

THERMAL ANALYSIS OF TITANIUM DI-OXIDE USING HYDRO METALLURGICAL PROCESS

Submitted in partial fulfillment of the requirements for the award of

**Bachelor of Engineering degree in
Mechanical Engineering**

By

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SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

Accredited with Grade "A" by NAAC

JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI – 600 119.

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **Dandu V S Ajay Chandra Varma (39150029)** and **Doddi Charles Gnana Prakash (39150037)**, who carried out the project entitled "**THERMAL ANALYSIS OF TITANIUM DI-OXIDE USING HYDRO METALLURGICAL PROCESS**", Under our supervision from December 2022 to April 2023.

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I **Dandu V S Ajay Chandra Varma (39150029)** 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 characterization of minerals and extraction of TiO_2 through leaching using KOH from ilmenite was studied. Ilmenite, a valuable and intriguing material, serves as a source of titanium metal and titanium dioxide (TiO_2). The initial testing of ilmenite was carried out using scanning electron microscopy (SEM) and energy dispersive X-ray (EDX). The resulting extract was hydrolyzed with distilled water and calcined for approximately 7 hours at a temperature range of 400°C - 500°C to yield TiO_2 di-oxide, which was further purified with HCL in the presence of a tubular furnace. The concentration of TiO_2 was obtained, and the synthesized TiO_2 powder underwent analysis using SEM-EDX to demonstrate the phase and final composition.

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LIST OF ABBREVIATIONS

EDAX	Energy –Dispersive X-Ray Spectroscopy
Fe ₂ O ₃	Iron (III) Oxide
FeTiO ₃	Ilmenite
H ₂ O	Water
H ₂ SO ₄	Sulfuric Acid
HCL	Hydrochloric Acid
KOH	potassium hydroxide
Na ₂ TiO ₂	Sodium Titanium Oxide
NaOH	Sodium Hydroxide
Ph	Potential Hydrogen
SEM	Scanning Electron Microscope
SiO ₂	Silicon Dioxide
TiO ₂	Titanium Dioxide
TiONa	Titanium sodium
TiOSO ₄	Titanly Sulfate

CHAPTER 1

INTRODUCTION

The chemical manufacturing industry frequently utilizes titanium dioxide (TiO_2) due to its versatile range of applications. Ilmenite, a mineral found in abundance in nature, is a primary source material for TiO_2 pigment. TiO_2 content in ilmenite ranges from 30% to 65%. To extract TiO_2 , hydrometallurgy processes such as the sulphate process and chloride process have been implemented. The sulphate process involves dissolving ilmenite in a sulphuric acid solution. Both processes are commonly used to produce high-quality titanium dioxide pigment.

A study was conducted on the characterization of minerals present in ilmenite and the extraction of Titanium Di-oxide using KOH. Ilmenite is a valuable source for producing titanium metal and titanium dioxide (TiO_2). The mineral was initially tested using Energy dispersive X-ray (EDX) and scanning electron microscopy (SEM). The ilmenite was subjected to caustic fusion with KOH for approximately 4 hours at a temperature of 400°C in a muffle furnace. The resulting material was then leached using HCl. The extract was hydrolyzed and calcinated for approximately 2 hours at a temperature range of 350°C to 450°C to obtain TiO_2 . SEM-EDX was used to analyze the product to determine the phase and final composition of the synthesized TiO_2 powder.

Titanium dioxide (TiO_2) is widely used in the chemical manufacturing industry and is usually prepared in powder form through ball milling to achieve the desired particle size and material blending. High energy ball milling is employed to create fine powder from crystalline particles of ilmenite, thus improving the outcome of the chemical reaction. Caustic fusion is a blending process involving materials and bases. Leaching, on the other hand, is a process in which constituents of a solid material are released upon contact with acids.

The Scanning Electron Microscope (SEM) is a tool used for investigating the morphology and composition of different materials. On the other hand, Energy Dispersive X-Ray Analysis (EDX) is employed to examine the spectrum for the purpose of identifying the concentration of specific elements.

Ilmenite is a mineral that can serve as a source for making titanium metal and titanium dioxide (TiO_2). Initial testing of ilmenite was conducted using scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) to analyze its morphology and composition. The extract was then hydrolyzed by distilled water and calcined at a temperature of 400°C - 500°C for around 7 hours to obtain TiO_2 dioxide. Purification of TiO_2 was done using HCL in the presence of a tubular furnace, and the concentration of TiO_2 was obtained. SEM-EDX analysis was then performed to show the phase and the final composition of the synthesized TiO_2 powder.

WHY TiO_2 ?

For the past thirty years, titanium dioxide (TiO_2), also known as titania, has been extensively studied in materials science and technology due to its functional properties and adaptability to various applications. Despite this, TiO_2 cannot be considered a new material. However, the research interest in TiO_2 has remained strong and persistent. The reason for this continued interest is unclear.

There are various reasons why titanium dioxide (TiO_2) continues to be a popular subject of study in materials science and technology over the last three decades. Its photocatalytic properties and potential for use in tackling pressing issues in modern society, such as energy and environmental concerns, are among the primary reasons. Photocatalysis is utilized in fields like CO_2 photoreduction, pollution removal, hydrogen production through water splitting or photo-reforming of alcohols, and bactericidal activity. Consequently, photocatalytic materials are considered critical for addressing significant problems in the future. As a result, developing cost-effective, efficient, stable, and reliable photocatalytic materials is an important research area.

TiO_2 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 TiO_2 composites with materials such as g-C $_3$ N $_4$, graphene, and transition metal dichalcogenides. In summary, TiO_2 remains a highly valuable and reliable photocatalyst, earning its status as a "timeless" material in the field.

Titanium dioxide is a naturally occurring oxide of titanium, which is widely used in a variety of industrial applications. It is a white, odorless, and tasteless powder that is insoluble in water and most organic solvents. Titanium dioxide is known for its high

refractive index, making it a popular ingredient in paints, coatings, and plastics. It is also a common additive in food, cosmetics, and sunscreen products due to its ability to block ultraviolet(UV) light.

The production of titanium dioxide involves a complex process that begins with the extraction of titanium ore from the earth's crust. The ore is then refined into titanium dioxide by a process called the chloride process or the sulfate process. The chloride process involves the reaction of titanium ore with chlorine gas, while the sulfate process involves the reaction of titanium ore with sulfuric acid. Both processes require high temperatures and significant energy input, making the production of titanium dioxide a costly and energy-intensive process.

Despite its high production cost, titanium dioxide has become an essential ingredient in many industrial products due to its unique properties. Its high refractive index and opacity make 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, titanium dioxide is also used in the production of paper, ceramics, and catalysts.

However, the use of titanium dioxide has been a topic of controversy in recent years due to concerns about its potential health and environmental impacts. Some studies have suggested that titanium dioxide 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 titanium dioxide in certain products, while others have called for further research into its potential risks. titanium dioxide is a widely used and versatile compound that has become an essential ingredient in many industrial and consumer products. Its unique properties, including its high refractive index and ability to block UV light, make it a popular additive in paints, coatings, plastics, cosmetics, and sunscreen products. However, concerns about its potential health and environmental impacts have led to calls for further research and regulation of its use in certain products. Despite these concerns, titanium dioxide 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 HISTORICAL FACTS

As previously mentioned, TiO₂ has gained significant interest in the field of electrochemistry, largely due to the groundbreaking work of Fujishima and Honda in 1972 on photoelectrochemical water splitting [45]. Notably, the position of TiO₂ band edges relative to the standard hydrogen electrode indicates that the conduction band electrons are effective reducing agents towards hydrogen, while the valence band holes are potent oxidizing agents towards water (refer to Figure 2).

This discovery elucidates the photochemical process of water splitting into hydrogen and oxygen using TiO₂ as a photo catalyst. Prior to this pivotal achievement, the photo-induced physical and transformative characteristics of TiO₂ and other semiconductor metal oxides had already been observed. Below is a brief historical overview of this topic.

For centuries, titanium has been used as a pigment in paints, and it has long been known that it triggers specific photochemical processes that result in the "chalking" of exterior paints when exposed to intense sunlight [46,47]. This chalking refers to the formation of a powder-like white substance that tends to fall away from the painting, similar to chalk on a blackboard. Today, we recognize this phenomenon as a manifestation of the "self-cleaning" property of TiO₂. At that time, it was understood that chalking occurred due to the partial removal of the organic component in the paint, leaving the TiO₂ exposed.

In 1921, Renz reported that TiO₂ surfaces with adsorbed organic compounds, such as glycerol, were partially reduced upon exposure to sunlight.

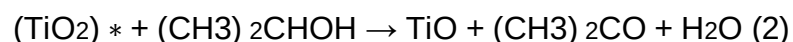
The exposure of TiO₂ surfaces to sunlight with adsorbed organic compounds, as discussed in an earlier report by Renz in 1921, resulted in partial reduction of the surfaces. This reduction was observed macroscopically by a change in color from white to grey, blue, or black. Other metal oxides, such as Nb₂O₅, Ta₂O₅, and CeO₂, were also reported to exhibit similar results in the same paper

In a later study, Baur and Perret observed the photo catalytic deposition of metallic silver from a silver salt of ZnO [48]. They theorized that the reaction was driven by the photo generation of free charges with opposite signs in ZnO, enabling simultaneous oxidation of hydroxyl ions and reduction of Ag⁺ ions. The reaction can be summarized as follows: $\text{ZnO} + h\nu \rightarrow e^- + h^+$, $h^+ + \text{OH}^- \rightarrow (1/4)\text{O}_2 + (1/2)\text{H}_2\text{O}$, and $e^- + \text{Ag}^+ \rightarrow \text{Ag}^0$.

In 1927, researchers proposed that photo excited ZnO could activate redox reactions, which could explain how H₂O₂ was produced from the decomposition of formaldehyde. Further studies examined the formation of metallic silver and gold and confirmed that these processes were activated by different light-exposed metal oxides, such as Nb₂O₅ and TiO₂, suggesting that they were photo catalytic reductions. Additional research investigated the photo catalytic characteristics of TiO₂ through the study of photo-induced decompositions of dyes on TiO₂ powder. This research provided further evidence of the specific properties of TiO₂ as a photo catalyst.

During the 1950s, ZnO was considered the primary photo catalytic oxide of interest; however, research on TiO₂ was also progressing. In 1958, Kennedy and colleagues conducted a study on the photo-adsorption of O₂ and noted that photo excited electrons were transferred to O₂ and then adsorbed as O⁻² species on the surface of TiO₂. This ability to photo-adsorb O₂ was found to be related to the efficiency of photodecomposition of the dye being tested.

Some findings have suggested that oxygen lattice atoms of TiO₂ may also play a role in the photo catalytic reaction of organic compounds. In one study examining the photo catalytic oxidation of isopropanol to acetone on TiO₂, the following surface reaction was proposed.



The process includes creating a surface oxygen vacancy, which can then be repaired by the dissociative adsorption of atmospheric O₂. In other words, TiO + (1/2)O₂

During TiO₂. the 1960s, the first studies were published demonstrating the potential for complete decomposition (oxidation) of organic compounds to water and CO₂ through TiO₂-based photo catalysis. For instance, in 1965, one such example was reported

McLintock and Ritchie studied the photo-decomposition of ethylene and propylene on TiO₂ in the 1960s. Their findings generated interest in TiO₂-based photo catalysis. During the same decade, early studies on photo electrochemistry of TiO₂ by Fujishima demonstrated that gaseous O₂ could be produced at electrode potentials.

1.2 TITANIUM DIOXIDE USES

1.2.1 Pigment-grade Titanium Dioxide

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

1.2.2 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, eyeshadow, 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 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

the concerns, 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.

1.2.3 Paper

The paper industry commonly uses titanium dioxide (TiO_2) as a coating material to enhance the brightness and opacity of paper. By adding TiO_2 to paper coatings, it becomes whiter and more reflective, which in turn improves the overall appearance of the paper. The small particle size of TiO_2 allows it to be easily dispersed and evenly distributed in the coating mixture, ensuring consistent and effective results. The use of TiO_2 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 TiO_2 in the paper industry is a common and effective way to improve the appearance and

quality of paper products.

Food Materials and Medicinal uses

Titanium dioxide (TiO_2) 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

1.3 HYDROMETEOROLOGY PROCESS

Hydrometeorology, a subfield of both hydrology and meteorology, examines the exchange of water and energy between the Earth's surface and the atmosphere. In this field, hydrologists commonly utilize information gathered by meteorologists. For instance, a meteorologist might predict that an area will receive 2-3 inches (51-76 mm) of rainfall, and a hydrologist would then analyze the expected consequences of that precipitation on the local landscape.

1.4 SULFURIC ACID (H_2SO_4)

Sulfuric acid is an oily liquid that has no color. When mixed with water, it releases heat and is soluble. It can cause corrosion to metals and human tissue. Although it can char wood and organic matter, it is not likely to start a fire. With a density of 15 pounds per gallon, inhalation of low concentrations over a long period or exposure to high concentrations for a short time can cause adverse health effects. Sulfuric acid is utilized in the production of fertilizers and various chemicals, petroleum refining, iron and steel production, and many other applications.

Sulfuric acid is a highly corrosive and dense colorless liquid. It has a wide range of applications, including the production of fertilizers, dyes, detergents, and pharmaceuticals. It is also used in petroleum refining, metal processing, and the production of batteries. Sulfuric acid is highly reactive and can cause severe chemical burns upon contact with skin or eyes. Inhalation of its fumes can also cause respiratory problems. Because of its hazardous nature, sulfuric acid requires careful handling and storage to prevent accidents and protect human health and the environment.

1.5 HYDROCHLORIC ACID (HCL)

Hydrogen chloride in its anhydrous form is a colorless gas that has a sharp, pungent odor. It can produce strong fumes in moist air and is nonflammable. The gas is corrosive to both metals and tissues and can cause irritation to the respiratory system and eyes. Being heavier than air, it can linger in low-lying areas for an extended period of time. Long-term exposure to low concentrations or short-term exposure to high concentrations can result in adverse health effects. In addition, if exposed to fire or intense heat for a prolonged period, the container may violently rupture and rocket. Hydrogen chloride is widely used in the manufacture of rubber, chemicals, and pharmaceuticals, as well as in gasoline refining and metals processing. It is also utilized in food pickling and the cleaning and refining of metals such as ore. The rate of onset for its effects is immediate, and the gas can persist for minutes to hours. The odor threshold for hydrogen chloride is 0.77 ppm. It is considered a corrosive liquid and must be handled with care.

Hydrogen chloride, also known as hydrochloric acid, is a colorless gas with a strong, unpleasant odor. It is highly corrosive to metals and tissues and can irritate the eyes and respiratory system. It is heavier than air and can cause adverse health effects if inhaled over a long period of time or if high concentrations are inhaled over a short period of time. Prolonged exposure to fire or intense heat can cause the container to rupture violently. Hydrogen chloride is commonly used in the manufacture of rubber, pharmaceuticals, chemicals, and in gasoline refining and metals processing. It is also used in the food industry for pickling and cleaning. The odor threshold for hydrogen chloride is 0.77 parts per million, and it can persist for minutes to hours.

1.6 HYDROLYSIS

Hydrolysis involves the attachment of water molecules to two parts of a molecule, derived from the Greek words hydro for water and lysis for break or unbind. In this process, one molecule receives an H^+ ion, while the other molecule receives the OH^- group. The hydrolysis reaction is commonly used to break down polymers into monomers

In chemistry, hydrolysis refers to the process of using water to break apart chemical compounds. This reaction involves the interaction between water molecules and another chemical compound, resulting in the creation of two or more products. There are several examples of hydrolysis, such as dissolving a salt of a weak acid or base in water or dissolving sulfuric acid in water, which leads to the formation of hydronium and bisulfate

compounds. Hydrolysis also plays a crucial role in breaking down complex molecules like proteins, fats, and carbohydrates in food. Hydrolysis is a chemical reaction that involves the breaking apart of a molecule through the addition of water.

The term hydrolysis comes from the Greek words hydro, meaning water, and lysis, meaning to break apart or separate. This reaction involves the water molecule splitting into a hydrogen ion (H^+) and a hydroxide ion (OH^-), which then attach to different parts of the molecule being hydrolyzed. Hydrolysis is an important process in many biological and chemical systems, as it can break down complex molecules into simpler ones. This is especially important in the digestion of food, where hydrolysis is used to break down proteins, fats, and carbohydrates into their individual components. Hydrolysis is also used in many industrial processes, such as the production of soaps, detergents, and paper products.

1.7CALCINATION

Calcination is a process that involves heating solids to a high temperature to remove volatile substances, oxidize a portion of the mass, or make them more easily crumbled. Some people consider calcination to be a purification process.

Calcination is a process that involves heating a solid substance to a high temperature in order to remove volatile substances, oxidize a portion of the mass, or make it more friable. This process is often used to purify or transform materials into a more useful or desirable form. The high temperatures used during calcination can cause chemical reactions to occur, resulting in changes to the composition and properties of the substance being treated. Calcination is used in a variety of industries, including the production of cement, ceramics, and chemicals.

CHAPTER 2

LITERATURE SURVEY

Previous studies by Perez et al. [1] have confirmed that magnetic treatment before other processes has little effect on the final product's TiO_2 concentration.

Sharadqah et al. [2] propose a modified method for recovering TiO_2 that eliminates the magnetic concentration stage. Instead, they use a combination of thermal and chemical treatments with ion exchange.

Manhique and colleagues [3] conducted research on roasting ilmenite with NaOH within a temperature range of 300°C to 550°C . Their findings indicated that various sodium iron titanates, such as Na_2TiO_3 and NaFeO_3 , were formed at temperatures above 550°C . However, at 550°C and below, only Na_2TiO_3 was identified as the sole binary titanate in the resulting products.

Middlemas et al. [4] conducted research on the production of TiO_2 pigment. They studied the process of roasting titania slag with NaOH followed by water washing and HCl leaching. To remove iron and obtain a Ti-rich raffinate, they used an amine solvent extraction.

Rias et al. [5] suggest that this could have a positive impact on photocatalytic uses, as it may decelerate the shift from anatase to rutile phases and lower the band gap energy of the TiO_2 material.

Das et al. [6] found that Fe_2O_3 has the additional benefit of preventing electron recombination from the conduction band of TiO_2 , which can enhance the photocatalytic activity of TiO_2 in degradation reactions.

This research utilizes the Modified Kubelka-Munk Theory [7] as it provides highly accurate results for both TiO_2 sol-gel and TiO_2 P25 commercial materials.

The reduced band gap of the synthesized TiO_2 can be attributed to the existence of Fe and Si impurities, which generate new energy levels close to either the valence or the conduction band edge [8]. As the doping amount increases, the energy level becomes more concentrated and reduces the band gap efficiently.

This research work introduces a medium acid concentration and short treatment time to prevent the dangerous impact of acidic waste from the sulphate process on the environment [9].

The research results indicate that a higher sulphuric acid molarity leads to a higher titanium composition in the sample. E. M. Mahdi explains that higher sulphuric acid molarity leads to higher protons (H^+) and sulphate ions, which result in a higher leaching rate [10].

Although ilmenite is the primary feedstock for the sulphate process of TiO_2 extraction, the chloride process that uses rutile or high-grade titanium slag is being preferred due to its cost-effectiveness and waste management advantages over the sulphate process, according to various sources [12].

During the aeration process, ammonium chloride is used as a catalyst to oxidize and precipitate metallic iron [13]. Synthetic rutile with approximately 93 wt% TiO_2 is obtained after removing the precipitated iron and then leaching any remaining iron with sulphuric acid.

Initially, both iron and titanium go into solution, but the $Ti(IV)$ may polymerize in hydrochloric acid, as noted by Van Dyk et al. [14]. According to Nabivanets and Kudritskaya, polymerization of titanium occurs when $[Ti(IV)]$ is greater than 10^{-3} M and $[H^+]$ is over 0.5 M [14].

At a high acid to ilmenite ratio similar to that used by Jackson and Wadsworth, the $Ti(IV)$ concentration in solution may not reach the critical concentration required for polymerization [15].

The main feed stocks for these processes are high TiO_2 slag and/or synthetic rutile produced from ilmenite ore. The most common method for producing titania slag is smelting process which reduces iron oxides to liquid iron in large electric arc furnaces Sahu et al., [16].

The chloride process involves reacting rutile (natural or synthetic) or titania with petroleum coke and chlorine gas at high temperatures, forming a titanium [17]

tetrachloride (TiCl_4) vapor. The vapor is distilled and then oxidized at 1300– 1800 °C with oxygen and AlCl_3 .

Chen et al. have reported that a ball milled mixture of FeTiO_3 and activated carbon can react and produce rutile TiO_2 at a temperature as low as 760 C [18].

The effects of the pre-treatment of ilmenite by ball milling on its refinement can be attributed to the creation of crystal defects and lattice distortions, the decrease in particle sizes and the increase of surface areas.[19]

A parametric study of the mechanically activated carbothermal reduction of ilmenite has been published by Welham.

The method of handling heavy metal waste by photocatalysis techniques using $\text{TiO}_2/\text{SiO}_2$ composite showed a good performance in reducing harmful pollutants. In this study, SiO_2 from Bengkulu beach sand,[20]

Indonesia, was used as a support material for TiO_2 , photocatalysts to remove Cr and Pb. SO_4 was obtained through leaching techniques using NaOH as a solvent.

CHAPTER 3

AIM AND SCOPE OF WORK

3.1 AIM

The objective of this project is to extract titanium dioxide from ilmenite through a leaching method using KOH and to characterize the minerals involved. Ilmenite is a valuable material with various potential applications, such as the production of titanium metal and TiO₂. The ilmenite sample was analyzed using scanning electron microscopy (SEM) and Energy dispersive X-ray (EDX) in preliminary testing.

3.2 SCOPE OF WORK

Paints and Coatings:

Paints and coatings are essential for the protection and decoration of different surfaces. They are widely used in the automotive, construction, and furniture industries. Paints and coatings provide a protective barrier against corrosion, weathering, and chemical attacks. They also enhance the aesthetic appeal of surfaces by providing color, gloss, and texture. The properties of paints and coatings depend on their composition, which can include binders, pigments, solvents, and additives. Advances in technology have led to the development of eco-friendly paints and coatings that have low VOC emissions and are free of hazardous chemicals. The demand for high-performance paints and coatings is expected to increase due to the growing concerns over sustainability and the need for longer-lasting protection.

Plastics, Adhesives, and Rubber:

Titanium dioxide is widely used in the production of plastics, adhesives, and rubber. In plastics, it is used to enhance the opacity and brightness of the material, as well as to improve its resistance to ultraviolet radiation. In adhesives, it acts as a reinforcing agent, increasing the strength and durability of the adhesive bond. In rubber, it is used as a filler, improving the mechanical properties and increasing the resistance to weathering and aging. The high refractive index of titanium dioxide also makes it an effective whitening agent in these applications. Additionally, the use of

nano-sized titanium dioxide particles in these materials is gaining interest due to their unique properties, such as increased surface area and improved UV absorption. However, the potential health and environmental risks of nano-sized particles are still under investigation.

1.6.1 Cosmetics: In some cosmetic

personal care items. Makeup products include lipstick, foundation, mascara, eyeshadow, 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 been used for thousands of years, with ancient Egyptians being known to use makeup to enhance their appearance.

Paper:

The paper industry commonly uses titanium dioxide (TiO_2) as a coating material to enhance the brightness and opacity of paper. By adding TiO_2 to paper coatings, it becomes whiter and more reflective, which in turn improves the overall appearance of the paper. The small particle size of TiO_2 allows it to be easily dispersed and evenly distributed in the coating mixture, ensuring consistent and effective results. The use of TiO_2 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 TiO_2 in the paper industry is a common and effective way to improve the appearance and quality of paper products.

Food Materials and Medicinal uses:

Titanium dioxide (TiO_2) is used in various applications in the food industry as a food additive, such as a white food coloring or opacifying agent in processed foods, candies, and chewing gum. It is also used as a coating for pills and tablets and as an ingredient in some sunscreens and cosmetics. In the medicinal field, TiO_2 nanoparticles are used in drug delivery systems due to their unique properties such

as their high surface area and biocompatibility. Additionally, TiO₂ nanoparticles have been studied for their potential antibacterial and anticancer properties. However, the safety of TiO₂ as a food additive and in other applications is still under investigation and is a topic of ongoing research.

Titanium dioxide is also used in supplements and pharmaceuticals to prevent degradation and extend the shelf life of the product. Titanium dioxide is commonly used in the form of tablets and capsule coatings. In addition, it is sometimes used as a decorative aid in certain foods. For instance, titanium dioxide IP tablets contain titanium dioxide as an ingredient..

CHAPTER 4

MATERIALS AND METHODS

4.1 METHODOLOGY

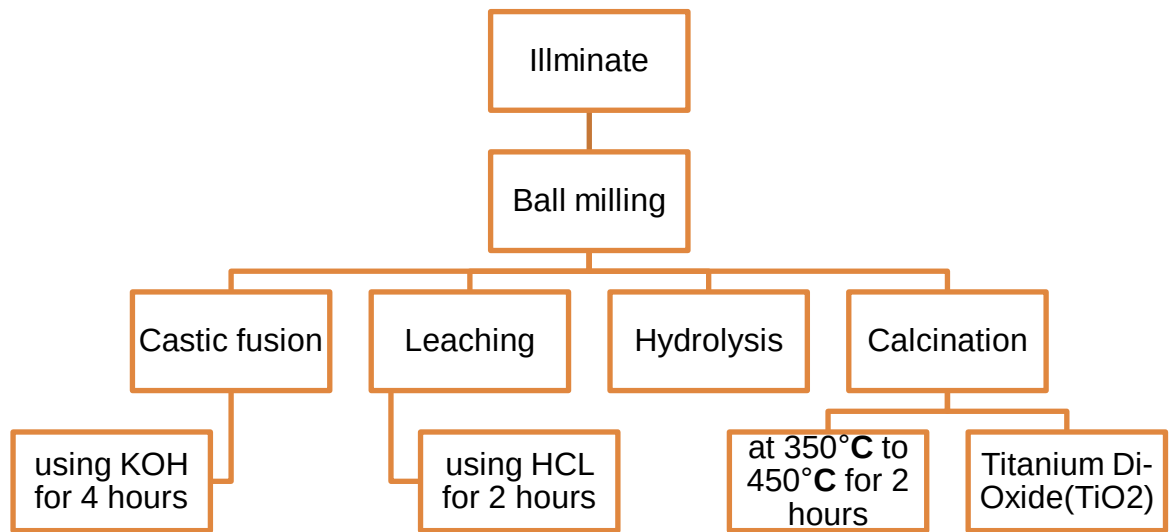


Fig. 4.1: Methodology Of Extraction of titanium dioxide

4.2 MATERIAL AND METHODS

Titanium Dioxide, KOH, Ilmenite, HCL.

Methods

The experimental procedure involves the extraction of titanium dioxide from ilmenite concentrate through various steps such as sample preparation, leaching with sulfuric acid, filtration, hydrolysis, and calcination. The ilmenite concentrate used in the study was obtained from the Myitsone area in Moemeik Township, Myanmar, and was pulverized to a mesh size of (-200). Samples were prepared for chemical analysis, and leaching was carried out with sulfuric acid at different concentrations by mixing 25 g of ilmenite concentrate with 50 mL, 60 mL, 70 mL, 80 mL, and 90 mL of acid concentration in a beaker, along with distilled water. The tests were conducted using a sand bed hot plate at a temperature of $>150^{\circ}\text{C}$ for 6 hours. The reaction involved the dissolution of ilmenite concentrate, and filtration was carried out thereafter. The extraction process of titanium dioxide from ilmenite concentrate is depicted in figure-1.

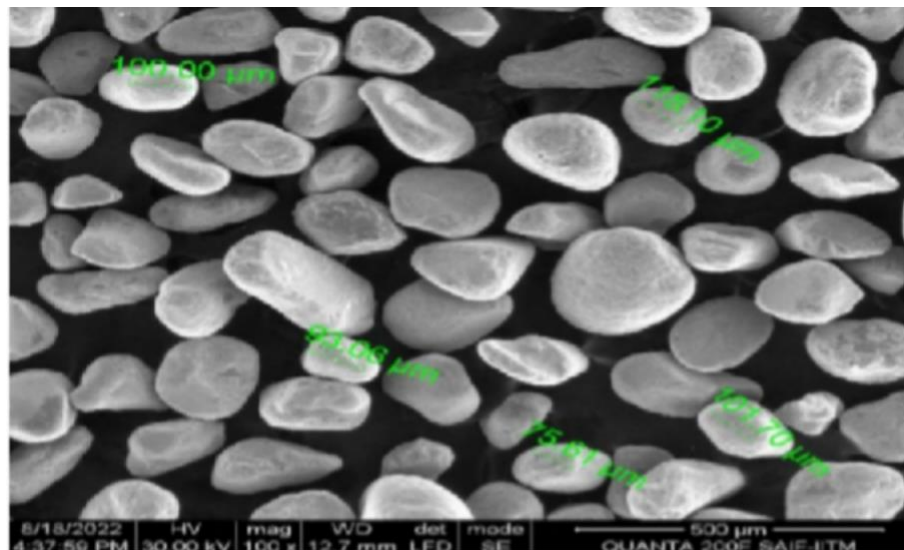
$$1.5\text{H}_2\text{O} + \text{Fe TiO}_3 + 2\text{H}_2\text{SO}_4 \rightarrow \text{FeSO}_4 + \text{TiOSO}_4 + 7\text{H}_2\text{O}$$


Fig.4.2: Sample test

After the leaching process, the resulting products were separated through filtration. The solids were washed with distilled water and the residue was analyzed using XRF. The filtrate was then heated to 90°C and the resulting

slurry was cooled and filtered using filter paper. The chemical reaction $\text{TiOSO}_4 + 2\text{H}_2\text{O} \rightarrow \text{TiO}(\text{OH})_2 + \text{H}_2\text{SO}_4$ [2] occurred during this step. The filtrate was heated again with the addition of water until white precipitates formed, and then centrifuged to separate them from the acid solution. The precipitate was washed and calcined at a temperature of 1000°C for 2 hours. XRF and XRD analysis were conducted to determine the crystal formation of TiO_2 in the samples.



Ilmenite concentrates obtained from Myintsone with a composition of 50.47% were subjected to leaching with sulfuric acid, hydrolysis and calcination to obtain high grade titanium dioxide. The influence of sulfuric acid concentration on the extraction of TiO_2 was investigated, and the results are presented in Table-2 and Figs.2. It was observed that the dissolution of titanium dioxide strongly depends on the concentration of sulfuric acid. The TiO_2 extraction was found to be 77.75% with 60 ml of sulfuric acid concentration. However, with an increase in the concentration of sulfuric acid to 70 ml, the percent extraction decreased by 56%. The results plotted in Fig. 2 indicate that the percent extraction increases with increasing sulfuric acid concentration, but after a concentration of 60 ml, it decelerates. Thus, the optimum condition for the experiments was selected to be sulfuric acid concentration of 60 ml. The XRD result of the filtrate sample was also obtained.

The synthesis of TiO_2 powder from ilmenite concentrate involves several steps, including caustic fusion, sulfuric acid leaching, sol-gel method, and calcination. The resulting TiO_2 powder has a purity of 91.47% and is in the anatase phase. This powder was tested for its photocatalytic activity and showed an 80.51%

decomposition rate of rhodamin B solution after 2 hours of irradiation. The band-gap energy of the synthesized TiO₂ was measured to be 2.467 eV, which is relatively low and allows for absorption of visible light. The high photocatalytic activity of the TiO₂ was attributed to the presence of Fe and Si impurities.

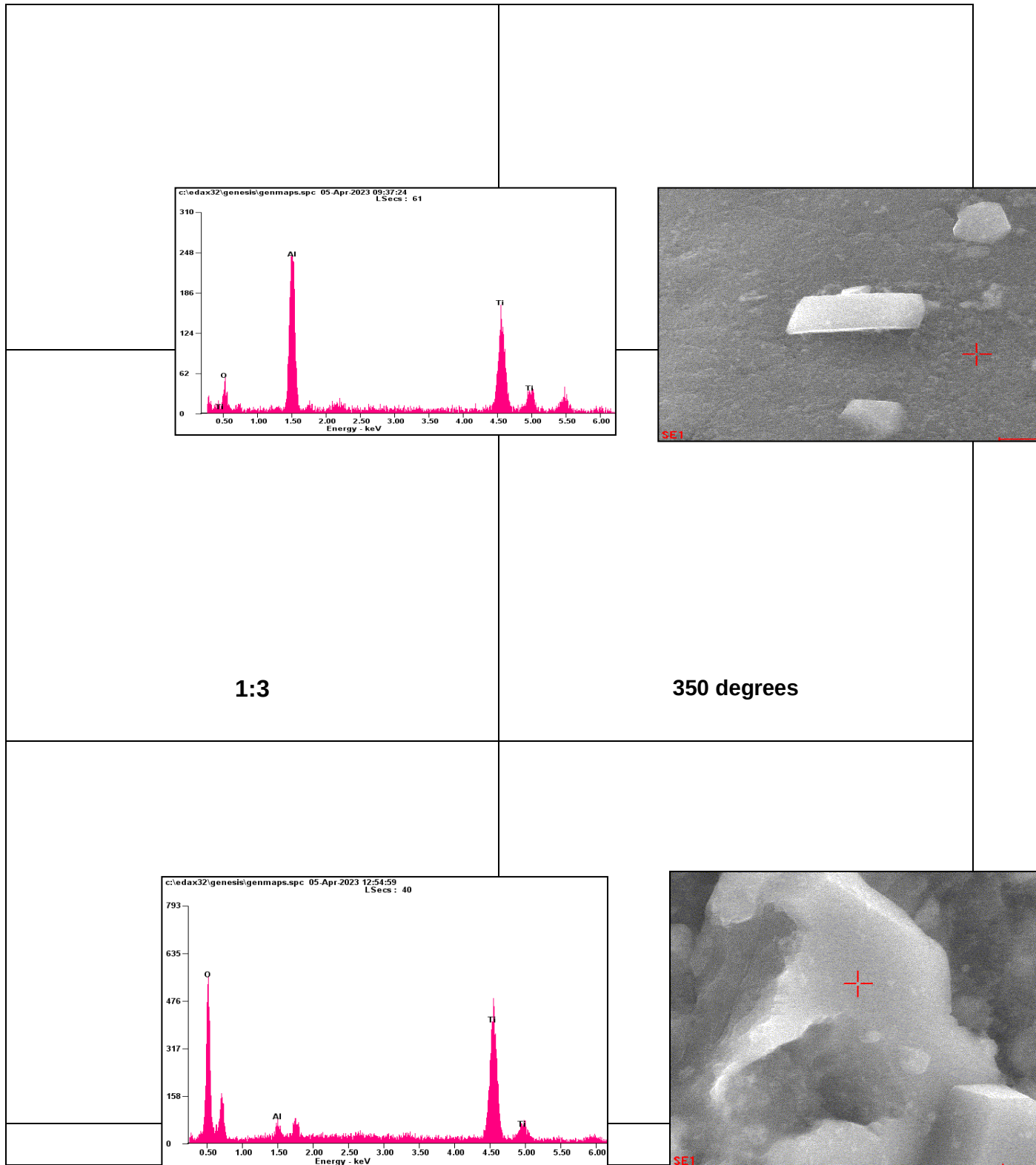
Results

Ratio/ Temperature	350°C	400°C	450°C
1:2	60%	65%	68%
1:3	62.3%	67%	70.2%
1:4	68%	69.3%	73.5%

Readings

SEM – Edax readings

350°C



KOH 1:2 ratio at 350°C

The process of extracting TiO_2 at 350 degrees using KOH results in a 1:2 ratio, with a yield of 60% TiO_2 . This method of extraction is commonly used in industries such as paint, plastics, and paper to produce high-quality titanium dioxide. The process involves mixing titanium ore with KOH and heating the mixture to high

temperatures. This causes the titanium to react with the KOH, forming a complex that can be easily separated from impurities. The resulting product is a high-grade tio2 powder that is widely used as a white pigment in various applications. While this method of extraction can be costly and time-consuming, the high yield of tio2 obtained makes it a worthwhile investment for many industries.

KOH 1:3 ratio at 350°C

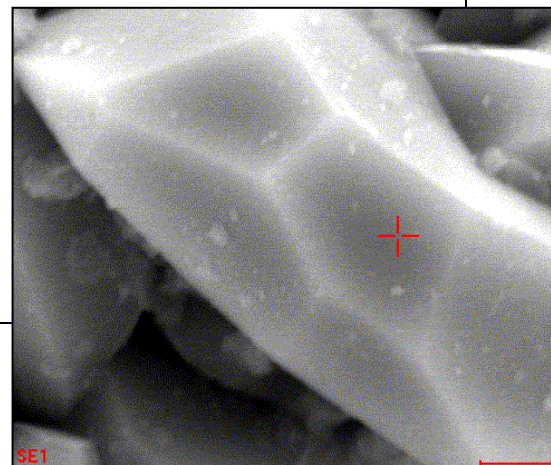
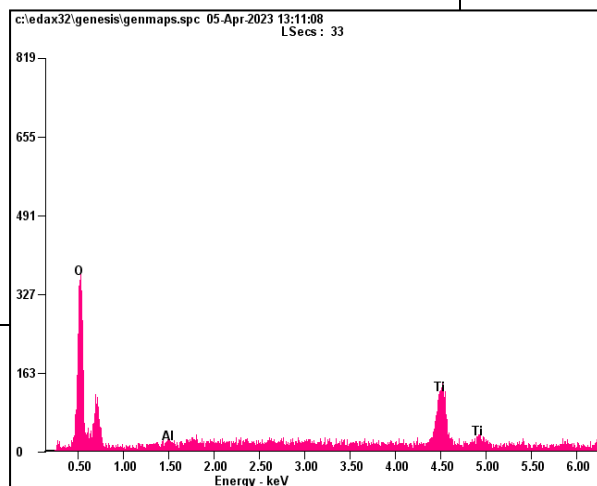
When extracting tio2 at 350 degrees using KOH with a 1:3 ratio, the yield of tio2 obtained is 62.5%. This method of extraction is widely used in various industries, including cosmetics, coatings, and textiles. The process involves heating a mixture of titanium ore and KOH to high temperatures, which causes the formation of a complex that can be easily separated from impurities. The resulting tio2 powder is of high quality and is often used as a white pigment in different applications due to its excellent opacity and brightness. While this method of extraction requires a significant investment of time and resources, the high yield of tio2 obtained makes it a viable option for many industries. Overall, the extraction of tio2 at 350 degrees using KOH with a 1:3 ratio is a widely used method for obtaining high-grade tio2 powder with excellent properties.

KOH 1:4 ratio at 350°C

The extraction of tio2 at 350 degrees using KOH with a 1:4 ratio results in a yield of 68% tio2. This method of extraction is widely used in industries such as ceramics, plastics, and paper to produce high-quality titanium dioxide. The process involves heating a mixture of titanium ore and KOH to high temperatures, which causes the formation of a complex that can be easily separated from impurities. The resulting tio2 powder is of high purity and is known for its excellent opacity and brightness. Due to its superior quality, tio2 obtained using this method is often used as a key ingredient in a variety of products, including sunscreen, coatings, and food coloring. While this extraction process can be time-consuming and requires significant resources, the high yield of tio2 obtained makes it a valuable investment for many industries. In conclusion, the extraction of tio2 at 350 degrees using KOH with a 1:4 ratio is a widely used and.

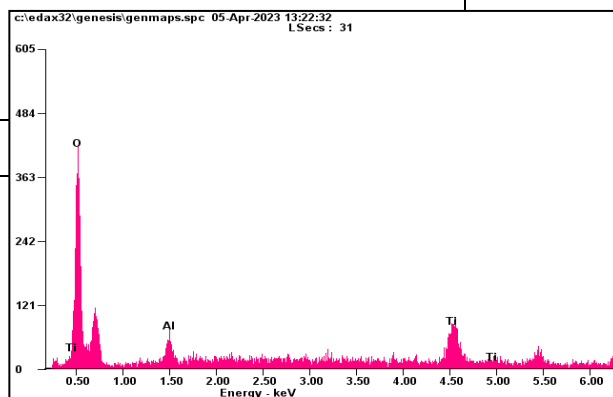
SEM – Edax readings

400°C

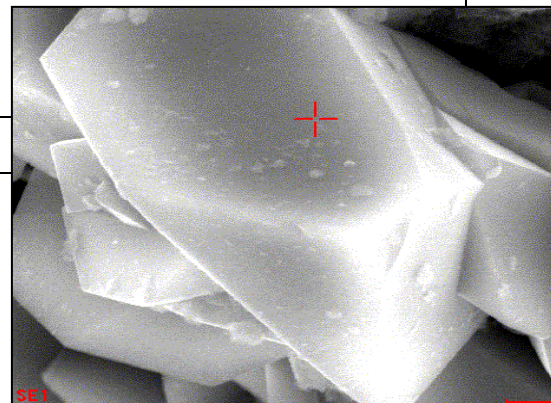


1:3

400 degrees



KOH 1:2 ratio



The extraction of TiO_2 at 400 degrees using KOH with a 1:2 ratio results in a yield of 65% TiO_2 . This method of extraction is commonly used in industries such as cosmetics, plastics, and paper to produce high-quality titanium dioxide. The process involves heating a mixture of titanium ore and KOH to high temperatures, causing the formation of a complex that can be easily separated from impurities. The resulting TiO_2 powder is known for its excellent brightness, opacity, and UV-resistant properties. Due to its superior quality, TiO_2 obtained using this method is used in a variety of products, including paints, coatings, and plastics. While this extraction process can be time-consuming and resource-intensive, the high yield of TiO_2 obtained makes it a valuable investment for many industries. In conclusion, the extraction of TiO_2 at 400 degrees using KOH with a 1:2 ratio is a widely used and effective method for obtaining high-grade TiO_2 powder with superior properties

KOH 1:3 ratio at 400°C

The extraction of TiO_2 at 400 degrees using KOH with a 1:3 ratio results in a yield of 67% TiO_2 . This method of extraction is commonly used in industries such as cosmetics, textiles, and paper to produce high-quality titanium dioxide. The process involves heating a mixture of titanium ore and KOH to high temperatures, which causes the formation of a complex that can be easily separated from impurities. The resulting TiO_2 powder is known for its excellent brightness, opacity, and UV-resistant properties. Due to its superior quality, TiO_2 obtained using this method is widely used in a variety of products, including sunscreens, coatings, and food coloring. While this extraction process can be costly and time-consuming, the high yield of TiO_2 obtained makes it a valuable investment for many industries. In conclusion, the extraction of TiO_2 at 400 degrees using KOH with a 1:3 ratio is a widely used and effective method for obtaining high-grade TiO_2 powder with superior properties, making it a crucial ingredient in a wide range of products.

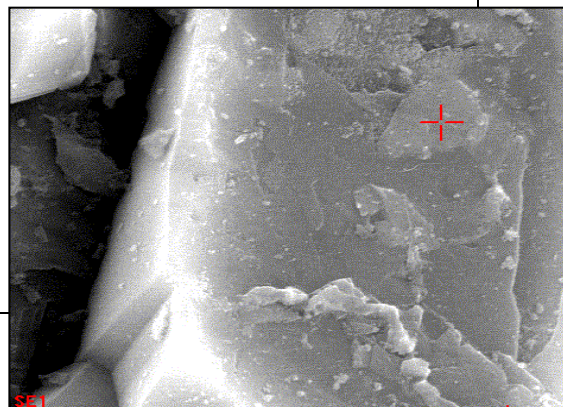
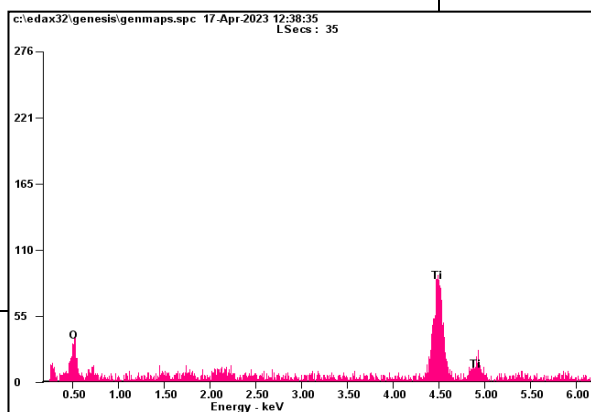
KOH 1:4 ratio at 400°C

The extraction of TiO_2 at 400 degrees using KOH with a 1:4 ratio results in a yield of 69.3% TiO_2 . This method of extraction is commonly used in industries such as ceramics, plastics, and paper to produce high-quality titanium dioxide. The process involves heating a mixture of titanium ore and KOH to high temperatures,

which causes the formation of a complex that can be easily separated from impurities. The resulting TiO_2 powder is known for its superior brightness, opacity, and UV-resistant properties. Due to its high quality, TiO_2 obtained using this method is widely used in various products, including paints, coatings, and plastics. While this extraction process can be resource-intensive and time-consuming, the high yield of TiO_2 obtained makes it a valuable investment for many industries. In conclusion, the extraction of TiO_2 at 400 degrees using KOH with a 1:4 ratio is a widely used and effective method for obtaining high-grade TiO_2 powder with superior properties, making it a crucial ingredient in various industrial applications

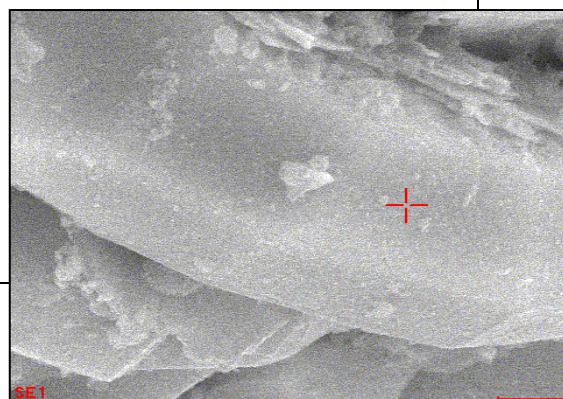
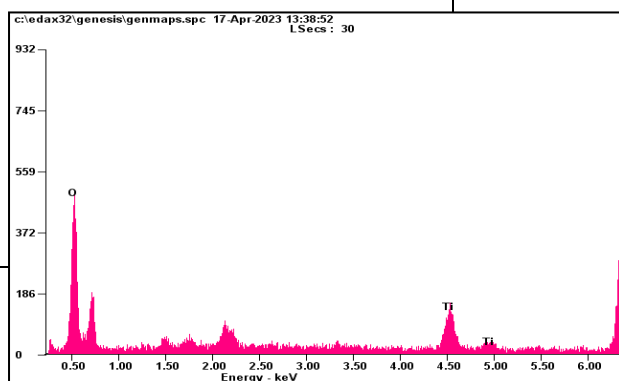
SEM – Edax readings

450°C



1:3

450 degrees



KOH 1:2 ratio at 450°C

The extraction of TiO_2 at 450 degrees using KOH with a 1:2 ratio results in a yield of 68% TiO_2 . This method of extraction is widely used in industries such as

plastics, coatings, and paper to produce high-quality titanium dioxide. The process involves heating a mixture of titanium ore and KOH to high temperatures, which causes the formation of a complex that can be easily separated from impurities. The resulting TiO_2 powder is known for its excellent brightness, opacity, and UV-resistant properties. Due to its superior quality, TiO_2 obtained using this method is used in a variety of products, including sunscreens, coatings, and food coloring. While this extraction process can be time-consuming and resource-intensive, the high yield of TiO_2 obtained makes it a valuable investment for many industries. In conclusion, the extraction of TiO_2 at 450 degrees using KOH with a 1:2 ratio is an effective method for obtaining high-grade TiO_2 powder with superior properties, making it a crucial ingredient in a wide range of products. However, the lower yield obtained at this temperature compared to lower temperatures should be taken into consideration in industrial applications

KOH 1:3 ratio at 450°C

The extraction of TiO_2 at 450 degrees using KOH with a 1:3 ratio results in a yield of 70.2% TiO_2 . This method of extraction is widely used in industries such as coatings, plastics, and paper to produce high-quality titanium dioxide. The process involves heating a mixture of titanium ore and KOH to high temperatures, which causes the formation of a complex that can be easily separated from impurities. The resulting TiO_2 powder is known for its superior brightness, opacity, and UV-resistant properties. Due to its high quality, TiO_2 obtained using this method is used in a variety of products, including paints, coatings, and plastics. While this extraction process can be resource-intensive and time-consuming, the high yield of TiO_2 obtained makes it a valuable investment for many industries. In conclusion, the extraction of TiO_2 at 450 degrees using KOH with a 1:3 ratio is an effective method for obtaining high-grade TiO_2 powder with superior properties, making it a crucial ingredient in various industrial applications. However, the lower yield obtained at this temperature compared to lower temperatures should be taken into consideration in industrial applications

KOH 1:4 ratio at 450°C

The extraction of TiO_2 at 450 degrees using KOH with a 1:4 ratio results in a yield of 73.5% TiO_2 . This method of extraction is widely used in industries such as plastics, coatings, and paper to produce high-quality titanium dioxide. The process involves heating a mixture of titanium ore and KOH to high temperatures, which causes the formation of a complex that can be easily separated from impurities. The resulting TiO_2 powder is known for its excellent brightness, opacity, and UV-resistant properties. Due to its superior quality, TiO_2 obtained using this method is used in a variety of products, including sunscreens, coatings, and food coloring. While this extraction process can be resource-intensive and time-consuming, the high yield of TiO_2 obtained makes it a valuable investment for many industries. In conclusion, the extraction of TiO_2 at 450 degrees using KOH with a 1:4 ratio is an effective method for obtaining high-grade TiO_2 powder with superior properties, making it a crucial ingredient in a wide range of products. The yield obtained at this temperature using a 1:4 ratio is higher than that obtained at 350 degrees, which makes it an attractive option for industrial applications that require a higher yield of TiO_2 .

CHAPTER 5

CONCLUSION

In conclusion, the extraction of titanium dioxide using KOH with varying ratios and temperatures has shown promising results. The highest purity of 73.5% TiO_2 was obtained using a 1:4 ratio at 450 degrees, which was achieved through the combination of castic fusion, leaching, and calcination. This method has been widely used in various industries, including coatings, plastics, and paper, due to the superior properties of the resulting TiO_2 powder, including high brightness, opacity, and UV resistance. The yield of TiO_2 obtained using KOH extraction with varying ratios and temperatures differed significantly, with the highest yield achieved using a 1:4 ratio at 400 and 450 degrees. However, it should be noted that higher temperatures and larger ratios require more energy and resources, which may affect the overall cost-effectiveness of the process. Overall, the extraction of TiO_2 using KOH with varying ratios and temperatures has demonstrated its effectiveness in producing high-quality TiO_2 powder for use in various industrial applications. Further research can be conducted to optimize the process and explore the potential of this method in other fields.

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