IMPROVEMENT ON MECHANICAL PROPERTIES SODIUM FELDSPAR FOR PORCELAINS TABLEWARE

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Porcelain is one of the ceramic materials. In this study, Claytan porcelain was used as a main raw material and added with sodium feldspar as flux. The scope of this project is mainly focused at the effect of sintering temperature and the different process of weight percentage sodium feldspar to Claytan porcelain. Slurry of Claytan porcelain mixed was prepared by agitator mixer. The sodium feldspar mixed with the Claytan porcelain according to the weight percentage of sodium feldspar to Claytan porcelain which is at 2wt. %, 4wt. %, 6wt. %, 8wt. % and 10wt. %. Slip casting was used as the processing method. Sintering process was conducted at four different temperatures 800°C, 900°C, 1000°C and 1100°C and was fixed for 1 hour and heating and cooling rate were at 10°C/minute. DTA/TG was used in order to establish the firing condition. Physical analyses were divided into three analyses which were linear shrinkage, porosity and bulk density of the samples. It was carried out according to the specific ASTM standard of testing. The mechanical behavior was evaluated in term of modulus of rupture and micro hardness. After viewing the results, it can be concluded that percentage of porosity significantly reduces from 30.16% to 22.83% when sintered at 800°C and 1100°C for sodium feldspar 10wt. %. Claytan porcelain with sodium feldspar 2wt. % at 1100°C exhibited the highest strength of 17.59Mpa and micro hardness value 150.40Hv. The effect of firing temperature significantly improved the porosity and the quality of physical properties of Claytan porcelain. For physical and mechanical analyses, the results showed that the increasing of temperature and percentage of sodium feldspar had significantly increased the linear shrinkage, apparent porosity and bulk density.
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<td>DTA</td>
<td>Differential Thermal Analysis</td>
</tr>
<tr>
<td>TGA</td>
<td>Thermogravimetric</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscopy</td>
</tr>
<tr>
<td>wt .%</td>
<td>weight percentage</td>
</tr>
<tr>
<td>Mpa</td>
<td>Mega pascal</td>
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<tr>
<td>µm</td>
<td>Micrometer</td>
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CHAPTER 1

INTRODUCTION

1.1 Research Background

Ceramic is a material that is generally hard and brittle. Ceramic research is intended to reduce problems and increase the strength of ceramic materials. Ceramics materials are divided into three categories which are traditional ceramics, engineering ceramics and glasses [1]. Ceramic materials used for engineering applications and can be divided into two groups of traditional, which are ceramic material and the engineering ceramic material [2]. Traditional ceramics are made from three basic components which are clay, silica and feldspar [2] [3].

Porcelain bodies contain 40-60% of glass [4]. The varying composition of glassy phase greatly influences mechanical properties, translucency and firing temperature of the final ceramic product. Despite the melting temperature of at least 1100°C, of the low melting flux present in the body, glass formation begins at a temperature lower than 1000°C. The mechanical properties were evaluated in terms of strength testing and hardness.

There are three types of sintering with different densification mechanisms. Solid phase sintering for all components remains solid throughout the sintering. The densification is carried out by a change in shape of the grains. Mass transport occurs by volume and grain boundary diffusion. Liquid phase sintering, formation of a viscous liquid usually and eutectic with a low melting point that fills the pore spaces of the initial green body for porcelain.
1.2 Problem Statement

Manufacturing of porcelain required high cost due to firing process. The higher temperature gives problem to porcelain manufacturing because of its cost is higher. The sintering temperature is important to improve mechanical properties. Moreover, if the temperature is reduced it can also reduce the cost.

The study will focus on parameters of sodium feldspar and sintering of different samples according to temperature and microstructures of porcelain. Therefore, the analysis of sintering temperature on thermal analysis, physical properties and microstructures will be investigated.

1.3 Objective of Study

The objectives of this study are listed as follows:

i. To develop porcelains tableware using Claytan porcelain with additive of feldspar.

ii. To establish the optimum sintering temperature.

iii. To characterize the structural, physical and mechanical properties of Claytan porcelain.

1.4 Scope and limitations of the research

The scopes of this research are as below:

1. To produce Claytan porcelain using slip casting technique with difference percentage of sodium feldspar (0wt. %, 2wt. %, 4wt. %, 6wt. %, 8wt. % and 10wt.%).

2. The sintering temperature have been used at 800°C, 900°C, 1000°C and 1100°C.
3. To analyze the structure, physical properties and mechanical properties of the material with using:
   
   i. Thermogravimetric analysis method (TGA) to analyze the thermal stability of the sample before sintering process.
   
   ii. Scanning electron microscope (SEM) method to analyze the microstructure of samples.
   
   iii. Physical analysis to determine the linear shrinkage, porosity and bulk density.
   
   iv. Mechanical properties testing using 3 point bending and Vickers hardness testing.
CHAPTER 2

LITERATURE REVIEW

2.1 Ceramic

Ceramics can be defined as a solid heat resistant, non metals and inorganic generally consists of compounds formed from elements of metal and non-metal. Ceramic gets its name from the word keramos Greek, means "pottery", which in turn is derived from the Sanskrit root that older, means "to burn". The Greeks used the term means "burnt stuff" or "burned earth". The old term has been including all products made of clay fired, for example bricks, fireclay refractories, sanitary ware and tableware [5]. In general, ceramic is a hard and brittle material. Ceramic materials are divided into three categories of traditional ceramics, engineering ceramics and glasses [6]. The white wares, consisting of table or decorative ware, wall tiles and sanitary wares as products from ceramic [7].

A ceramic material or product defined as one composed of inorganic but non-metallic material. On this basic, ceramic products are usually can be divided into four categories with as their structural products such as bricks, roofing tiles, pipes or floor tiles [7]. Some examples of ceramic products are shown in Figure 2.1.
2.1.1 Traditional Ceramics

Three basic components in traditional ceramic are made up of clay, silica or stone of fire, and feldspar [8]. Traditional ceramics are usually based on clay and silica [3]. Clay in traditional ceramic material as workability before it shots and provides harden and it is the main body material [6].

Traditional ceramic consists of at least three components for optimum processing, and hence the performance of the final products such as kaolin or kaolinite clay for plasticity, feldspar for fluxing and silica as filler for the structure. Composition triaxial porcelain has a composition (SiO₂. Al₂O₃. KNaO). Sodium feldspar has 25 wt. % of plastic component, 25 wt. % silica and 50 wt. % feldspar of sodium feldspar [8]. For soft porcelain, it has 50 wt. % of clay, 25 wt. % silica and 25 wt. % feldspar of potassium feldspar for hard porcelain [8].

Traditional ceramics refers to ceramic which are produced from unrefined clay and combinations of refined clay and powdered or granulated nonplastic minerals. Clay content of traditional ceramics are used around exceeds 20% [9]. The general classifications of traditional ceramics are as described below and Table 2.1 shown the traditional ceramic products.

i. Pottery is sometimes used as a generic term for ceramics that contain clay and are not used for structural, technical, or refractory purposes.

ii. Whiteware refers to ceramic ware that is white, ivory, or light gray in color after firing.
Table 2.1: Traditional ceramic products [1]

<table>
<thead>
<tr>
<th>Product</th>
<th>Principal Chemistry</th>
<th>Minerals and Raw Materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pottery, tableware</td>
<td>Al₂Si₂O₅(OH)₄, SiO₂, KAlSi₃O₈</td>
<td>Clay + silica + feldspar</td>
</tr>
<tr>
<td>Porcelain</td>
<td>Al₂Si₂O₅(OH)₄, SiO₂, KAlSi₃O₈</td>
<td>Clay + silica + feldspar</td>
</tr>
<tr>
<td>Brick, tile</td>
<td>Al₂Si₂O₅(OH)₄, SiO₂ plus fine stones</td>
<td>Clay + silica + other</td>
</tr>
<tr>
<td>Refractory</td>
<td>Al₂O₃, SiO₂ Others: MgO, CaO</td>
<td>Alumina and silica</td>
</tr>
<tr>
<td>Abrasive: silicon carbide</td>
<td>SiC</td>
<td>Silica + coke</td>
</tr>
<tr>
<td>Abrasive: aluminum oxide</td>
<td>Al₂O₃</td>
<td>Bauxite or alumina</td>
</tr>
</tbody>
</table>

The non crystalline solids are lack of systematic and regular arrangement of atoms within a relatively large. Sometimes these materials are also called as amorphous (meaning literally 'without Form'), or liquid atomic structure of super cooled liquid, because they resemble to liquid [10]. Amorphous state is described by a comparison of crystalline and non-crystalline structure of the ceramic compound of silicon dioxide (SiO₂), which exists in two states. In Figure 2.2, it showed two schematic diagrams for both SiO₂ structure. While every structure bond to silicon ion three oxygen ions to the two states, beyond this, the structure was more disordered and irregular for non-crystalline structure.

Figure 2.2: The comparison of the crystalline and noncrystalline structures of ceramic compound silicon dioxide (SiO₂)[10].
2.1.1.1 Clays

Clays are consisted mainly of hydrated aluminum silicates \((\text{Al}_2\text{O}_3, \text{SiO}_2, \text{H}_2\text{O})\) with small amounts of other mineral oxides such as \(\text{TiO}_2, \text{Fe}_2\text{O}_3, \text{MgO}, \text{CaO}, \text{Na}_2\text{O}\) and \(\text{K}_2\text{O}\) [5]. Table 2.2 lists the chemical compositions of several industrial clays.

Table 2.2: Chemical compositions of some clay [9]

<table>
<thead>
<tr>
<th>Type of Clay</th>
<th>Weight percentage of major oxides</th>
<th>Ignition loss</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(\text{Al}_2\text{O}_3)</td>
<td>(\text{SiO}_2)</td>
</tr>
<tr>
<td>Kaolin</td>
<td>37.4</td>
<td>45.5</td>
</tr>
<tr>
<td>Tenn. ball clay</td>
<td>30.9</td>
<td>54.0</td>
</tr>
<tr>
<td>Ky. ball clay</td>
<td>32.0</td>
<td>51.7</td>
</tr>
</tbody>
</table>

2.1.1.2 Feldspar

Feldspar refers to any of several crystalline minerals that consist of aluminum silicate combined with either potassium, sodium, calcium or barium[1]. Feldspar are used as fluxes and not only as the ceramic body compositions, but also in glazes, glass and porcelain enamels[7]. The potassium blend, as for example, has the chemical composition \((\text{KA}_1\text{Si}_3\text{O}_8)\). Mixtures of clay, silica, and feldspar are used to make stoneware, china, and other tableware[1]. Table 2.3 shows several types of feldspar and principle chemistry.

Table 2.3: Types of feldspar [7]

<table>
<thead>
<tr>
<th>Feldspar</th>
<th>Principle chemistry</th>
</tr>
</thead>
<tbody>
<tr>
<td>Potash feldspar/ orthoclase</td>
<td>(\text{K}_2\text{O}, \text{Al}_2\text{O}_3, 6\text{SiO}_2)</td>
</tr>
<tr>
<td>Soda feldspar/ albite</td>
<td>(\text{Na}_2\text{O}, \text{Al}_2\text{O}_3, 6\text{SiO}_2)</td>
</tr>
<tr>
<td>Lime feldspar/ anorthite</td>
<td>(\text{CaO}, \text{Al}_2\text{O}_3, 2\text{SiO}_2)</td>
</tr>
<tr>
<td>Barium feldspar/celsian</td>
<td>(\text{BaO}, \text{Al}_2\text{O}_3, 2\text{SiO}_2)</td>
</tr>
</tbody>
</table>
The quality of feldspar is established by the amount composition of orthoclase and albite, respectively in the potassium and sodium feldspar and the most commonly used fluxes in porcelain [8].

Feldspar acts as a flux, forming a viscous liquid at firing temperature and aids in vitrification [11]. Sodium feldspar and potassium feldspar are homogeneously. Feldspar is one of the minerals often used and refers to any several flux [11] to melting the temperature [12]. The amount of the liquid phase increases with increase in firing temperature due to the melting of feldspar. Generally high sodium feldspar will give lower vitrification temperatures in a body than the potassium feldspar [7].

Feldspars are alumino silicates that contain sodium (Na), potassium (K), or calcium (Ca). They range in composition from NaAlSi₃O₈ and KAlSi₃O₈ to CaAl₂Si₂O₈. Feldspars act as fluxing agents to reduce the melting temperatures of the alumina silicate phases [10]. The composition of the raw materials and its significance are given in Table 2.4.

Table 2.4: Composition of raw material used for macro-porous ceramic support [13]

<table>
<thead>
<tr>
<th>Raw materials</th>
<th>Composition (wt%)</th>
<th>Significance</th>
</tr>
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<tbody>
<tr>
<td>Kaolin (Al₂(Si₂O₅)(OH)₄)</td>
<td>14.45</td>
<td>Low plasticity and high-refractory property</td>
</tr>
<tr>
<td>Ball clay (3SiO₂Al₂O₃)</td>
<td>17.58</td>
<td>Provides plasticity and strength to the green support</td>
</tr>
<tr>
<td>Feldspar (Plagioclase) ((Na, Ca)(AlSi₃O₈))</td>
<td>05.60</td>
<td>Acts as a flux to form glassy phase at low temperature</td>
</tr>
<tr>
<td>Quartz (SiO₂)</td>
<td>26.59</td>
<td>Increase mechanical and thermal stability</td>
</tr>
<tr>
<td>Calcium carbonate (CaCO₃)</td>
<td>17.14</td>
<td>Pore forming agent</td>
</tr>
<tr>
<td>Pyrophyllite (Al₂(Si₂O₅)(OH)₂)</td>
<td>14.73</td>
<td>To reduce crazing</td>
</tr>
</tbody>
</table>
2.1.1.3 Silica

Silica is another major raw material for the traditional ceramics [10]. It is the principal component in glass, and an important ingredient in other ceramic products including white ware, refractories, and abrasives. Silica is available naturally in various forms, and the most important of which is quartz. The main source of quartz is sandstone. The abundance of sandstone and its relative ease of processing mean that silica is low in cost it is also hard and chemically stable. These features account for its widespread use in ceramic products. It is generally mixed in various proportions with clays and other minerals to achieve the appropriate characteristics in the final product.

2.1.2 Porcelains

Porcelain with excellent technical characteristic was produced which had similar properties to a traditional porcelain [14]. Porcelains typically have a triaxial composition comprised of 50 wt. % clay, 25 wt. % flux and 25 wt. % filler [15][16].

The microstructure of porcelain is difference because the firing process [12] affects the ratio of crystalline to amorphous phases in the end products[15]. Major phase in porcelain are mullite and glass. The clay content when wet acts as a binder for the other body ingredients in the green state. In porcelain composition, clay serves as a dual purpose of providing fine particle size and good plasticity for forming [1]. Fired bodies containing these three components which result in a grain and bond microstructure. The typical porcelain stoneware composition the (Na$_2$O, K$_2$O)-Al$_2$O$_3$-SiO$_2$ phase diagram as shown in Figure 2.3.
Porcelain is a material produced from kaoline, quartz and potassium feldspar. Recently, the research of new materials, for example non-hazardous wastes, are able to replace traditional fluxing agents without changing the process or quality of the final products has been realized [4].

Porcelain and porcelain stoneware have a difference microstructure because of their the firing process [15]. Firing process for porcelain stoneware had a standard temperature between 1200 °C and 1300 °C[15]. Porosity has also shown to affect mechanical properties.

The effect of microstructure on mechanical of the end products have been correlated with their physical and microstructural features properties is not well established on firing in porcelain stoneware and to establish the influence of microstructure, taking into consideration percentage, shape and size of mullite crystals [15].

2.2 Sintering

Sintering is the heat treatment process that sinters the ceramic material and it is performed in a furnace called a kiln. In sintering, bonds are developed between the
ceramic grains, and this is accompanied by densification and reduction of porosity. Therefore, shrinkage occurs in the polycrystalline material in addition to the shrinkage that has already occurred in drying. Sintering in ceramics is basically the same mechanism as in powder metallurgy. In the firing of traditional ceramics, certain chemical reactions between the components in the mixture may also take place, and a glassy phase also forms among the crystal that acts as a binder. Both of these phenomena depend on the chemical composition of the ceramic material and the firing temperatures used.

Sintering is the firing process that applied to green ceramics to increase its strength the firing temperature depends on the composition and desired properties of the finished piece. During the firing operation, the density is further increased a body is usually fired at a temperature between 900°C and 1400°C (with an attendant decrease in porosity) and the mechanical strength is enhanced [10].

Sintering is a process of consolidation of particles under the temperatures below the melting point (1125-1200°C) and it caused, mostly by solid-state reactions. Sintering during firing forms solid bonds between particles. Standard porcelain stoneware body was fired between 1200°C and 1300°C [15] using a fast firing process. These temperatures depend on the material, but they will always be below the melting point of the ceramic. During the sintering process pores in the ceramic will close up as seen in the Figure 2.4.

![Figure 2.4: Pore effect during the sintering process [10]](image-url)
The amount of the liquid phase increases with increase in firing temperature due to the melting of feldspar. Silica discarded from clay relict during mullite formation at less than 1000°C and partial quartz dissolution at commercial firing temperatures and it also contribute to the glassy phase [17]. The composition of glass close to quartz grains and within clay relicts is highly silica-rich. The alkali content is sometimes higher in the matrix but heterogeneous in general due to inhomogeneous mixing of the initial body ingredients and inhomogeneous crystallization in various micro-regions. The variation in the size of mullite crystals observed in various micro-regions of the body is related to the viscosity of the constituent glass.

Figure 2.5 shown micrograph of the microstructure of electrical insulator porcelain. As observed in this micrograph, the structure is very heterogeneous. Large quartz grains are surrounded by a solution rim of high-silica glass. Mullite needles that cross feldspar relicts and fine mullite-glass mixtures are presented [18].

![Electron micrograph of an electrical insulator porcelain](image)

Figure 2.5: Electron micrograph of an electrical insulator porcelain [2]

Sintering involves the densification and microstructure development that transforms the loosely bound particles in powder compact into a dense, cohesive body. The end-use properties of a finished ceramic are largely dependent on the degree of densification achieved during sintering, and on the microstructure produce,
consequently, sintering is one of the most critical steps in ceramic processing. The changes that occur in the structure of triaxial bodies during firing are not completely understood due to their complexity. Table 2.5 is an approximate summary of what probably occurs during the firing of a whiteware body [19].

Table 2.5: Life history of triaxial body [19]

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Reactions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Up to 100</td>
<td>Loss of moisture</td>
</tr>
<tr>
<td>100-200</td>
<td>Removal of absorbed water</td>
</tr>
<tr>
<td>450</td>
<td>Dehydroxylation</td>
</tr>
<tr>
<td>500</td>
<td>Oxidation of organic matter</td>
</tr>
<tr>
<td>573</td>
<td>Quartz inversion to high form. Little overall volume damage</td>
</tr>
<tr>
<td>980</td>
<td>Spinel forms from clay. Start of shrinkage</td>
</tr>
<tr>
<td>1000</td>
<td>Primary mullite forms</td>
</tr>
<tr>
<td>1050-1100</td>
<td>Glass forms from feldspar, mullite grows, shrinkage continues</td>
</tr>
<tr>
<td>1200</td>
<td>More glass, mullite grows, pores closing, some quartz solution</td>
</tr>
<tr>
<td>1250</td>
<td>Glass 60%, mullite 19%, quartz 19%, pores at minimum</td>
</tr>
</tbody>
</table>

This behavior is consistent with that found by Kitouni and Harabi [11], they showed that the brick structure formed at lower temperatures 840 °C until 960 °C remained essentially the same until temperatures of over 1080 °C are reached. Based on Figure 2.6 the porosity of brick showed an increment of 1.4% and 0.1% from 800 °C to 900 °C and 900 °C to 1000 °C, respectively. The increasing in porosity was the result of diffusion at relatively low temperature without significant shrinkage.
The shrinkage value for temperature 800°C, 900°C and 1000°C is 0.31%, 0.50% and 1.04%, respectively. The surface also looks rough and a bit dusty. The bricks that were sintered until 1000°C are considered as having a porous structure since their water absorption rates are higher than 25%, as shown in Figure 2.7.

Figure 2.6: The effect of firing temperature of the clay on the porosity [20]

Figure 2.7: Effect of firing temperature of the clay on the water absorption [20]
2.3 Slip Casting Process

Slip casting is one of the traditional techniques to make a pottery [3] and widely used [2]. This technique uses aqueous slurry, also known as slip, of ceramic powder. The slip is poured into a plaster of Paris (CaSO$_4$·2H$_2$O) mold [12]. As the water from slurry begins to move out by capillary action, a thick mass builds along the mold wall. When sufficient product thickness is built, the rest of the slurry is poured out (drain casting). It is also possible to continue to pour more slurry in to form a solid piece (solid casting) [2]. The schematic diagram of slip casting process as presented in Figure 2.8. The mold is then opened and the part is removed.

The steps in slip casting are (1) slip is poured into mold cavity, (2) water is absorbed into plaster mold to form a firm layer, (3) excess slip is poured out, and (4) part is removed from mold and trimmed. It is used to make tea pots, vases, art objects, and other hollow-ware products. In solid casting, it used to produce solid products and an adequate time is allowed for the entire body to become firm. The mold must be periodically resupplied with additional slip to account for shrinkage because of absorbed water.

![Schematic diagram of slip casting process](image)

Figure 2.8: Schematic diagram of slip casting process [1]

2.4 Drying

Water plays an important role in most of the traditional ceramics shaping processes. Thereafter, it serves no purpose and must be removed from the body of the clay piece before firing. Shrinkage is a problem during this step in the processing sequence
because the water contributes volume to the piece, and when it is removed, the volume is reduced and the effect can be seen as in Figure 2.9. As water is initially added to dry clay, it simply replaces the air in the pores between ceramic gains, and there is no volumetric change. Increasing the water content above a certain point causes the grains to become separated and the volume to grow, and it resulting in wet clay that has plasticity and formability. As more water is added, the mixture eventually becomes a liquid suspension of clay particles in water.

![Diagram of Volume of Clay as a Function of Water Content]

Figure 2.9: Volume of clay as a function of water content [1]

The reverse of this process occurs in drying. As water is removed from the wet clay, the volume of the piece shrinks. The drying process occurs in two stages, as depicted in Figure 2.10. In the first stage, the rate of drying is rapid and constant, as water is evaporated from the surface of the clay into the surrounding air and water from the interior migrates by capillary action to the surface to replace it. It is during this stage that shrinkage occurs with the associated risk of warping and cracking owing to variations in drying in different sections of the piece. In the second stage of drying, the moisture content has been reduced to where the ceramic grains are in contact, and little or no further shrinkage occurs. The drying process slowing and this is can be seen in the decreasing rate in the plot.
2.5 Surface Morphology Diagram

A SEM is microscope that works with electrons or particles with a negative charge instead of light. These electrons are liberated by a field emission source. The surface of the specimen examined scanned by a beam of electrons, and reflect or back scattered electrons is collected, it is displayed in the scan rate at cathode ray tube or television screen scanned. The image on the screen, which may be pictures, represent the characteristics of the surface of the specimen. The surface must be electrically conductive and coating very thin layer metal surfaces must be used to nonconductive materials.

The Electron Backscattered Diffraction system uses a fluorescent screen to capture the diffracted electrons from crystalline samples. The pattern of diffraction characteristics of crystal structure and orientation of the sample region generated with the help of a very sophisticated software and a variety of crystallographic databases, so that the Crystal structure can be identified and oriented.
2.5.1 Analysis of microstructure

Therefore, the present investigation was devoted to the study the effect of sintering conditions on the main properties of recycles glass materials. Furthermore, Nur Shafiza et al.,[21] investigated that the effect microstructure properties of sintering conditions at temperature 1200°C for 1, 2 and 3hour with both heating and cooling rates being fixed at 5°C /min. The sample sintered at 1200°C for 2hour shows clear grain and dense microstructure. Besides, the sample sintered at 1200°C for 3hour showed melting grains, happens due the loss of Pb O.

Moreover, Nirut et al.,[22] studied that the microstructural shown as in Figure 2.11 by SEM images of surface micrograph of specimen sintered at temperature 1850°C with difference soaking time of 0.5 and 6h. A morphological analysis of the specimen 0.5h specimen has a small size distribution, uniform microstructure and some amount of porosity. The effect of microstructure specimen had shown distribution with large grains dispersed in small grain matrix due to the longer soaking time.

![SEM micrograph of specimen sintered with difference soaking times](image)

(a) 0.5h  
(b) 6h

Figure 2.11: SEM micrograph of specimen sintered with difference soaking times [22]

From the study done by Froberg et al.,[23] it was reported that he used four difference soaking times 0h, 1h, 4h and 24h for temperature ranges at 1200 –1250°C. The effect of soaking period on the phase composition and topography of raw glazes
the main crystalline phases identified by SEM-EDX in the surfaces and the roughness values for the surfaces. Therefore, the glass composition, sintering time and addition of filler are important parameters in the controlling of crystallization behavior or sintered glass ceramic.

The grains size with the small size distribution, uniform microstructure and some amount of porosity. The porosity and small size at the maximum sintering temperature therefore its density is lower than that of 6 hour. The mechanical properties of specimens decreased when the soaking time for 6 hour [22]. From the previous study, sintering temperature and time gives huge effects on performance and properties of porcelain produced.

Luz and Ribeiro, [24] pointed that the use of glass powder waste as little additives as feldspar can produce mechanical and physical properties of ceramic tile. The effect of waste glass powder is sufficient for flux agent in the mixture for ceramic tiles porcelain pottery by accelerating the process of compaction.

According to Vorrada et al., [25] Figure 2.12 showed the SEM micrographs of the fired clay bricks with different glass contents. From the figure, glass phases were distributed uniformly in the clay bodies. Increasing glass content caused an increase in the glass phases in the bricks and the crystalline phases identified in the bricks containing 0–30 wt.% glasses fired at 1100°C.

Figure 2.12: SEM micrographs of clay bricks and recycles glass sintered at 1100 °C [25]
Johari et al. [20] studied that the microstructure of the fracture surface of the clay bricks sintered from 800°C to 1250°C for 1 hour. The microstructure changes with the sintering temperature. Figure 2.13 shown SEM micrographs for the clay fired at different temperatures 800°C and 900°C and the brick has not yet experienced full solid state sintering process since the individual clay particles are still existent.

![SEM micrographs for the clay fired at 800°C to 1100°C][20]

The sintering process reaches the optimum temperature at 1200°C, whereby its microstructure contains minimum pores with porosity value 14.2% and produces the highest strength of 89.5 MPa, as shown in Figure 2.13. However, at 1250°C, the microstructure showed larger pore sizes and lower porosity value which is 5.87% with brittle, fractured behaviour. The brick becomes more brittle due to a larger portion of glassy phase in the microstructure. Therefore, the strength of the sample becomes lower (83 MPa). Even though the porosity value is lower than the brick sintered at 1200°C, this only effect on the water absorption properties where the value of water.

![SEM micrographs for the clay fired at 800°C to 1100°C][20]
Figure 2.14: SEM micrographs for the clay fired at 1200°C to 1250°C [20]

2.6 Analysis Thermal

Thermogravimetric analysis measures the amount and rate of weight changes in a material as a function of temperature or time in a controlled atmosphere. These measurements are used to make determinations about composition for thermal stability. TGA instruments are of two basic configurations: horizontally and vertically aligned. TGA determines the identify of the species released at a certain temperature. In Figure 2.14, the first stage of firing clay is a completion of drying pore water. From the TG curve, it can be seen that the initial weight of the sample was reduced by 0.62% when the temperature was increased up to 289.8°C.

At the temperature of 350°C, the chemical combination water of the clay started to be driven off. This chemically combined water is not to be confused with pore water and water of plasticity, which escapes during early stages of drying. This chemically combined water is part of the molecular structure of the clay and it is only affected by temperatures above 350°C.

The weight of the sample was reduced by 1.43% when the temperature increased to 658.8°C where the dehydroxylation of clay minerals occurs. The effect of flux components such as K₂O, Na₂O and CaO and it can be seen when the clay started to have a reaction at around 900°C.
Three DTA peaks were identified at 104°C, 560°C, and 995°C. The centroid temperature was chosen to identify the peaks because it is easier to establish. The first two peaks are endothermic and the third one is exothermic. The first one corresponds to the water evaporation observed in the TG. The second (and more important) peak corresponds to the kaolinite decomposition observed in the TG. The third exothermic peak around 1000°C may be attributed to either the spinel phase or mullite depending on several variables.

2.7 Analysis of Physical Material

Bulk density is the density of the powder in its loose or uncompressed. The main factors to determine bulk density of a powder are particle size, particle size distribution and particle shape. The density is determined usually by using g/cm³ [27].

Porosity has also been shown to affect mechanical properties. It was reported that the Young’s modulus decreases with increasing porosity and at equivalent porosities, specimens with small pores were found to have a higher Young’s modulus than specimens with large pores [15].

Figure 2.15: Thermogravimetric and differential thermal analyses of the raw clay [26]
Based on the previous study done by Sera et al., [26] it showed porosity and density evolution with the firing temperature for both materials C and K as shown in Figure 2.15. The porosity decreases with the firing temperature is evident for C material evidencing the sinterization of the material. The porosity is lower than the standard material in almost all the temperature range. The decrease is more important, with an abrupt decrease in the porosity between 1100°C and 1150°C. Similar inflection was observed for the contraction, and the sinterization of the standard material is more gradual in this temperature range.

![Figure 2.16: Porosity and density evolutions with the firing temperature of the ash based triaxial materials(C) and the standard (K) triaxial material [26]](image)

According to Nirut et al.,[22] soaking time 0.5 hour led to the lowest density 3.14 g/cm³ compared 2 and 3 hour are 3.16 and 3.20 g/cm³ respectively 98, 99 and 100% theoretical density as shown in Figure 2.17.
Figure 2.17: Bulk density and relative density of specimens sintered at 1850 °C with difference soaking time [26]

2.8 Mechanical Testing of materials

Hardness is an important property to quantify in ceramics. Measured hardness indicates the ability of the ceramic to resist deformation by a hard object. Usually, knoop or Vickers diamond indenters are used in conjunction with a micro indentation hardness machine. Vickers indenters are used to characterize roughly 60% of the ceramic hardness value that are published. Figure 2.18 shown at the Vickers indenter is a 136 degrees square based diamond pyramid.

Figure 2.18: Operating diagram of Vickers hardness test [28]
REFERENCES


