Synthesis of Barium Titanate using Microwave Heating Method at Different Heating Time

by

AIDI ADAM BIN MOHD RAOF
J20A0409

A proposal submitted in fulfillment of the requirements for the degree of Bachelor of Applied Science (Materials Technology) with Honours

FACULTY OF BIOENGINEERING AND TECHNOLOGY UMK

2023

DECLARATION

I declare that this thesis entitled "SYNTHESIS AND CHARACTERIZATION OF BARIUM TITANATE USING MICROWAVE HEATING METHOD" is the result of my own research except as cited in the reference. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature:

Name: Aidi Adam Bin Mohd Raof

No.matric: J20A0409

Date: 29/2/2024

Signature:

Name: Associate Prof. Dr Muhammad Azwadi Bin Sulaiman

Stamp:

Assoc. Prof. Dr. Muhammad Azwadi Sulaiman
Senior Lecturer
Faculty of Bioengineering and Technology,
Universiti Malaysia Kelantan

Date: 29/2/2024

ACKNOWLEDGEMENT

Bismillahirrahmanirrahim. In the name of Allah, The Most Beneficent and The Most Merciful. First and foremost, I want to thank Allah SWT for providing me with the health and strength I needed to finish the final project. My sincere gratitude also goes out to the Faculty of Biotechnology and Engineering at Universiti Malaysia Kelantan and particularly to Associate Prof. Dr. Muhammad Azwadi bin Sulaiman, who had been a very helpful supervisor who guided, supervised, and gave me many insightful advice over the course of the project.

In addition, I would like to thank my UMK friends Muhamad Afif Zaafarani Bin Muhamad Aidi for his unwavering support and assistance in helping me finish this project. I would also like to thank to lab assistants, Ts Pn Hanisah Izzati and En.Qamal who has assisted me throughout the experiment.

Lastly, I would like to express my sincere gratitude to my parents, Mr. Mohd Raof Bin Mohd Nasir and Mrs. Rusnah Binti Kassim, for their support throughout this project both in financially, morally and for their understanding and unwavering love.

ABSTRACT

Barium titanate (BaTiO3) is a ceramic material with significant applications in electronic, optical, and energy-related fields due to its exceptional dielectric constant, ferroelectric, and piezoelectric properties. But conventional synthesis techniques often produce low-purity materials at high temperatures and with hazardous chemicals. Microwave-aided synthesis has surfaced as a potentially advantageous substitute, providing enhanced purity, quicker reaction rates, and larger yields. The primary objectives of this work are to synthesise and characterise barium titanate by means of microwave heating. The goals include figuring out the microstructure, dielectric constant, and crystallographic phases of BaTiO3, as well as investigating how radiation duration affects this formation. Titanium oxide and barium carbonate were combined, ground, dried, and microwave-heated. After that, the material was sintered and packed into pallets. The synthesised materials were analysed using characterization methods including as optical microscopy (OM), X-ray diffraction (XRD), density and porosity studies, and dielectric testing. The results demonstrate that microwave heating is a sustainable and effective method for manufacturing high-quality barium titanate, which has potential uses across numerous sectors. This advances the development of synthetic procedures for complicated oxide materials.

ABSTRAK

Barium titanate ($BaTiO_3$) ialah bahan seramik dengan aplikasi penting dalam bidang elektronik, optikal dan berkaitan tenaga kerana sifat pemalar dielektrik, feroelektrik dan piezoelektrik yang luar biasa. Tetapi teknik sintesis konvensional sering menghasilkan bahan ketulenan rendah pada suhu tinggi dan dengan bahan kimia berbahaya. Sintesis bantuan gelombang mikro telah muncul sebagai pengganti yang berpotensi berfaedah, memberikan ketulenan yang dipertingkatkan, kadar tindak balas yang lebih cepat dan hasil yang lebih besar. Objektif utama kerja ini adalah untuk mensintesis dan mencirikan barium titanat melalui pemanasan gelombang mikro. Matlamatnya termasuk memikirkan struktur mikro, pemalar dielektrik, dan fasa kristalografi $BaTiO_3$, serta menyiasat bagaimana tempoh sinaran mempengaruhi pembentukan ini. Titanium oksida dan barium karbonat telah digabungkan, dikisar, dikeringkan, dan dipanaskan dengan gelombang mikro. Selepas itu, bahan itu disinter dan dibungkus ke dalam palet. Bahan yang disintesis dianalisis menggunakan kaedah pencirian termasuk sebagai mikroskop optik (OM), pembelauan sinar-X (XRD), kajian ketumpatan dan keliangan, dan ujian dielektrik. Hasilnya menunjukkan bahawa pemanasan gelombang mikro ialah kaedah yang mampan dan berkesan untuk menghasilkan barium titanat berkualiti tinggi, yang mempunyai potensi kegunaan merentas pelbagai sektor. Ini memajukan pembangunan prosedur sintetik untuk bahan oksida yang rumit.

Table of Contents

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

BaTiO3	BariumTitanate
BaCo	Barium Carbonate
TiO2	Titanium Oxide
XRD	X-ray Diffraction
OM	Optical Microscope

LIST OF SYMBOLS

°C	Degree Celsius
%	Percentage
g	Gram
MHz	Mega Heartz

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Barium titanate $BaTiO_3$ is a well-known ceramic material with important applications in electronic, optical, and energy-related fields because to its high dielectric constant, ferroelectric, and piezoelectric properties. These properties make barium titanate a promising material for capacitors, sensors, actuators, and energy storage devices (Carter & Norton, 2013).

However, the traditional methods for synthesizing barium titanate, such as solid-state reaction, hydrothermal synthesis, and sol-gel method, are energy-intensive and often involve the use of toxic or hazardous chemicals, which can pose environmental and health risks. In addition, these methods often result in low purity and poor reproducibility of the synthesized material (Carter & Norton, 2013).

In recent years, microwave-assisted methods have emerged as a promising alternative for synthesizing various ceramic materials, including barium titanate. Microwave-assisted methods use microwave energy to initiate and drive chemical reactions, which results in faster reaction rates, higher product yields, and improved purity compared to traditional methods. In addition, microwave-assisted methods are often healthier for the environment and human health, as they require less energy and produce fewer waste by-products (Carter & Norton, 2013).

Therefore, the synthesis and characterization of barium titanate using a microwave heating method is a topic of growing interest, as it offers a more sustainable and efficient approach to producing high-quality barium titanate. The optimization of the synthesis parameters and the characterization of the resulting material using various techniques can provide valuable insights into the crystal structure, morphology, and vibrational modes of barium titanate, which are important for its technological applications (Carter & Norton, 2013).

1.2 Problem Statement

The synthesis of barium titanate is traditionally carried out at high temperatures, which can lead to the formation of impurities and the consumption of a large amount of energy which is 900 to 1200 °C. Microwave-assisted synthesis has emerged as a promising alternative that can reduce energy consumption and improve the purity of the resulting material. This studied aim to synthesize barium titanate using a heating microwave method and characterize its structural and physical properties.

1.3 Objectives

This research was be carried out with the following objectives:

1. To study the effect of radiation time on the formation of $BaTiO_3$
2. To determine the crystallographic phases of the synthesized barium titanate, and dielectric constant, and the microstructure.

1.4 Scope of Study

The scope of this study is to investigate the feasibility and effectiveness of the microwave heating technique in producing barium titanate. The synthesis will determine the suitable power level, to achieve high-quality barium titanate. The prepared barium titanate will be characterized for microstructure, density, and dielectric properties.

1.5 Significances of Study

The result and discovery from this study are hoped to give contribute to the advancement of synthetic techniques for complex oxide materials. Traditional methods often require high temperatures, long reaction times, and multiple processing steps. The microwave heating method offers a promising alternative that can streamline the synthesis process, reduce energy consumption, and improve overall productivity. The comparative analysis of microwave heating with conventional synthesis methods enables a comprehensive evaluation of the microwave technique's advantages and limitations. It is also expected from this study to prove that can open the way for improved material properties, enhanced device performance, and more sustainable and efficient synthesis processes.

CHAPTER 2

LITERATURE REVIEW

2.1 Ceramic

Ceramics are a kind of inorganic material with properties that stem from the interplay between its metallic and non-metallic constituents. Ceramics are the most flexible material class. Its malleability stems from the fact that its bonds are a mixture of ionic and covalent connections of varying strengths (Misra & Misra, 2022). These features, such as relatively high fusion the temperatures, high modulus, high wear power, poor thermal properties, high hardness and fragilities combined with tenacities, and low ductility, dictate a number of ceramic material characteristics, including bonding. They lack conduction electrons and serve as effective electrical insulators because of the chemical bonds that hold them together (Eliche-Quesada et al., 2019).

There are two primary types of ceramics, namely traditional ceramics and modern ceramics. Traditional ceramics based on silicates include cement, clay products, and refractories. There is a considerable demand for and supply of traditional pottery. Traditional ceramics are often made using clay and other materials mined from the earth (Karimi-Jafari et al., 2017). Advanced ceramics are fabricated from synthetic raw materials that have been subjected to intensive chemical processing in order to achieve a high degree of purity and improve upon their physical qualities. Therefore, sophisticated and forward-thinking methods are used in their production. Applications in magnetism, ferroelectricity, piezoelectricity, and superconductivity are found in a broad variety of ceramics, including carbides, nitrides, borides, pure oxides, and others (Eliche-Quesada et al., 2019).

On the other hand, because they don't corrode, ceramic materials have a significantly longer usable life than other types of materials (Ayode Otitoju et al., 2020). As such, it can be said that ceramic materials have a wide range of uses since they possess extremely distinctive qualities that are unique to them and cannot be found in any other material (Eliche-Quesada et al., 2019).

2.2 Electroceramic

Electroceramics, a kind of high-tech ceramic, are used in several electronic, optical, and magnetic systems. The high electrical resistance of ceramics was a major factor in their first use in the electrical industry. Insulating bodies were developed to protect electrical wires in power lines from the elements. Later on, it became clear that the spectrum of potential qualities was exceedingly broad. Excellent ionic conductors include certain ceramics known as rapid ion conductors, which have received a lot of attention over the past 20 years due to their critical roles in fuel cells, batteries, and sensor technologies (Setter, 2001).

There are several electrical, optical, and magnetic uses for electroceramics. Low dielectric constant electroceramics are utilized to create integrated circuit substrates, whereas high dielectric constant electroceramics are used to create capacitors. Transformer cores can be made of electroceramics with good magnetic characteristics, and transducers for microphones, for example, can be made of electroceramics that display piezoelectricity. Optical phenomena like luminescence and lasing, which are used in fluorescent lighting and lasers, may be seen in some electroceramic materials (Setter, 2001).

2.3 Barium Titanate ($BaTiO_3$)

$BaTiO_3$ is one of the most thoroughly researched ferroelectric materials and is being used in a range of sectors thanks to its outstanding dielectric and ferroelectric characteristics include among others, field-effect transistors, thermistors, electro-optical devices, electromechanical devices, transducers, actuators, and multilayer capacitors. Ferroelectricity of $BaTiO_3$ bulk physical characteristics and crystal structure of $BaTiO_3$ have been the subject of much research. The four crystal phases of cubic, tetragonal, orthorhombic, and rhombohedral bulk $BaTiO_3$ predominate (Jiang et al., 2019). It exhibits a conventional perovskite structure (i.e., cubic phase) at high temperatures 120 ℃, where the Ba_2+ and O_2 ions form the FCC lattice and the smaller Ti_4+ cation sits in the octahedral interstitial spaces, creating (TiO_6) within the FCC array. Because the cation and anion centres of this cubic phase structure coincide, it possesses a high degree of symmetry and lacks ferroelectricity (also known as paraelectricity). The crystal structure undergoes a net spontaneous polarisation and changes from the paraelectric to the ferroelectric phase when the temperature is lowered to below 120 ℃, accompanied by the deviation of the cation centre from the anion centre. $BaTiO_3$ displays a tetragonal structure when the temperature is between 120 ℃ and 51 ℃, where the Ti_4+cation deviates from the centre of the TiO_6 octahedron. This causes a net polarisation along the edge direction since the cation centre is now further away from the anion centre (Jiang et al., 2019).

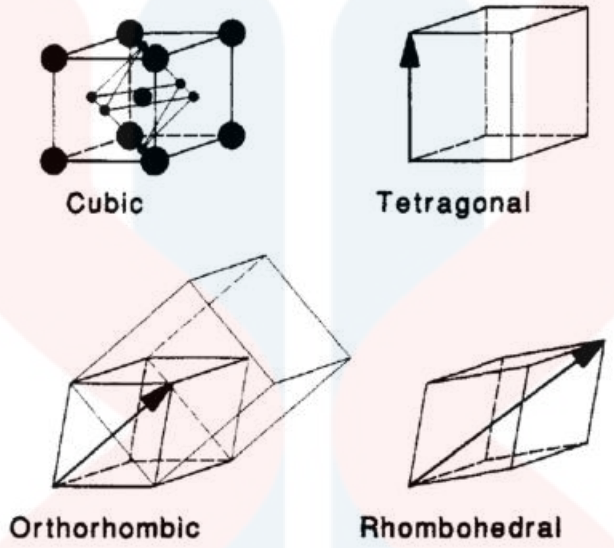


Figure 2.1: Four different crystalline structure of $BaTiO_3$

(Source from: Jiang et al., 2019)

The structure transitions to the orthorhombic phase, which may be thought of as the extension of the cubic unit cell in the face diagonal direction, with additional cooling to between 51 ℃ and 90 ℃. Last but not least, the cubic unit cell elongates along the body diagonal direction at to 90 ℃, signaling the beginning of the rhombohedral phase. With the cation centre diverging from the anion centre as a result of distortions along these axes, the crystal structure exhibits non-symmetry, resulting in ferroelectric phases with a net spontaneous polarisation (Kwei et al., 1993). There has been a lot of interest in the study of the production and property analysis of nanoscopic ferroelectric $BaTiO_3$. To create molecular electronics, such as random access memory and logic circuits, it is necessary to be able to alter the characteristics of nanostructured ferroelectric materials. $BaTiO_3$ nanocrystals (NCs) behave differently from both their bulk counterparts and several other well-known nanocrystals (NCs) systems and their ferroelectric and dielectric characteristics are strongly influenced by the crystal size (Jiang et al., 2019).

Several theories have been put out to further clarify the underlying process of $BaTiO_3$ dielectric and ferroelectric behaviour at the nanoscale. $BaTiO_3$/polymer nanocomposites must also be able to homogeneously scatter within the polymer matrix without phase separation or particle agglomeration for them to be used in practical applications. In contrast to conventional simple mixing methodologies, this has spurred recent considerable research efforts toward the development of surface modification techniques that enable high-quality $BaTiO_3$/polymer nanocomposites with uniform dispersion and favourable morphology (Ferro et al., 2021).

2.4 Application of Barium Titanate

Barium titanate is extensively used as a ferroelectric ceramic, especially in piezoelectric gadgets such undersea transducers, sensors, heaters with positive heat coefficients, and multilayer capacitors (Sufiiarov et al., 2021). Tiny amounts of materials with high dielectric constants and outstanding resistivity are needed for these uses. Barium titanate is an example of a material having a perovskite crystalline structure. Cubic in structure at its Curie temperature of 130 °C, it may also be tetragonal, orthorhombic, or rhombohedral at lower temperatures. Since barium titanate is often produced as a powder, spark plasma sintering (SPS) may prove to be an excellent method of sintering such crystalline structures in comparison to extended conventional sintering techniques, which result in undesired grain formation. Synthesis of the powder, powder preparation (grinding/ball milling), sintering, and annealing at a lower temperature for a longer length of time (5-12 hours) are the four common steps in the production of barium titanate (Buscaglia et al., 2021).

The impact of grain size on the ferroelectric characteristics of barium titanate was examined in one of the earliest research on the sintering of barium titanate samples. It has been demonstrated that samples with typical grain sizes of roughly 1 micrometer (µm) exhibit the greatest values of relative permittivity ′ (6079), piezoelectric coefficient $_{=}$ 519 pC $N-_{1}$, and planar electromechanical coupling factor kp (39.5%). As grain sizes fluctuated, their values changed as well. Consequently, the ferroelectric and piezoelectric characteristics of barium titanate are greatly influenced by regulated grain development (Buscaglia et al., 2021).

2.5 Synthesis of Barium Titanate

Due to the high dielectric constant and ferroelectric characteristics of barium titanate, microwave-based heating is being investigated for application in both its production and sintering. Electrical characteristics and microstructure of $BaTiO_3$ ceramics produced by microwave sintering at temperatures between 1300 and 1400 °C. It showed that high-performance ceramics with a positive temperature coefficient of resistivity may be produced using the microwave sintering method and a dopant that develops donor levels even at high doping levels (Vinothini Venkatachalam & Jon Binner, 2020).

As a consequence, at temperatures over 700 °C, heating in a microwave field totally converts the cubic $BaTiO_3$ into the tetragonal form. Since microwaves have several benefits over traditional heating techniques, including the ability to heat objects quickly and selectively, these experiments used them as a new heating source. Microwaves may be used to process materials, and their frequency can be changed to regulate the quantity of energy given(Vinothini Venkatachalam & Jon Binner, 2020).

Tetragonal barium titanate, $BaTiO_3$, a nanocrystalline material, has been created by Vinothini Venkatachalam & Jon Binner, (2020). They examined the effects of microwave frequency, microwave bandwidth sweep time, and ageing time on the microstructure, particle size, phase purity, surface area, and porosity of the produced $BaTiO_3$ sample using microwaves with frequencies of 3-5.5 GHz and various bandwidth sweep durations (Kashimura, 2016). According to Sugawara (2016), if one wants to understand the chemical mechanisms underlying each of the steps involved in the synthesis, one has to look at the temperature dependency of the microwave absorption characteristics of $BaTiO_3$ particles. To explain the benefits of these processes, it seems sense to determine the microscopic heat distribution. Based on their absorption characteristics, various materials are heated by microwaves in different ways, creating an uneven thermal distribution at the micron level. These heat distributions might cause abnormal chemical behaviours, which have been seen before when ceramics were chemically created from powdered mixes (Vinothini Venkatachalam & Jon Binner, 2020).

CHAPTER 3

MATERIALS AND METHODS

3.1 Introduction

Materials that were use in this research are barium carbonate, which served as the source of barium ions. Titanium oxide, provided titanium ions. Methanol acted as a solvent and a stabilizing agent. Microwave reactor is a specialized vessel designed for microwave heating. Microwave source is a microwave generator capable of delivering the required power. The stirring apparatus is to ensure thorough mixing during the synthesis process. A thermometer is to monitor the temperature during the reaction. Last is analytical instruments, various characterization techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR) can be used to analyse the properties of the synthesized barium titanate (Qian et al., 2020).

3.2 Barium Titanate Preparation

Because barium oxide is a chemically volatile element that highly reactive to surroundings, barium carbonate was used as a starting material to synthesize BaTiO. BaTiO was provided by R&M Chemicals (CAS:513-7-9, 500g) with purity 98.5% purity and analytical reagent (A.R.) grade. The molecular weight for $BaCO_3$ is 84.63 g. The powder has a white colour an actual look. TiO_2 was supplied by R&M

Chemicals (CAS:13463-67-7,500g) with 99.5% purity and analytical reagent (A.R.) grade. The molecular weight of TiO_2 is 34.25 g/mol, and the power appears in white coloured powder.

3.2.1 Mixing and Milling

Reducing particle size and avoiding aggregation will be largely dependent in the mixing and milling procedure. It also aims to produce a very homogeneous blend of basic materials. For compound formation to occur during calcination or burning, the matter of adjacent particles must interdiffusion, and the process completion time will be equal to the square of the particle size. The smaller particles have more surface area than the bigger ones. The smaller size hence has a quicker rate of diffusion than the size. A lab Roll Mill (model no. QM-5) from Magna 33 Value Sdn Bhd was used to mix thoroughly using ball milling in a sealed plastic bottle with Zirconium beads with powder to ball ratios of 1:10 for 24 hours at 120 rpm, giving enough time for mixing.

3.2.2 Drying and Sieving

The grey-colored wet mixture was ground and combined, and it was then left to dry in a glass plate under a fume hood about twenty-four hours. When the combination was totally dry, it was sieved through a regular sieve. The big particle was ultimately crushed up rising an agate mortar to produce finer particles.

3.2.3 Microwave Heating

The barium titanate was placed in a microwave heating machine. The microwave was set to high temperature, and the durations of microwave testing were 10 minutes, 20 minutes, 30 minutes, and 40 minutes. Equipment, reaction kinetics, and microwave system capabilities determined the conditions. Starting with a lower power setting, adjustments were made to minimize overheating or aggressive reactions. The microwave heating was started, and the reaction temperature was closely checked using a thermometer or temperature probe. Reaction conditions were maintained by adjusting microwave power or heating time. Futhurmore, the precursor solution absorbed microwave radiation, raising the temperature rapidly. Barium titanate formed as barium carbonate and titanium oxide reacted at higher temperatures. The vessel was carefully removed from the microwave after heating, and heat-resistant gloves were used due to the vessel's temperature. The reaction mixture was cooled. (Kashimura et al., 2016).

3.2.4 Pressing Process

An agate mortar was used to grind the outcome into a fine powder. The mould wall was cleaned with WD-40 to remove any contaminants and corrosion. After mixing the powdered barium carbonate and titanium oxide, the mixture was pressed using uniaxial compression at 300 MPa pressure for 2 minutes, resulting in pallets that had a diameter of 2 mm and size of 10 mm.

FYP FBKT

3.2.5 Sintering Process

The pallets were sintered in air ambient at 1050℃ for 10 hours. For sintering process, the heating and cooling rate also was set as 5℃/min.

3.3 Characterization of Different Radiation Time of Microwave Processing

The sample will characterized by using XRD, OM, Density and Porosity measurement, and Dielectric Testing of the sample.

3.3.1 X-ray diffraction

The Bruker D2-Phaser XRD machine, located in the UMK X-ray laboratory, was used to perform the analysis. First, powders were ground into fine powder. Next, in order to preserve the crystal's preferred orientation, the powder of barium titanate was placed on the sample holder and gently flattened within the sample holder cavity without compressing them. After that, the sample was exposed to CuKα radiation and scanned for 2θ between 10 and 90. Finally, the powder diffraction pattern results were analysed using DIFFRAC.EVA software.

3.3.2 Optical Microscope

Optical microscopy provides important information about the microstructure and characteristics of sintered barium titanate pellets. Grain structure, porosity, phase composition, flaws, favoured orientations, and surface characteristics are all shown.

3.3.3 Density Measurement

The density was measured using a density measurement apparatus. The Archimedes Principle, or buoyancy method, was used by the testing apparatus. To weigh the sample, both liquid and air media were used. This illustrates the Archimedes Principle, which states that the weight of the liquid's displacement volume equals the reduction in apparent weight of an item immersed in liquid. Water was chosen as the liquid medium for this study because one millilitre has exactly one gramme of mass.

Then, the relative density of each sample was calculated by using Equation 3.1.

$$\rho s = \rho_{fl} \frac{m\,(a)}{m(a) - m(fl)} \quad \text{Equation 3.1}$$

Where,

ρs = Density of body

ρ_{fl} = Density of Liquid

$m_{(a)}$ = Mass of Body

$m_{(fl)}$ = Mass of Liquid

After each sample was weighed in both air and water, its bulk density was determined. The relative density of every sample was then calculated in order to ascertain the overall trend of all the samples.

The porosity calculated by using Equation 3.2

$\pi_a = \frac{m3-m1}{m3-m2}$ x 100 Equation 3.2

π_a = porosity

m_1= mass of the dried sample

m_2=apparent mass of saturated sample in water

m_3=Mass of saturated sample weight in air

CHAPTER 4

RESULT AND DISCUSSION

4.1 Microwave heating

Figure 4.1, which shows the temperature rising during microwave absorption in low power mode, illustrates the outcome. The aim of this investigation was to assess the sample's ability to retain heat during the firing procedure. Throughout the testing period of 10, 20, 30, and 40 minutes, the potential for heat absorption was measured every 30 seconds. Throughout the test, the temperature of each sample was recorded using a microwave oven that was connected to a temperature controller. According to the figure 4.1, all the sample were begun at an average 25℃ to 30 ℃, and the graph showed an unstable continuous temperature. The Barium Titanate 40M sample having the highest microwave absorption at 611℃ and Barium Titanate 10M was the lowest microwave absorption this due to the sample reinforcement and how well the material absorb heat according time 10, 20, 30, and 40 minutes.

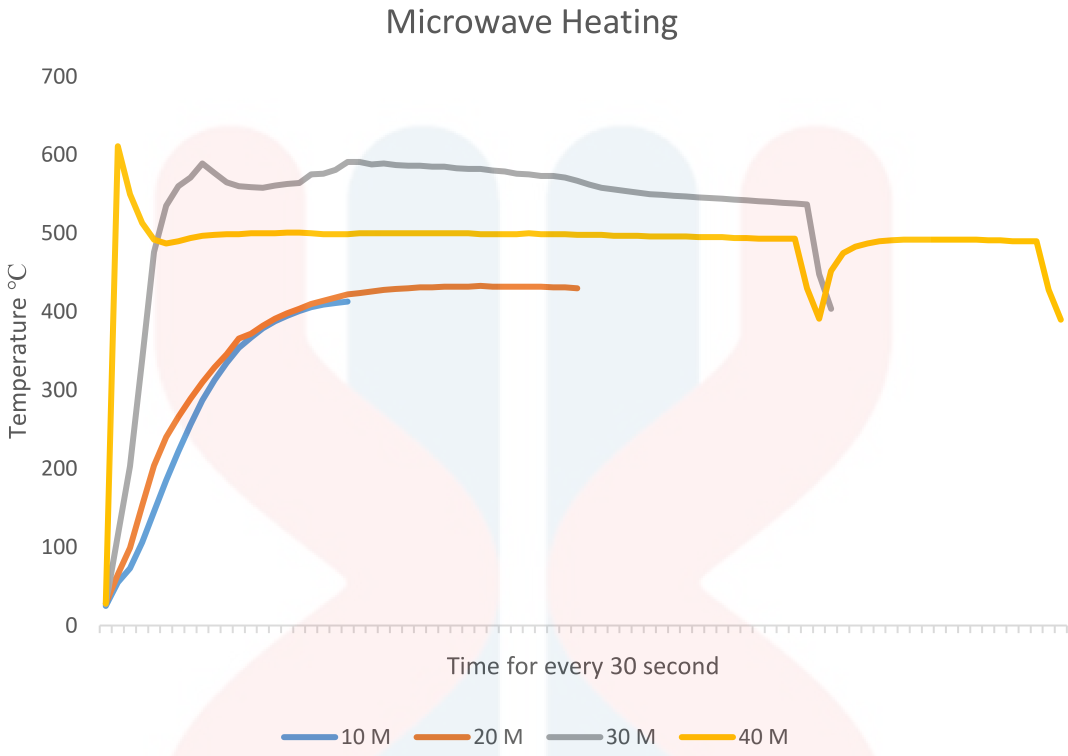


Figure 4.1: Temperature vs time for firing process in 10, 20, 30, and 40 minutes in high power.

4.2 XRD

This section will be clarify the crystalline of barium titanate using X-ray Diffraction.

4.2.1 X-ray Diffraction Barium Titanate (10M)

This study used X-ray diffraction (XRD) to characterise the raw materials, barium carbonate and titanium oxide. Both materials are tested in this way in order to determine the element and characteristic. The measurement range for the barium titanate 10M (COD1507757) XRD spectra was 2θ = 20° to 100°. Sample 10M lattice design was cubic. From the figure 4.2, it shown a few clear peaks that have sharp and weak diffraction peak. The highest peak contains in the graph was at 2ϴ = 31.55 ℃ (101) while the lowest peak was at 2ϴ = 50.91 ℃ (201).

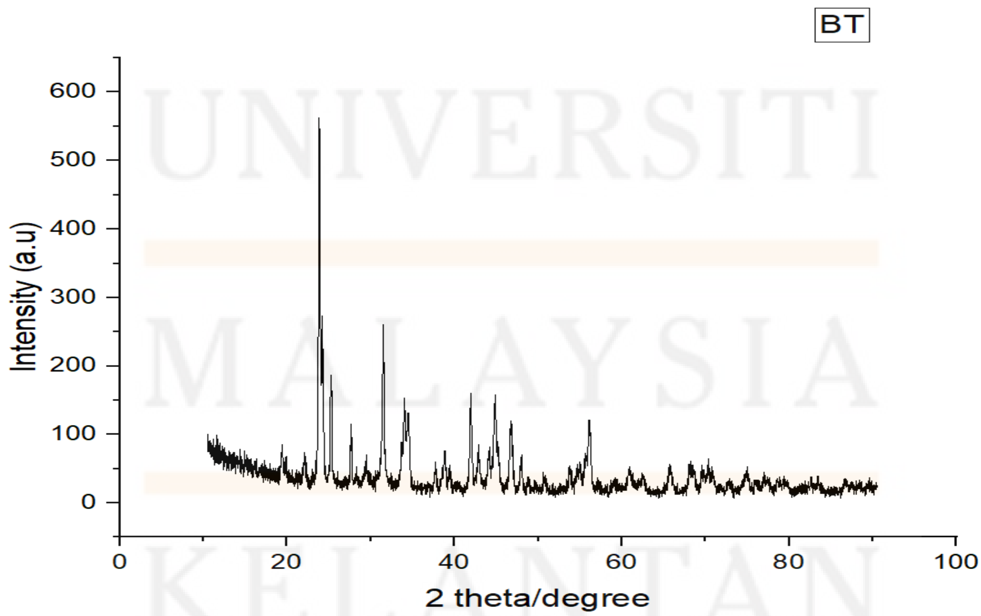


Fig2ure 4.2: XRD pattern for Baium Titanate (10M)

4.2.2 X-ray Diffraction Barium Titanate (20M)

The XRD spectra of Barium Titanate (20M) was measure in range of 2θ from 20° to 40°. Barium Titanate (20M) with COD2100863 has crystalline structure cubic. In addition, the lattice parameter of Barium Titanate of a was 4.00600. According to figure, the highest peak was at 2θ = 31.55° and the lowest peak at 2θ = 50.93°.

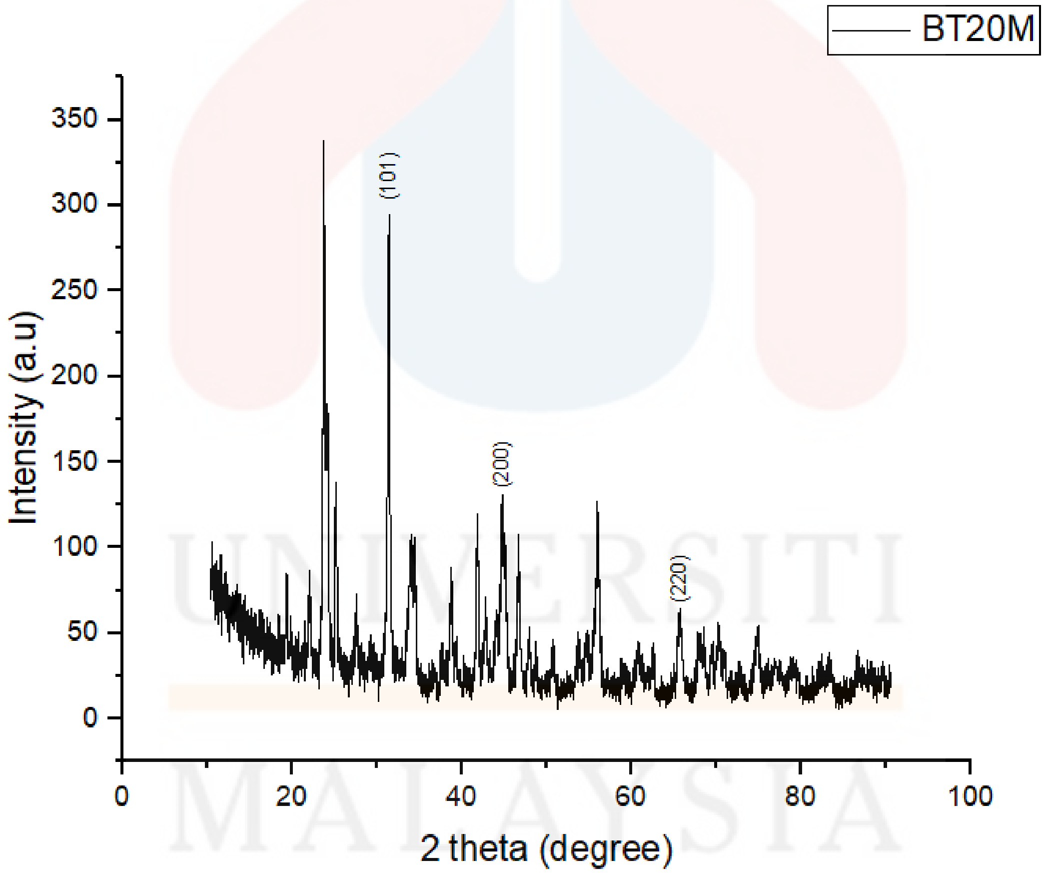


Figure 4.3: XRD pattern for Barium Titanate (20M)

4.2.3 X-ray Diffraction Barium Titanate (30M)

Based on figure 4.4, the XRD spectra of Barium Titanate (30M) was measured in range of 2θ from 20° to 80°. With pattern cubic of COD1507757 the parameter of a was 4.00730. From figure 4.4, it shown that the highest peak were recorded at $2\theta = 31.54^\circ$ and the lowest peak recorded at $2\theta = 65.87^\circ$.

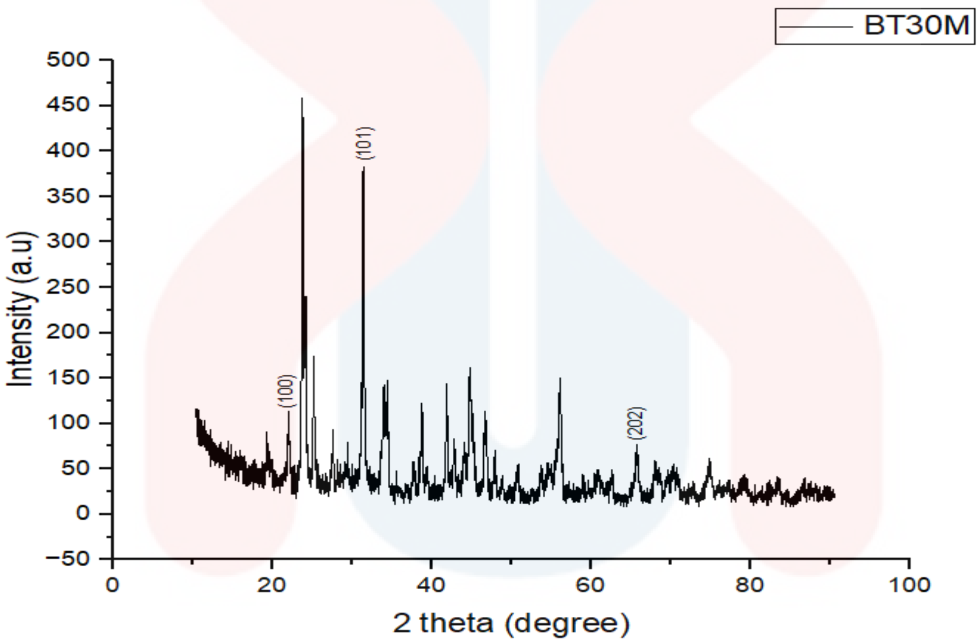


Figure 4.4: XRD pattern for Barium Titanate (30M)

4.2.4 X-ray Diffraction Barium Titanate (40M)

Based on figure 4.4, the XRD spectra of Barium Titanate (40M) was measured in range of 2θ from 20° to 70°. Barium Titanate (COD2100803), Barium Carbonate (COD9013850), and Titanium Oxide (COD1537224) has crystalline structure orthorhombic, cubic and hexagonal. From figure 4.4, we can see that the highest peak is Barium Carbonate at $2\theta = 23.88^\circ$, the second highest peak was

Barium Titanate at 2θ = 31.55°, and the lowest peak was Titanium Oxide at 2θ = 24.22°.

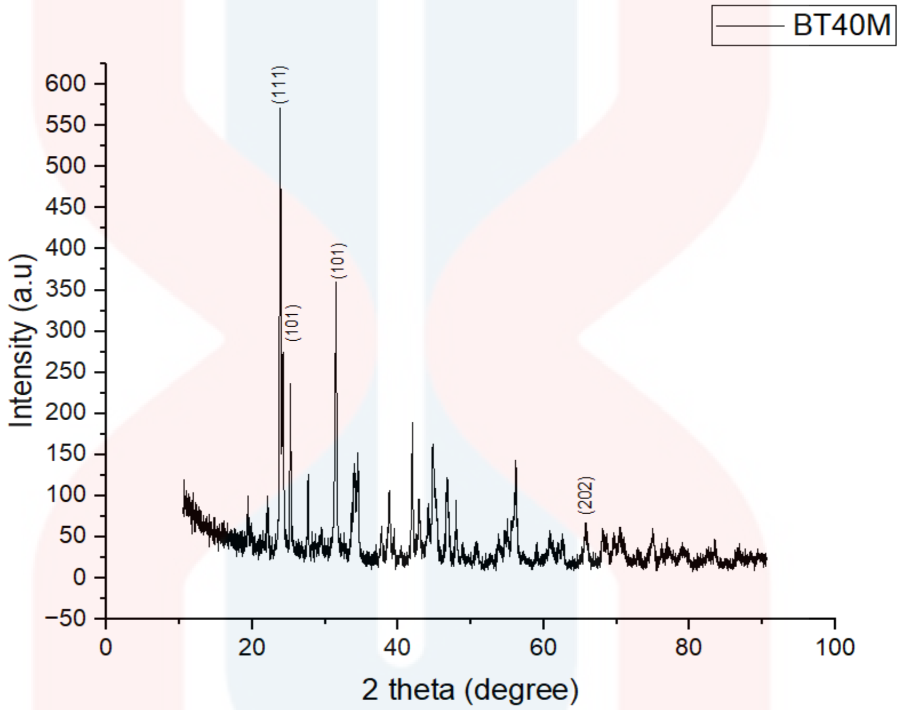


Figure 4.5: XRD pattern for Barium Titanate (40M)

4.2.5 Barium Titanate characterization after sintered pallet

The XRD pattern of a sintered pellet of barium titanate after 10, 20, 30, and 40 minutes is shown in Figure 4.6. The highest peak on the graph is located at 2θ = 31.33° count. Barium titanate cubic structure is shown on each peak. BaTiO3 is represented by the peaks (COD Number: 4124842).

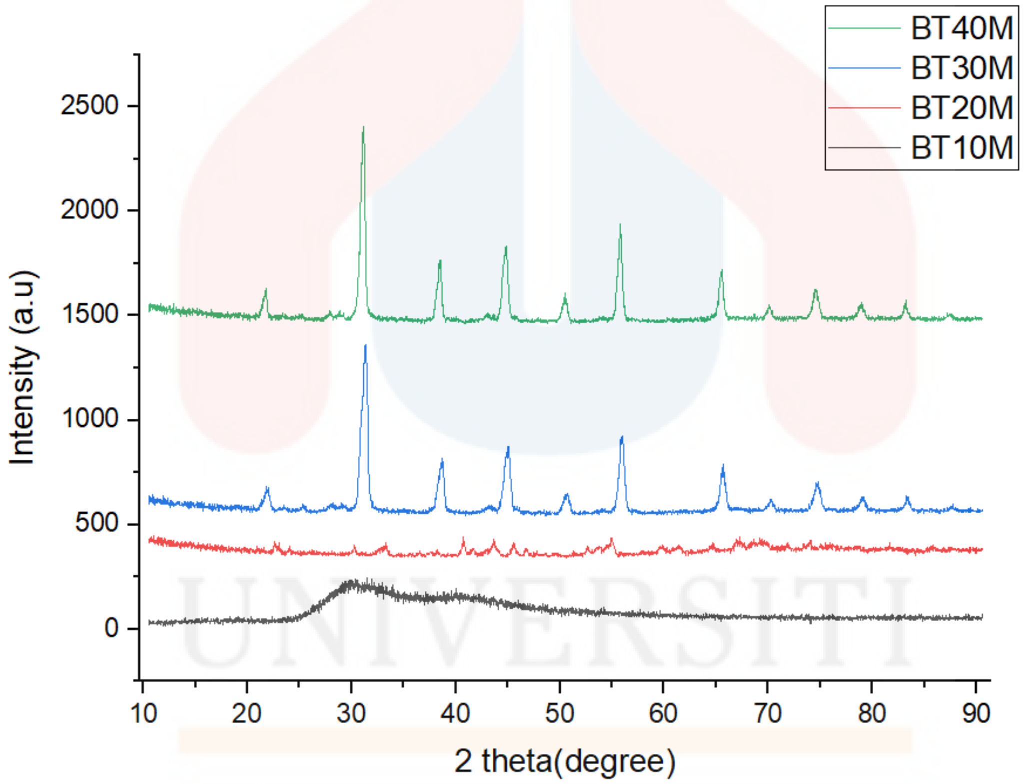


Figure 4.6: XRD pattern of sintered pellet Barium Titanate 10,20,30, and 40 minutes.

4.3 Density

A density test machine was used to perform the test. The particle was weighted in water first, then in air. The pellet's bulk density was then determined. Next, each sample's relative density was computed. The Archimedes principle was employed in the density measurement. The initial equilibrium state, as defined by Archimedes' theory, is when a body submerged in a liquid experience an upward force equal to the weight of the fluid being displaced.

Table 4.1 shows the density results for all sample. The bulk density has achieved on ly the highest at 1.99 g cm -3. The sample density has been decreased by increasing temperature.

Table 4.1: Table density and porosity of Barium Titanate sample

Sample	In Air	In Water	Bulk Density (g cm $^{-3}$)	Relative Density (%)	Porosity
BT 10M	0.858	0.528	1.99	60.78	60.78
BT 20M	0.823	0.509	1.99	62.43	62.43
BT 30M	0.824	0.506	1.98	60.65	60.65
BT 40M	0.855	0.521	1.95	61.28	61.28

From the figure 4.7 we can see that the trend relative density is increasing at BT20M at 62.43 %, but decrease at BT30M at 60.65 %, and increase back at BT40M at 61.28 %. The relative density was calculated by using equation 1. The highest relative density achieved is BT20M pallet at 62.43 % and the lowest relative density is BT30M pallet at 60.65 %.

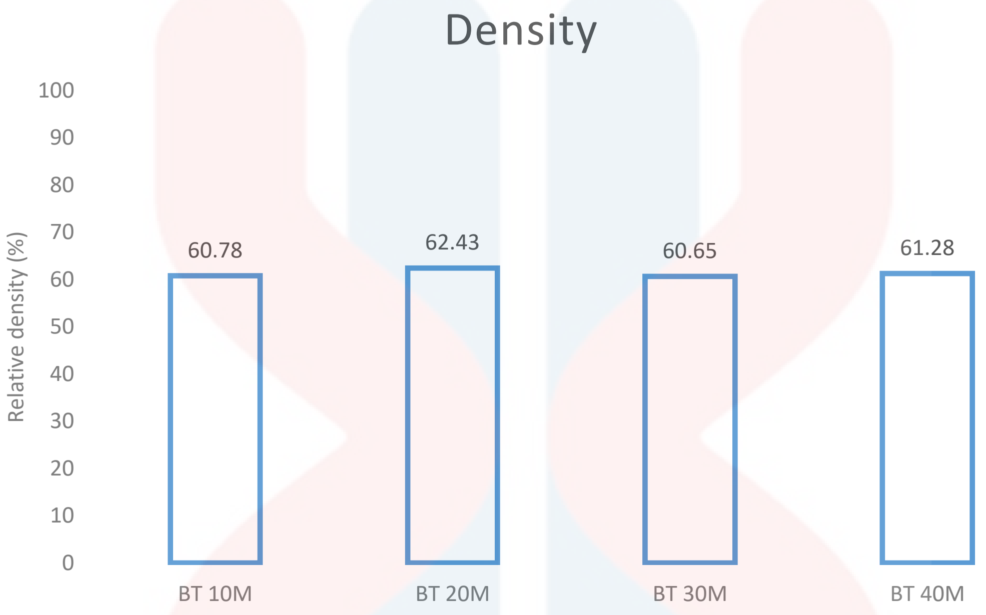


Figure 4.7: Graph of relative density of Barium Titanate from different heating time

The porosity that resulted from the samples being sintered is shown in Figure 4.8. Among all the samples, the BT30M sample had the lowest porosity. It is clear from the graph that samples with greater densities suggested lower porosities.

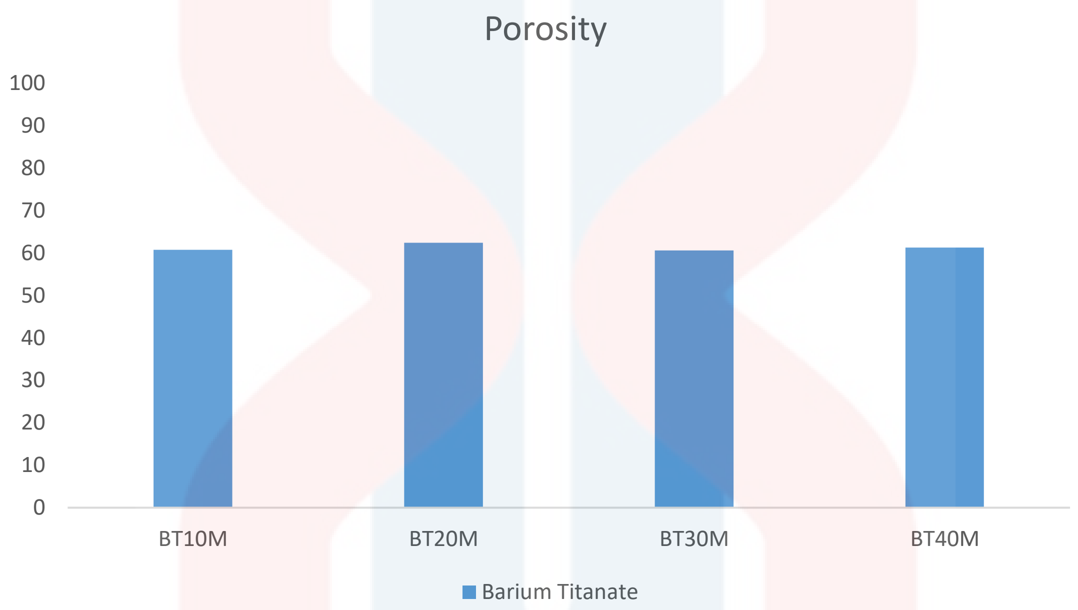
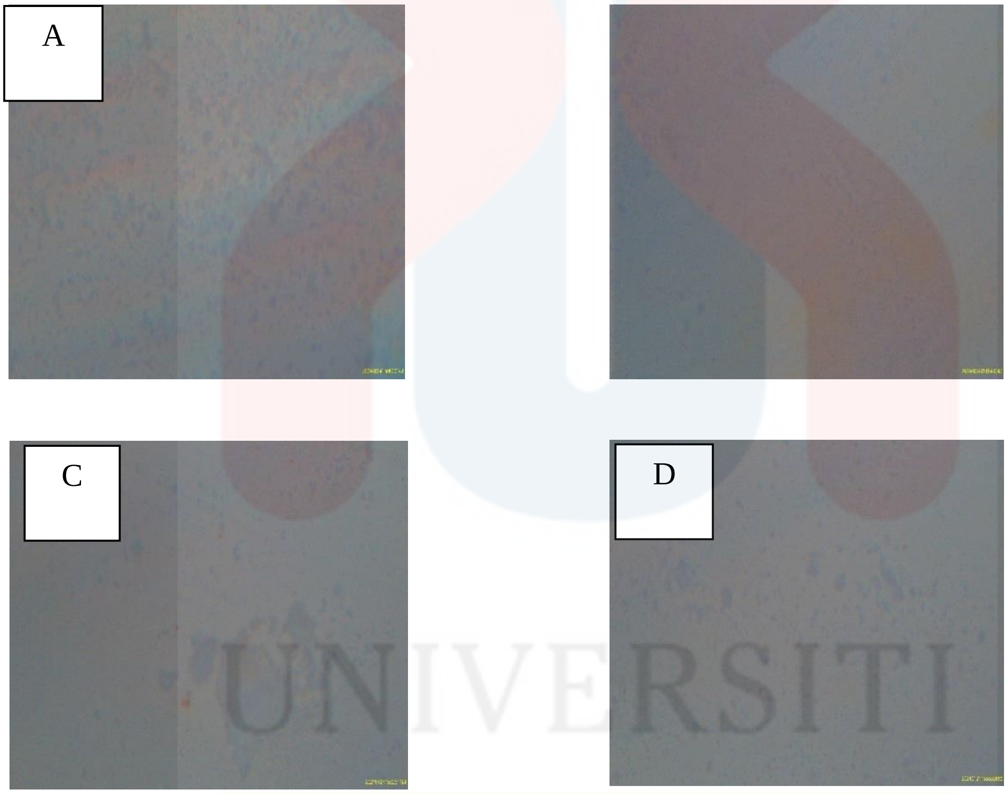


Figure 4.8: Graph of porosity of Barium Tintanate after sintered pellet

4.4 Morphology Properties

The surface morphology of the Barium titanate composite cross section was investigated using an optical microscope. Because sample preparation affects imaging quality, it is the most crucial step before analysis. Poor image quality will result from ,improper sample preparation because of the charging effect.



Surface morphology on Barium Titanate 10, 20, 30, and 40 minutes using Optical Microscope

4.5 Dielectric

Using an LCR meter, the dielectric constant and dielectric loss of BT were measured at room temperature and at a frequency of 10 MHz at the Institute of Nanoelectric and Electronic (INEE), University Malaysia Perlis (UniMAP), Perlis.

The figure 4.8 showed a BT content that increased with dielectric constant at 10 MHz, ranging from 7.54 to 50.5. Theoretically, a material's dielectric characteristics will grow with composition, leading to the production of massive dipoles in the ceramic particle via space charge polarisation at the particle level. Additionally, it demonstrates how the constant of dielectric steadily decreases as measurement frequency increases. These effects occur because heterogeneous dielectric materials have weak contact polarisation, or the Maxwell-Wagner-Sillar impact, and ceramic grains' dielectric relaxation.

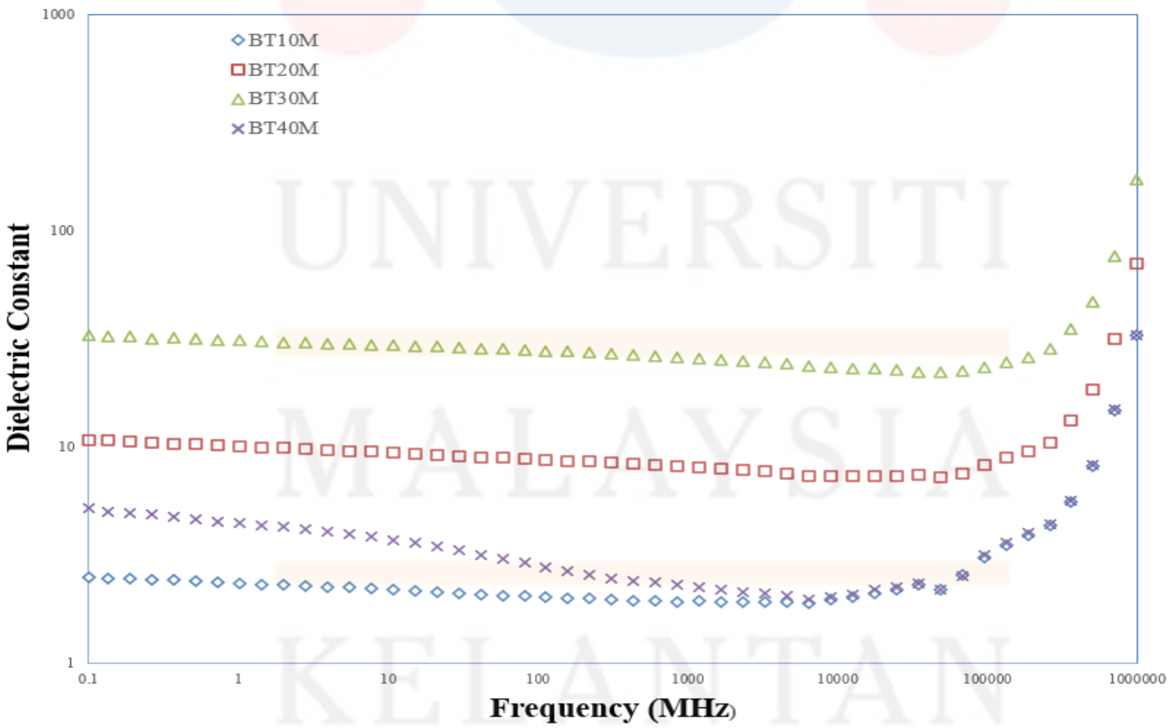


Figure 4.9: Dielectric Constant of BariumTiatanate from different heating time

Figure 4.9 depicts the BT composite's dielectric loss characteristics, which drop through 6.1 to 1.0 at 10 MHz while rising in frequency from 11 to 31 MHz until the relaxation phenomena occurs. Energy charged polarisation is the cause of this, which results in a decreased ability to keep up with a high frequency alternating electric field. Additionally, when filler loading increases, the composite's dielectric loss likewise decreases.

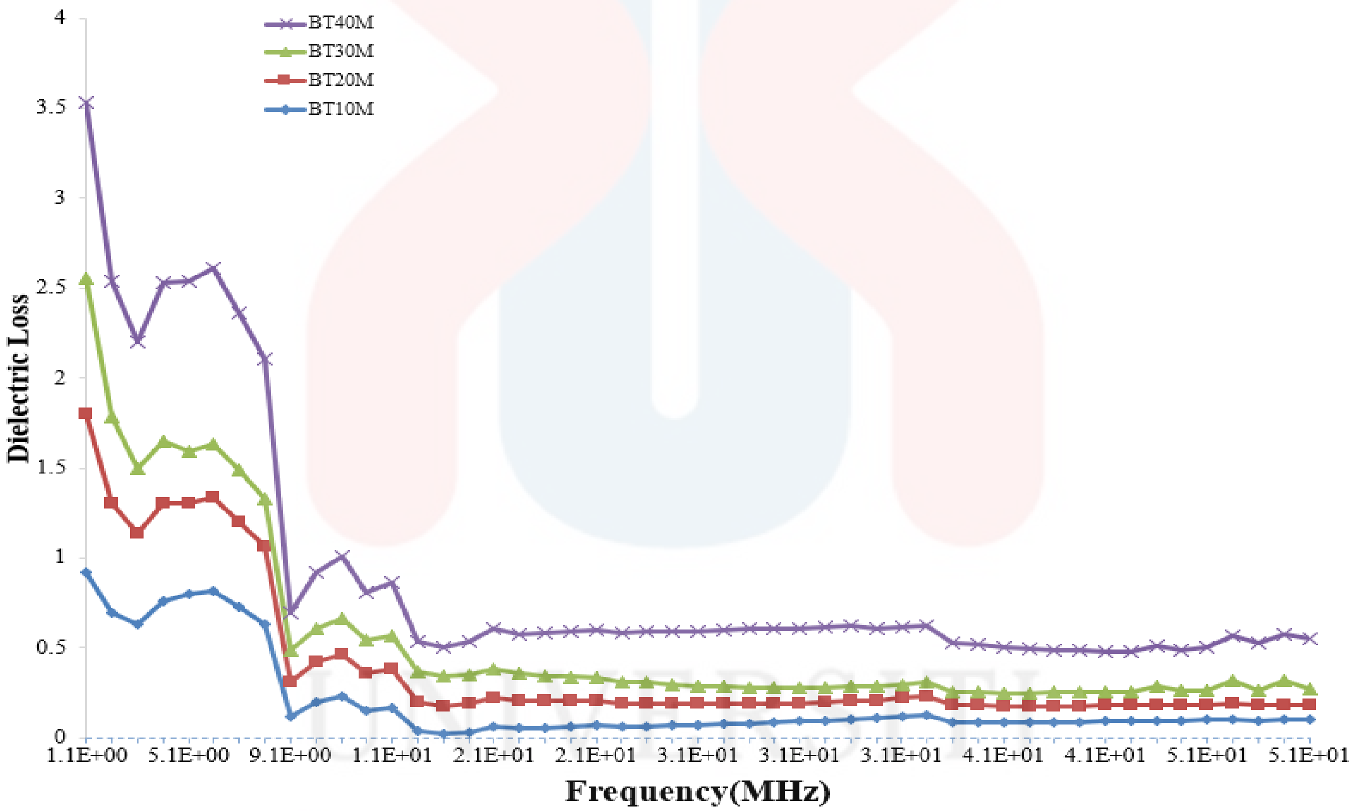


Figure 4.10: Dielectric loss of Barium Titanate from different heating time

CHAPTER 5

CONCLUSION

In conclusion, the investigation into the properties of Barium Titanate (BT) samples subjected to various heating times has provided valuable insights into its characteristics. The study revealed significant variations in microwave absorption, crystalline structure, density, porosity, morphology, and dielectric properties among the samples. Microwave absorption capabilities differed notably, with Barium Titanate 40M exhibiting the highest absorption at 611℃ and Barium Titanate 10M showing the lowest. X-ray Diffraction (XRD) analysis elucidated distinct diffraction patterns for each sample, providing insights into their crystalline structures and compositions. Density measurements indicated changes in bulk and relative density, with denser samples exhibiting lower porosity. Morphology analysis underscored the importance of proper sample preparation for accurate imaging. Dielectric constant and loss measurements highlighted the electrical characteristics of BT samples, showing an increase in dielectric constant with BT content and a decrease in dielectric loss with increasing frequency. Overall, these findings deepen our understanding of BT's behavior under different conditions and have implications for its potential applications across various industries.

RECOMMENDATION

For future research, It is advised to investigate how different synthesis factors, including precursor concentrations, microwave power levels, and reaction times, affect the characteristics of barium titanate (BT). Further research on a larger range of heating periods beyond the purview of this work may provide further light on the behaviour and characteristics of the material. Furthermore, carrying out more thorough examinations using techniques like atomic force microscopy (AFM) and transmission electron microscopy (TEM) may provide a better comprehension of the morphology and microstructure of BT samples produced by microwave heating. Additionally, experimenting with various sintering methods and settings—like temperature and time—may aid in refining the densification process and improving the material's qualities even more. Furthermore, it would be beneficial to look at the possible uses of BT in certain systems or devices, such energy storage units or sensors, in order to comprehend its effectiveness and practicality.

REFERENCES

Ayode Otitoju, T., Ugochukwu Okoye, P., Chen, G., Li, Y., Onyeka Okoye, M., & Li, S. (2020). Advanced ceramic components: Materials, fabrication, and applications. In *Journal of Industrial and Engineering Chemistry* (Vol. 85, pp. 34–65). Korean Society of Industrial Engineering Chemistry. https://doi.org/10.1016/j.jiec.2020.02.002

Buscaglia, V., Buscaglia, M. T., & Canu, G. (2021). BaTiO3-Based Ceramics: Fundamentals, Properties and Applications. In *Encyclopedia of Materials: Technical Ceramics and Glasses* (pp. 311–344). Elsevier. https://doi.org/10.1016/B978-0-12-803581-8.12132-0

Cahyono, Y., Ramadhanti, S., Rasmianti, Dewi, Y. N. K., & Asrori, M. Z. (2021). Effect of annealing hold time on thickness and optical properties of barium titanate solar cell material. *Journal of Physics: Conference Series*, *1816*(1), 012062. https://doi.org/10.1088/1742-6596/1816/1/012062

Carter, C. B., & Norton, M. G. (2013). Introduction. In *Ceramic Materials* (pp. 3–15). Springer New York. https://doi.org/10.1007/978-1-4614-3523-5_1

Eliche-Quesada, D., Pérez-Villarejo, L., & José Sánchez-Soto, P. (2019). Introductory Chapter: Ceramic Materials - Synthesis, Characterization, Applications and Recycling. In *Ceramic Materials - Synthesis, Characterization, Applications and Recycling*. IntechOpen. https://doi.org/10.5772/intechopen.84710

Ferro, G., Ganjam, D., Gibson, M., Partington, K., Trikha, A., Wu, M., Domrzalski, J., Dato, A., & Monson, T. (2021). Investigating the dielectric properties of barium titanate nanocomposites using transmission electron microscopy image processing. *MRS Advances*, *6*(25), 631–635. https://doi.org/10.1557/s43580-021-00095-0

Gómez Batres, R., Guzmán Escobedo, Z. S., Gutiérrez, K. C., Leal Berumen, I., Hurtado Macias, A., Pérez, G. H., & Orozco Carmona, V. M. (2021). Impact Evaluation of High Energy Ball Milling Homogenization Process in the Phase Distribution of Hydroxyapatite-Barium Titanate Plasma Spray Biocoating. *Coatings*, *11*(6), 728. https://doi.org/10.3390/coatings11060728

Jiang, B., Iocozzia, J., Zhao, L., Zhang, H., Harn, Y. W., Chen, Y., & Lin, Z. (2019). Barium titanate at the nanoscale: Controlled synthesis and dielectric and ferroelectric properties. In *Chemical Society Reviews* (Vol. 48, Issue 4, pp. 1194–1228). Royal Society of Chemistry. https://doi.org/10.1039/c8cs00583d

Karimi-Jafari, M., Kowal, K., Ul-Haq, E., & Tofail, S. A. M. (2017). 3.22 Spark Plasma Sintering of Lead-Free Ferroelectric Ceramic Layers. In *Comprehensive Materials Finishing* (pp. 347–357). Elsevier. https://doi.org/10.1016/B978-0-12-803581-8.09203-1

Kashimura, K., Sugawara, H., Hayashi, M., Mitani, T., & Shinohara, N. (2016). Microwave heating behavior and microwave absorption properties of barium titanate at high temperatures. *AIP Advances*, *6*(6), 065001. https://doi.org/10.1063/1.4953325

Kwei, G. H., Lawson, A. C., Billinge, S. J. L., & Cheong, S. W. (1993). Structures of the ferroelectric phases of barium titanate. *The Journal of Physical Chemistry*, *97*(10), 2368–2377. https://doi.org/10.1021/j100112a043

Misra, R. D. K., & Misra, K. P. (2022). Fundamentals of ceramics: introduction, classification, and applications. In *Ceramic Science and Engineering* (pp. 5–20). Elsevier. https://doi.org/10.1016/B978-0-323-89956-7.00020-6

Qian, H., Zhu, G., Xu, H., Zhang, X., Zhao, Y., Yan, D., Hong, X., Han, Y., Fu, Z., Ta, S., & Yu, A. (2020). Preparation of tetragonal barium titanate nanopowders by microwave solid-state synthesis. *Applied Physics A*, *126*(4), 294. https://doi.org/10.1007/s00339-020-03472-y

Setter, N. (2001). Electroceramics: looking ahead. *Journal of the European Ceramic Society*, *21*(10–11), 1279–1293. https://doi.org/10.1016/S0955-2219(01)00217-5

Sufiiarov, V., Kantyukov, A., Popovich, A., & Sotov, A. (2021). Structure and Properties of Barium Titanate Lead-Free Piezoceramic Manufactured by Binder Jetting Process. *Materials (Basel, Switzerland)*, *14*(16). https://doi.org/10.3390/ma14164419

Vinothini Venkatachalam, & Jon Binner. (2020). Synthesis of nanocrystalline barium titanate: Effect of microwave power on phase evolution. *Journal of the European Ceramic Society*, *40*(12), 3974–3983.