CHARACTERIZATION AND MECHANICAL PROPERTIES OF POROUS CERAMICS WITH DIFFERENT CARBON BLACK CONTENTS

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BACHELOR OF MECHANICAL ENGINEERING TECHNOLOGY WITH HONOURS

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Faculty of Mechanical and Manufacturing Engineering Technology

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A thesis submitted
in fulfillment of the requirements for the degree of
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DECLARATION

I declare that this thesis entitled “**Characterization and Mechanical Properties of Porous Ceramics with Different Carbon Black Contents**” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :
Name : Muhamad Ammar Baqir Bin Zol Nadzruddin
Date : 20 January 2023

APPROVAL

I hereby declare that I have checked this thesis and, in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Mechanical Engineering Technology with Honours.

Signature :

Supervisor Name : Associate Professor Ts. Dr. Umar Al-Amani Bin Haji Azlan

Date : 20 January 2023

DEDICATION

First and foremost, author would like to praise and thank the Almighty God and with heartfelt gratitude to the Prophet Muhammad S.A.W., for giving us the strength and because of His blessing, author finally managed to accomplish this Bachelor Degree Project (BDP). Without His blessing, author wouldn't have gone this far. This Bachelor Degree Project (BDP) cannot complete without effort and co-operation. Author would want to take this moment to offer my heartfelt gratitude and heartfelt appreciation to author’s mother, Nang Rohani Binti Long Ismail, and author’s father, Zol Nadzruddin Bin Mansor, also to author’s sibling for their support and sacrifice throughout this journey, both mentally and physically.

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ABSTRACT

Nowadays, energy conservation in structural applications is a serious challenge that has a substantial impact on the environment in Malaysia and around the world. One of the most important issues in this challenge of energy conservation is the loss of heat by convection through the use of traditional solid building materials. To address this problem, controlled porosity ceramics are utilised in a variety of functional and structural applications, such as filters, thermal insulation, bio-scaffolds, and composite preforms. High porosity, in general, leads to poor thermal conductivity, which is required for thermal insulation. Increased porosity, on the other hand, lowers the strength of ceramics in a reciprocal manner. As a result, different types, amounts, and forms of pore-forming agents (porogen) must be incorporated into a porcelain-type tile ceramic body composition to control the creation of pores. Porous ceramics are known to be extremely reliant on pore structure, with pore size, shape, and distribution all having an impact. However, it was also discovered that a phase with modest peaks and an unknown phase was present in XRD. This research demonstrated that peak formation is not much impacted by rising CB concentrations. Pores in porous ceramics were primarily created by CB microspheres losing their shape at high temperatures from SEM. The strength of sintered samples decreased from 60 MPa to 55 MPa on average as a result of the presence of CB. It is generally known that a high porosity caused a decrease in mechanical characteristics. The number of closed pores was generally decreased and more open porosity was produced by the CB. The PFA's particle size distribution had no effect on the amount of porosity, but it did have a significant impact on the number of pores, as well as their size and quantity. However, because the CB were burned off below the sintering temperature and the pores were created, the CB also did not affect any tests or results that were also clearly demonstrated in SEM.

ABSTRAK

Pada masa kini, penjimatan tenaga dalam aplikasi struktur merupakan cabaran serius yang memberi kesan besar kepada alam sekitar di Malaysia dan di seluruh dunia. Salah satu isu terpenting dalam cabaran penjimatan tenaga ini ialah kehilangan haba secara perolakan melalui penggunaan bahan binaan pepejal tradisional. Untuk menangani masalah ini, seramik keliangan terkawal digunakan dalam pelbagai aplikasi berfungsi dan struktur, seperti penapis, penebat haba, bio-perancah, dan prabentuk komposit. Keliangan yang tinggi, secara amnya, membawa kepada kekonduksian haba yang lemah, yang diperlukan untuk penebat haba. Peningkatan keliangan, sebaliknya, merendahkan kekuatan seramik secara timbal balik. Akibatnya, jenis, jumlah dan bentuk agen pembentuk liang (porogen) yang berbeza mesti dimasukkan ke dalam komposisi badan seramik jubin jenis porselin untuk mengawal penciptaan liang. Seramik berliang diketahui sangat bergantung pada struktur liang, dengan saiz, bentuk dan pengedaran liang semuanya mempunyai kesan. Walau bagaimanapun, ia juga mendapati bahawa fasa dengan puncak sederhana dan fasa yang tidak diketahui terdapat dalam analisis pembelauan sinar-x. Penyelidikan ini menunjukkan bahawa pembentukan puncak tidak banyak dipengaruhi oleh peningkatan peratusan karbon hitam. Liang-liang dalam seramik berliang dicipta terutamanya oleh mikrosfera karbon hitam yang kehilangan bentuknya pada suhu tinggi melalui pengimbasan mikroskop elektron. Kekuatan sampel menurun daripada 60 MPa kepada 55 MPa secara purata akibat kehadiran karbon hitam. Umumnya diketahui bahawa keliangan yang tinggi menyebabkan penurunan kekuatan. Bilangan liang tertutup secara amnya berkurangan dan lebih banyak keliangan terbuka dihasilkan oleh karbon hitam. Pembentukan saiz liang oleh agen pembentukan liang tidak mempunyai kesan ke atas jumlah keliangan, tetapi ia mempunyai kesan yang ketara ke atas bilangan liang, serta saiz dan kuantitinya. Walau bagaimanapun, kerana karbon hitam telah dibakar di bawah suhu pensinteran dan liang-liang dicipta, ia juga tidak menjejaskan sebarang ujian atau keputusan yang juga jelas ditunjukkan dalam pengimbasan mikroskop elektron.

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LIST OF SYMBOLS AND ABBREVIATIONS

% - Percentage
°C - Celsius
°C/min - Celsius per Minute
°F - Fahrenheit
µm - Micron
D,d - Diameter
g - Gram
kN - kilo Newton
kV - kilo Volt
m - Meter
mm - Millimetre
mm/min - Millimetre per Minute
MPa - Mega Pascal
s - Second
BCE - Before Common Era
CB - Carbon Black
Cw - Waterproof Silicon Carbide
FRP - Fibre Reinforced Plastic
PFA - Pore Forming Agent
SEM - Scanning Electron Microscope
XRD - X-ray Diffraction Analysis

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CHAPTER 1

INTRODUCTION

1.1 Background

In today's world, energy conservation in structural applications is a serious concern that has a significant impact on the environment in Malaysia and around the world. The loss of heat by convection due to the use of typical solid construction materials is one of the most important components of this energy conservation issue. Ceramic tiles are commonly found on floors, walls, roofs, and table tops. These good’s dimensional stability and appearance, water absorption, hardness and load resistance, scratch resistance, and temperature resistance to shock, frost, and crazing are all crucial properties (Dhir et al., 2017).

In Medieval Italy, the earliest ornamental tiles, usually of tiny proportions, were used in public and ecclesiastical structures. Since the beginning of the 12th century, ceramic tile development has attempted to make manufacturing more efficient (Jakub Piorkowski., 2020). During the "golden age of engineering ceramics" in the 1970s and 1980s, new discoveries in ceramics production were primarily performance driven, giving a great playground for material scientists to create new materials with consistently new and improved features (Gadow & Kern, 2014).

The existence of microscopic gaps or spaces inside a material is referred to as porosity. Porosity is defined as the open spaces between grains or trapped in grains in a microstructure. Porosity is a number that ranges from less than 0.01 for solid granite to more than 0.5 for peat and clay. By multiplying the fraction by 100, it may also be expressed in %

terms (Corrosionpedia., 2018). Porosity and pore characteristics such as size, connectivity and distribution have a significant impact on material properties. In general, the lower the porosity, the fewer linked pores there are in the same substance. As a result, the strength will be stronger, water absorption will be lower, permeability and frost resistance will be better. (Pezeshki et al., 2018). However, increased porosity reduces the strength of ceramics in a reciprocal manner.

Carbon Black (CB) is a manufactured substance made out of a thin black powder of virtually pure elemental carbon that has been in use for over a century (Tofighy & Mohammadi, 2019). Theoretically, spherical micro-pores and nano-pores can increase the mechanical characteristics of porous ceramics while lowering their heat conductivity. CB is distinguished from ordinary pore-forming agents by its nano-scale particle size and spherical shape (Liu et al., 2016). CB grades with varying qualities such as specific surface area, particle size and structure, conductivity, and colour are produced by partial combustion or thermal breakdown of gaseous or liquid hydrocarbons under regulated circumstances.

Furthermore, after burning out at high temperatures, such an agent leaves no contaminants behind. However, because it is reliant on the depletion of petroleum, the manufacturing and processing of CB is dangerous (Tofighy & Mohammadi, 2019). As a result, CB is use as the pore producing agent into a porcelain-type tile ceramic body composition to regulate pore formation.

1.2 Problem Statement

Nowadays, energy conservation in structural applications is a serious challenge that has a big influence on the environment in Malaysia and throughout the world. One of the most important issues in this challenge of energy conservation is the loss of heat by convection through the use of traditional solid construction materials. Heat capacity, thermal expansion coefficient are all thermal characteristics of ceramic materials. Thermal energy may be stored or transmitted by a solid. The ability of a substance to absorb heat from its surroundings is referred to as heat capacity. This highlights one of the primary reasons why the surfaces of ceramics reached high temperatures when exposed to external environments that are exposed to solar radiation (Živcová et al., 2009).

The porosity of ceramic tiles may be controlled by adding pore-forming agents, which helps to limit heat conductivity. The presence of pores can have a detrimental influence on mechanical strength in general. As a consequence, heat conductive ceramic may be reduced while maintaining mechanical strength. Pore-forming agents have showed the capacity to reach high porosity levels in particular (Liu et al., 2016). During the heating process to the firing temperature, organic particles are burnt away, leaving gaps in the ceramic body. The pore-forming agent utilized will influence the morphology of these voids, which may be controlled by incorporation percentage and pore size.

Carbon black (CB) was used as pore-forming agents (PFA's). CB was chosen because of its large-scale production and low cost, making it economically viable for industrial uses. As previously stated, the goal of this study is to evaluate the performance of ceramics based on a variety of characteristics utilizing the PFA of CB.

1.3 Research Objective

The main aim of this research is to develop generation of tiles in buildings structural applications. Specifically, the objectives are as follows:

a) To fabricate the ceramic tiles with carbon black at different ratios.

b) To study the mechanical properties of ceramic tiles.

1.4 Scope of Research

The research was look at the manufacture of powder, the fabrication of ceramics, the properties of the powder or ceramic, and the performance of ceramics. All of this research was carried out in order to create porous ceramics for use in applications and structures. There are many different types of powder that may be used to produce ceramics and one of the processes in the powder production process is to choose which type of powder will be used.

The optimal forming process for fabricating ceramics was studied or analysed in this study. There are a few different types of forming methods. Due to the equipment provided in the lab, it is necessary to limit the types of PFA's that can be used. However, use the strategy that was result in a favourable consequence in order to attain the goal. X-ray diffraction analysis (XRD), scanning electron machine (SEM), three-point flexural strength test, and density porosity are among the tests and analyses that were performed. All of this testing and analysis were done to help determine the best appropriate parameter or approach to utilise.

Furthermore, by obtaining the results of the test and analysis, it is possible to enhance and eliminate the issue. The most crucial stage in completing the analysis was to study the performance of ceramics. Following the completion of the test, this project was examined the performance of ceramics to determine what porous ceramics are required.

1.5 Significant of Research

The aim of the research was learned more about the functions and properties of PFA’s such as CB, which can be used to enhance porosity in ceramics. The importance of understanding the pore forming agent functions because, in general, the pore forming agent can have a negative effect on ceramic, but if the pore forming agent is used incorrectly, it can lead ceramic to have a large-scale porosity. Furthermore, when the scale porous ceramic is higher, the mechanical strength of the ceramic is reduced. As a result, the ceramic will be more easily fractured.

Controlling the porosity ceramic requires a detailed understanding of the properties of the pore forming agent. It is thus possible to enhance heat conductivity by establishing a correct percentage of porosity ceramic. By conducting this research, were be able to gain a better understanding of the ceramic tile that has been used in construction. The ceramic surface will become hotter as a result of the sun's radiation, which causes a high-temperature exterior environment. The heat from the atmosphere and solar radiation will be absorbed by the ceramic. Understanding the functions and properties of the pore producing agent can help to improve and resolve these issues in this scenario.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter will discuss the historical research on this issue, which is structured ceramic. There are various research papers addressed within the literature review which will be compared to the present project. This chapter covers the history and structure of ceramics generally. Journals, books, newspapers, and other materials is used as guides additionally to the current chapter. This chapter emphasizes the necessity of evaluating past research and therefore the evolution of the difficulty through time. Literature research also gives a method and structure for creating this effort a hit. It's critical to try and do so in order to reinforce ceramic.

2.2 History of Ceramics

Ceramics is an ancient industry, extending back thousands of years. When humans discovered that clay could be easily acquired and shaped by mixing it with water and heating it, a huge business formed. According to the American Ceramic Society, the earliest known ceramic artefact goes back to 28,000 BCE. The Venus of Doln Vstonice is a woman figure from a little village near Brno, Czech Republic. Hundreds of Ice Age animal sculptures were uncovered among the remnants of a horseshoe-shaped kiln (Darvell, 2018).

Several thousand years later, the earliest samples of pottery were discovered in Eastern Asia. Pot pieces dating from 18,000-17,000 BCE have been discovered in China's Xianrendong cave. It is thought that pottery was first used in China and then migrated to

Japan and the Russian Far East, where archaeologists discovered fragments of ceramic artefacts going back to 14,000 BCE.

During the Neolithic era, pottery usage increased fast as permanent populations devoted to cultivation and farming. From 9,000 BCE, clay-based ceramics were used as food and water containers, art, tiles, and bricks across Asia, the Middle East, and Europe. Early objects were sun-dried or burned in rudimentary kilns below 1,000°C. Simple linear or geometric motifs were painted on monotone pottery.

The earliest blast furnaces in Europe were erected in the 15th century. Originally composed of natural materials, they melted iron. Synthetic materials with better heat resistance (refractories) initiated the industrial revolution in the 16th century. These refractories enabled industrial metal and glass melting, as well as coke, cement, chemicals, and ceramic manufacture (Fuchs, 2017).

Since then, the ceramic sector has seen significant changes. Traditional ceramics and glass have grown widespread, but new products have been developed over time to take use of these materials' particular features, such as low thermal and electrical conductivity, good chemical resistance, and high melting point. The first porcelain electrical insulators were developed about 1850, kicking off the technical ceramic's era.

After WWII, ceramics and glass helped build advanced industries including electronics, optoelectronics, medical, energy, automotive, aerospace, and space exploration. Advances in ceramic processing and characterization have made it feasible to manufacture materials with customized properties. Nanotechnology has boosted ceramic processing, enabling manufacturers to make translucent ceramics, ductile ceramics, hyper elastic bones, and microscopic capacitors. According to Figure 2.1, there are example of ceramic product.

Figure 2.1: Ceramics product (https://www.civilengineeringweb.com, 2021)

2.3 Ceramic Materials

In general, there are two types of ceramic materials utilized in engineering applications. There are two types of ceramics which is traditional and advanced. Traditional ceramics are often created from three basic ingredients as shown in Figure 2.2, likes clay, silica (flint), and feldspar. Bricks, tiles, and porcelain pieces are examples. Advanced ceramic materials, on the other hand, are made up of very pure aluminium oxide (Al_2O_3), silicon carbide (SiC), and silicon nitride compounds (Si_3N_4).

Figure 2.2: Raw materials of ceramics: (a) Clay, Feldspar, (c) Silica (Shree Charbhuja Exim, 2020).

2.3.1 Properties of Ceramics

The sorts of atoms present, the types of bonding between the atoms, and the manner the atoms are packed together determine the characteristics of ceramic materials, as do all materials. The majority of ceramics are composed of two or more parts. This is referred to as a compound. Alumina (Al_2O_3), for example, is a chemical made composed of aluminium and oxygen atoms.

A chemical link holds the atoms in ceramic materials together. Covalent and ionic chemical bonding are the most frequent in ceramic materials. The chemical link between metals is known as the metallic bond. The covalent and ionic bonds between atoms are stronger than metallic bonds. That is why metals are ductile and ceramics are brittle in general. Ceramic materials are employed in a broad variety of applications due to their diverse characteristics. The majority of ceramics are:

- Hard
- Wear-resistant
- Brittle
- Refractory
- Thermal insulators
- Nonmagnetic
- Oxidation resistant
- Prone to thermal shock
- Chemically stable

Clay is soft and flexible because, like graphite, its atoms are made up of flat sheets that may glide past one another with just weak bonds binding them together. When water is introduced to clay, the polar water molecules (positively charged on one end and negatively charged on the other) help to break down the bonds, making the clay more malleable. When clay is burnt, the water evaporates, and the aluminium, silicon, and oxygen atoms bind together in a rigid structure called aluminium silicate, which explains why fired clay is so tough (Chris Woodford, 2021).

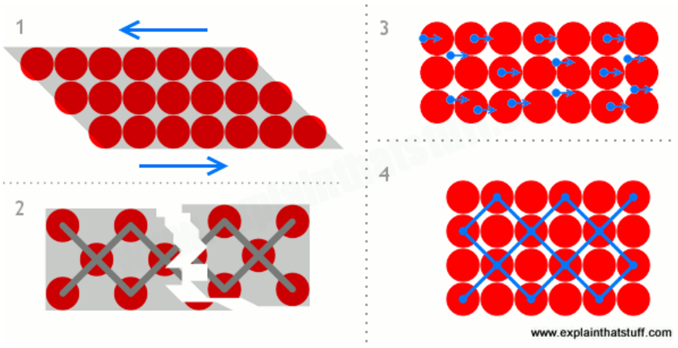
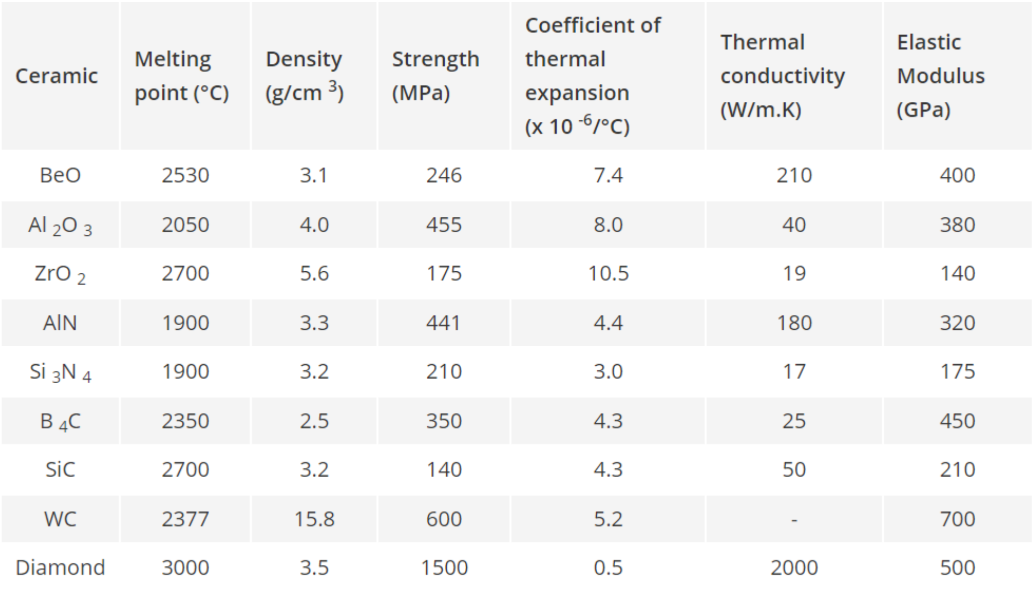


Figure 2.3: Structure of ceramic atoms(www.explainthatstuff.com, 2022).

Ceramic has high melting temperatures, making them heat resistant, as well as incredible hardness and strength, great resilience, making them long-lasting and hard-wearing, and low electrical and thermal conductivity. They are effective insulators and chemically inert, which means they do not react with other substances. Controlling the microstructure may overcome intrinsic stiffness, allowing ceramic springs to be made, and ceramic composites with a fracture toughness half that of steel have been created.

A ceramic's porosity may affect qualities such as heat conductivity and hardness. This is due to the gaseous that fills the pore in the ceramic having a limited heat conductivity. As a consequence, thermal insulators in ceramics have a greater influence. Porosity, on the other hand, affects the strength and durability of the material. As a result, the porosity of the ceramic must be adjusted in order to solve this issue. The oxides, nitrides, and carbides are the three primary compositional classes of engineering ceramics. The Table 2.1 lists the general characteristics of the most often used ceramics.

Table 2.1: Properties of ceramic(Pfeifer, 2009).



Ceramic	Melting point (°C)	Density (g/cm ^{3})	Strength (MPa)	Coefficient of thermal expansion (x 10 $^{-6}$/°C)	Thermal conductivity (W/m.K)	Elastic Modulus (GPa)
BeO	2530	3.1	246	7.4	210	400
Al_2O_3	2050	4.0	455	8.0	40	380
ZrO_2	2700	5.6	175	10.5	19	140
AlN	1900	3.3	441	4.4	180	320
Si_3N_4	1900	3.2	210	3.0	17	175
B_4C	2350	2.5	350	4.3	25	450
SiC	2700	3.2	140	4.3	50	210
WC	2377	15.8	600	5.2	-	700
Diamond	3000	3.5	1500	0.5	2000	500

2.4 Applications of Ceramic

Crystalline and glass ceramics are split into two groups based on their applications which is conventional and advanced.

2.4.1 Conventional Ceramic

The following are the categories of traditional ceramics:

- ❖ Bricks, tiles, and pipes are structural clay products. These goods are mostly made up of silica and alumina, with minor percentages of other oxides such as iron oxide, magnesia, titania, potassium oxide, and sodium oxide.
- ❖ Stoneware (tableware, artware, tiles, and cookware), china (tableware, artware, cookware, toilets), porcelain (tableware, artware, electrical insulators, and dentures), and electrical insulators. The same components that are used in structural clay goods are used in these items.
- ❖ Cements are synthetic mineral combinations used in the construction of roads, bridges, and buildings, such as concrete and mortars. Cements are formed into a hard,

bonded mass via a heatless hydration process at room temperature. Tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetra calcium aluminoferrite are the four major components of cement.

- In high-temperature furnaces, refractory materials are utilized as thermal insulation. They can withstand corrosive gases, liquids, and solids at high temperatures. Silica, aluminium silicate, and magnesite are examples of refractory materials.

Traditional ceramics and glassware are typically oxide compositions and come in both crystalline and amphorous forms. They are not entirely dense in many forms and have porosity in the micron and larger size range. Porosity may have a specific technical role in certain applications (e.g., thermal insulation, filters), such as influencing thermal characteristics and determining mechanical strength (Pfeifer, 2009).

Based on TWI Ltd, Ceramics are durable, non-combustible, and inert when compared to metals and polymers. As a result, they're suitable for high-temperature, corrosive, and tribological applications. These uses depend on a unique set of qualities found only in industrial ceramics, including:

- property preservation at extreme temperatures
- low expansion coefficient
- resistance to corrosion
- insulation (thermal)
- insulation from electricity
- low population density

Engineering ceramics are used to create components for a variety of industrial applications, including electronic device ceramic substrates as shown in Figure 2.4, turbocharger rotors as shown in Figure 2.5, and automobile engine tappet heads. Oil-free bearings in food processing equipment, aerospace turbine blades, nuclear fuel rods, lightweight armor, cutting tools, abrasives, thermal barriers, and furnace/kiln furnishings are all examples of where advanced ceramics are employed.

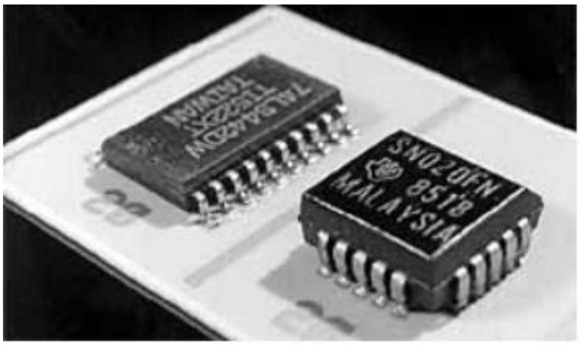


Figure 2.4: Ceramic substrates for electronic devices (https://www.twi-global.com, 2018).

Figure 2.5: Ceramic turbocharger rotor assembly made from silicon nitride courtesy of NGK/NTK Spark Plug Co (https://www.twi-global.com, 2018).

2.4.2 Advanced Ceramics and Glasses

Advanced ceramics and glasses are materials that have been meticulously created to have certain mechanical, electrical, magnetic, or optical qualities. Chemical compositions, crystal phases, and microstructures vary widely in advanced ceramics and glasses (Pfeifer, 2009). They may be divided into the following categories:

- Metals fail in high-stress, high-temperature, or corrosive situations, hence structural ceramics are used instead. Engines, wear parts, heat management, processing equipment, and biomedical and dental components are among the applications.

Alumina, silicon nitride, silicon carbide, zirconia, boron nitride, mullite, cordierite, and molybdenum disilicide are examples of this kind of material.

- Electronic ceramics for applications such as capacitors, sensors, semiconductors, superconductors, ionic conductors, varistor, and substrate/packaging. Electronic substrates are made of alumina, aluminium nitride, and boron nitride. Capacitors, sensors, actuators, and ferroelectrics all employ titanate compositions.
- Solid-state lasers, optical data transfer, photoelectric, wavelength-tuned windows, and tailored mirrors are all examples of optical ceramics and glasses for information display. Titanates, rare-earth oxides, and chalcogenide glasses are common composition families.
- Pump components (seals, bearings, plungers), faucets (seals, valves), and rotating equipment wear and corrosion resistance (bearings). Alumina, zirconia, silicon carbide, and silicon nitride are common compositions.
- Magnetic ceramics are used in actuators, sensors, data storage, transformers, recording, sonar, and microwave applications. Ferrites, rare earth oxides, and copper oxide compositions are among them.
- Diamond, silicon carbide, tungsten carbide, silicon nitride, and alumina are used in machining and grinding tools for extended life and great efficiency.

2.5 Bi-layered Structure Ceramics

Bi-layered ceramic structures are extensively employed in a number of applications, whether they are composed of a dense layer on a dense substrate, a porous layer on a dense substrate, or a dense layer on a porous substrate (Chen et al., 2017). Bi-layered ceramic tiles have a changeable thickness and are made up of two layers with different densities. They are dense and porous.

A twofold pressing motion is used in the new production method, which is rapid and easy to utilize in an industrial context and allows for faultless interface bonding between layers. The bi-layered ceramic tile has an upper layer with a density comparable to standard porcelain stoneware tiles and a porous bottom layer that allows for weight reduction while maintaining mechanical strength.

This bi-layer structure offers an advantage in ceramics due to its structure. As porosity rises, so does thermal conductivity. A ceramic's porosity may diminish its heat conductivity even further. Increasing a ceramic's porosity, on the other hand, reduces its strength, making it unsuitable for structural uses. To solve the problem, the ceramic production process must be managed. The denser layer of the ceramic may overcome the ceramic's weak strength owing to porosity by integrating a bi-layer structure ceramic (Liu et al., 2018).

2.6 Pore Forming Agent

Ceramic filters and membranes, fuel cell electrodes, catalytic supports for biomaterials, piezoelectric materials, and thermally or acoustically insulating bulk materials are just a few of the uses for porous ceramic materials.

There have been many known ways for making porous ceramics. Pore-forming chemicals have showed the capacity to reach high porosity levels in particular. During the heating process to the firing temperature, organic particles are burnt away, leaving gaps in the ceramic body (Živcová et al., 2009). The pore-forming agent utilized will influence the morphology of these voids, which may be controlled by incorporation content and particle size distribution. Pore-forming substances have been researched extensively, including starch, graphite, Lycopodium, and polymethyl methacrylate (Li et al., 2022).

The pore generating agent in this experiment will be CB. Because of its particular qualities, this performance agent was selected to help boost heat conductivity and mechanical strength.

2.6.1 Carbon Black

Carbon black (CB) is a kind of carbon that results from the incomplete combustion and thermal degradation of gaseous or liquid hydrocarbons in a controlled environment as shown in Figure 2.6. This method produces a thin, powdery black dust that is one of the top 50 compounds on the planet. CB is used as a filler in the rubber business, but it's also found in a variety of colours and inks. It's a conductive substance used to manufacture electrodes and carbon brushes in the electrical industry (Katsoufis et al., 2020).

CB is a porous, strewn material. Given this, CB's effective thermal conductivity must be interpreted as a value equal to the thermal conductivity of any homogeneous substance through which, given similar dimensions and temperatures on the borders, the same amount of heat travels as goes through the carbon black.

There are many industries employ technical-grade CB as a technological component, thermal insulator, thermostable material, and so on. The polydisperse granules 0.2-3.0 mm in size used in the tyre industry are made in granulators from amorphous carbon black and a molasses-in-water binder solution comprising 3 percent molasses by weight. After that, the granules are dried at temperatures ranging from 350 to 500 °F. When evaluating heat transfer processes in this and other processes, knowing the effective thermal conductivity coefficient of such carbon black particles is critical (Tofighy & Mohammadi, 2019).

The size and form of the pores and cavities are known to alter the effective thermal conductivity of dispersed and porous materials. Some pores and cavities may be closed volumes, while others may connect to create open channels. Heat transfer occurs in such materials due to a number of causes. Conductivity inside the particles and at points of direct contact are used to transfer information. Both conductivity and radiation cause transfer in the air medium of the pores and cavities. Radiant heat transfer contributes more when pore and cavity sizes grow (Long et al., 2013).

The size of the cavities between particles grows as the particle size grows, and the direct contact area between particles is larger with coarser particles than with small particles. As a consequence, the contact thermal resistance decreases and the effective thermal conductivity rises.

Figure 2.6: Powder of CB (http://divyaint.com, 2012).

2.7 Raw Materials

In this project, four distinct kinds of raw materials are utilized to make porous ceramic samples. Raw ingredients will include clay, feldspar, silica, and a pore forming agent.

2.7.1 Clay

Clay has long been an important part of human existence. Clay raw materials are utilized and valued in a variety of industries, including agriculture, civil engineering, and environmental research as shown in Figure 2.7. This is because to their diverse features, great resilience to atmospheric conditions, geochemical purity, ease of access to deposits near the earth's surface, and cheap cost (Konta, 1995).

Figure 2.7: Types and clay product (https://www.sciencelearn.org.nz, 2019)

The word "clay" is used in many different ways. It's a geological word for a fine-grained, earthy material that shows flexibility when mixed with a tiny quantity of water. It's also a particle size term that's been around for a long time in the study of sedimentary rocks and soils, referring to particles bigger than 2 m. This limit provides a natural distinction between clay minerals, which are commonly found in 2 m of diameter particles, and non-clay minerals, which are found in bigger particles.

The clay mineral theory, presented in the 1920s and 1930s that the clay portion of a soil or rock is formed of tiny particles of a limited number of crystalline minerals, was confirmed between about 1930 and 1960. At the time, cutting-edge research methods were used to find the minerals and characterize their structures.

Clay minerals are frequently employed as inert fillers in polymers, paints, and the paper industry, and their worth stems from their capacity to substitute for more costly materials or their physical utility (arising from the anisotropic particle shape). Chemically, clay minerals, on the other hand, are reactive. Phase transitions and intercrystallite reactivity, for example, drive their use in ceramics (Wolf et al., 2022).

Clay minerals are also used to make zeolites, which are used for ion exchange, catalysts, and catalytic supports. They've even been linked to the genesis of life, since they use catalytic surface formation to make complex organic compounds. Finally, although clays are normally found in nature, specific ionic substitution patterns may be used to synthesize them synthetically. Rheology modifiers, for example, are made from several of these elements. Figure 2.8 shows the types of clay.

Figure 2.8: Types of clay (Rebecca Ruger-Wightman, 2018)

2.7.2 Earthenware Clay

Earthenware clays are the oldest and most widely used clays among potters today. It's made of plastic and is suitable for beginners. The colour of pottery is generally warm. Brown, red, orange, or grey are the tones of wet clay. Earthenware may be brown, orange, red, grey, or white after burning. Colours in pottery emerge after firing. The most common colour that may get is terracotta. Paints and glazes may simply be applied on earthenware.

Sculpting, hand-building, and wheel throwing are all done using earthenware. It can make sculptures, flowerpots, and other garden art. Water cannot get trapped within and break if it freezes. In compared to other clay kinds, earthenware burns at the lowest temperatures, allowing for the required hardness to be achieved. For bisque firing, most earthenware is burned between 1828 and 2088 °F (998 and 1142 °C), Cone 06 to Cone 2. Glaze was fired from Cone 08 to Cone 04 at temperatures ranging from 1728 to 1945 °F (942 to 1063 °C). This is related to the compound's impurities, which include minerals. Earthenware is non-vitreous once burned, meaning it absorbs 7 % or more moisture (water). Earthenware remains soft even after firing and may be scraped with a sharp tool. Figure 2.9 shows the example of product in earthenware clay.

Figure 2.9: Product in earthenware clay (https://www.dreamstime.com, 2016)

2.7.3 Ball Clay

The most plastic clays are ball clays, which have relatively little mineral imperfections. They include a lot of kaolinite and quartz, as well as 10 to 25 % mica. Ball clays are extremely fine minerals that occur naturally as sediments or deposits. Ball clays include lignite and other materials. Ball clays lose their colour when fired. Ball clays are grey in hue when they are in the greenware stage. They take on a light buff tint after firing. When burned properly, they give a beautiful white tone, making them popular among potters (Burst, 1991).

Ball clays are often used for floor tiles, toilet bowls, vases, kiln furniture, and dinnerware as shown in Figure 2.10 because to their excellent plastic and binding qualities. Ball clay is too fine and slippery to use on its own. It may be used for wheel throwing, but it's best known for slip casting. Ball clays harden at roughly 2345 °F (1285 °C) Cone 10 after firing. They have one major flaw, and it occurs when firing. During the fire process, ball clays shrink dramatically. It can't utilize ball clay on its own unless you have the necessary skills or a big enough kiln. When combined with different clays, ball clay is the ideal material to use for pottery (like kaolin).

Figure 2.10: Product in ball clay (Tony Hansen, 2017)

2.7.4 Stoneware Clay

Stoneware clays are nonporous, hard, and mildly flexible. It got its name from its stone-like properties. When wet, it can variety in colour from white to grey to brown. Kaolinite is present in stoneware clays, along with minor amounts of mica and quartz. Illite and smectite are also commonly found. The coloring is also affected by the type of firing and the temperature. Figure 2.11 shows the product from stoneware clay.

This clay is easy to work with and paint with underglazes, glazes, overglazes, enamels, and other materials. As a result, stoneware clay is a popular dinnerware clay. Hand-building and wheel-throwing are the most common applications. The firing temperatures for stoneware clays are 2106 to 2262 °F (1152 to 1239 °C). Cones 3 through 7 High fire temperatures ranging from 2280 to 2345 °F (1249 to 1285 °C). Cones 8 through 10.

Fire clays are a form of refractory clay used mostly as a filler. These are heat resistant, allowing stoneware clays to mature at higher temperatures. They also add a little roughness to stoneware because they include mineral impurities like iron. After being burned, iron frequently leaves black stains on ceramics.

Figure 2.11: Stoneware clay product

2.7.5 Porcelain Clay

Porcelain or China ceramics, particularly for plates, are quite popular. They're made out of mostly pure mineral kaolin, sometimes known as "China Clay," and other minerals. Porcelain does come in a variety of colours, but they are all quite delicate and light. They'll have a very light grey-ish tone at the greenware stage. They are off-white to white after fire. Glazes and enamels on porcelain can be used just for colour and do not require glazing to repel water (Dondi et al., 2014).

Kaolin clays are the least flexible of the clays and thus the most difficult to deal with. They burn at extremely high temperatures. To lower the firing temperature and improve workability, porcelain is combined with various minerals. Wheel throwing and cast sliding are the most common methods for creating dinnerware, vases, and other decorative pieces.

Hard-paste, soft-paste, and bone China are the three varieties of porcelain ceramics which is: -

- The most prevalent type is hard-paste or "real" porcelain. It commonly has feldspar or mica as an additional material to the kaolin. True" porcelain is burned at extremely high temperatures (2345 °F or 1285 °C) in Cone 10 to produce more durable products.
- The least frequent type of porcelain is soft-paste porcelain. Cone 5 was invented by Europeans and shot at lower temperatures (about 2167 °F or 1186 °C). Soft paste porcelain is regarded as a fragile material. It does not require the creation of a fixed mineral. This type is made up of kaolin, bone ash, quartz, glass, and soapstone, with ball clay thrown in for good measure.
- In current times, bone China has mostly superseded genuine porcelain. It's the most durable type of porcelain. It has a high physical strength and is resistant to chip damage. It frequently results in a white or translucent appearance. Kaolin, bone ash, feldspar, and phosphates are all present.

The four principal types of clay covered in this study were Earthenware, Stoneware, Ball Clay, and Porcelain. These clays differ in terms of firing temperatures, colours, textures, and applications. Even if the clays are largely made up of the same minerals. The first step in manufacturing pottery is to choose the correct clay. Advanced potters may work with any clay type and even mix them together to change the plasticity. Example of Porcelain clay product is shown in Figure 2.12.

Figure 2.12: Porcelain clay product (https://civiljungle.com, 2016)

2.8 Feldspar

Feldspar is the most common mineral group in the earth's crust, making up around 60% of all terrestrial rocks based on Britannica. Sodium feldspar, potassium feldspar, and mixed feldspars make up the bulk of deposits. Feldspars are mostly employed in industrial applications due to their high alumina and alkali content. The word feldspar is used to describe a broad range of materials. Glass for drinking, glass for protection, fiberglass for insulation, bathroom floor tiles and shower basins, and tableware are all made of feldspar. Feldspar is a mineral that come across often. Figure 2.13 shows the types of feldspar which is Orthoclase, Na-Plagioclase and Ca-Plagioclase.

Feldspar, after clay, is the second most important component in the production of pottery. Feldspar has no set melting point and melts slowly over a broad temperature range. This greatly facilitates the melting of quartz and clays, allowing for modification of this crucial phase in ceramic manufacture with proper mixing.

Feldspars are used as fluxing agents to form a glassy phase at low temperatures, as well as a source of alkalise and alumina in glazes. They soften, melt, and moisten other batch materials to improve the strength, hardness, and durability of the ceramic body, as well as cement the crystalline phase of other ingredients.

Figure 2.13: Types of feldspar (KenResearch, 2020)

2.9 Silica

Silica is a mineral made up of silicon dioxide. In the body, silicon dioxide (as quartz crystal) nearly always occurs as unmelted particles embedded in the fired matrix (although finer one dissolve into the inter-particle glass). In glazing chemistry, though, talking with silica, an oxide. All glazes that melt completely and re-solidify include silicon dioxide, an oxide. Many of them are at least 70 %. Figure 2.14 shows the example of silica powder.

Materials leak silicon dioxide into the glaze melt as the kiln temperature rises. Different materials dissolve into the melt at different temperatures. The size of the particles in the components affects how rapidly they dissolve in the melt. Glazes' main glass-forming component is silicon dioxide. Glass may be formed by combining silicon dioxide with almost any other oxide (Darvell, 2018).

Ceramics and refractories need silica to be manufactured. In the production of ceramics, silica silicon dioxide is used to help control thermal expansion, regulate drying and shrinkage, and enhance structural integrity and aesthetics. Silica is necessary for the glazing and body composition of items such as dinnerware, sanitary ware, and floor and wall tiles.

The skeletal framework of the ceramic body is formed by silica, on which clays and flux components bind. Aside from skeleton formulation, Silica is widely used to improve the properties of other goods, such as heat stability, durability, and even pigment extension, reducing the necessity for costly pigments like titanium dioxide. The brightness, reflectance, colour uniformity, and oil absorption of ceramics manufactured with silicon dioxide have been improved.

Furthermore, in the production of shaped and monolithic refractories, silica minerals are also used as main aggregates. Silica provides acid resistance in high-temperature situations such as industrial furnaces. Silica allows ceramics and refractories to maintain important qualities at a lower cost while also providing extra advantages to their makers.

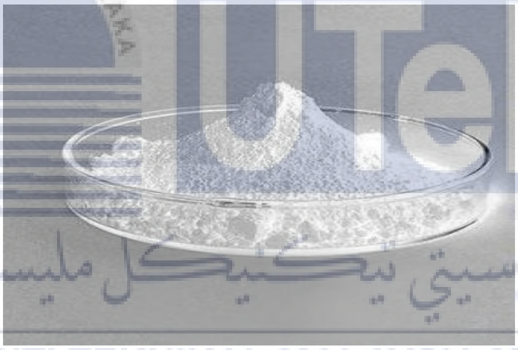


Figure 2.14: Silica powder

2.10 Concluding Remarks

The main goal of this chapter is to understand the characterization of powders and porous ceramics that have been impacted by various pore forming agents based on previous research. The role of pore forming chemicals in porous ceramics is explained in this general chapter. In addition, assist in the research of methods to improve the strength of porous ceramics when adding of PFA’s.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This research study was conducted based on the methodology. This methodology plays an important role in implementing this research study accordingly. The details of the methodology are explained in detail in this chapter. The literature reviews have assisted the researcher to focus on the type of research method that be most suitable for this study.

In addition, the first subtopic in this chapter was an explanation of design of experimental which is the process flow chart of preparation and fabrication. The next subtopic was cover parameters of study and fabrication of porous ceramic with the explanation step or procedure of the process. There were many various types of powder that may be used to make ceramics, and choosing which type of powder to use is one of the steps in the powder production process.

Furthermore, this chapter were look into or analyze the best forming procedure for producing ceramics. The sorts of forming agents that can be utilized in the lab must be limited due to the equipment available. However, in order to achieve the goal, apply the method that was provide a favorable outcome. The tests and analyses were including X-ray diffraction analysis (XRD), scanning electron microscope (SEM), three-point flexural strength test, density and porosity. All of this testing and analysis were carried out in order to identify the most appropriate parameter or method to use.

3.2 Process of Fabrication Ceramics

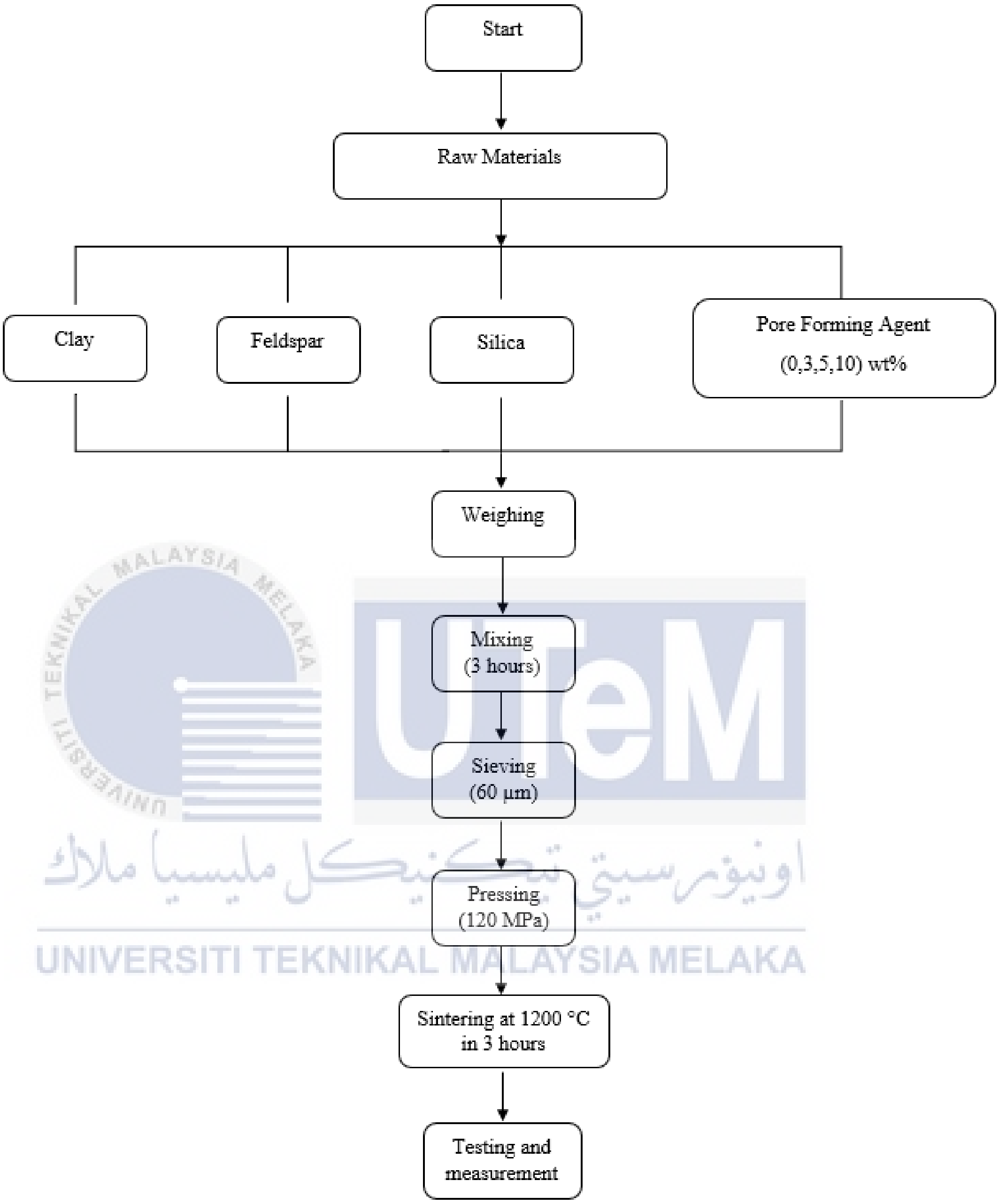


Figure 3.1: Process flowchart of preparation and fabrication of ceramics

3.3 Parameters of Study

Clay (kaolinite), silica (silicon dioxide), and feldspar were used to make the sample ceramic combination (microcline). This porosity ceramic mixture has the chemical formula $Al_2O_3.2Si_2O_2.\ 2H_2O + SiO_2 + KAlSi_3O_8$. The total weight of the composition can be calculated using a chemical equation to be 310 g. As a result, each material's weight may be calculated using the composition's overall weight. Referring to Appendix A, the weights for clay are 24.84 g, feldspar is 40.64 g, and silica is 4.51 g.

The sintering procedure was used to create a structural sample. Sintering is an important step of producing densified porous ceramic. The compact powder was partially sintered in this study by firing at 1200 °C. These temperatures were based on a single sintering temperature ramp-up curve as shown in Figure 3.2. Each temperature was rise 5 °C/min until it reaches sintering temperature, after which it was naturally cool down to room temperature, which is typically 25-28 °C. This pre-heat phase configuration oxidizes the compact porous ceramic before powder consolidation. This prevents the sample from bursting out of the structure.

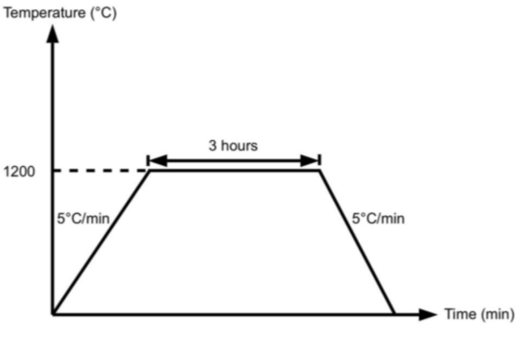


Figure 3.2: Sintering temperature profile at 1200 °C

3.4 Fabrication of Porous Ceramics

Ceramic structure sample manufacturing was done by disc shape and bar shape. The bar shape sample was 75 mm in length, 10 mm in width and 5 mm in height on average while for the disc shape sample was 5 mm in diameter on average. There were four various types of ceramic mixtures in the proportions of 0,3,5, and 10 % of 70 g. Clay and feldspar were in solid phase while silica in powder state. So, clay and feldspar were crunched by stone mortar and pestle. Each variety of sample were transformed into 7 sintered samples. As a result, the overall sample’s quantity was 34 sintered of ceramic samples. For testing purposes, each type ratio mixture creates 10 pieces.

3.4.1 Process of Weighing and Mixing

Every raw ingredient powder was calculated by weight. This weighing procedure ensures that each sample contains a consistent percentage of raw powder in the porous ceramic composition. After that, porous ceramic agent was added to the composition at a rate of 0,3,5,10 %, as shown in the Table 3.1.

Table 3.1: The weight of raw materials powder

Percentage	Type of materials				
	Clay	Feldspar	Silica	Sodium Silicate	Carbon Black
0%	24.84g	40.64g	4.51g	5.00g	0g
3%	24.84g	40.64g	4.51g	5.00g	2.10g
5%	24.84g	40.64g	4.51g	5.00g	3.50g
10%	24.84g	40.64g	4.51g	5.00g	7.00g

After that, ball milling was used to combine the porous ceramic powder composite as shown in Figure 3.3. Ball milling is a method of reducing composite powders to ultra-fine particles. Every porous ceramic powder was mixed for 3 hours at a constant rotational speed at the Ball Mill Machine. Meanwhile, 30 milling balls were placed in the jar with porous ceramic powder to assist the mixing process run more smoothly. Figure 3.4 shows the ball mill machine that was used to mix the powder. Based on Figure 3.5, weighing process of the raw material were used A&D FZ-300i-EC Precision balance. The weight of the raw material as stated in Table 3.1 above.

Figure 3.3: Ball milling with powder composite

Figure 3.4: Ball Mill Machine

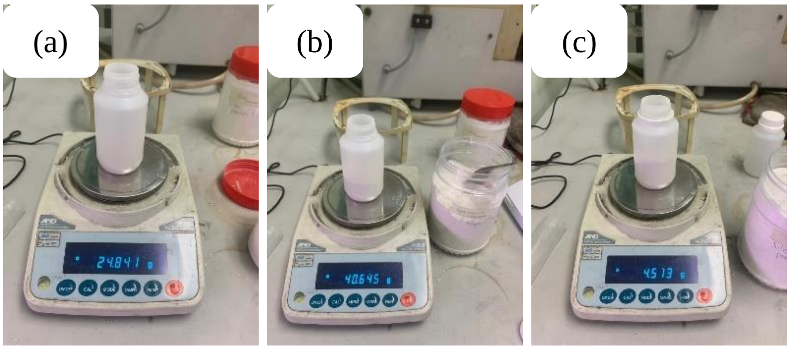


Figure 3.5: Weighing process with different of raw material contents: (a) Clay, (b) Feldspar, (c) Silica.

3.4.2 Process of Sieving

After the mixing process, some granulate powder forms, necessitating the sifting procedure. The sieving method was helped to extract larger particles from a sample's smaller particle size range. Sieving was used to mechanically separate a powder sample with a particle size of 60 micron as shown in Figure 3.5. The sample was shaked in tap sieve shakers in either a vertical or horizontal motion.

Figure 3.6: Sieving process at 60 µm

3.4.3 Process of Pressing

As previously stated, the ceramic fabrication sample were created on a bar shape and disc shape. Under a uniaxial charge, a bar shape of the simplest form and size was known to successfully compact the powder in a uniform density. The bar shape’s dimension was 75 mm in length, 10 mm in width and 5 mm in height on average. In comparison to the square form of the tile, this form uses a minimal amount of material. Based on Figure 3.7, the SM100-Universal Test Machine was used to make the porous ceramic structure in this procedure.

The procedure began with the addition of porous ceramic powder to the disc shape and bar shape mould as shown in Figure 3.8, with the application of low pressure to the plunger in order to form a first compact. After that, place the mould in the Universal Test Machine and press for 60 seconds at 120 MPa. The powder was form a bar or disc shape like structure with sufficient force. This demonstrates that one of the most essential steps in determining the sample force is dry pressing.

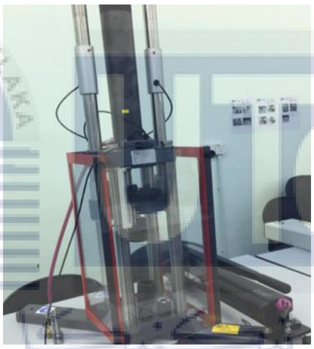


Figure 3.7: SM100-Universal Test Machine

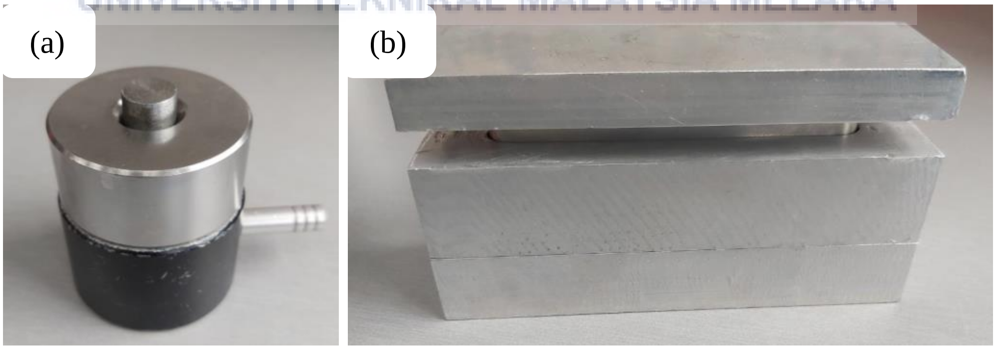


Figure 3.8: Mould of sample for different shape: (a) Disc shape and (b) Bar shape.

3.4.4 Process of Sintering

Sintering have been done after pressing to help the compact porous ceramic powder become a densified ceramic. Sintering was a process of hardening ceramic powder particles by heating a "green" compact to a temperature below the melting point as the substance of individual particles diffuses into adjacent powder particles. The sintering process was carried out in this research using a heat treatment furnace machine as shown in Figure 3.10. These fire processes were based on a single ramp-up sintering temperature profile, according to the parameters of study. Every sample also underwent the drying process, which was heated to 60 °C and increased in temperature at a rate of 5 °C/min until the temperature was reached as shown in Figure 3.9, drying temperature profile.

All samples from the ratio sample batch were heated at 1200 °C. The temperature of the heat treatment furnace equipment was raised to the previously specified sintering profile temperature of 1200 °C at a rate of 5 °C/min. To burn all of the combination composition, the compact ceramic was stay at this temperature for 3 hours.

The compact porous ceramic was cooled in the heat treatment furnace with room temperature at 26 °C ± 2 °C after being heated to profile sintering temperature. After sintering, the compact porous ceramic was achieved the densified ceramic state as stated in Figure 3.11 and Figure 3.12.

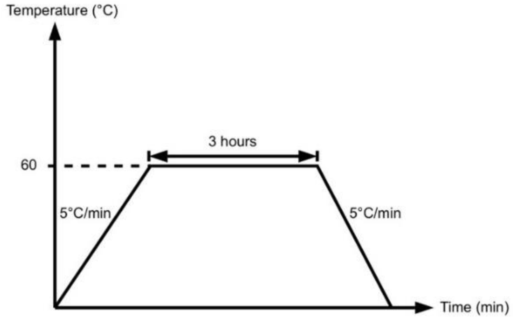


Figure 3.9: The drying temperature profile

Figure 3.10: Heat Treatment Furnace Machine

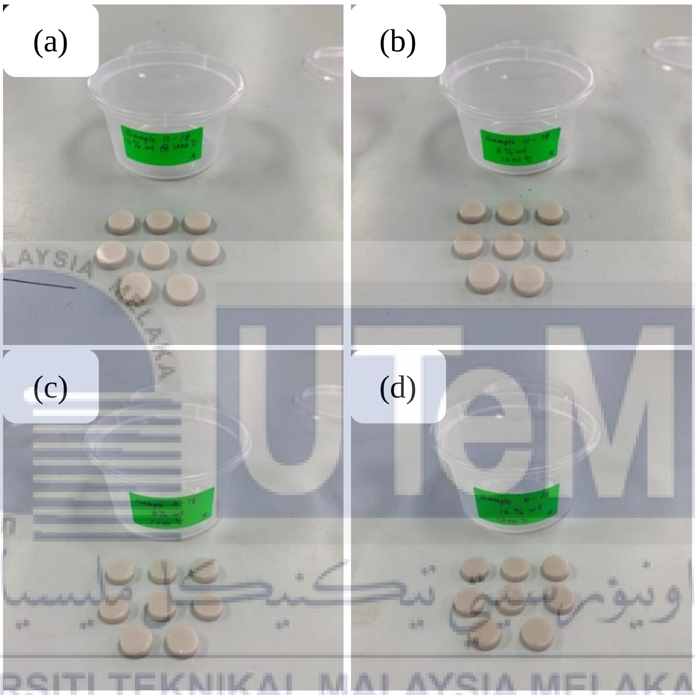


Figure 3.11: Sintered disc shape samples with different CB contents: (a) 0 %, (b) 3 %, (c) 5 %, (d) 10 %.

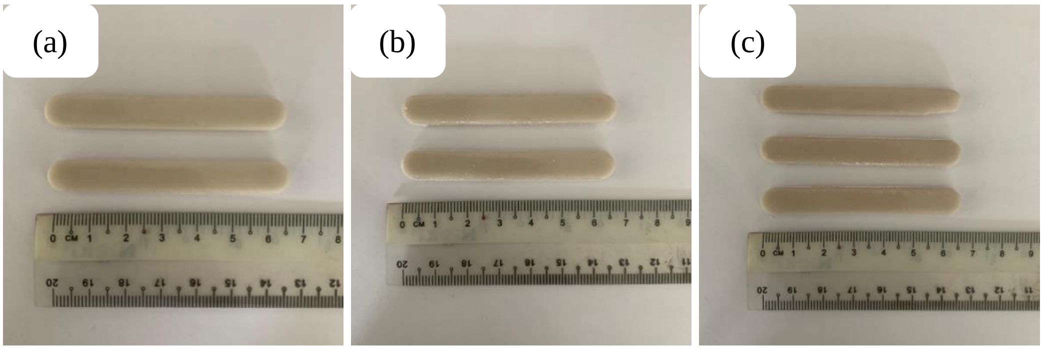


Figure 3.12: Sintered bar shape samples with different CB contents: (a) 3 %, (b) 5 %, (c) 10 %.

3.5 Testing of Powder and Porous Ceramics

Porosity, density, and mechanical properties such as strength were used to evaluate the performance of porous ceramic materials. The investigation also included flexural strength tests and a scanning electron microscope (SEM). CB at concentrations of 0 %, 3 %, 5 %, and 10 % were used in this study to evaluate the features of porous ceramic.

3.5.1 X-Ray Diffraction Analysis (XRD)

The study of a substance's crystal structure, known as XRD analysis, was used to determine the crystalline phases present in a material and, as a result, to reveal information about its chemical composition. It was able to identify the stages of the process by comparing the data recorded with that found in reference databases.

The scattered intensity of an x-ray beam was measured as a function of the direction from which the beam was emitted in this test method. The scattering pattern, also known as a diffraction pattern, can be seen after the beam has been separated, revealing the crystalline structure of the substance being investigated in the experiment. The Rietveld refinement method was then used to establish the crystal structure that is most likely responsible for the pattern that has so far been observed by PANalytical X’Pert PRO as shown in Figure 3.13.

X-ray diffraction is a technique that can be used to figure out the composition of minerals, polymers, corrosion products, and other unknown materials, among other things. Element uses powder diffraction on finely powdered particles at its testing facility to evaluate the vast majority of products.

Figure 3.13: PANalytical X’Pert PRO Serial No. DY2706

3.5.2 Porosity and Density (Archimedes’ Principle)

The buoyant force exerted by a submerged item was equal to the weight of the fluid displaced by the object, according to Archimedes' Principle. The Archimedes buoyancy technique was used to determine apparent porosity and density. Wa represents dry mass, Wc represents saturated mass, and Wb represents mass suspended in water.

The dry mass (Wa) of sample’s was measured using a precision balance A&D FZ-300i-EC as shown in Figure 3.10 after heating with an accuracy of 0.0001 g. For a few minutes, the samples were placed in distilled water. The saturated mass (Wc) and mass suspended in water (Wb) were measured using the same scale after impregnation. The data was evaluated in order to determine the apparent porosity and density formula.

Figure 3.14: A&D FZ-300i-EC Precision Balance

3.5.3 Scanning Electron Microscope (SEM)

Scanning electron microscopy (SEM) was a complex technique for collecting images of material microstructures. Due to the potential of gas molecules to disturb the electron beam and the secondary and backscattered electrons required for imaging, SEM is usually performed in a vacuum environment. The microstructure pictures were captured using a JEOL JSM-6010PLUS/LV as shown in Figure 3.17 to characterize the samples in this study.

Top surface and cross section samples were tested in this testing. Before started the SEM, a few steps of preparation the sample such as grinding, polishing and thermal etching. According to Figure 3.15 (a), (b) and (c), the grit sizes used to grind each sample were 1000 Cw, 1500 Cw, and 2000 Cw. Following the grit size of adhesive paper, the grinding procedure was carried out and must carry out this procedure to ensure that the sample's surface is free from scratches. For each sample, a brief period of grinding was made. The samples were then polished using a polishing micro pad and 0.05 µm of $Al_{2}0_{3}$ as shown in Figure 3.15 (d).

Following the completion of the grinding and polishing, the drying process took place in the oven at a temperature of 60 °C in less than a minute. All samples were placed in the furnace for the thermal etching process once drying was complete. As shown in Figure 3.16, this procedure involved soaking for one minute at a temperature of 1100 °C. Before scanning, samples must be dried and cleaned. An adhesive tab affixed to the aluminium stub was used to mount the sample. Run the vent process to ensure the vacuum chamber were ventilated. When it finished, placed the samples in the sample holder and close the chamber door. After then, leave the pump running until it reaches its maximum vacuum level. The samples were ready to be put to the test.

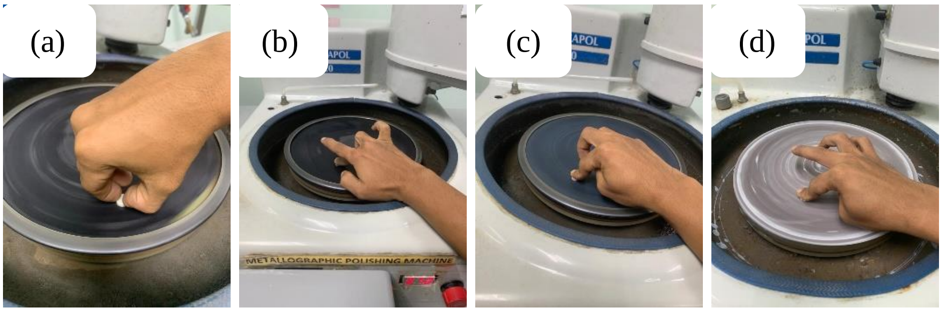


Figure 3.15: Grinding and polishing with different grit size: (a) 1000 Cw, (b) 1500 Cw, (c) 2000 Cw, (d) Polishing with Al_20_3

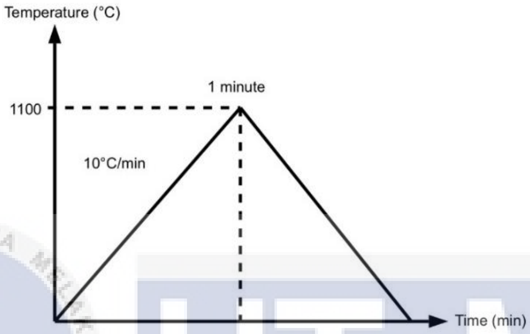


Figure 3.16: The thermal etching profile at 1100 °C for SEM

Figure 3.17: JEOL JSM-6010PLUS/LV Model

3.5.4 Flexural Strength Testing

A flexural test, also known as a bending tensile test was a method of determining the flexural strength and other significant properties of materials. Plastics, fiber-reinforced plastics (FRP), metals, and ceramic materials were subjected to destructive materials testing. The sequence of flexural tests is comparable. The amount of pressure points and the support of the test sample was varied, but in this case, a 3-point flexural test was used as shown in Figure 3.18.

The middle of the checking fixture was filled with standardized, typically cylindrical samples for flexural testing. The rounded support rollers (bearings) were spaced at a specific distance from each other (support width). The bearing's support width and the diameter of the cylindrical sample was proportionate. The sample was loaded with increasing force until it breaks or reaches the previously set deformation by the test punch, which goes down gently and at a consistent speed. Breaking force refers to the maximum load applied during a flexural test.

Figure 3.18: Shimadzu AG100kN-X Plus

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

The results of the flexural test, Scanning Electron Microscopy (SEM), and Microstructure Microscope analysis tests were shown in this chapter. In addition, the Shimadzu AG100kN-X plus was used to perform a three-point flexural test with a maximum force of 100 kN. Lastly, for porosity and density test was applied by using Archimedes' Principle and an XRD analysis. In this chapter, there are a few data achieved from the flexural strength test, SEM and Microstructure Microscope analysis that was analyzed.

4.2 Optimization of Sintering Temperature

Initially, the exact temperature to heat up the samples was not determined due to various melting point of raw material used in the sample. Therefore, the sample was burnt in a furnace at a temperature of 1075 °C but it was unsuccessful as the sample's surface being in a powdery state. Furthermore, when the sample in an unsuitable temperature condition, it affected its weak point structure, making it easily breakable. In order to address this problem, rising the temperature to 1250 °C was a solution. According to Table 4.1, the sample changed its shape from disc to oval but the sample's surface was bright and smooth compared with other samples burnt in lower temperature. Sample without CB served as a reference temperature to burn other samples containing of CB.

Table 4.1: Physical changes on ceramic samples with different sintering temperature

No.	Sample specification	Before sintering	1075°C	1200°C	1250°C
1.	Image	 (a)	 (b)	 (c)	 (d)
2.	Diameter	13.08mm (initial)	12.42mm (-5.05%)	10.70mm (-18.20%)	7.65mm (-41.51%)
3.	Thickness	4.82mm (initial)	4.72mm (-2.07%)	4.10mm (-14.94%)	5.70mm (18.26%)
4.	Surface	Powdery	Powdery	Not powdery	Not powdery
5.	Weight (g)	0.871 (-8.32%)	0.842 (-11.37%)	0.849 (-10.63%)	0.747 (-21.37%)
6.	Shape	Disc	Disc	Disc	Oval
7.	Remarks	The sample in powder conditions that easily occur debris or break	The sample still in a dusty and rough state on the surface	Significant rate of contraction and a rather smooth surface on the sample	The sample is in a very shiny state but has changed its original shape

One of the few noticeable changes at the sample burnt at the different temperature is the dimensions such as diameter, thickness and shape. As the temperature used to burnt the sample increases, there was occurrence of shrinking. According to Table 4.1, the diameter of sample burnt at temperature 1075 °C is 12.42 mm while the temperature at 1250 °C is 7.65 mm. This shows a reduction of 38 % of firing shrinkage in diameter length when compared with sample sintered at 1075 °C. The sample shrinks for a variety of reasons. First, the shrinkage of the sample is due to water loss during firing. Clay typically contains 20 % water and 9 % organic material. Even while clay feels dry before going into the kiln, it really contains some water (Marie, 2022).

This water is both residual moisture and water that has chemically bound to the clay particles. The water that is chemically bound to the clay is forced out of it as it is burnt, and any remaining moisture evaporates. This drying out process causes the clay to contract. Additionally, the shrinkage is a result of both chemical and physical changes. The clay loses some of its volume as a result of the burning off of organic material during the fire process. During the burning process, clay also goes through a vitrification process.

Based on the Table 4.1, the weight of sample burnt at temperature 1075 °C is 0.842 g while the temperature at 1250 °C is 0.747 g. When compared to a sample sintered at 1075 °C, this indicates a reduction of 11 % in the weight of the sample during sintering. There were several factors that led to the sample shrinking. Weight changes mostly result from the vaporization of water and carbon dioxide from the basic materials. The majority of weight loss in samples exposed to temperatures up to about 600 °C is attributed to water evaporation, but weight loss at temperatures over 600 °C is a result of both evaporation and the breakdown of the material’s contents. Based on preliminary experiment and observation from the data, the suitable temperature for sintering samples were 1200 °C.

Table 4.2: Physical changes of bar shape samples

Percentage of CB	Type of sample	Original size (mm) $l \times w \times h$	Sintered size (mm) $l \times w \times h$	Percentage of shrinking
0 %	Sample 1	75 × 10 × 4.8	66.4 × 8.9 × 3.9	-35.98 %
	Sample 2	75 × 10 × 4.9	66.5 × 9.0 × 3.8	-38.11 %
3 %	Sample 1	75 × 10 × 4.7	66.6 × 9.1 × 3.8	-34.67 %
	Sample 2	75 × 10 × 4.8	66.3 × 9.0 × 3.9	-35.36 %
5 %	Sample 1	75 × 10 × 4.8	66.1 × 8.9 × 3.8	-37.90 %
	Sample 2	75 × 10 × 4.9	66.1 × 9.0 × 3.8	-38.49 %
10 %	Sample 1	75 × 10 × 4.8	66.2 × 9.1 × 3.9	-35.98 %
	Sample 2	75 × 10 × 4.7	66.0 × 9.2 × 3.9	-32.82 %

According to Table 4.2, all samples were sintered in a furnace at a temperature of 1200 °C, and sample 2 with 5 % of CB has the highest shrinkage rate, which is 38.49 %. With a shrinkage percentage rate of 38.11 %, sample 2 for 0 % of CB behind sample 2 for 5 % of CB, nevertheless. The sample with the lowest shrinkage percentage (32.82 %) for 10 % of CB is sample 2. The samples shrank as a result of a number of circumstances. The evaporation of water and carbon dioxide from the raw materials is primarily responsible for changes in sample size. In addition, the grain size was increased during the sintering process by filling in the existing pores and strengthening the bond between the elements in the samples.

As a result of decreasing the vapour-solid interfaces of the particles, the sintering process driving force is the reduction of surface energy of the constituents. The part becomes denser and its mechanical characteristics are improved as a result of the pores reducing or even closing during the diffusion process.

4.3 Results and Analysis of Experimental Data

The sample's density, porosity and water absorption were all measured using the Archimedes method. SEM was utilized to examine the microstructural characteristics of the surfaces of sintered samples as well as to describe the materials as-received. The mechanical strength was assessed using three-point flexural test measurements. Shimadzu AG 100kN-X plus was used to conduct tests on bar shape made from sintered samples.

4.3.1 Peak Formation of Ceramics by X-ray Diffraction Analysis (XRD)

XRD was used to analyze the phase and structure of ceramics. An XRD pattern of porous ceramics made with 0 %, 3 %, 5 % and 10 % of CB were depicted. As can be observed, the XRD patterns of common ceramics including kaolinite ($Al_2O_3.2Si_2O_2.\ 2H_2O$), silica (SiO_2), and feldspar ($KAlSi_3O_8$) were easily identifiable. This proves that the primary ceramic structure and phase were successfully created following sintering. However, it was also discovered that a phase with modest peaks and an unknown phase was present. It is thought that the unidentified phase might be the intermediary phase of the particular ceramic component.

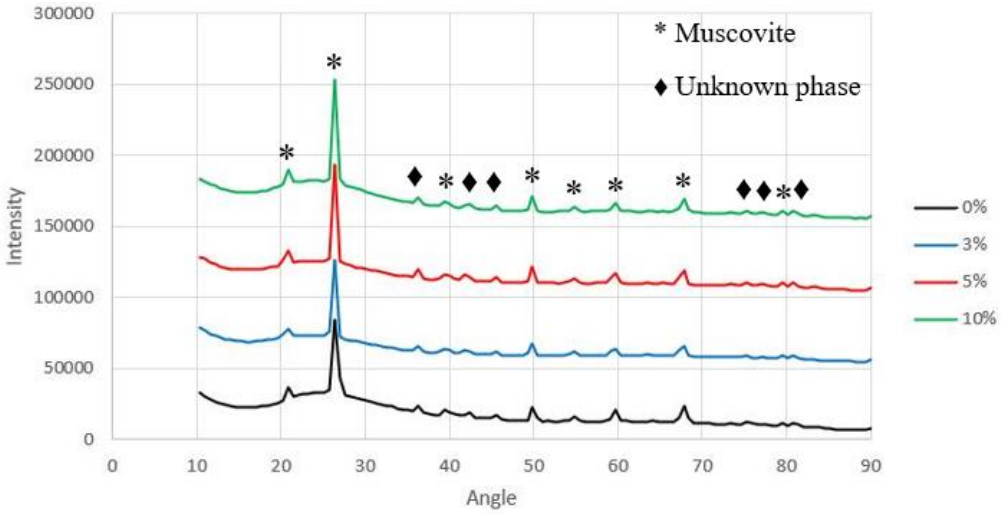


Figure 4.1: XRD patterns of ceramics with different CB contents

The peak creation of ceramics with various CB contents is depicted in Figure 4.1. As can be seen, the muscovite phase is represented by a few of peaks with significant intensities, while the rest peaks are of undetermined phase. The strongest peak can be plainly seen at a 26° of 2θ angle, suggesting the presence of potassium and aluminium in the form of silicate minerals. Additionally, from samples with 3 % CB concentration to those with 10 % CB content, the trend of peak formation is relatively constant across all samples. This research demonstrated that peak formation is not much impacted by rising CB concentrations. However, there is a minor difference between samples with and without CB in terms of peak intensity, especially at 26° of 2θ angle.

4.3.2 Relation of Porosity and Density

The porosity and density of the samples were calculated using Archimedes' Principle. Two analyses for Wa, Wb, and Wc were performed on each sample of CB with concentrations of 0 %, 3 %, 5 %, and 10 %. The gathered information was examined in order to use the apparent porosity and density formula.

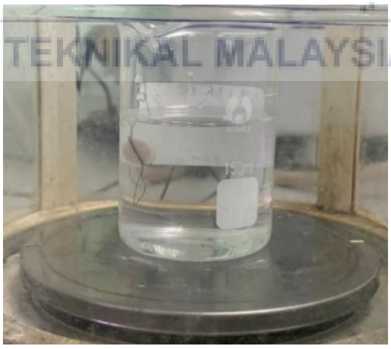


Figure 4.2: Sample testing using Archimedes' Principle

The apparent porosity of each sample was then calculated by multiplying the result by Archimedes' basic formula.

$$P = \frac{Wc - Wa}{Wc - Wb} \times 100$$

Where, P is the apparent porosity, Wa is the dry mass, Wc is the saturated mass and Wb is mass suspended in water.

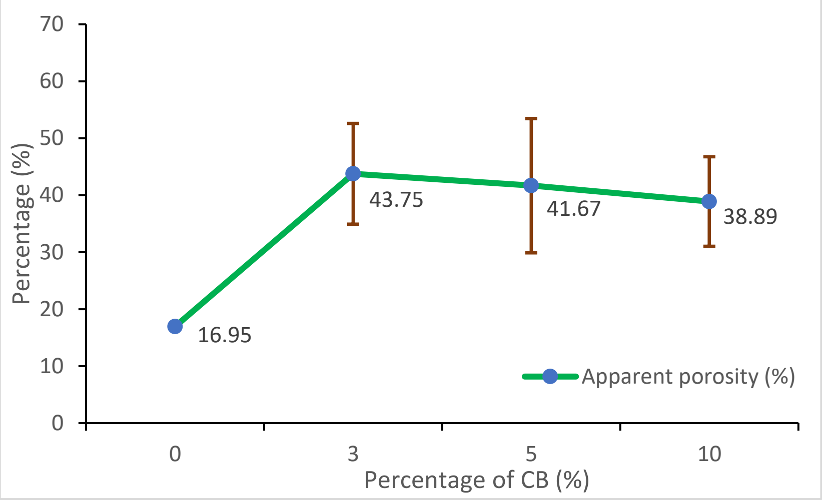


Figure 4.3: The percentage of apparent porosity in different contents of CB

Each CB sample had concentrations of 0 %, 3 %, 5 %, and 10 % of Wa, Wb, and Wc that were twice evaluated. The sample used to measure apparent porosity is depicted in the water in Figure 4.2. The greatest value of all the samples examined is 43.75 % for 3 % of CB, according to Figure 4.3, while the apparent porosity for 0 % of CB is 16.95 %. The 5 % of CB sample has decreased to 41.67 %, nevertheless it is still less than the 3 % mark. The reading for apparent porosity percentage, which was 38.89 % in the sample with 10% CB, decreased in a similar manner.

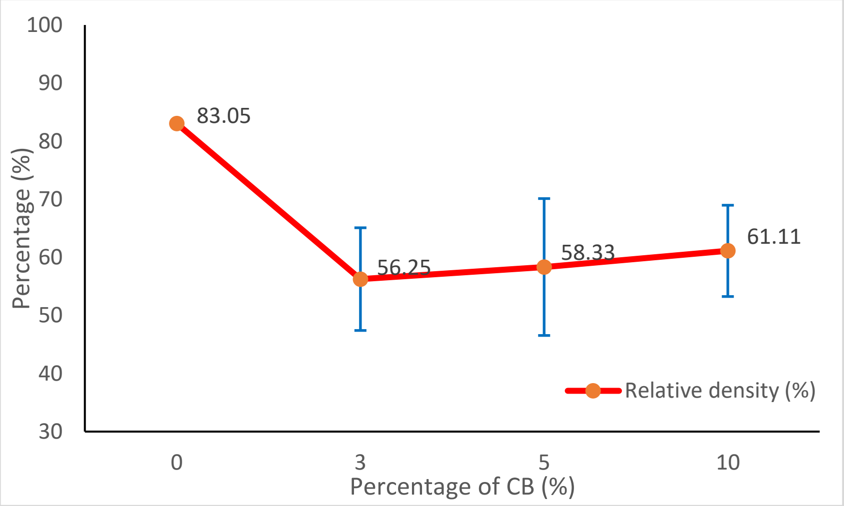


Figure 4.4: The percentage of relative density in different CB contents

According to the graph in Figure 4.3, the percentage for 0 % of CB is the lowest and has substantially increased when having 3 % of CB, but has reduced steadily as the percentage of CB increases. Based on graph in Figure 4.4, the relative density of 0 % of CB is the highest value compared to others sample but for 3 % of CB was proportional decrease. However, sample 5 % and 10 % of CB were higher than 3 % of CB in the relative density. In addition, the value of standard deviation for 5 % of CB was the highest compared to others sample. The amount of CB utilized in the composite is one of the elements responsible for the apparent porosity seen in the samples. By referring to Appendix B, the result of this testing was compiled.

4.3.3 Scanning Electron Microscopy (SEM)

The microstructure image of the samples used in this investigation was taken with a JEOL JSM-6010PLUS/LV Serial No. MP1010000530053 to characterize them with a 25 kV acceleration voltage and a working distance of roughly 10.0 mm.

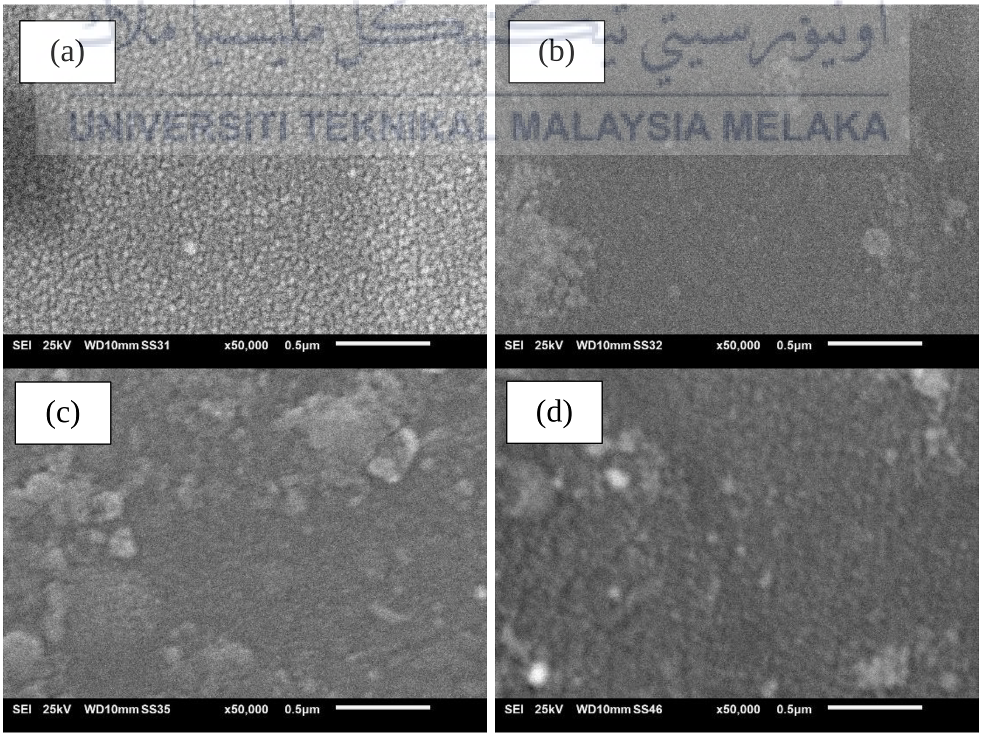


Figure 4.5: SEM images (top surfaces) of sintered samples with different CB contents: (a) 0 %, (b) 3 %, (c) 5 % and (d) 10 %.

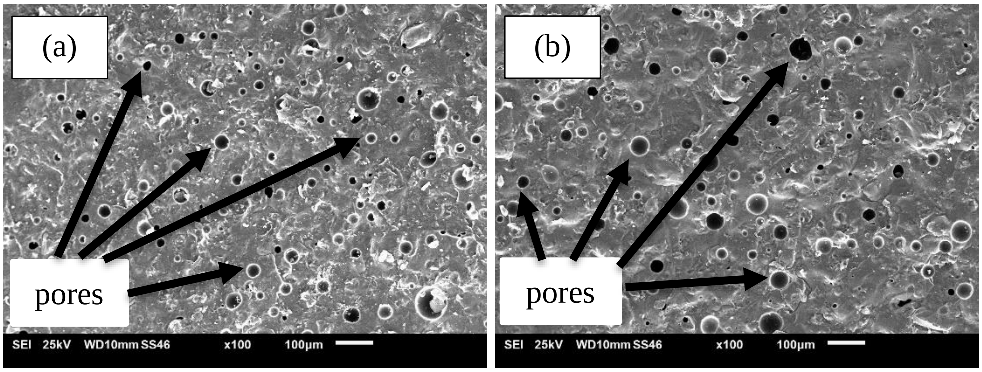


Figure 4.6: SEM images (cross section) of sintered samples with different CB contents: (a) 0 % and (b) 10 %.

The SEM image of the sintered CB samples at 1200 °C is shown in Figures 4.5 (top surface) and Figure 4.6 (cross section). The material was successfully integrated, as can be observed. Sample (a) in Figure 4.5 is on the top surface and exhibits lower grain size. The sample in (a) (0% without CB, Figure 4.6) had a compact structure with a low level of porosity and uniform micro-pore distribution, but when 10% more CB was added, larger pores began to emerge in Figure 4.6 (b). Pores in porous ceramics were primarily created by CB microspheres losing their shape at high temperatures.

Therefore, the apparent porosity of the material increased as the number of CB microspheres increased. Additionally, the apparent porosity of porous ceramics increased to more than 21 % when the addition levels of CB were increased from 0 % to 10 %. Therefore, it might be concluded that CB can ensure high porosity in porous ceramics acting as pore-forming agents. Figure 4.6 illustrates how the sample's macroscopic structure is still present after the CB microspheres have burned away.

4.3.4 Flexural Strength Test

The mechanical strength was assessed using three-point flexural test measurements. To assess the impact of pore structure on the strength of the samples, bars manufactured from sintered samples with porosities ranging from 16 % to 43 % were subjected to testing using the Shimadzu AG-100kN-X plus. This machine could handle a maximum load of 100 kN. This testing was conducted at a 1 mm/min pace. Each CB sample underwent this experiment twice, with concentrations of 0 %, 3 %, 5 %, and 10 % of CB. By referring to Appendix C, the results of these mechanical tests were compiled.

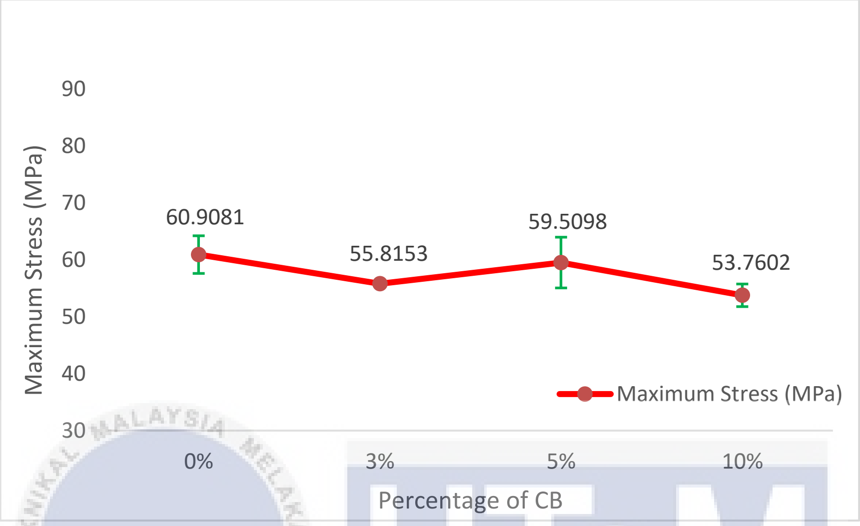


Figure 4.7: The result of flexural strength test with different percentage of CB

Flexural strength of sintered samples with and without CB addition is shown in Figure 4.7. The sample without CB had the maximum strength, 60.9081 MPa at 1200 °C. The strength of sintered samples decreased from 60 MPa to 55 MPa on average as a result of the presence of CB. It is generally known that a high porosity caused a decrease in mechanical characteristics. In this instance, the densification of sintered samples involved the liquid phase sintering process and CB breakdown. The CB particles initially totally disintegrated above 550 °C, which increased the size of the holes in the samples. In general, a sample's strength will decline as its porosity increases.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

In this experiment, the porous ceramics with micro-pores and microstructure were successfully fabricated using CB as the PFA’s. Ceramics porosity, mechanical, and physical properties were assessed, as well as the impact of the PFA’s composition, content, and particle size distribution. The development of porosity in ceramic composites for the preparation and manufacturing of porous ceramics is the main focus of this research. The findings demonstrate that porosity levels on the ceramic can be easily regulated by the quantity of PFA. In actuality, it was found that the amount of PFA incorporation correlated roughly linearly with the sample's open porosity. The number of closed pores was generally decreased and more open porosity was produced by the PFA. The PFA's particle size distribution had no effect on the amount of porosity, but it did have a significant impact on the number of pores, as well as their size and quantity.

The addition of CB in the sintered sample were enhanced the porosity of samples fired at 1200 °C comparing to the samples without added CB at the same temperature. Furthermore, PFA's successfully help form and increase pores. This is clearly proven in the SEM image on the cross section with and without of CB. Pores in porous ceramics were primarily created by CB microspheres losing their shape at high temperatures. The strongest sample was the one without CB, measuring 60.9081 MPa at 1200 °C. Due to the presence of CB, the strength of sintered samples dropped from 59 MPa to 53 MPa based on the percentage of CB. It is well-known that a high porosity led to a reduction in the sample's mechanical properties, including strength. However, because the CB were burned off below the sintering temperature and the pores were created, the CB also did not affect any tests or results that were also clearly demonstrated in SEM.

5.2 Recommendations

There are various recommendations for potential future enhancements to help this research improve and generate better outcomes. Future researchers who are interested in this study can improve it by utilising different PFA’s or materials that can increase the sample's strength, like glass fibre. However, the researcher also can improve by using either cold isostatic pressing (CIP) or hot isostatic pressing (HIP) when pressing the samples.

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APPENDICES

APPENDIX A Calculation for weight materials

Clay (Kaolinite) + Silica (Silicon Dioxide) + Feldspar (Microcline)

$Al_2O_3.2Si_2O_2.\ 2H_2O + SiO_2 + KAlSi_3O_8$

= [2(13) + 3(8) + 2(2+8)] + [16+16] + [19+13+52+64]

= 310g

$$\frac{Clay}{Clay + Silica + Feldspar} \times 70g$$

Clay = $\frac{110}{310} \times 70g = 24.84g$

Feldspar = $\frac{180}{310} \times 70g = 40.64g$

Silica = $\frac{20}{310} \times 70g = 4.51g$

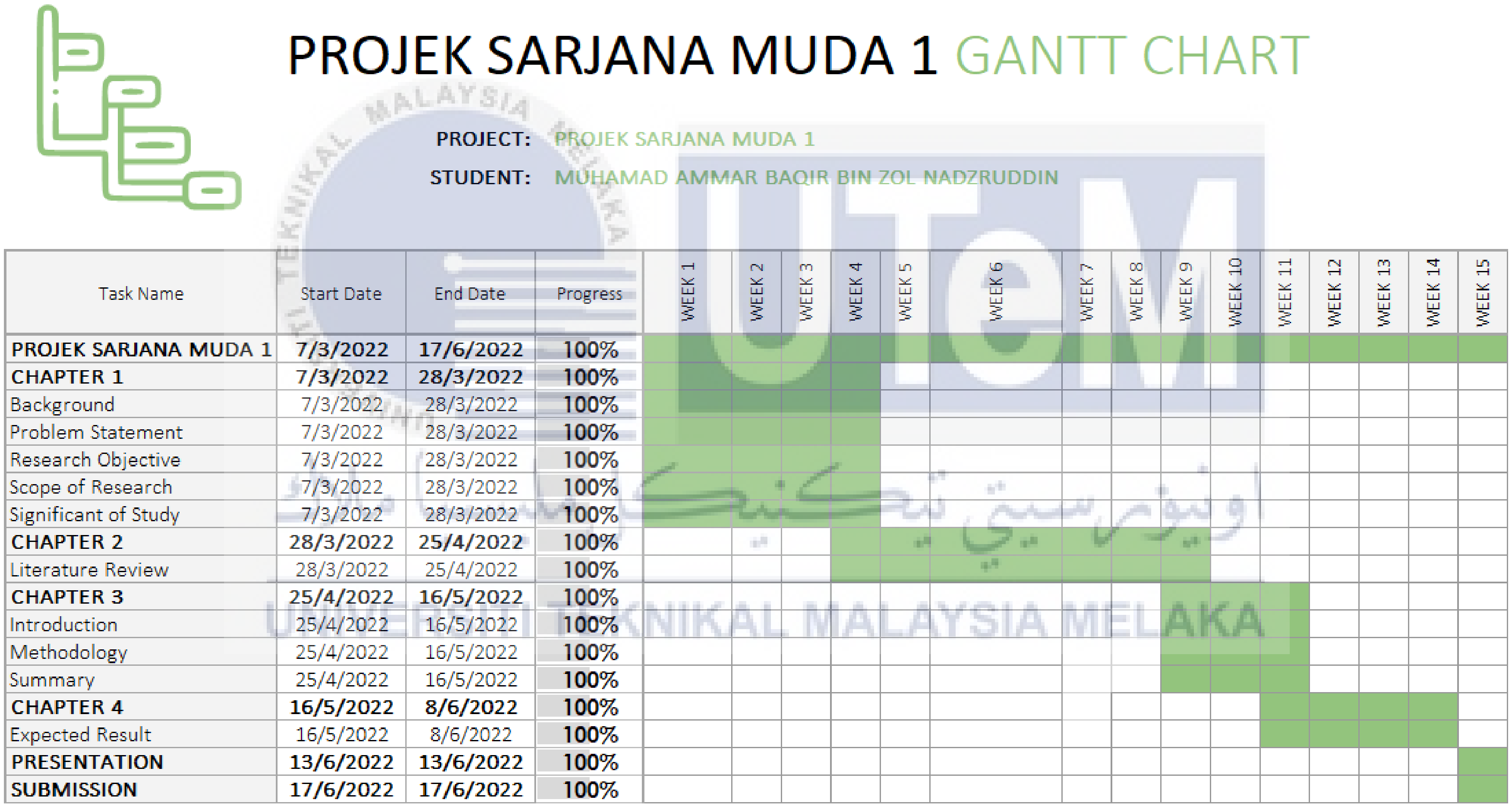
APPENDIX B Results of relative density and apparent porosity with different contents of CB

Percentage of CB	Type of sample	Dry mass (W_a)	Mass suspended in water (W_b)	Saturated mass (W_c)	Apparent porosity	Relative density	Average apparent porosity	Average relative density	Standard deviation
0%	Sample 1	0.842g	0.827g	0.845g	16.67%	83.33%	16.96%	83.05%	0.004
	Sample 2	0.848g	0.824g	0.853g	17.24%	82.76%			
3%	Sample 1	0.865g	0.861g	0.869g	50%	50%	43.75%	56.25%	0.088
	Sample 2	0.871g	0.866g	0.874g	37.50%	62.50%			
5%	Sample 1	0.862g	0.856g	0.865g	33.33%	66.67%	41.67%	58.34%	0.118
	Sample 2	0.880g	0.876g	0.884g	50%	50%			
10%	Sample 1	0.850g	0.842g	0.854g	33.33%	66.67%	38.59%	61.12%	0.079
	Sample 2	0.843g	0.838g	0.847g	44.44%	55.56%			

APPENDIX C Results of flexural strength test for different contents of CB

Percentage of CB	Type of sample	Maximum force (N)	Average maximum force (N)	Maximum stress (MPa)	Average maximum stress (MPa)	Elastic force (MPa)	Average elastic force (MPa)
0%	Sample 1	85.8466	83.2782	63.2453	60.9081	2261.61	2033.17
	Sample 2	80.7098		58.5708		1804.72	
3%	Sample 1	75.5469	75.7615	55.6572	55.8153	2162.90	2068.91
	Sample 2	75.9761		55.9734		1974.91	
5%	Sample 1	76.5006	80.7763	56.3598	59.5098	621.42	1369.00
	Sample 2	85.0519		62.6598		2116.58	
10%	Sample 1	74.8793	72.9720	55.1654	53.7602	863.73	1122.88
	Sample 2	71.0646		52.3550		1382.02	

APPENDIX D Gantt Chart for PSM 1



PROJEK SARJANA MUDA 1 GANTT CHART

PROJECT: PROJEK SARJANA MUDA 1

STUDENT: MUHAMAD AMMAR BAQIR BIN ZOL NADZRUDDIN

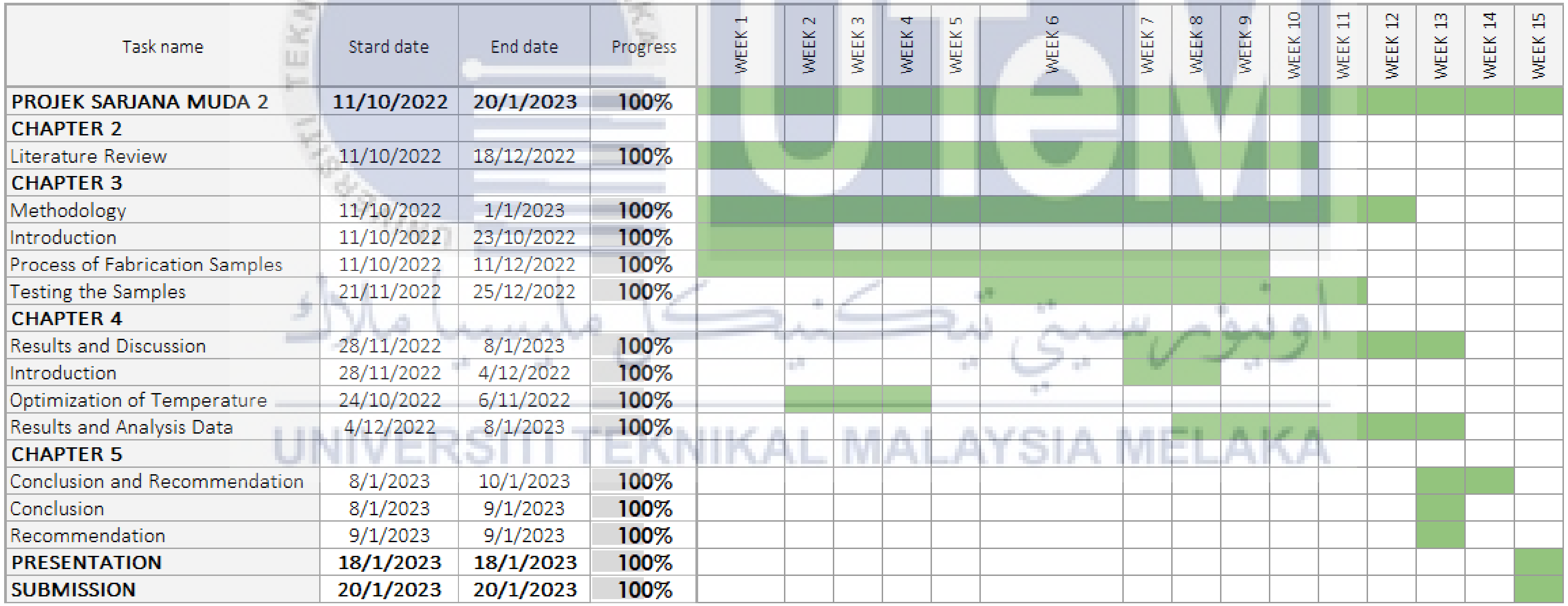
Task Name	Start Date	End Date	Progress	WEEK 1	WEEK 2	WEEK 3	WEEK 4	WEEK 5	WEEK 6	WEEK 7	WEEK 8	WEEK 9	WEEK 10	WEEK 11	WEEK 12	WEEK 13	WEEK 14	WEEK 15
PROJEK SARJANA MUDA 1	**7/3/2022**	**17/6/2022**	**100%**	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩
CHAPTER 1	**7/3/2022**	**28/3/2022**	**100%**	🟩	🟩	🟩	🟩											
Background	7/3/2022	28/3/2022	100%	🟩	🟩	🟩	🟩											
Problem Statement	7/3/2022	28/3/2022	100%	🟩	🟩	🟩	🟩											
Research Objective	7/3/2022	28/3/2022	100%	🟩	🟩	🟩	🟩											
Scope of Research	7/3/2022	28/3/2022	100%	🟩	🟩	🟩	🟩											
Significant of Study	7/3/2022	28/3/2022	100%	🟩	🟩	🟩	🟩											
CHAPTER 2	**28/3/2022**	**25/4/2022**	**100%**				🟩	🟩	🟩	🟩	🟩	🟩						
Literature Review	28/3/2022	25/4/2022	100%				🟩	🟩	🟩	🟩	🟩	🟩						
CHAPTER 3	**25/4/2022**	**16/5/2022**	**100%**									🟩	🟩	🟩				
Introduction	25/4/2022	16/5/2022	100%									🟩	🟩	🟩				
Methodology	25/4/2022	16/5/2022	100%									🟩	🟩	🟩				
Summary	25/4/2022	16/5/2022	100%									🟩	🟩	🟩				
CHAPTER 4	**16/5/2022**	**8/6/2022**	**100%**											🟩	🟩	🟩	🟩	
Expected Result	16/5/2022	8/6/2022	100%											🟩	🟩	🟩	🟩	
PRESENTATION	**13/6/2022**	**13/6/2022**	**100%**															🟩
SUBMISSION	**17/6/2022**	**17/6/2022**	**100%**															🟩

APPENDIX E Gantt Chart for PSM 2

PROJEK SARJANA MUDA 2 GANTT CHART

PROJECT: PROJEK SARJANA MUDA 2

STUDENT: MUHAMAD AMMAR BAQIR BIN ZOL NADZRUDDIN



Task name	Stard date	End date	Progress	WEEK 1	WEEK 2	WEEK 3	WEEK 4	WEEK 5	WEEK 6	WEEK 7	WEEK 8	WEEK 9	WEEK 10	WEEK 11	WEEK 12	WEEK 13	WEEK 14	WEEK 15
PROJEK SARJANA MUDA 2	**11/10/2022**	**20/1/2023**	**100%**	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩
CHAPTER 2																		
Literature Review	11/10/2022	18/12/2022	100%	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩					
CHAPTER 3																		
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Introduction	11/10/2022	23/10/2022	100%	🟩	🟩													
Process of Fabrication Samples	11/10/2022	11/12/2022	100%	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩						
Testing the Samples	21/11/2022	25/12/2022	100%						🟩	🟩	🟩	🟩	🟩	🟩				
CHAPTER 4																		
Results and Discussion	28/11/2022	8/1/2023	100%							🟩	🟩	🟩	🟩	🟩	🟩	🟩		
Introduction	28/11/2022	4/12/2022	100%							🟩	🟩							
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Results and Analysis Data	4/12/2022	8/1/2023	100%								🟩	🟩	🟩	🟩	🟩	🟩		
CHAPTER 5																		
Conclusion and Recommendation	8/1/2023	10/1/2023	100%													🟩	🟩	
Conclusion	8/1/2023	9/1/2023	100%													🟩		
Recommendation	9/1/2023	9/1/2023	100%													🟩		
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20 January 2023
Date