FUSION OF GROUNDED ILMENITE TO SYNTHESIS TITANIUM DIOXIDE WITH KOH

Submitted in partial fulfillment of the requirements for the award of

Bachelor of Engineering Degree in
Mechanical Engineering

By

BALAJI C.P - 39150017

DEPARTMENT OF MECHANICAL ENGINEERING

SCHOOL OF MECHANICAL ENGINEERING

SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)
Accredited with Grade "A" by NAAC I12B Status by UGC I Approved by AICTE
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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **BALAJI C.P - 39150017** who carried out the project entitled **"FUSION OF GROUNDED ILMENITE TO SYNTHESIS TITANIUM DIOXIDE WITH KOH".** The Project work carried out by them under our supervision and guidance during December 2022 to April 2023.

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I **BALAJI C.P. (39150017)** hereby declare that the project report entitled **“THERMAL ANALYSIS OF TITANIUM DI-OXIDE USING HYDRO METALLURGICAL PROCESS”** done by me under the guidance of **Dr.J.HEMANANDH, M.E.,Ph.D.,** is submitted in partial fulfillment of the requirements for the award of Bachelor of Engineering degree In Mechanical Engineering.

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ABSTRACT

The aim of the work is to extract Titanium di-oxide from ilmenite with sulphuric acid solution. Caustic fusion of ilmenite with H_2SO_4 is carried out for 160 mins at 500'C in the ratio 1:4 Acid leaching of ilmenite with NaOH is carried out for 90 mins at 85'C to obtain crystalline structure. Hydrolysed and calcinated at 400'C to obtain TiO_2 The properties and purity of sample were analysed by X-Ray diffraction and Scanning Electron Microscope(SEM).

The SEM-EDX analysis was used to determine the phases and final composition of the synthesized TiO_2 powder. The analysis showed that the 1:3 ratio of ilmenite and NaOH calcined at a temperature of 500°C gave the highest TiO_2 percentage of 72.56 in rutile form. Rutile is one of the three naturally occurring forms of TiO_2 and is considered to be the most stable and preferred form for industrial applications.

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LIST OF ABBREVIATIONS

EDAX	Energy-Dispersive X-Ray Spectroscopy
Fe2O3	Iron (111) Oxide
FeTiO3	Ilmenite
H2O	Water
H2SO4	Sulfuric Acid
HCL	Hydrochloric Acid
KOH	Potassium Hydroxide
Na2TiO2	Sodium Titanium Oxide
NaOH	Sodium Hydroxide
Ph	Potential Hydrogen
SEM	Scanning Electron Microscope
SiO2	Silicon Dioxide
TiO2	Titanium Dioxide
TiONa	Titanium Sodium
TiOSO4	Titanyl Sulfate

CHAPTER 1

INTRODUCTION

Titanium dioxide is a naturally occurring oxide of titanium, with a chemical formula of TiO2 and a pigment color index of Cl 77891. Apart from its use in paint, it has a wide range of applications in food, paper coatings, plastics, cosmetics and more. It is mainly obtained from minerals such as ilmenite, rutile and anatase, but can also be found in brookite. In certain high-pressure conditions, titanium dioxide can exist in monoclinic baddeleyite-like and orthorhombic a-PbOrlike forms. TiO2 is a widely used bulk commodity that serves as a white pigment for products like paints, plastics, paper, and rubber. It is manufactured by two primary industrial methods using either ilmenite, a ferrous titanate ore, or natural/synthetic rutile as the starting material. The wet sulphuric acid process utilizes ilmenite-based raw materials, while the high-temperature chloride process relies on rutile as the main input. Despite years of advancement, both manufacturing methods are still widely utilized and are considered to be both economically efficient and environmentally responsible industrial practices. In the following examples, the combined reaction rate- multiphase calculation is employed in analyzing two representative unit processes involved in TiO2production.

1.1 ADVANTAGES OF $Ti0_2$

Titanium dioxide (TiO2) is a widely used and versatile compound that has become an essential ingredient in many industrial and consumer products. It is a white, odorless, and tasteless powder that is insoluble in water and most organic solvents. TiO2 is known for its high refractive index, making it an effective whitening agent in paints, coatings, and plastics. Its ability to block UV light also makes it a popular additive in sunscreen products, protecting the skin from harmful rays. In addition to these industrial and cosmetic applications, TiO2 is also used in the production of paper, ceramics, and catalysts. The production of TiO2 involves a complex process that requires high temperatures and significant energy input, making it a costly and energy-intensive process. However, despite its high production cost, TiO2 remains an important compound in many industries due to its unique properties.

One of the reasons for the continued interest in TiO2 in materials science and technology is its photocatalytic properties and potential for use in addressing pressing issues in modern society such as energy and environmental concerns. Photocatalysis is utilized in fields like CO2 photoreduction, pollution removal, hydrogen production through water splitting or photo-reforming of alcohols, and bactericidal activity. TiO2 is a highly researched and utilized photocatalyst, often viewed as a standard in the field due to its unique properties. Recent advancements in energy conversion and light-based materials have focused on TiO2 composites with materials such as g-C3N4, graphene, and transition metal dichalcogenides. Developing cost-effective, efficient, stable, and reliable photocatalytic materials is an important research area.However, the use of TiO2 has been a topic of controversy in recent years due to concerns about its potential health and environmental impacts. Some studies have suggested that TiO2 nanoparticles may be harmful to human health when inhaled and that they may also have negative effects on aquatic organisms and ecosystems. As a result, some countries have restricted the use of TiO2 in certain products, while others have called for further research into its potential risks. Despite these concerns, TiO2 remains an important compound in many industries and will likely continue to be used in a variety of products for the foreseeable future.

1.1.1 Uses of Ti02

Titanium dioxide pigment, also known as pigment-grade titanium dioxide, is utilized in various applications that require high opacity and brightness. It is commonly used in white, pastel, and dark-colored surfaces and items. Below are some detailed applications of pigment-grade titanium dioxide. Paints and Coatings

The paint industry utilizes titanium dioxide for coating purposes due to its opacity and durability properties. It helps to ensure that the paint lasts longer and provides protection to the painted surfaces. Plastics, Adhesives, and Rubber.

The exposure of plastics and other materials to light can cause cracking, fading, and brittleness. Titanium dioxide can help reduce these issues. Cosmetics are products that are designed to enhance or alter the appearance of the face, body, or hair. The cosmetic industry includes a wide range of products such as makeup, skincare, haircare, fragrances, and personal care items. Makeup products include lipstick, foundation, mascara, eye shadow, and more, and are used to enhance facial features and create different looks. Skincare products include moisturizers, cleansers, toners, and serums, which are used to improve the overall appearance and health of the skin. Haircare products include shampoos, conditioners, and styling products that are used to cleanse, condition, and style the hair. Fragrances are used to enhance personal scent, and personal care items include products such as deodorants, toothpaste, and body have been used for thousands of years, with ancient Egyptians being known to use makeup to enhance their appearance.

Today, the cosmetic industry is a multi-billion dollar industry that continues to grow as people seek to enhance their physical appearance. With the rise of social media and the popularity of beauty influencers, the use of cosmetics has become even more widespread. Concerns have been raised about the safety and potential harm of certain cosmetic products. For example, some cosmetics may contain harmful chemicals or allergens that can cause skin irritation or other health issues. As a result, many consumers are now turning towards natural or organic cosmetics that are made from natural ingredients and are considered to be safer and more environmentally friendly. Cosmetics remain a popular and important part of many people's lives. They offer a way to enhance one's appearance, express one's personal style, and boost confidence. As the industry continues to evolve, it is likely that we will see new and innovative cosmetic products that cater to the ever-changing needs and desires of consumers.

Pigment-grade titanium dioxide is a common ingredient in cosmetic products, particularly those designed to hide blemishes and even out skin tone. It is also used to add brightness and luminosity to the skin. The small particle size of titanium dioxide allows it to adhere well to the skin, providing a long-lasting effect. Additionally, the opacity and reflective properties of titanium dioxide help to create a smooth and flawless appearance on the skin. Due to its non-toxic and non- irritating nature, it is commonly used in cosmetics for sensitive skin. In addition to its use in traditional cosmetics such as foundation, concealer, and powder, titanium dioxide can also be found in sunscreens, lipsticks, and eye shadows. Its ability to provide broad-spectrum UV protection makes it an essential ingredient in many sunscreens. In lipsticks and eye shadows, it is used to add pigment and shine to the product. Overall, the versatility and effectiveness of titanium dioxide make it a valuable ingredient in the cosmetic industry.

The paper industry commonly uses titanium dioxide (TiO2) as a coating material to enhance the brightness and opacity of paper. By adding TiO2 to paper coatings, it becomes whiter and more reflective, which in turn improves the overall appearance of the paper. The small particle size of TiO2 allows it to be easily dispersed and evenly distributed in the coating mixture, ensuring consistent and effective results. The use of TiO2 in paper coatings not only improves the aesthetic properties of the paper but also enhances its durability and resistance to aging and yellowing. This is particularly important for high-quality printing applications, where the longevity and clarity of the printed material are essential. Overall, the use of TiO2 in the paper industry is a common and effective way to improve the appearance and quality of paper products.

Titanium dioxide (TiO2) provides protection to food items, beverages, supplements, and pharmaceutical products by offering opacity to visible and ultraviolet light, thus preventing premature degradation and increasing their shelf life. In the pharmaceutical industry, titanium dioxide is commonly used in the form of tablets, including titanium dioxide IP tablets, capsule coatings, and as a decorative aid in certain foods.

1.2 EXTRACTION OF TiO_2

1.2.1 Ilmenite

Ilmenite is a widely distributed accessory mineral found in igneous rocks, sediments, and sedimentary rocks across the globe. It was also discovered in abundance in lunar rocks and regolith by the Apollo astronauts. With a chemical composition of $FeTi0_3$, ilmenite is a black iron-titanium oxide and is the primary source of titanium, a metal that is necessary for the production of various high-performance alloys. The majority of ilmenite extracted worldwide is utilized in the manufacturing of titanium dioxide (Ti02), a significant pigment, whitening agent, and polishing abrasive. Ilmenite is highly resistant to weathering, and as rocks containing ilmenite break down, ilmenite grains scatter with the sediment. The heavy specific gravity of these grains leads to their segregation and accumulation as "heavy mineral sands" during stream transport, which geologists can easily recognize due to their black color. "Black sand prospecting" has long been used as a means of locating heavy mineral placer deposits. Most of the ilmenite that is commercially produced is extracted by excavating or dredging these sands, which are then processed to separate the heavy mineral grains such as ilmenite, leucoxene, rutile, and zircon. The composition of the Ilmenite is shown in table 1.1.

1.2.2 Sodium Hydroxide (NaOH)

Sodium hydroxide, also known by its chemical formula NaOH, is a versatile and widely used chemical compound in various industries. It is a strong base that is highly reactive and can dissolve in water to form a highly alkaline solution. Due to its corrosive and reactive nature, caution should be exercised when handling it. In the soap and cleaning industries, sodium hydroxide is used to saponify fats and oils, which converts them into soap. It is also used in household cleaning products, such as drain cleaners and oven cleaners, due to its ability to dissolve organic materials. The paper industry is a significant user of sodium hydroxide, where it is used to break down lignin in wood pulp and separate it from cellulose fibers. It is also used in the production of aluminum, where it is used to dissolve aluminum oxide to produce alumina.

Sodium hydroxide is also used in the food industry to process various foods such as cocoa, chocolate, and olives. It is used to adjust the pH of foods and to remove impurities. In medicine, sodium hydroxide is used as an ingredient in certain medications such as pain relievers and anticoagulants. It is also used in cholesterol-reducing medications and in the treatment of certain skin conditions. In addition to its various industrial and medical applications, sodium hydroxide is used in water treatment to adjust the pH of water and to remove heavy metals and impurities. It is also used in the manufacture of various chemicals, including dyes, detergents, and textiles.

1.2.3 Hydrochloric Acid (HCl)

Hydrogen chloride is a gas that has a pungent odor and is colorless. It reacts with the chlorides formed by active metals, their oxides, hydroxides, and carbonates, but only readily in the presence of humidity. If the hydrogen chloride is completely dry, it is unreactive. Hydrochloric acid has the properties of a common strong acid, including reactions with metals that displace hydrogen gas, reactions with simple (metal) oxides and hydroxides that produce a metal chloride and water, and reactions with weak acid salts in which the heavy acid is displaced. When hydrogen chloride dissolves in water, HCl is formed, which is a simple diatomic molecule with a polar bond due to chlorine's greater electronegativity than hydrogen. HCl is strongly acidic, colorless, viscous, and corrosive, with a distinct smell. It is commonly used as a laboratory reagent and in industry, including the processing of leather and production of gelatin. The physical properties of HCl, such as density, melting point, pH, and boiling point, depend on its molarity or concentration.

1.3 STEPS INVOLVED

1.3.1 Ball Milling

The process of Ball Milling is a mechanical technique that is widely used to grind powders into fine particles. Unlike the traditional method where reactants are broken apart using solvent molecules, ball milling utilizes mechanical forces to break them apart. This technique has been recently introduced and is known as mechanochemistry. The synthesis and reactions of organic compounds using ball milling have been extensively reviewed. However, the use of solvent-free ball milling in organic synthesis is not yet widely adopted. A rotating hollow cylindrical shell is a common structure used for this purpose. The axis of rotation can be horizontal or at a slight angle to it. Inside the shell, there are balls made of materials like steel (including chrome steel), stainless steel, ceramic, or rubber, which are used as grinding media. The inner surface of the shell is typically lined with an abrasion-resistant material.

1.3.2 Caustic Fusion

Caustic fusion is a chemical process that involves heating a sample with a strong alkaline solution, typically a mixture of sodium hydroxide (NaOH) and potassium nitrate (KN03), to fuse and dissolve it. The resulting solution is then further processed for analysis or testing. This method is commonly used in analytical chemistry and materials science to extract and analyze the mineral content of geological samples such as rocks, soils, and sediments. It is also used in the recycling of electronic waste to recover valuable metals such as gold, silver, and copper.

1.3.3 Leaching

Leaching is a process of extracting a substance, typically a metal or mineral, from a solid material by dissolving it in a liquid. The liquid used for leaching is called the leaching agent or solvent, and it can be water, acid, or other chemicals, depending on the substance being extracted and the desired outcome. Leaching is commonly used in mining and metallurgical processes to extract valuable metals such as copper, gold, and silver from ores. In this process, the ore is crushed and then treated with a leaching agent, which dissolves the metal and forms a solution. The solution is then separated from the solid residue and processed further to recover the metal. Leaching can also be used to remove harmful substances or contaminants from soils and other solid materials. For example, soil contaminated with heavy metals can be treated with a leaching agent to dissolve the metals and remove them from the soil.

1.3.4 Calcination

Calcination is a process of heating a substance to high temperatures in the absence or limited presence of air or oxygen, to bring about a thermal decomposition or a phase transition. The process involves converting a solid material into a powder or porous mass, and is often used in metallurgical, chemical, and material science applications. One of the most common uses of calcination is in the production of cement, where limestone and other minerals are heated to a high temperature to produce a powdered form of calcium oxide, which is then mixed with water and other materials to create concrete. Calcination is also used in the production of ceramics, where clay and other materials are heated to high temperatures to create a hard, durable material.

Calcination can also be used to change the physical properties of a material, such as its particle size or porosity. This can be useful in applications such as catalysis, where a material's surface area and reactivity are important factors. While calcination can be a useful process, it can also have environmental impacts if not managed properly. High temperatures and the release of gases during the process can contribute to air pollution, and the waste products produced can pose a disposal challenge. Therefore, it is important to use appropriate safety measures and environmental controls when carrying out calcination processes.

1.4 ANALYSIS

Scanning Electron Microscope (SEM)

SEM stands for Scanning Electron Microscopy, which is a type of microscopy used to produce highly magnified images of materials at high resolution. In SEM analysis, a focused beam of electrons is scanned across the surface of a sample, and the resulting signals are used to generate an image of the sample's surface. Unlike traditional optical microscopes, SEM uses electrons instead of light to image the sample. This allows for much higher magnification and resolution, as electrons have much shorter wavelengths than visible light. SEM can produce images with magnifications ranging from 1Ox to over 100,000x, depending on the type of sample and the specific SEM used. In addition to producing high-resolution images, SEM can also be used to analyze the composition and structure of materials. By analyzing the signals produced by the electrons as they interact with the sample, researchers can gain insight into the sample's chemical composition, crystal structure, and other properties.

1.4.2 Energy-Dispersive X-Ray Spectroscopy

EDX, or Energy Dispersive X-ray Spectroscopy, is a technique used to identify the elemental composition of a material. It is often paired with Scanning Electron Microscopy (SEM), where the SEM is used to image the sample while EDX analyzes the elemental composition.

During EDX analysis, a beam of electrons is directed at the sample, which causes it to emit X-rays. These X-rays are then collected and analyzed to determine the elements present in the sample. The energy of the X-rays corresponds to the atomic structure of the elements, allowing for accurate identification of the elements present. EDX analysis is used in a variety of fields, including materials science, chemistry, geology, and biology. It is non-destructive, meaning that the sample remains intact and can be further analyzed or used for other purposes after analysis.

CHAPTER 2

LITERATURE SURVEY

Mills et al. [1] discovered that a sufficient decrease in the band gap energy of the TiO2 photocatalyst can enhance its ability to absorb energy in the visible light range, potentially increasing utilization of the solar spectrum up to 40%, as opposed to the previous limit of less than 1%, before band gap alteration. The decreased band gap of Ag-TiO, from 3.20eV to 2.98eV, resulted in improved photoactivity. Therefore, they measured the band gap of their high-grade TiO2 and high crystalline Ag-TiO2, and studied their effects on photoactivity.

In this study Pelaez et al. [2] concluded that a photocatalysis technique utilizing TiO2/SiO2 composite was employed to handle heavy metal waste, resulting in effective reduction of harmful pollutants. The support material tor TiO2 photocatalysts was SiO2 sourced from Bengkulu beach sand in Indonesia. To obtain SiO2, leaching techniques were utilized with NaOH serving as a solvent. The resultant SiO2 was then used to remove Cr and Pb.

Chunlei Ma [3] has developed a cost-effective process that involves minimal use of hydrochloric acid at a low concentration (6M HCI in a volume of only 350 ml for 25g of ilmenite) and a lower temperature of 170°C. The closed hydrothermal system utilizes a revolving autoclave to maintain the desired pressure and temperature conditions, making it an innovative method for breaking down hard inorganic solids at considerably lower temperatures. This marks the first instance of an autoclave being used to break the crystalline structure of such materials.

As per the findings of Rias and Das [4], incorporating $Fe2O_3$ in TiO2 can yield positive results tor photocatalytic applications. This is because $Fe2O_3$ can hinder the phase transformations from anatase to rutile, and reduce the band gap energy of TiO_2. Moreover, Fe_2O_3 can also impede the recombination of electrons from the conduction band to the valence band, which can enhance photocatalytic activity.

According to Perez and Sharadqah [5], the concentration of TiO_2 in the final product is not significantly affected by the magnetic treatment prior to other processes. They presented a modified method of TiO_2 recovery that eliminates the stage of magnetic concentration and instead uses a combination of thermal and chemical treatments with ion exchange.

In this study, Feixiang Wu [6] used milling to obtain finely sized particles of ilmenite. To maintain the oxidation process during roasting, activated carbon was added. The roasted ilmenite was then leached with sulfuric acid and the resulting titanyl sulfate solution was hydrolyzed using EDTA to extract the solvated iron. After calcination, TiO_2 was produced.

In this experiment, Huzaikha Binti Awang [7] concluded that the use of diluted sulfuric acid was found to be effective in reducing waste acid emissions and minimizing the adverse effects of high viscosity concentrated sulfuric acid, as it helps to increase the dissolution of ilmenite. However, increasing the leaching time or temperature can lead to a decrease in the dissolution of titanium due to polymerization and hydrolysis, without any effect on iron.

In a study by Manhique et al., [8], ilmenite was roasted with NaOH within a temperature range of 300°C to 950°C. The study showed that at temperatures above 550°C, different sodium iron titanates such as Na_2TiO_3 and $NaFeO_2$ are formed, while at temperatures of 550°C and below, Na_2TiO_3 is the only binary titanate present in the products. Middlemas et al. found that TiO_2 pigment production could be achieved by roasting titania slag with NaOH, followed by water washing and HCl leaching, and then applying solvent extraction with an amine to remove iron and leave a Ti-rich raffinate.

The acidic waste from the sulfate process can create a danger to our environment. Therefore, only a medium acid concentration and short treatment time is introduced in this research work. The results also show that the higher the acid molarity, the higher the titanium composition on that sample. E. M. Mahd.,[9] stated that the higher the sulphuric acid molarity, the higher the protons (H+) and sulfate ions which lead to the higher leaching rate.

The smaller band gap can be explained by the presence of Fe and Si impurities in the synthesized TiO2, which are creating new energy levels either near the valence or the conduction band edge. With the increase in the amount of doping, the energy level becomes denser and effectively decreases the band gap. Modified Kubeika-Munk Theory [10] is used in this research due to its highly accurate results obtained with the TiO2 sol-gel and TiO2 commercial.

According to Linus Pauling's principle of parsimony [11], minerals with a complex composition and limited structural positions may need to place a certain number of ions. As a result, chemical elements substitute each other in specific atomic sites, resulting in what is known as a solid solution. This principle is applicable to Chromite Minerals.

Munammed Nurdin [12] stated that Titanium is a lightweight and highly heat-resistant metal with a high melting point, making it desirable for many applications. It can be extracted through the hydrometallurgical process, which involves leaching ilmenite with hydrochloric or sulfuric acid. This method is an efficient way of separating minerals from other elements. The resulting titanium extract can then be further processed tor specific applications, such as serving as a base material in the production of TiO2.

Middlemas et al. [13] have reported that in the chloride process, $TiCl_4$ vapor is produced by chlorinating either high grade rutile or titanium slag in a fluidized bed reactor with the presence of coke. This vapor is then converted to titanium dioxide using oxygen in the presence of $AlCl_3$.

Gazquez et al. concluded that while the sulfate process is straightforward and can handle lower grade ores, it yields low-quality products and generates a significant amount of waste iron sulfate. In a separate study, Saji et al.
[14] explored the use of Cyanex in xylene for the extraction of Ti{IV) from various synthetic binary and ternary mixtures in HCl solutions. They used aqueous

solutions containing metal ions and 0.3M HCI, and an organic solution containing 0.2M Ti was extracted from Cyanex, almost entirely into the organic phase.

Li et al., Urakaev, and Wei et al. [15] emphasized the importance of mechanically activating ilmenite with a ball mill prior to leaching. Hydrochloric acid leaching is considered the primary technology for processing ilmenite or titanium slag to produce titanium-rich materials due to its excellent impurities removal capability, especially for MgO and CaO, as well as its high leaching speed and acid regeneration technology has been concluded by Mahmoud et al, Samal, Walpole et al, Zhang et al.

Van Dyk et al. [18] reported that both iron and titanium initially dissolve in hydrochloric acid, but titanium may polymerize in the solution. Nabivanets and Kudritskaya established criteria to determine when titanium polymerization would occur. At a high acid to ilmenite ratio, as in the case of Jackson and Wadsworth's study, the concentration of titanium in the solution may not reach the critical concentration required for polymerization.

The purpose of this experiment [19] is to address the issue of diminishing supply of rich ferruginous ores and increasing dependence on lower- grade ores with higher concentrations of minor and trace impurity oxides. The new technique has the potential to reduce waste generation by minimizing the need for chlorination, during which most of the iron, minor, and trace impurities are removed via fractional distillation.

According to research conducted by Leone et al.,[20], using a molten-salt bath as an electrolyte for the electrolysis of titanium tetrachloride has great potential for industrial applications in the production of high-purity titanium metal. During the process, the titanium tetrachloride is continuously fed below the surface of the molten electrolyte, which is a eutectic mixture of salts and aluminum oxide.

CHAPTER 3

AIM AND SCOPE OF THE WORK

3.1 AIM

In this experiment a procedure with reduced time and temperature is followed and testing the process to give the pure Ti02 and analyze the product and develop the procedure.

Ti02 is the rare mineral which cannot be found on the earth surface. It needs to be extracted from Its ores. There are different types of ore on the earth surface and these ore have different composition of materials in it. The primary ore used in the extraction process is "Ilmenite". There are many methods of extraction carried out in this process. In this project our main aim is to reduce the reaction time and temperatures and to find the best way for extraction of Titanium dioxide and give a brief analysis on the work.

3.2 SCOPE OF THE WORK

The scope of this work is to develop a procedure for the extraction of titanium dioxide (Ti02) from ilmenite, a mineral containing titanium and iron. The aim is to reduce the reaction time and temperature required for the extraction process. The work involves a series of chemical processes, including caustic fusion, acid leaching, hydrolysis, and calcination, to extract and analyze the components of ilmenite. The final products obtained from calcination at different temperatures and ratios are analyzed using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) to determine their composition. The study aims to identify the optimal conditions for the extraction of Ti02 from ilmenite and provide a brief analysis of the results.

CHAPTER 4

MATERIALS AND METHODS

4.1 METHODOLOGY

In this process Titanium dioxide is obtained by using a different process. Ilmenite is the major source of Ti02. The hydrometallurgical process is used in this experiment in which hydrolysis is done after every single module of work and the ph values will be monitored constantly as shown in fig 4.1.

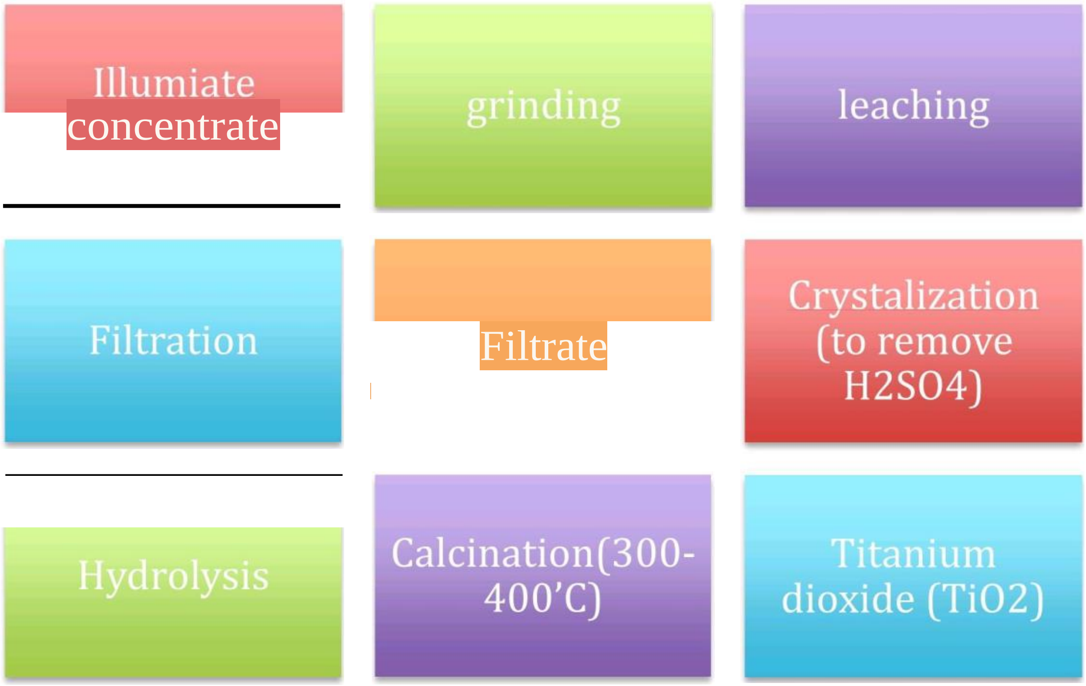


Fig. 4.1: Methodology

4.1.1 *Hydrometallurgical Process*

The entire process for this extraction is based on the Hydrometallurgical Process, Which is also known as "A process for extracting metals from ores and waste materials involves the use of aqueous media, with or without pressurized conditions, in combination with water, oxygen, and other chemical reagent.

It involves mainly three different procedures based on the extraction material and Ore. In this process Caustic Fusion, Acid Leaching and Calcination are followed. The caustic fusion technique, more properly known as caustic dissolution, involves fusion of ore with the base to remove the ferrous particles from the ore and form an intermediate product. Acid Leaching was the process by which constituents of a solid material were dissolved into the contacting acid and converted into liquid phase. To remove the excess material present in the Firt. Calcination is the heating process where solids to high temperature for the purpose of removing volatile substances, oxidizing to a portion of mass. In this experiment calcination is performed to remove the excess water and obtain the purest form.

4.2 MATERIAL AND METHODS

4.2.1 Materials

Ilmenite, (HCL), H2SO4 ,Sodium Hydroxide, Distilled Water.

4.3 EXPERIMENTAL PROCEDURE

Caustic Fusion: In this step, ilmenite is mixed with sodium hydroxide (NaOH) at different ratios of 1:2, 1:3, and 1:4, and heated to 450° C tor about 4 hours as shown in fig 4.2 and fig 4.3. The purpose of this step is to convert the ilmenite into a more soluble form that can be extracted easily in the subsequent steps. The reaction between ilmenite and NaOH produces sodium titanate and iron oxide.

Leaching: The output of the caustic fusion process is then subjected to leaching using hydrochloric acid (HCl). 36 ml of 2M HCl is added to 200 ml of distilled water and stirred with the past output using an electric burner, as shown in Fig. 3. The purpose of this step is to dissolve the sodium titanate and iron oxide and separate them from the remaining insoluble impurities.

Hydrolysis: After the leaching step, the output is subjected to hydrolysis to reduce its pH value and maintain it below 4. This is achieved by adding a base such as sodium hydroxide or ammonia to the output until the desired pH level is reached. The purpose of this step is to prepare the output for the next step of calcination.

Calcination: In this step, the output from the hydrolysis process is calcinated at different temperatures of 400° C, 500° C, and 600° C for 1 hour, as shown in Fig. 5. The purpose of this step is to transform the remaining sodium titanate and iron oxide into more stable phases such as titanium dioxide and magnetite. The calcination temperature and time are critical factors that determine the quality and properties of the final product.

SEM and EDS Analysis: Finally, the 9 samples obtained from calcination at different temperatures and ratios are separated and analyzed using Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) techniques. SEM is used to study the morphology and structure of the samples, while EDS is used to identify the elemental composition of the samples.

Fig. 4.3: Electric Burner

The experiment described involves a series of chemical processes to extract and analyze the components of ilmenite, a mineral containing titanium and iron.The first step is Caustic Fusion, in which ilmenite and sodium hydroxide are mixed at a ratio of 1:2 and heated to 450°C for approximately 4 hours. This process is repeated at ratios of 1:3 and 1:4 to obtain multiple samples. Caustic fusion is a process that involves the reaction of an oxide with a strong base, resulting in the formation of soluble alkali metal salts. In this case, the reaction produces sodium titanate and sodium ferrate.

Next, the output of the caustic fusion process undergoes acid leaching, where hydrochloric acid is used to dissolve the sodium titanate and sodium ferrate. 36 ml of 2M hydrochloric acid is added to 200 ml of distilled water and stirred with the output of the caustic fusion process using an electric burner as shown in fig 4.4. The acid leaching process dissolves the sodium titanate and sodium ferrate, leaving behind any insoluble materials. After the acid leaching process, the resulting output undergoes hydrolysis to reduce its pH to less than 4. Hydrolysis is the process of adding water to a substance to break down its chemical bonds. In this case, water is added to the output of the acid leaching process to neutralize any remaining acid and reduce the pH. The final step is Calcination, where the three samples obtained from the caustic fusion process are divided into 9 smaller samples. Each of these samples is calcinated at different temperatures (400°C, SOO^{0}C, and 600°C) for 1 hour in a high-temperature muffle furnace as shown in fig 4.5. Calcination is a process of heating a material to a high temperature to bring about a chemical change. In this case, calcination is used to convert the sodium titanate and sodium ferrate into titanium dioxide (TiO2) and iron oxide (Fe2O3). Finally,all 9 samples are separated based on their ratios and temperatures and analyzed using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) to determine the composition of the resulting compounds. SEM is used to image the samples at high magnification and EDS is used to analyze the chemical composition of the samples.

Fig. 4.4: Leaching at 110°C for 2 hours

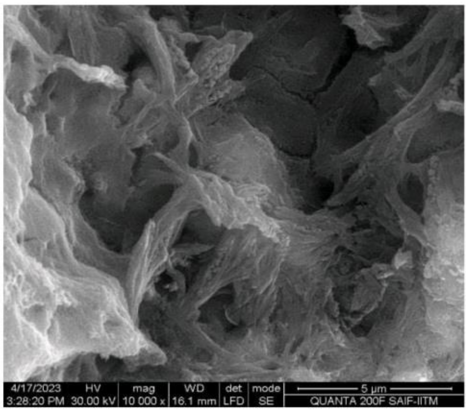
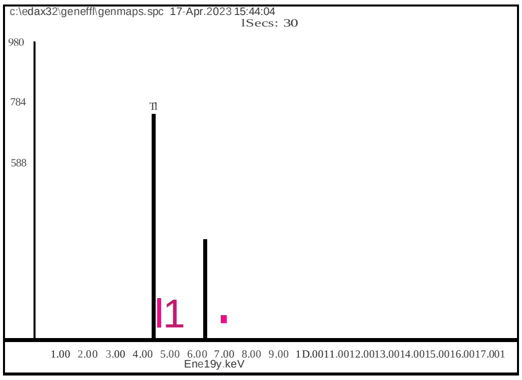
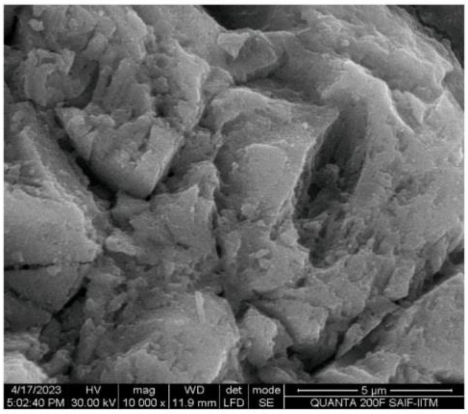
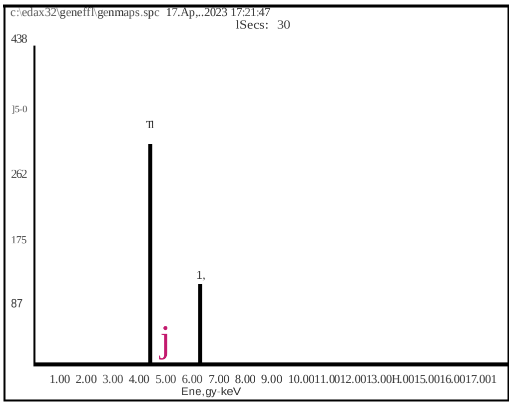
Fig. 4.5: Calcination

CHAPTER 5

RESULTS AND DISCUSSION

The high grade TiO2 can be obtained and the purity is checked using Scanning Electron.

Microscope and Electron Dispersive spectroscopy. The EDS and SEM analysis of the products are as follows.

SEM	***EDS***
 ***Fig. 5.1***	 ***Fig. 5.2***
 ***Fig. 5.3***	 ***Fig. 5.4***

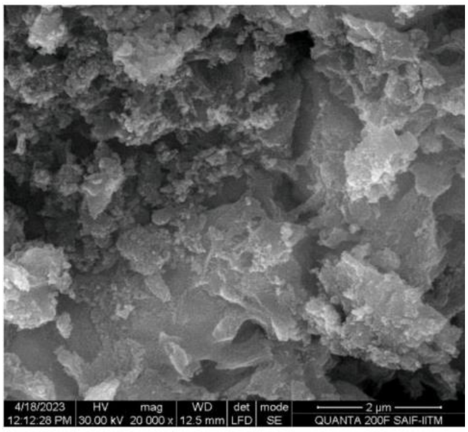


Fig. **5.5**

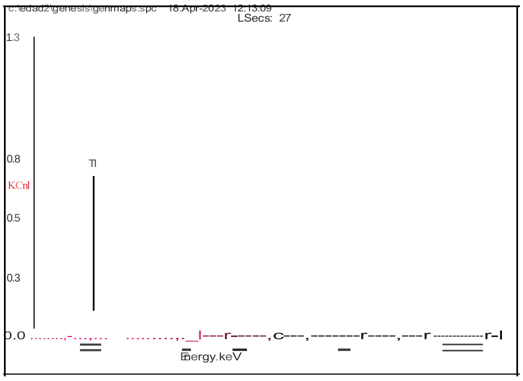


Fig. **5.6**

5.1 ANALYSIS OF 1:2 RATIO

The SEM and EDS analysis figures for the obtained high-grade TiO2 have been named as Fig. 5.1 to Fig. 5.6. Among these, Fig. 5.1 shows the SEM and EDS analysis of the product obtained, whereas Fig. 5.2, Fig. 5.3, Fig. 5.4, Fig. 5.5, and Fig. 5.6 represents the SEM and EDS analysis of the product obtained at different temperatures with a ratio of 1:2. According to the EDS analysis, the weight by percentage and atomic percent of TiK and OK in the products obtained at different temperatures with a ratio of 1:2 are as follows:

Based on the EDS analysis, it can be inferred that the ratio 1:2 at 500°C provided the best result for TiK, with a weight by percentage of 59.09% and an atomic percent of 32.55%, which is better than the results obtained at 400°C and 600°C. Therefore, it can be concluded that the product obtained with a ratio of 1:2 at 500°C is the most suitable for obtaining high-grade TiO2 with better purity.

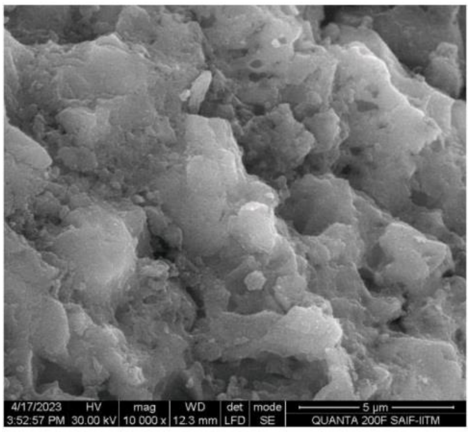


Fig. 5.7

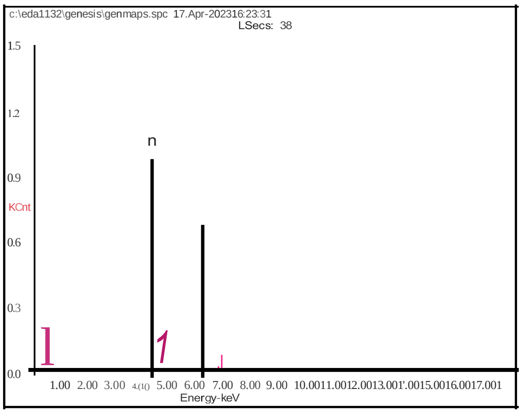


Fig. 5.8

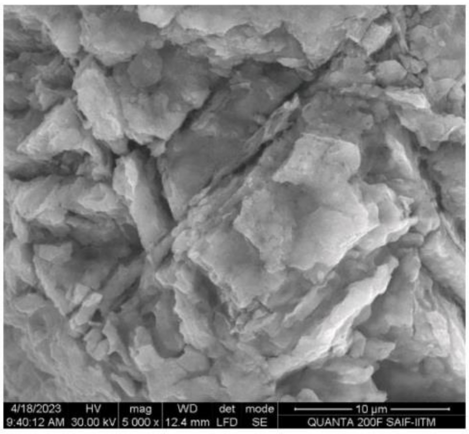


Fig. 5.9

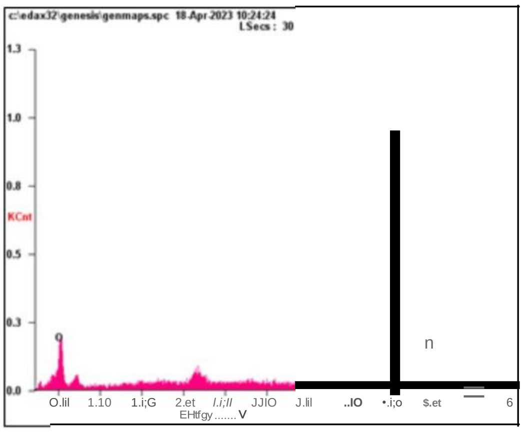


Fig. 5.10

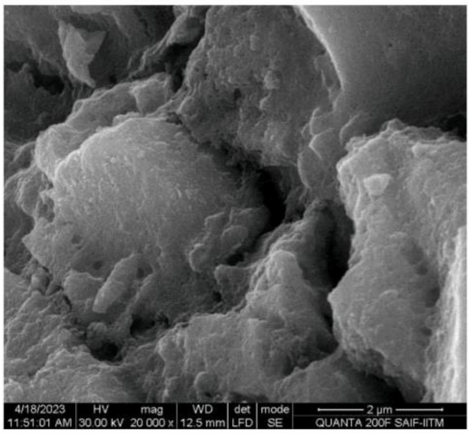


Fig. 5.11

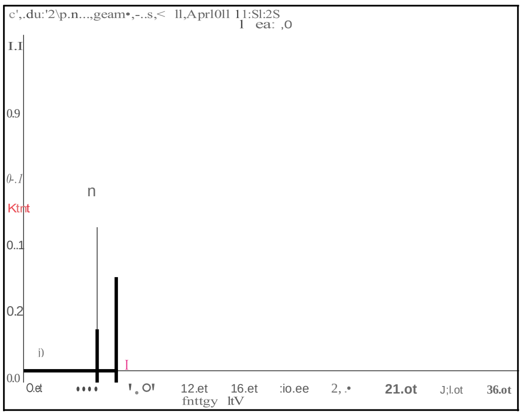


Fig. 5.12

5.2 ANALYSIS OF 1:3 RATIO

The SEM and EDS analysis figures for the obtained high-grade TiO2 have been named as Fig. 5.7, to Fig. 5.12. Among these, Fig. 5.7 shows the SEM and EDS analysis of the product obtained, whereas Fig. 5.8, Fig. 5.9 Fig. 5.10, Fig. 5.11 and Fig. 5.12 represents the SEM and EDS analysis of the product obtained at different temperatures with a ratio of 1:3. According to the EDS analysis, the weight by percentage and atomic percent of TiK and OK in the products obtained at different temperatures with a ratio of 1:3 are as follows:

Based on the EDS analysis, it can be inferred that the ratio 1:2 at 500°C provided the best result for TiK, with a weight by percentage of 59.09% and an atomic percent of 32.55%, which is better than the results obtained at 400°C and 600°C. Therefore, it can be concluded that the product obtained with a ratio of 1:2 at 500°C is the most suitable for obtaining high-grade TiO2 with better purity.

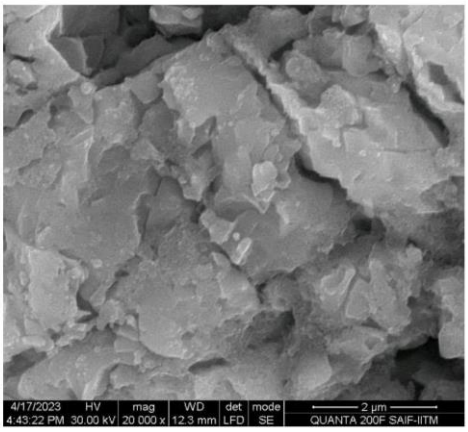
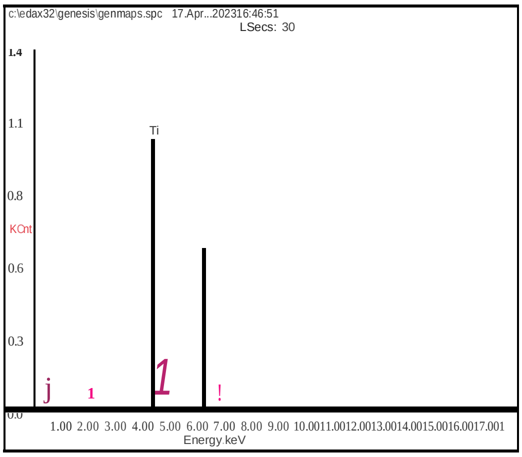
	

Fig. 5.13	***Fig. 5.14***

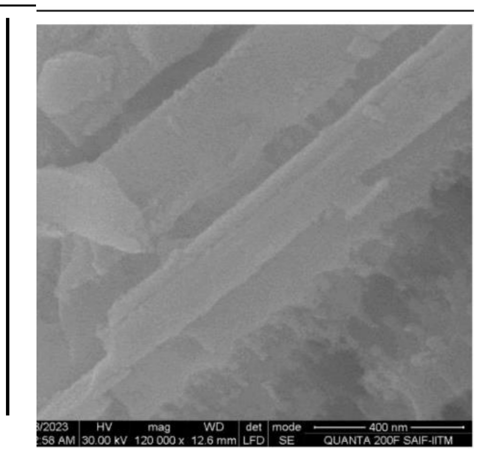


Fig. 5.15

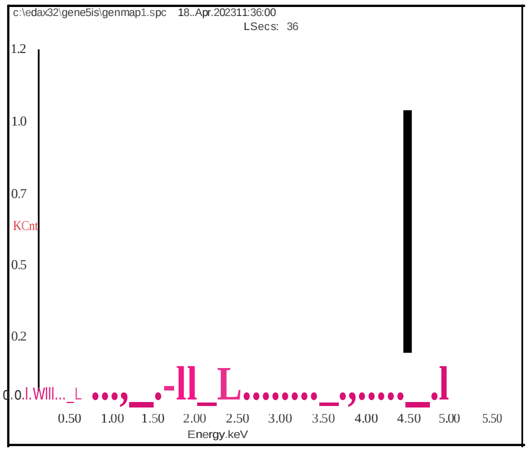


Fig. 5.16

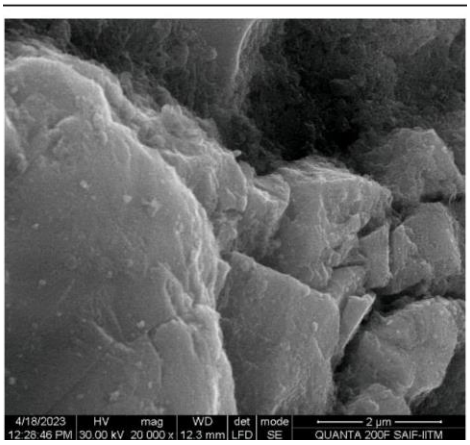


Fig. 5.17

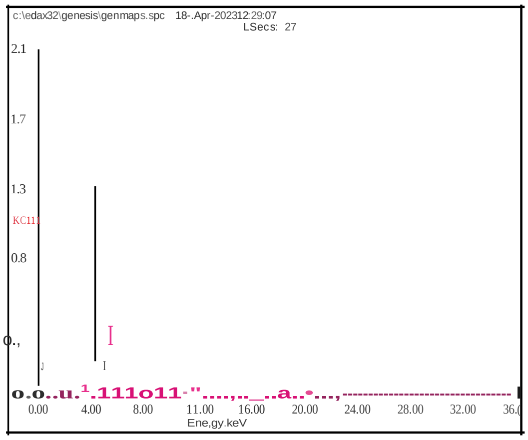


Fig. 5.18

5.3 ANALYSIS OF 1:4 RATIO

The SEM and EDS analysis figures for the obtained high-grade Ti02 have been named as Fig. 5.13,to Fig. 5.18. Among these, Fig. 5.13 shows the SEM and EDS analysis of the product obtained, whereas Fig. 5.14, Fig. 5.15 Fig. 5.16, Fig. 5.17 and Fig. 5.18 represents the SEM and EDS analysis of the product obtained at different temperatures with a ratio of 1:4. According to the EDS analysis, the weight by percentage and atomic percent of TiK and OK in the products obtained at different temperatures with a ratio of 1:4 are as follows:

Based on the EDS analysis, it can be inferred that the ratio 1:2 at 500°C provided the best result for TiK, with a weight by percentage of 59.09% and an atomic percent of 32.55%, which is better than the results obtained at 400°C and 600°C. Therefore, it can be concluded that the product obtained with a ratio of 1:2 at 500°C is the most suitable for obtaining high-grade TiO2 with better purity.

The below formula provided is used to calculate the weight percentage of TiO2 in a sample based on the EDX analysis of Titanium (Ti) and the matrix (other elements in the sample). TiK represents the weight percentage of Ti in the sample, and OK represents the weight percentage of the matrix in the sample. The formula works by dividing the weight percentage of Ti by the weight percentage of the matrix, then multiplying it by 100 divided by the sum of the weight percentages of Ti and the matrix. This gives the weight percentage of TiO2 in the sample.

Table 5.1 Percentage of TiO2 at Different Temperatures

RATIO/TEMPERATURE	400°C	SOO^{0}C	600°C
1:2	49.71	68.11	48.78
1:3	65.22	66.09	51.14
1:4	64.96	71.9	57.77

The results obtained from the EDS and SEM analysis clearly indicate that the 1:3 ratio of Ilmenite and Sodium Hydroxide at 500°C calcination temperature is the most effective condition for producing pure Titanium Dioxide. At this condition, the EDS analysis revealed that the TiK content is 47.77 (wt%) and
23.40 (At %), which is the highest compared to the other conditions studied. Additionally, the SEM images for this condition (Fig. 5.9 and 5.10) show a high degree of crystallinity and uniformity in the TiO2 particles, indicating that the reaction is proceeding effectively.

On the other hand, the other conditions studied, such as 1:2 ratios at 450°C, 500°C, and 600°C, and 1:3 ratios at 400°C and 600°C, show lower TiK content and less uniform particle distribution. Therefore, these conditions are not as effective as the 1:3 ratio at 500°C for producing pure TiO_2.

In conclusion, the results suggest that the 1:3 ratio of Ilmenite and Sodium Hydroxide at 500°C is the optimal condition for obtaining high-purity TiO_2. These findings can be useful in designing and optimizing the process for large- scale production of TiO2.

CHAPTER 6

CONCLUSION

Based on the information provided, it appears that the extraction of high-grade Titanium di-oxide from ilmenite concentrate is a complex process that involves multiple steps, including sample preparation, caustic fusion, leaching, hydrolysis, and calcination. The efficiency of the extraction process is strongly influenced by the concentration of hydrochloric acid used for leaching. The synthesized TiO2 powder showed a purity of 71.9% in anatase phase, and impurities such as Fe and Si contributed to the high photocatalytic activity of the synthesized TiO2. However, the recombination of Fe and TiO2 resulted in poor photocatalytic properties for TiO2, which can be improved by optimizing the calcination temperature.

Interestingly, while the study by Y Aristanti and Y I Supriyatna [4] suggested that a calcination temperature of 650°C was optimal for achieving maximum photocatalytic activity of TiO2, the experiment mentioned in this question observed that a temperature of 550°C was best for calcination for a 1:3 ratio of ilmenite and NaOH. This discrepancy could be due to differences in the specific experimental conditions, such as the ratio of reactants and the type of ilmenite concentrate used.

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