declare 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 ABREVIATIONS

Al_2O_3	Alumina
$CaCO_3$	Calcium Carbonate
CO_2	Carbon Dioxide
CTE	Coefficient of Thermal Expansion
DTA	Differential Thermal Analysis
Fe_2O_3	Iron Oxide
FESEM	Field Emission Scanning Electron Microscope
ICDD	International Center for Diffraction Data
MOR	Modulus of Rupture
$3Al2O_3 \cdot 2SiO_2$	Mullite
OM	Optical Microscope
PSA	Particle Size Analysis
SiO_2	Quartz
TG	Thermogravimetry
XRF	X-ray Fluorescence
XRD	X-ray Diffraction

LIST OF SYMBOLS

α	Alpha
β	Beta
^{o}C	Degree Celsius
^{o}C/min	Degree Celsius per Minute
g	Gram
g/cm^{3}	Gram per Centimeter Cube
h	hour
<	Less than
>	More than
µm	Micron Meter
MPa	Mega Pascal
Mm	Millimeter
min	Minute
%	Percentage
RT	Room Temperature
S	Second
wt %	Weight Percent

PENGHASIALAN DAN PENCIRIAN JUBIN SERAMIK RINGAN TIGA LAPISAN

ABSTRAK

Kajian ini menerangkan proses penghasilan jubin seramik ringan tiga lapisan, terbentuk hasil daripada tiga lapisan dengan struktur padat berada di lapisan atas dan bawah serta struktur berliang pada lapisan tengah untuk membantu mengurangkan berat jubin seramik yang dihasilkan. Parameter yang dikaji ialah kesan nisbah tiga lapisan dan suhu pembakaran bagi penghasilan jubin seramik ringan tiga lapisan. 5% berat kalsium karbonat ($CaCO_3$) digunakan di dalam formula penghasilan jubin seramik bagi menghasilkan lapisan tengah yang berliang. Serbuk seramik dan $CaCO_3$ dianalisa. Pendarfluor Sinar-X (XRF), FESEM dan Pembelauan Sinar-X (XRD) telah digunakan untuk menganalisa komposisi kimia, morfologi dan komposisi fasa sementara TG-DTA digunakan untuk menentukan perlakuan haba serbuk seramik ini. Mikrograf optik bagi keratan rentas jubin seramik ringan tiga lapisan menunjukkan bahawa ikatan antara muka yang baik di antara lapisan dapat dihasilkan. Selain itu, pekali pengembangan terma bagi sampel yang belum dan sudah dibakar menunjukkan tiada isu bagi ketidakpadanan haba berlaku. Maklumat ini menunjukkan bahawa penggunaan serbuk $CaCO_3$ dengan jumlah bersesuaian di dalam formula lapisan tengah bagi jubin seramik yang dibakar akan mengurangkan berat jubin seramik sebanyak 7.3% pada suhu 1150^{o}C dan 3.7 % pada suhu 1050^{o}C. Selain daripada itu, kekuatan mekanik bagi jubin seramik ringan tiga lapisan akan berkurangan dengan penambahan nisbah lapisan pada lapisan tengah yang berliang. Justeru, kaedah tekanan tiga lapisan adalah calon yang bersesuaian bagi kaedah penghasilan jubin seramik ringan.

FABRICATION AND CHARACTERIZATION OF TRI-LAYER LIGHTWEIGHT CERAMIC TILE

ABSTRACT

This study describes the fabrication process of tri-layered lightweight ceramic tiles, formed from three layers with dense structure at upper and lower layers and porous structure at middle layer, which helps the weight reduction of tile. The parameter studied were the effect of tri-layer ratio and firing temperature on fabrication of tri-layer lightweight ceramic tile. 5 wt% of calcium carbonate ($CaCO_3$) was introduce into the ceramic tile formulation in order to produce porous structure middle layer. The ceramic powder and $CaCO_3$ was characterized. X-Ray Fluorescence (XRF), Field Emission Scanning Electron Microscope (FESEM) and X-Ray Diffraction (XRD) were used to analyse the chemical composition, morphology and phase composition while Thermogravimetry- Differential Thermal Analysis (TG-DTA) was used to determine the thermal behaviour of these powder. Optical micrograph for cross section of tri-layer lightweight ceramic tile indicate that good interfacial bonding between the layers were obtained. Also, thermal expansion coefficient of green bodies and sintered sample shows that there is no thermal mismatch issue exist. This information indicates that the incorporation of $CaCO_3$ powder in the middle layer formula for sintered ceramic tile reduced weight of ceramic tile by 7.3% (1150 ^oC) and 3.7% (1050 ^oC). Also, the mechanical strength of the lightweight ceramic tiles were reduced when increasing the layer ratio of the porous structure middle layer. Therefore, tri-layer pressing approach is suitable candidate method for the fabrication of lightweight ceramic tile.

CHAPTER 1

INTRODUCTION

1.1 Background

Ceramic tile is made up of clay, feldspar, quartz and talc (Leonelli *et al.*, 2001). It has been molded into a shape before sintered in a furnace. Ceramic tiles can either be glazed or unglazed depends on its application, but the majority of the glazed ceramic tiles used for household application such as kitchens, bathrooms and laundry rooms. Today, it is being used more often in areas that were previously covered with carpet or resilient floor covering (Fleming, 1997).

The porcelain tile is a clay body composition with the triaxial body (kaolin-feldspar-quartz) components. The term “clay” indicates the raw materials that provide plasticity and green strength during the forming of porcelain production. It contributes substantially to the color of the fired ware. Kaolinite, most significant part of ceramic tile, with chemical formula of $Al_2Si_2O_5OH_4$. It helps in forming, offering plasticity, and green body mechanical strength during processing. It contributes to the formation of mullite and vitreous phase during sintering. The fired colour of the kaolin is white as it has a high degree of purity. Ball clays often are referred to as plastic clays, because they possess a finer particle size, which produces greater plasticity (Carty and Senapati, 1998)

Feldspar is an important and common fluxing material to lower the melting temperature of the ceramic and thus to reduce cost (Carty and Senapati, 1998). It helps to attain vitreous nature of the body and the high mechanical resistance at the end of the firing stage. During sintering, it melts and forms glassy liquid that bonds the other components together, hence, reduces the open porosity and low level of closed porosity. Potash feldspar has been the most commonly used fluxes in porcelain (Kingery, 1960). Potash feldspars rarely are pure, usually containing the mineral albite (sodium feldspar) and anorthite (calcium feldspar) (Carty and Senapati, 1998).

Fillers are generally the coarsest-particle-size fraction of a porcelain body. The coarse particle size provides resistance to cracking during drying and forms a skeletal network during firing to mitigate pyroplastic deformation (bending during heat treatment). Quartz is the most commonly used fillers in porcelain bodies (Lundin, 1959). Addition of quartz decreases its unfired strength and plasticity but assist to facilitate escape of gases during drying and sintering. It also reduces drying shrinkage and increases the whiteness of the fired body (Ryan, 1999). Alumina can be substituted for quartz to increase the mechanical strength of the fired ware, and nepheline syenite can be substituted for feldspar (Carty and Senapati, 1998). Table 1.1 indicates the primary raw materials used in manufacturing of commercial ceramic tiles.

Table1.1 Primary raw materials used in manufacturing of commercial ceramic tiles (Carty and Senapati, 1998).

Raw material	Composition	Impurities
Ball (plastic) clay	$Al_2O_3.2SiO_2.2H_2O$	Quartz *, TiO_2, Fe_2O_3
Kaolin (chine) clay	$Al_2O_3.2SiO_2.2H_2O$	Montmorillonite, quartz
Soda feldspar	$Na_2O.Al_2O_3.6SiO_2$	K_2O, CaO, MgO, quartz
Potash feldspar	K_2O. $A1_2O_3.6SiO_2$	Na_2O, CaO, MgO, quartz
Nepheline syenite	$K_2O.3Na_2O.4A1_2O_3.9SiO_2$	CaO, MgO, quartz
Alumina	Al_2O_3	Na_2O
Quartz	SiO_2	TiO_2, Fe_2O_3

* Quartz impurity in ball clay may be as high as 35 wt%.

Traditional ceramics are ceramic materials that derived from naturally occurring raw materials such as clay minerals and quartz sand (Manfredini and Hanuskova, 2012). Conventional ceramic tiles are made up of raw material manufactured through industrial processes, for instance, china clay, ball clay, feldspar and quartz. The properties of conventional ceramic tile are much better than the traditional ceramic tile and can be manipulated by varying the composition of the raw materials, process route and the sintering profile. However, there are a number of disadvantages that conventional ceramic tile suffered. The mechanical properties of a fired tile often are not sufficiently good for particular applications. In particular, the thermal shock resistance of the tile body may be insufficiently low. This can lead to crazing when the tile subjected to rapid temperature changes. In addition, the moisture absorption can be too high, in turn leading to cracking problems. During sintering, tiles shrink in a variable manner, leading to size variations (Scarth and Evitt, 2000).

Recently, thermally insulates building facades include lightweight structural panels, and mostly consist of porcelain stoneware with controlled porosity. Ventilated facades are energy efficient systems that can build up the thermal and acoustic insulation of buildings. In ventilated facades, an external facade cladding is installed in front of the common building facade, creating an air space in between (Patania *et al.*, 2010). This gives rise to the invention of the lightweight porcelain tiles. However, according to Novais *et al.* (2015) the current lightweight tiles have high water absorption caused by the larger pore size. The reduction in weight also causes loss of mechanical strength of the tiles and their pyroplastic deformation (bending during heat treatment) also indicated in García-Ten *et al.* (2012) studies.

1.2 Problem Statement

Porous ceramics have a wide range of applications, ranging from the filtration and separation of liquid or gas to thermally or acoustically insulating bulk materials or coating layers. Lightweight tile is one of the porous ceramic. The preparation of lightweight tiles with controlled microstructure like porosity, pore size and pore space topology has been a subject of constant interest. Pore forming agents (PFA), Pore forming agent is a pyrolysable material which burns out during sintering. Different pore forming agents such as wheat particles, starch, PMMA, poppy seed and sawdust can be used to prepare several porous ceramics with controlled microstructure (Manap and Jais, 2009).

Novais *et al.* (2015) has been investigated previously about the possibility of incorporating wood waste (sawdust) as a pore forming agent into ceramic tiles for the production of lightweight bi-layered porcelain stoneware tiles. The results demonstrate that sawdust incorporation promotes a reduction of product weight (up to 7.5%), while maintaining suitable mechanical strength when compared to commercial stoneware tiles. Meanwhile, (García-Ten *et al.* (2012)) reported that the weight porcelain stoneware tile was decrease by 2% after incorporate size silica carbide (SiC) particles into the body formula. Based on these studies, they are more focusing on decreasing of ceramic tile weight. Nowadays, ceramic tile companies classified their tile product based on water absorption properties (ANSI Ceramic Tile Standard, 2008). Therefore, in our study, we attempted to fabricate lightweight ceramic tile with low water absorption property.

In this study, we propose a new approach for fabrication of lightweight ceramic tile by using tri-layer approach. Top and bottom layers of lightweight ceramic tile are formed by den sense layer with a density similar to conventional ceramic tile and the middle layer is formed by porous structure layer, which reduce the weight of the product and at the same time maintain suitable mechanical strength. The introduction of porosity is accomplished by incorporating pore forming agent– calcium carbonate ($CaCO_3$) into the tile formulation, then this mixture was used at middle porous structure layer. We expect that the produced tri-layered ceramic tiles are lighter than conventional ceramic tiles, thus provide thermal insulation, at the same time, decreasing their transport and distribution costs.

1.3 Objectives

The objectives of this research work are:

i. To investigate the tri-layer approach in fabricating of lightweight ceramic tile

ii. To investigate the effect of different firing temperature on the properties of tri-layer lightweight ceramic tiles.

1.4 Scopes of Work

This research work covered the study of the fabrication of tri-layer lightweight ceramic tile body incorporated with 5 wt% of calcium carbonate ($CaCO_3$) as porosity agent. Tri-layer lightweight ceramic tile made up of three different layer compositions, combining dense structure at upper and bottom layers with a porous structure at middle layer. The layer ratio specimen were denoted as T1 (35 wt% (top) :30 wt% (middle) :35 wt% (bottom)), T2 (30 wt% (top):40 wt% (middle):30 wt% (bottom)) and T3 (25 wt% (top):50 wt% (middle):25 wt% (bottom)).

Based on these ratios, the powder compacted into a button shape (weight: 24grams, diameter: 50mm). The hydraulics pressing machine was selected for the compaction with a pressure of 9 MPa and holding time of 5 seconds. The ceramic powder was placed inside the die and pressed with selected pressing parameter, then a mixture of ceramic powder and pore forming agent ($CaCO_3$) was added and pressed before the addition of the ceramic powder into the die. After the compaction, the green bodies were dried in oven for 24 hours in 100 ^{o}C to remove the excess moisture. Then, green bodies with different layer ratio were then sinter at 1050 ^{o}C and 1150 ^{o}C with total soaking time of 4 hours and heating rate of 10^{o}C per minute.

Several characterizations were done to validate raw materials. Thermogravimetric-Differential Thermal Analysis (TG-DTA), particle size analysis, X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD) and Field Emitting Scanning Electron Microscope (FESEM) were conducted to characterize the raw materials. Phase analysis of fired test piece was determined by X-Ray Diffraction (XRD) while surface morphology of fired test piece cross-sectional area was determined by Optical Microscope (OM). The bulk density moisture absorption ability, modulus of rupture (MOR), and coefficient of thermal expansion (CTE) of the fired test piece was tested as well. Figure 1.1 shows the overall process for the fabrication of the tri-layer lightweight ceramic tiles.

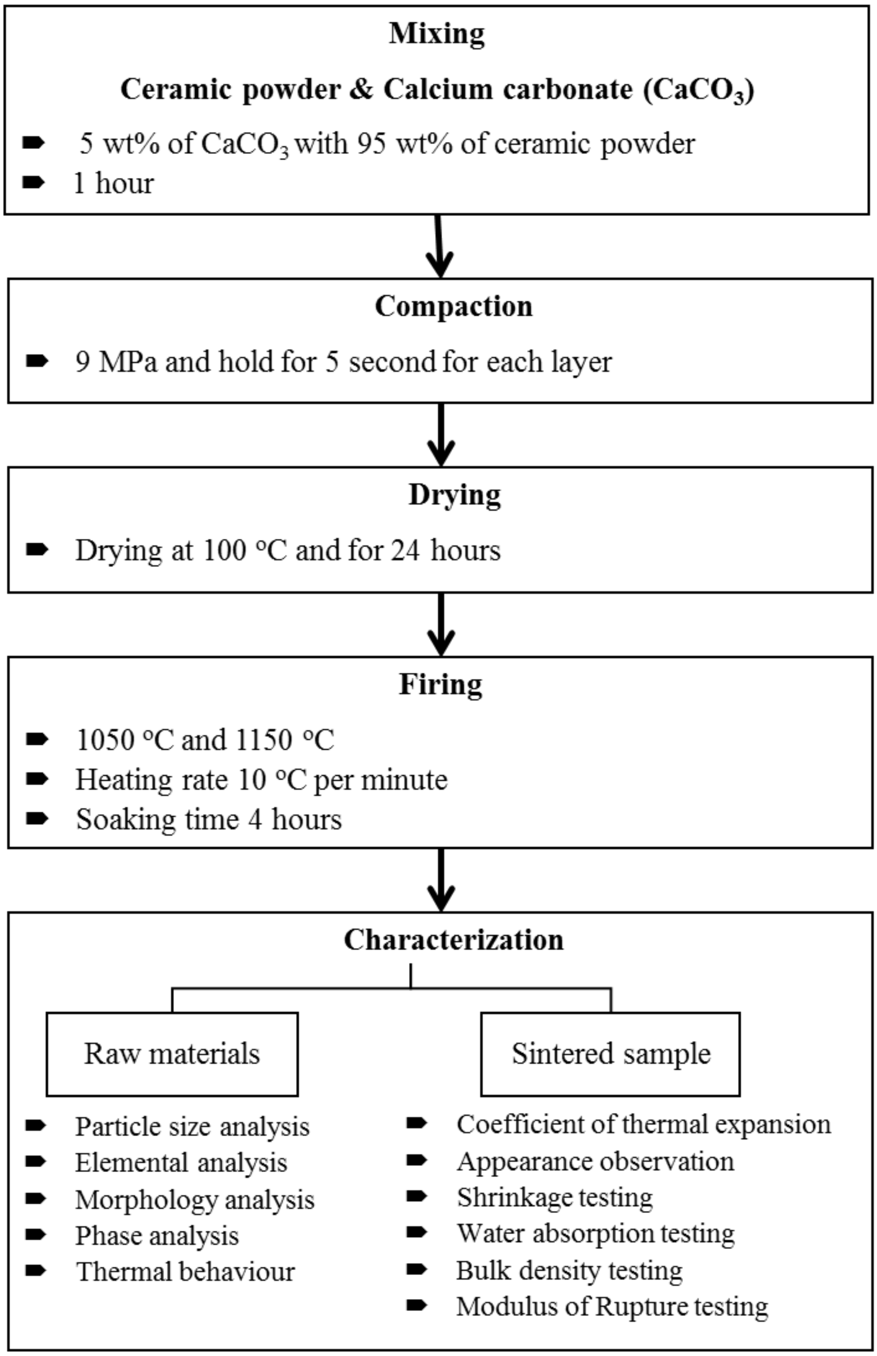


Figure 1. 1 The overall process for the fabrication of the tri-layer lightweight ceramic tiles

1.5 Outline of Chapters

This thesis consists of five chapters. Chapter One consists of introduction, objectives, research motivation, problem statement and study scopes of the project. Meanwhile, the concept, theory and literature review related to the fabrication of tri-layer lightweight ceramic tile we explained in Chapter Two. In Chapter Three, experimental details and characterization approaches are explained. Chapter Four focuses on presenting the results and discussion of this project. Finally, the conclusion of this project and recommendations for future work is stated in Chapter Five.

CHAPTER 2

LITERATURE REVIEW

2.1 Historical of ceramic tile

Ceramic tiles are thin pieces of clay or other inorganic materials, hardened by oven firing. The term of “tile” come from the French word “tuile”, which is also from the Latin word tegula, means a tile is made up of fired clay. By contrast the use of tiles goes back a very long way (Morariu, 2011). Many sources independently proved that the actual known history of clay-based tiles have been in existence as far as the fourth millennium BC (4000 BC) to Egypt. At Wittenham in Oxfordshire plenty of Roman roof tiles were discovered in 2004 revealing that large Roman buildings became a part of this important landscape when the Iron Age gave way to the Roman period some 2000 years ago (Morariu, 2011). Tile is a fabricated piece of hard-wearing material, produced by firing natural geomaterials such as clay, feldspar, quartz etc. at high temperatures.

In building, the history is closely related to the use of bricks and tiles for roof and the decoration of important features. The early Mesopotamian builders and Chinese civilisations all used terra cotta tiled roofs. Hollow pottery components in walls or dome type elements have been used to reduce dead weight for structural reasons. Earthenware pipes for drainage have been found in archaeological excavations (De Feo *et al.*, 2014). Decorative glazed tiles and porcelain were first manufactured in Europe about 15th century after learning the techniques and developments in China and the Islamic civilisations (Ward-Harvey, 2009).

During the medieval period, tile was mostly used in monasteries and palaces. The raw materials of tiles at that time were the local clays. The tile can be shaped by simple methods, such as flattening the clay and cutting pieces into shape. The only mechanical aid available during that time was a wooden mould carved in relief which indented a pattern on the clay slab. The slab would be dried and the impression filed with white pipe clay. The tiles would be trimmed flat before it is completely dried. A layer of lead ore glaze splashed on the tile surface before fired. These encaustic tiles were created from the 12th to the 16th centuries, was then disappeared until the Victorian era of the 19th century (Morariu, 2011).

In 1843, Herbert Minton developed modern tile industry after he revived the lost art of encaustic tile-making in England. The industry was further transformed in the 1840s by a method named "dust-pressing”. This method involved of compacting the almost dry clay powder by two metal dies. Dust-pressing substituted tile-making by hand with wet clay, and facilitated mechanization of the tile-making industry. Dust-pressing enabled higher productivity and reduce the cost of better quality tiles in a wide range of colours and designs (Grimmer and Konrad, 1996). In the last three decades in Egypt, ceramic tiles have established as one of the very fast growing heavy clay-based industries (Hanan, 2013). Nowadays, a very wide range of wall and floor tiles that come from many industrialised countries, as well as from local firms, including modern wall tiles for building interiors. Many ceramics are made of coarse-grained and color clays that occurred widely throughout the world where color is not a critical factor (Ward-Harvey, 2009).

2.2 Category of ceramic tile

Ceramic tiles are originated from mixtures of clay, sand and other natural materials that are formed into a mold and sintered at high temperatures, up to 1250 ℃. Ceramic tile products offered in a broad range of textures, patterns and sizes. Moreover, they are frequently used as different applications from tableware and sanitary ware to roofing tiles (Hanan, 2013). Ceramic tiles possess the characteristic like all ceramic material, for example, durable, hygienic, non-combustible, fire-resistant and easy to maintain. Water absorption is the main property to be counted when classifying ceramic tiles of any type, as the percentage indicate the open porosity of the tile and reveals the degree of vitrification (Amin *et al.*, 2017). Besides that, ceramic tiles are also classified according to their shaping or production method (either dry pressed or extruded).

In general, both porcelain and non-porcelain tile are called ceramic tile. Water absorption is the consideration when classifying the tiles. Porcelain tile possesses low water absorption (<0.5%) due to higher feldspar content. Porcelain tiles primarily consist of clay, feldspar and quartz, heat treated to form a mixture of glass and crystalline phases (Leonelli *et al.*, 2001). Porcelain endowed with high bending strength, lower water permeability (<0.5%), excellent frost and chemical resistant and optimal abrasion resistant (Novais *et al.*, 2015). These outstanding properties make porcelain suitable as material for floor and wall tiles in high demanding environments such as industries, hospital and shopping centers. They come in various sizes, colors, and shapes. Along with mosaics, they are suitable for use almost anywhere for use almost everywhere and anyplace (Bridge *et al.*, 1992).

2.2.1 Porcelain tiles

Porcelain is a vitrified product of mixtures of clay, quartz and feldspar (Martín-Márquez *et al.*, 2008). Porcelain microstructures are grain and bond type with large particles of filler (usually quartz) held together by a finer matrix, which is almost fully dense, composed of mullite crystals and a glassy phase (Lee and Rainforth, 1994). Porcelain sintered for several hours process with the aim of promoting a high mullite formation (Martín-Márquez *et al.*, 2008). Because of the complex connection between raw materials, processing paths and approaches and the kinetics of the sintering process, porcelains represent some of the most complicate ceramic systems (Carty and Senapati, 1998).

Porcelain tiles are the hardest and densest tiles available. Because the color in porcelain tile usually goes all the way through, they are non-porous, resist scratching and can resist temperature extremes. Tile manufactured for outside use has a really low water absorption, minimizing the cracking, chipping and other effects of expansion when the temperature goes down below freezing. Porcelain tile also defines as a ceramic tile that has a water absorption of 0.5% or less that is generally made by the pressed or extruded method. They do not include the materials with very little or no crystallinity such as glass tile (ANSI Ceramic Tile Standard, 2008).

Figure 2.1 Glazed and unglazed ceramic tiles (King, 2003)

2.2.2 Floor tiles

Floor tiles are specifically used on floors, but also suitable to be used on walls and countertops. This tile fabricated by either pressing, extrusion or the plastic method, from clays that made a dense body into distinctive textured appearance (ANSI Ceramic Tile Standard, 2008). Floor tiles contain a carrier layer and a superimposed floor surface, forming layer which is offered by aesthetic and wear resistant surface (Nemeth, 1980). According to the International Standard, floor tiles are classified as either having percent water absorption stipulated at 6 % to 10 % (Martín-Márquez *et al.*, 2008). There are several types of floor tile suitable for various applications, which are glazed floor tiles, quarry tile and so on.

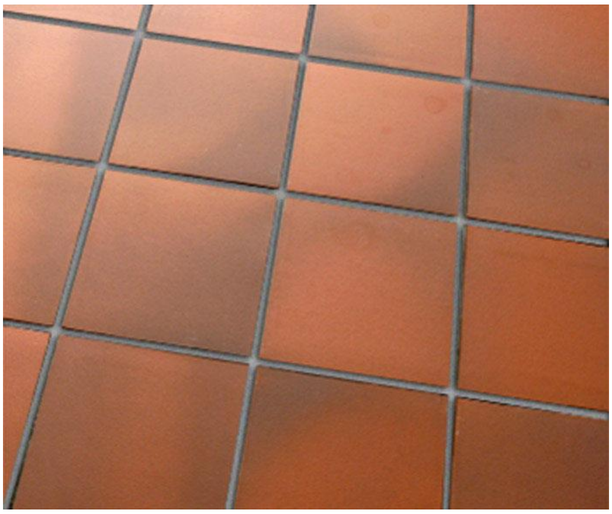
2.2.2.1 Glazed floor tiles

Glazed floor tiles are covered with glass-forming minerals and ceramic stains. Normally, these tiles have either a matte, semi-gloss or high-gloss finish and offer better stain and moisture resistance than unglazed tile. Glazed floor tiles also have different finishes. High gloss finishes can be more slippery and scratches can become more visible, while matte or textured finishes help with traction and scratches, and dirt is less visible. Glazed tile is a tile with a fused impervious facial finish composed of ceramic materials, fused to the body of the tile. This tile usually used for the floors in the living rooms as it expected to have lesser exposure to the mechanical loads and dirt. Figure 2.2 shows image of glazed floor tile.

Figure 2.2 Image of glazed floor tile (King, 2003)

2.2.2.2 Quarry tiles

Quarry tile typically made by extrusion method from clay or shale (Ritchie and Company, 1986). It is manufactured from raw material and the method is similar to the making industry of brick products. Quarry tiles are hard and durable due to the extrusion process. These tiles suitable for the outdoor application even though the quarry tiles are unglazed. Quarry tiles get the fired color from the raw material composition that created the tile. Generally, quarry tiles with shale based are red or brown in color. Fired tiles with base of clay have grey or beige color (Ritchie and Company, 1986).



(a)

(b)

Figure 2. 3 Images of quarry tile with a base of (a) shale and (b) clay (Ritchie and Company, 1986)

2.2.3 Wall tiles

Wall tiles designed to the similar visual characteristic as floor tile. This tile is either glazed or unglazed. Wall tile suitable for interior use or which is commonly non-vitreous for improved adhesion to vertical facades (ANSI Ceramic Tile Standard, 2008). Typically, wall tiles have attached spacing lugs. Thus, water absorption values are slight higher compared to porcelain and floor tiles recorded by wall tiles is with a range of 10% to 18%. These tiles do not necessary to tolerate with excessive impact, abrasion, or be subjected to freeze/thaw cycling (ANSI Ceramic Tile Standard, 2008, Byrne, 2008). Few considerations should be taken into account, especially for kitchen and bathroom.

Wall tile with resistance to the impact of weak acid, weak alkaline and stains would be selected when considering the wall tile in kitchen. The wall of bathroom exposed to water and in contact with stain and chemicals after everyday use. Therefore, the tiles should be stain and chemical resistant. In fact, the wall tiles for the bathroom should be able to be cleaned easily with common detergents (United States Tariff Commission, 1970). Figure 2.4 and Figure 2.5 display the images of wall tiles used for kitchen and bathroom respectively.

Figure 2.4 Wall tiles used for kitchen

Figure 2.5 Wall tiles used for bathroom

2.2.4 Roof tiles

Roof tiles are made from locally available materials such as terracotta or slat and designed primary to exclude rain. Roof tiles installed from the framework of roof by fixing them with nails. Generally, roof tiles are usually set up in parallel rows, with the upper row overlapping the row below it to keep out rainwater and prevent the nail that hold the row below from corrosion. Other than this, the tiles used to offer the protective weather envelope of the building. Nowadays, many shapes of roof tiles have developed such as flat tiles, interlocking roof tiles and so on.

Flat roof tile is the simplest type, which are laid in regular overlapping rows. One of the example of flat roof tile is the clay plain tile. Plain tile made from clay has been used for more than eight hundred years. This tile available at a range of colours, texture and option which meet the aesthetic and performance demands of all types of roofing applications. Interlocking roof tiles set up in side and top locking to improve protection from water and wind. This interlocking roof tile provides durability and weather protection. There are other advantages of interlocking, such as easy to install, flexible gauge, low minimum pitch and higher aesthetic value. Figure 2.6 displays the images of (a) clay plain tile and (b) interlocking roof tile.

(a)

(b)

Figure 2.6 Images of (a) clay plain tile and (b) interlocking roof tile

2.3 Ceramic Tile Body Formulation

Ceramic tiles are manufactured by sintering a mixture of kaolin, white-firing plastic clays, feldspars and sands. Sintering temperatures range between 1000 ℃ to 1020 ℃ for porous tiles, 1050 ℃ to 1200 ℃ for single-fired dense tiles (De Noni Jr *et al.*, 2010). A ceramic tile manufacturing consists of raw material of unique characteristics, which can be categorised into three main groups, which are plastic, fluxing agents and inert materials.

Plastic materials used are clay and kaolin, which key role is to provide plasticity and therefore green strength to the pressed body. The major fluxing materials used are feldspar, talc and pyrophyllite, whose main function is to generate liquid phase during sintering. In the role of inert raw materials, quartz is definitely the most common because of its availability. Moreover, quartz is used essentially to improve the dimensional stability after firing and remain inert during the firing cycle (Acchar and Dultra, 2015).

2.3.1 Clay

Santos (1989) stated that clay is a natural, earthy and fine-grained material. Clay develops some plasticity when moistened with water and composed of hydrated silicates of aluminum ($Al_2O_3.2SiO_2.H_2O$), iron (Fe) and magnesium (Mg), with small amounts of alkali and alkaline earth elements. There is considerable variation in the terminology of clay minerals in various scientific and technological fields that use this material (Acchar and Dultra, 2015). As discussed before, plasticity is the key purpose of clays for ceramic tile. Based on Santos (1989), plasticity in clays formed as result of attraction forces between clay particles and the lubricating action of water between anisometric lamellar particles. It could be expected that plasticity grows when the clay has sufficient water to cover the entire surface with a film that behave like lubricant, enable the sliding of the planes on each other during application of shear stress. Water molecules confined to the surface of the clay minerals through hydrogen bonds to combine clay particles to each other in the wet clay form (Acchar and Dultra, 2015). This gives rise to various mechanical ability of the green body.

2.3.2 Kaolin

The kaolin was derives from Kau-ling (a place in China) as Kau-ling is the location from which the first samples were extracted. Kaolin is mostly composed of kaolinite. Kaolinite is a clay mineral with chemical formula is $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$ and has extremely refractory behavior after sintering (Acchar and Dultra, 2015). In order to obtain light color during sintering, kaolinite clays are preferably used.

Kaolin used in most of the production ceramic tile in order to lessen the content of impurities and increase the proportion of kaolinite. Significantly, the key feature of kaolin for use in production of ceramic tiles is due to its whiteness. Kaolinite is a major supplier of aluminum oxide (Al_2O_3), which is a regulator of the equilibrium of reactions during the glazing process. Furthermore, alumina involved in the formation of glassy phase when associate with alkaline flux or also found for the most part at the end of the sintering process formed mullite ($3Al2O_3 \cdot 2SiO_2$). As a result of its needle morphology, alumina acts as “skeleton” of products promoting the increment of the mechanical strength. The light color of the porcelain tile samples is desired due to the reaction of Fe_2O_3 and TiO_2 with pigments and dyes cause deviations in color of glazed specimens. Based on Moraes (2007) study, kaolin must have kaolinite content at range of 75-85 %. However, minerals that affect the firing color such as hematite (Fe_2O_3) content need to be less than 0.9 %. As a result, the whiteness index of the ceramic tile after firing stay between 85 and 92%.

2.3.3 Feldspar

Biffi (2002) stated that feldspars are silicon aluminates of alkali and alkaline earth metals. Feldspars are important ceramic tile production. In fact, feldspar provide mechanical ability through high vitrification at the end of the sintering process.

The addition of fluxing materials in manufacturing of ceramic tile is to achieve certain water absorption and mechanical strength values with control open porosity and reduce closed porosity after sintering. The use of flux enable liquid phase to form before reaching final firing temperature. The liquid phase formed during firing has certain quantity and viscosity to enable the filling of the pores exist, at the same time, enhance the reactivity between elements, so that the onset of sintering happens at lower temperatures. The basic element for the fluxing properties is the content of alkali in the minerals. Theoretically, K_2O and Na_2O, respectively, in potassium and sodium feldspars are approximately 16.9 and 11.8 % (Acchar and Dultra, 2015).

2.3.4 Pyrophyllite

Ceramic pyrophyllite or only pyrophyllite, is a very fine metasedimentary rock mainly consists of sericite, kaolinite and quartz, with alkali content of aproximately 7 %, providing it fluxing characteristics (Acchar and Dultra, 2015). Attributable to its mineralogical and chemical nature, pyrophyllite presents no plastic and plastic properties (Motta *et al.*, 1998).

2.3.5 Talc

Talc is a magnesium silicate with a chemical formula of $3MgO \cdot 4SiO_2 \cdot H_2O$ used in varying amounts up to 4 %. The addition of talc is to increase the fusibility because of the formation of eutectic between talc and feldspar, improving the resistance to staining and flexural modulus. The use of talc led to some improvement such as resistance to stains, flexural modulus, lowers the thermal expansion coefficient and increased whiteness when in the presence of zirconium (Acchar and Dultra, 2015).

2.3.6 Quartz

Quartz (SiO_2, with crystalline structure), also known as sand is added with the purpose of maintaining a siliceous skeleton because when the temperature increased, the other components such as clay, kaolin, and feldspars soften. Furthermore, it is a critical component of correcting the ratio between SiO_2 and Al_2O_3 during formation of mullite ($3Al_2O_3 \cdot 2SiO_2$).

Quartz is also one of the component in the mineralogical composition in other raw materials, such as clays, kaolin, and feldspars. Therefore, some attention needed to be given during the allotropic change that happens when heated up to 573 ^{o}C. α-quartz with rhombohedral structure transform to β-quartz with hexagonal structure, and during cooling at the same temperature and the inverse transformation arises. Expansions and contractions occur during the allotropic transformations. This is due to changes in the crystalline structure of quartz that leading in internal defects such as microcracks.

2.4 Properties of ceramic tile

Ceramic tiles is a ceramic surfacing unit, usually relatively thin in relation to facial area, having either glazed or unglazed face and fired above red heat in the course of manufacture to a temperature sufficiently high to produce specific physical properties and characteristics (ANSI Ceramic Tile Standard, 2008). They possess a broad range of properties, and certain tiles are better suited for some installations than others. Consequently, precise knowledge of the properties is critical for the consumer to get the desired and anticipated value of the tile (ANSI Ceramic Tile Standard, 2008).

The porcelain tile is a ceramic product of high performance made from a triaxial mixture: clay or kaolin, quartz and feldspar. The clay fraction helps forming providing plasticity and dry mechanical strength during processing and forming mullite and vitreous phase during firing. The feldspars develop a liquid phase at low temperatures and assist the sintering process, allowing a virtually zero (<0.5%) open porosity and a low level of closed porosity (<10%). The quartz promotes thermal and dimensional stability thanks to its high melting point (Leonelli *et al.*, 2001). Figure 2.7 is a diagram that represents compositions of triaxial ceramic products (Norton, 1974).

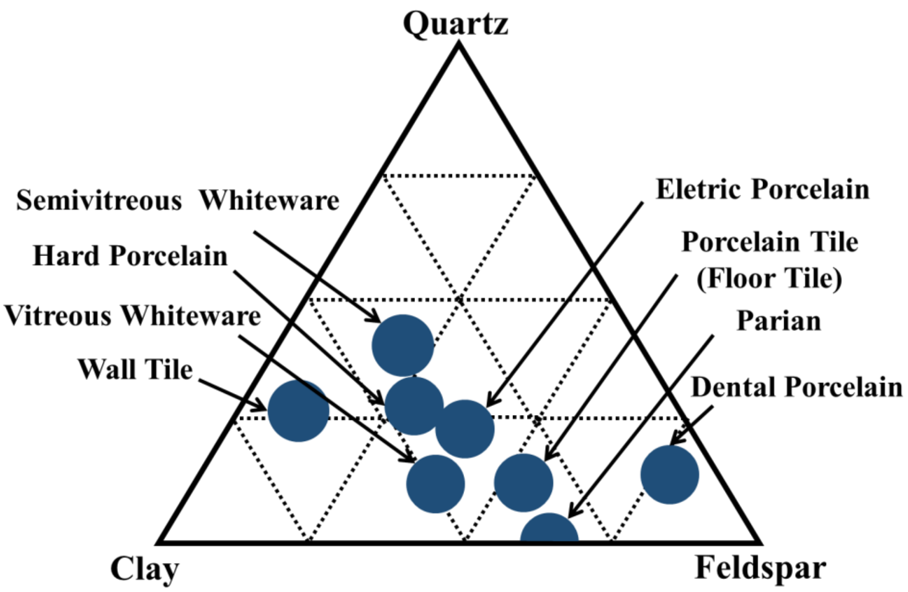


Figure 2.7 Compositions diagram of triaxial ceramic products (Norton, 1974).

2.4.1 Water absorption

Water absorption is a critical test is to examine the ability of a sintered tile to absorb water or moisture. The test reveal the porosity of the tile or how likely water is to infiltrate into the body. The more water that penetrate into the body, higher possibility of the tile to crack if the temperature drops below freezing when the tile soaked with water. It does not matter for indoor installation, but for outdoor installation. This is because most of the tile installations are constructed around or near water. Porous ceramic materials can help absorb moisture and unwanted organisms. Therefore, water absorption is one of the key properties, as in wet-area applications, it can include health and safety matters, while in exterior applications, it can introduce substantial freeze or thaw damage (ANSI Ceramic Tile Standard, 2008).

2.4.2 Visual abrasion resistance

A measure of the resistance of tile surfaces to visible surface abrasion. Final selection for all applications should take into account expected traffic, conditions, and maintenance. Final selection should also consider whether there is exposure to water (especially standing water), oil, grease, etc., which create slippery conditions. Floor applications with exposure to these elements require extra caution in product selection (Byrne, 2008).

2.4.3 Aesthetic class

Tiles may vary in color, texture, or appearance according to the manufacturer's design for that particular tile series or product line. Manufacturer may communicate the aesthetic characteristics of a particular tile product according to the aesthetic class designation (ANSI Ceramic Tile Standard, 2008).

2.4.4 Deep abrasion resistance

Deep abrasion resistance is measure of the resistance to wear of an unglazed tile intended for floor covering. (Byrne, 2008). Porcelain Enamel Institute (PEI) test is a measurement of the deep abrasion resistance of a tile, meaning how much "rubbing" it takes to physically impact the look of the tile.

2.4.5 Chemical resistance

All type of ceramic tiles are subject to chemical attack due to cleaning process, environment pollution and products spills on flooring. The chemicals comprising this class include the most popular cleaning solutions and materials used for residential and light commercial tile installations. Chemical aggression lead to loss of gloss, change in