

# **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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**(DEEMED TO BE UNIVERSITY)**

**Accredited with Grade "A" by NAAC**

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**APRIL– 2023**



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## DEPARTMENT OF MECHANICAL ENGINEERING

### 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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## DECLARATION

We Dandu V S Ajay Chandra Varma (39150029) and Doddi Charles Gnana Prakash (39150037), 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.

DATE :26.04.2023  
PLACE: Chennai

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SIGNATURE OF THE CANDIDATE

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## ABSTRACT

Study on minerals characterization and  $\text{TiO}_2$  extraction by leaching method using  $\text{H}_2\text{SO}_4$  ilmenite. Ilmenite is an economically important and interesting material. This mineral can be used as the source of making titanium metal, titanium dioxide ( $\text{TiO}_2$ ). Results of initial testing using scanning electron microscopy (SEM) and Energy dispersive X-ray (EDX) on ilmenite. The extract was hydrolysis by distilled water and calcined for  $\pm 7$  hours at temperature of  $400^\circ\text{C}$ - $500^\circ\text{C}$  to obtain  $\text{TiO}_2$  di-oxide then purified with HCL in the presence of tubular furnace and the concentration of  $\text{TiO}_2$  is obtain and the analysis of the product is done using (SEM-EDX) analysis is include to show the phase and the final composition of the synthesized  $\text{TiO}_2$  powder.

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## LIST OF ABBREVIATIONS

|                                  |   |                                      |
|----------------------------------|---|--------------------------------------|
| EDAX                             | - | Energy-Dispersive X-Ray Spectroscopy |
| Fe <sub>2</sub> O <sub>3</sub>   | - | Iron (III) Oxide                     |
| FeTiO <sub>3</sub>               | - | Ilmenite                             |
| H <sub>2</sub> O                 | - | Water                                |
| H <sub>2</sub> SO <sub>4</sub>   | - | Sulfuric Acid                        |
| HCL                              | - | Hydrochloric Acid                    |
| KOH                              | - | Potassium Hydroxide                  |
| Na <sub>2</sub> TiO <sub>2</sub> | - | Sodium Titanium Oxide                |
| NaOH                             | - | Sodium Hydroxide                     |
| Ph                               | - | Potential Hydrogen                   |
| SEM                              | - | Scanning Electron Microscope         |
| SiO <sub>2</sub>                 | - | Silicon Dioxide                      |
| TiO <sub>2</sub>                 | - | Titanium Dioxide                     |
| TiNa                             | - | Titanium Sodium                      |
| TiOSO <sub>4</sub>               | - | Titanly Sulfate                      |

# CHAPTER 1

## INTRODUCTION

Titanium dioxide ( $\text{TiO}_2$ ) is one of the most commonly required materials in the chemical manufacturing industry due to its wide variety of applications. The mineral ilmenite abundantly in nature has major source material of  $\text{TiO}_2$  pigment. Ilmenite contains  $\text{TiO}_2$  to revolve 30% until 65%. Hydrometallurgy process has been carried out by acidic solution condition as a sulphate process, chloride process. Sulphate process was done through to dissolve ilmenite into sulfuric acid solution. This two processes are most widely used to produce the high quality titanium dioxide pigment.

Titanium dioxide ( $\text{TiO}_2$ ) is one of the most commonly used minerals in the chemical manufacturing industry. Ball Milling is used to make the powder to the finest form and blend materials. The High energy ball milling process is used in ilmenite to make the crystalline particles into fine powder to enhance the results in the chemical reaction. Now, Caustic Fusion is the process of blending the materials and bases. Leaching is a process by which constituents of a solid material are released when contacting with Acids.

Scanning Electron Microscope (SEM) allows both Morphology and Composition of various materials. Energy Dispersive X-Ray Analysis (EDX) used to analysis the spectrum in order to determine the abundance of specific elements.

This mineral can be used as the source of making titanium metal, titanium dioxide ( $\text{TiO}_2$ ). Results of initial testing using scanning electron microscopy (SEM) and Energy dispersive X-ray (EDX) on ilmenite. The extract was hydrolysis by distilled water and calcined for  $\pm 7$  hours at temperature of  $400^\circ\text{C}$ - $500^\circ\text{C}$  to obtain  $\text{TiO}_2$  di-oxide then purified with HCL in the presence of tubular furnace and the concentration of  $\text{TiO}_2$  is obtain and the analysis of the product is done using (SEM-EDX) analysis is include to show the phase and the final composition of the synthesized  $\text{TiO}_2$  powder.

## 1.1 WHY TiO<sub>2</sub>:

In the last three decades, titanium dioxide (TiO<sub>2</sub>, also named as titania) has been a major subject of study in materials science and technology for its functional properties and its versatility toward different applications. Hence, it can be reasonably said that TiO<sub>2</sub> is—by itself—not a “novel” material. Nevertheless, the many interests in it have neither waned nor slowed down over the past few years. Why is this?

Several answers to such a question can be given, but ultimately most of them are related to its photocatalytic properties and to the potentialities of photocatalytic technologies as a tool to tackle some of the most important challenges in modern society, such as environmental and energy issues. Photocatalysis finds application in important fields such as—to name some of the most important examples—CO<sub>2</sub> photoreduction [1,2], pollutant removal and pollution mitigation [3,4], hydrogen production via water splitting [5-7] or via photo-reforming of alcohols [8,9] and bactericidal activity [10-12]. Hence, photocatalytic materials are widely regarded as keystones for addressing some major problems we will face in the near future. Developing reliable, efficient, stable and unexpensive photocatalytic materials is thus regarded as a research field of great relevance.

TiO<sub>2</sub> is undoubtedly one of the most widely studied and exploited photocatalysts, an often considered as a sort of benchmark in the field for the reasons that will be discussed next. Some of the contemporary “hot topics” in energy conversion and in light-based transformational materials / systems involve composites based on TiO<sub>2</sub> (e.g., composites with g-C<sub>3</sub>N<sub>4</sub>, with graphene, with transition metal dichalcogenides, etc). In short, it can be stated that TiO<sub>2</sub> represents an “evergreen” photocatalyst and—most importantly—tha.

## 1.2 HISTORICAL FACTS

As mentioned, the interest in TiO<sub>2</sub> has been massively fostered by research in electro chemistry, and in particular by the mentioned 1972

achievement of photoelectrochemical water splitting by Fujishima and Honda [45]. It is to be noted that the position of the  $\text{TiO}_2$  band edges (vs. standard hydrogen electrode) indicates that conduction band electrons are good reducing agents toward hydrogen, whereas the valence band holes are strong oxidizing agents toward water (see Figure 2).

This explains the light-induced decomposition of water into oxygen and hydrogen without the application of an external bias. Before that breakthrough, the photophysical and photo-transformative properties of  $\text{TiO}_2$  (and of other semiconductor metal oxides as well) had been already evidenced: here we briefly sketch an historical outline of this subject.

Titania has been used for centuries as pigment in paints. As a consequence, it has been recognized since long ago that it activates specific photochemical processes, manifesting in the “chalking” of exterior paints when exposed to intense sunlight [46,47]. By chalking it is meant here that a powder-like white substance tended to be formed and to fall away from the painting, in a fashion similar to the chalk on a blackboard. Even from this simple description we can recognize this phenomenon as a manifestation of what is today called “self-cleaning” property of  $\text{TiO}_2$ . At the time, the chalking was correctly recognized as the effect of a partial removal of the organic component in the paint, leaving the  $\text{TiO}_2$  exposed.

For example, an earlier report by Renz in 1921 discussed how  $\text{TiO}_2$  surfaces with adsorbed organic compound (such as glycerol) were partially reduced when exposed to sunlight.

The reduction macroscopically manifested itself via a change in the color of titania, turning from white to grey, blue or black. Similar results were described in the same paper also for other metal oxides, such as  $\text{Nb}_2\text{O}_5$ ,  $\text{Ta}_2\text{O}_5$  and  $\text{CeO}_2$  [46].

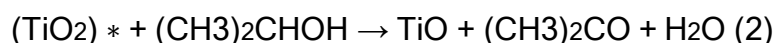
Few year later, Baur and Perret reported about the photocatalytic deposition of a silver salt of  $\text{ZnO}$  to produce metallic silver [48], hypothesizing

that the key element of the reaction was the photogeneration of free charges of opposite sign in ZnO that provided simultaneously oxidation of hydroxyl ions and reduction of Ag<sup>+</sup> ions, i.e.,  $\text{ZnO} + \text{H}_2\text{O} \rightarrow \text{e}^- + \text{h}^+$ ,  $\text{h}^+ + \text{OH}^- \rightarrow (1/4)\text{O}_2 + (1/2)\text{H}_2\text{O}$  and  $\text{e}^- + \text{Ag}^+ \rightarrow \text{Ag}^0$

In 1927, the ability of photoexcited ZnO to activate redox reaction was again invoked to explain the production of H<sub>2</sub>O<sub>2</sub> from decomposition of formaldehyde [49]. Later studies on formation of metallic silver and gold confirmed that these observed processes were indeed photocatalytic reductions activated by different light-exposed metal oxides, including Nb<sub>2</sub>O<sub>5</sub> and TiO<sub>2</sub> [50]. Other evidence of the specific photocatalytic characteristics of TiO<sub>2</sub> were provided by study of photo-induced decompositions of dyes on TiO<sub>2</sub> powder [51,52].

While during the 1950s the photocatalytic oxide of major interest was considered ZnO [53,54], the research on TiO<sub>2</sub> was also developing. Kennedy and coworkers in a 1958 study on photo-adsorption of O<sub>2</sub> underlined that photoexcited electrons were transferred to O<sub>2</sub> then adsorbed as O<sub>2</sub><sup>-</sup> species on the TiO<sub>2</sub> surface [55]. Such an ability to photo-adsorb the O<sub>2</sub> was found to be correlated to the efficiency of photodecomposition of tested dye.

Interestingly, some results also evidenced the possible involvement of oxygen lattice atoms of TiO<sub>2</sub> in photocatalytic reaction of organic compounds. For example, a study of the photocatalytic oxidation of isopropanol to acetone on TiO<sub>2</sub> suggested the following surface reaction [56];



This process involves the formation of a surface oxygen vacancy, which can next be healed by dissociative adsorption of atmospheric O<sub>2</sub>, i.e.,  $\text{TiO} + (1/2)\text{O}_2 \rightarrow \text{TiO}_2$ .

The 1960s witnessed the publication of the first studies demonstrating the possibility to fully decompose (oxidize) organic compounds to water and CO<sub>2</sub> via TiO<sub>2</sub>-based photo catalysis. One example was reported in 1965 by

McLintock and Ritchie, who investigated the photo-decomposition of ethylene and propylene on  $\text{TiO}_2$  [57]. In the same decade, early photoelectrochemistry studies of  $\text{TiO}_2$  by Fujishima triggered interest as they evidenced that gaseous  $\text{O}_2$  could be evolved at electrode potentials referred vs. standard hydrogen.

### **1.3 TITANIUM DIOXIDE USES**

#### **1.3.1 Pigment-grade Titanium Dioxide**

Pigment-grade type of titanium dioxide or titanium dioxide pigment is used in a wide range of uses that need high opacity and brightness. In fact, most surfaces and items that are coloured in white, pastel, and dark shades contain this oxide. The applications of pigment-grade titanium dioxide are given below in detail.

#### **1.3.2 Paints and Coatings**

It is used for the purpose of coating in the paint industry because its properties of opacity and durability of titanium dioxide help to ensure the longevity of the paint and protection of the surfaces which are painted.

#### **1.3.3 Plastics, Adhesives, and Rubber**

Titanium dioxide is helpful to reduce cracking, fading and the brittleness occurring in plastics and other materials as a result of exposure to light.

#### **1.3.4 Cosmetics**

In some cosmetic products pigment-grade titanium dioxide is used to help in order to hide blemishes and brighten the skin. For having the same effect it is used in the thinner coatings of make-up material.

#### **1.3.5 Paper**

Titanium dioxide ( $\text{TiO}_2$ ) is used in the paper industry for coating paper, brighter, making it whiter and more opaque.

### 1.3.6 Food Materials and Medicinal uses

The opacity to visible and ultraviolet light offered by titanium dioxide protects products of food items, beverages, supplements and pharmaceuticals from degradation in premature periods and enhances the longevity of the product. Titanium dioxide in medicine is continuously used by pharmaceuticals in the form of titanium dioxide tablets. This type of titanium oxide is used in titanium dioxide IP tablets, uses capsule coatings and as a decorative aid in some foods.

## 1.4 HYDROMETEOROLOGY PROCESS

**Hydrometeorology** is a branch of meteorology and hydrology that studies the transfer of water and energy between the land surface and the lower atmosphere. Hydrologists often use data provided by meteorologists.<sup>1</sup> As an example, a meteorologist might forecast 2-3 inches (51-76 mm) of rain in a specific area, and a hydrologist might then forecast what the specific impact of that rain would be on the local terrain.

## 1.5 SULFURIC ACID (H<sub>2</sub>SO<sub>4</sub>)

Sulfuric acid is a colorless oily liquid. It is soluble in **water** with release of heat. It is corrosive to metals and tissue. It will char wood and most other organic matter on contact, but is unlikely to cause a fire. Density 15 lb / gal. Long term exposure to low concentrations or short term exposure to high concentrations can result in adverse health effects from inhalation. It is used to make fertilizers and other chemicals, in petroleum refining, in **iron** and steel production, and for many other uses.

## 1.6 HYDROCHLORIC ACID (HCL)

Hydrogen chloride, anhydrous appears as a colorless gas with a sharp, pungent odor. Fumes strongly in moist air. Nonflammable. Corrosive to metals and tissues and irritating to the eyes and respiratory system. Heavier than air. Long-term inhalation of low concentrations or short-term inhalation of high

concentrations has adverse health effects. Prolonged exposure to fire or intense heat may result in the violent rupture and rocketing of the container. Used in the manufacture of rubber, pharmaceuticals, chemicals, and in gasoline refining and metals processing. Rate of onset: Immediate Persistence: Minutes to hours Odor threshold: 0.77 ppm Source/use/other hazard: Ore, other metal refining/cleaning; food/pickling; petroleum; corrosive liq.

## **1.7 HYDROLYSIS**

Hydrolysis is derived from a Greek word hydro meaning water and lysis which translates to the word break or to unbind. Usually in hydrolysis the water molecules get attached to two parts of a molecule. One molecule of a substance will get  $H^+$  ion and the other molecule receives the  $OH^-$  group. Hydrolysis reaction is mainly used to break down polymers into monomers.

So while studying chemistry, hydrolysis basically means the act of setting apart chemicals by adding water. The reaction of water with another chemical compound results in the formation of two or more products. Some examples of hydrolysis include dissolving a salt of a weak acid or base in water or dissolving sulphuric acid in water where hydronium and bisulfate compounds are formed. Hydrolysis also helps in breaking down proteins, fats, and complex carbohydrates in food.

## **1.8 CALCINATION**

Calcination, the heating of solids to a high temperature for the purpose of removing volatile substances, oxidizing a portion of mass, or rendering them friable. Calcination, therefore, is sometimes considered a process of purification.

## CHAPTER 2

### LITERATURE SURVEY

1. Perez et al. [1] previous studies have corroborated that the magnetic treatment prior to other processes does not significantly influence the concentration of the  $\text{TiO}_2$  in the final product.
2. Sharadqah et al. [2] Present a modified method of  $\text{TiO}_2$  recovery omitting the stage of magnetic concentration, applying the thermal and chemical treatments in combination with ion exchange
3. Manhique et al. [3] have studied ilmenite roasting with  $\text{NaOH}$  in the range of  $300^\circ\text{C}$  to  $50^\circ\text{C}$  and have indicated that different sodium iron titanates including  $\text{Na}_2\text{TiO}_3$  and  $\text{NaFeO}_3$  are formed above  $550^\circ\text{C}$  while at  $550^\circ\text{C}$  and below,  $\text{Na}_2\text{TiO}_3$  would be the unique binary titanate in the products.
4. Middlemas et al. [4] have studied the production of  $\text{TiO}_2$  pigment by roasting titania slag with  $\text{NaOH}$  and after water washing and  $\text{HCl}$  leaching solvent extraction using an amine was applied to remove iron leaving a Ti - rich raffinate.
5. Rias et al. [5] this will have a good effect on photocatalytic applications because it can slow down phase transformations from anatase to rutile and also reduce the band gap energy of the  $\text{TiO}_2$  itself.
6. Das et al. [6] In addition,  $\text{Fe}_2\text{O}_3$  can also prevent recombination of electrons from the conduction
7. Modified Kubelka-Munk [7]. Theory is used in this research due to its highly accurate results obtained with the  $\text{TiO}_2$  sol-gel and  $\text{TiO}_2$  P25 commercial.

The smaller band gap can be explained by the presence of Fe and Si impurities in the synthesized  $\text{TiO}_2$  which are creating new energy levels either near the valence or the conduction band edge [8]. With the

increase in the amount of doping, the energy level becomes denser and effectively decreases the band gap.

8. However, the acidic waste from the sulphate process can create a danger to our environment. [9] Therefore, only a medium acid concentration and short treatment time are introduced in this research work.
9. The results also show that the higher the acid molarity, the higher the titanium composition on that sample. [10] E. M. Mahdi, the higher the sulphuric acid molarity, the higher the protons (H<sup>+</sup>) and sulphate ions which lead to the higher leaching rate.
10. The ilmenite was mechanically activated by a ball mill, which is very important before leaching Li et al.,[11]
11. Ilmenite is the main feedstock for the extraction of TiO<sub>2</sub> pigment via the sulphate process, but increasingly the chloride process,[12] which uses rutile or high- grade titanium slag, is being favoured due to the fact that it appears to be more favourable in terms of cost and waste management, when compared to the sulphate process.
12. The metallic iron is oxidised and precipitated using an aeration process where ammonium chloride is used as a catalyst [13]. After the removal of precipitated iron, any remaining iron is removed by leaching with sulphuric acid, leaving behind synthetic rutile with around 93 wt.% TiO<sub>2</sub>.
13. According to Van Dyk et al.,[14] both iron and titanium go into solution initially but the Ti(IV) may polymerize in hydrochloric acid. Nabivanets and Kudritskaya determined the following criteria to indicate when polymerization of titanium would occur: [Ti(IV)] $\geq 10^{-3}$  M and [H<sup>+</sup>] $\geq 0.5$  M.
14. At a high acid to ilmenite ratio such as that used by Jackson and Wadsworth et.,[15] the Ti (IV) concentration in solution may never reach the critical concentration required for polymerization.

15. The main feed stocks for these processes are high  $\text{TiO}_2$  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].
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 ( $\text{TiCl}_4$ ) vapor. The vapor is distilled and then oxidized at 1300- 1800 °C with oxygen and  $\text{AlCl}_3$ .
17. Chen et al. have reported that a ball milled mixture of  $\text{FeTiO}_3$  and activated carbon can react and produce rutile  $\text{TiO}_2$  at a temperature as low as 760 C [18].
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]
19. A parametric study of the mechanically activated carbothermal reduction of ilmenite has been published by Welham.
20. 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,  $\text{SiO}_2$  from Bengkulu beach sand,[20]
21. Indonesia, was used as a support material for  $\text{TiO}_2$ , photocatalysts to remove Cr and Pb.  $\text{SO}_2$ , was obtained through leaching techniques using NaOH as a solvent.

## CHAPTER 3

### AIM AND SCOPE OF WORK

#### 3.1 AIM

The Aim of the project is to get the titanium dioxide Study on minerals characterization and  $\text{TiO}_2$  extraction by leaching method using  $\text{H}_2\text{SO}_4$  ilmenite. Ilmenite is an economically important and interesting material. This mineral can be used as the source of making titanium metal, titanium dioxide ( $\text{TiO}_2$ ). Results of initial testing using scanning electron microscopy (SEM) and Energy dispersive X-ray (EDX) on ilmenite

#### 3.2 SCOPE OF WORK

**Paints and Coatings:** It is used for the purpose of coating in the paint industry because its properties of opacity and durability of titanium dioxide help to ensure the longevity of the paint and protection of the surfaces which are painted.

**Plastics, Adhesives, and Rubber:** Titanium dioxide is helpful to reduce cracking, fading and the brittleness occurring in plastics and other materials as a result of exposure to light.

**Cosmetics:** In some cosmetic products pigment-grade titanium dioxide is used to help in order to hide blemishes and brighten the skin. For having the same effect it is used in the thinner coatings of make-up material.

**Paper:** Titanium dioxide ( $\text{TiO}_2$ ) is used in the paper industry for coating paper, brighter, making it whiter and more opaque.

**Food Materials and Medicinal uses:** The opacity to visible and ultraviolet light offered by titanium dioxide protects products of food items, beverages,

supplements and pharmaceuticals from degradation in premature periods and enhances the longevity of the product. Titanium dioxide in medicine is continuously used by pharmaceuticals in the form of titanium dioxide tablets. This type of titanium oxide is used in titanium dioxide IP tablets, uses capsule coatings and as a decorative aid in some foods

## CHAPTER 4

### MATERIALS AND METHODS

#### 4.1 METHODOLOGY

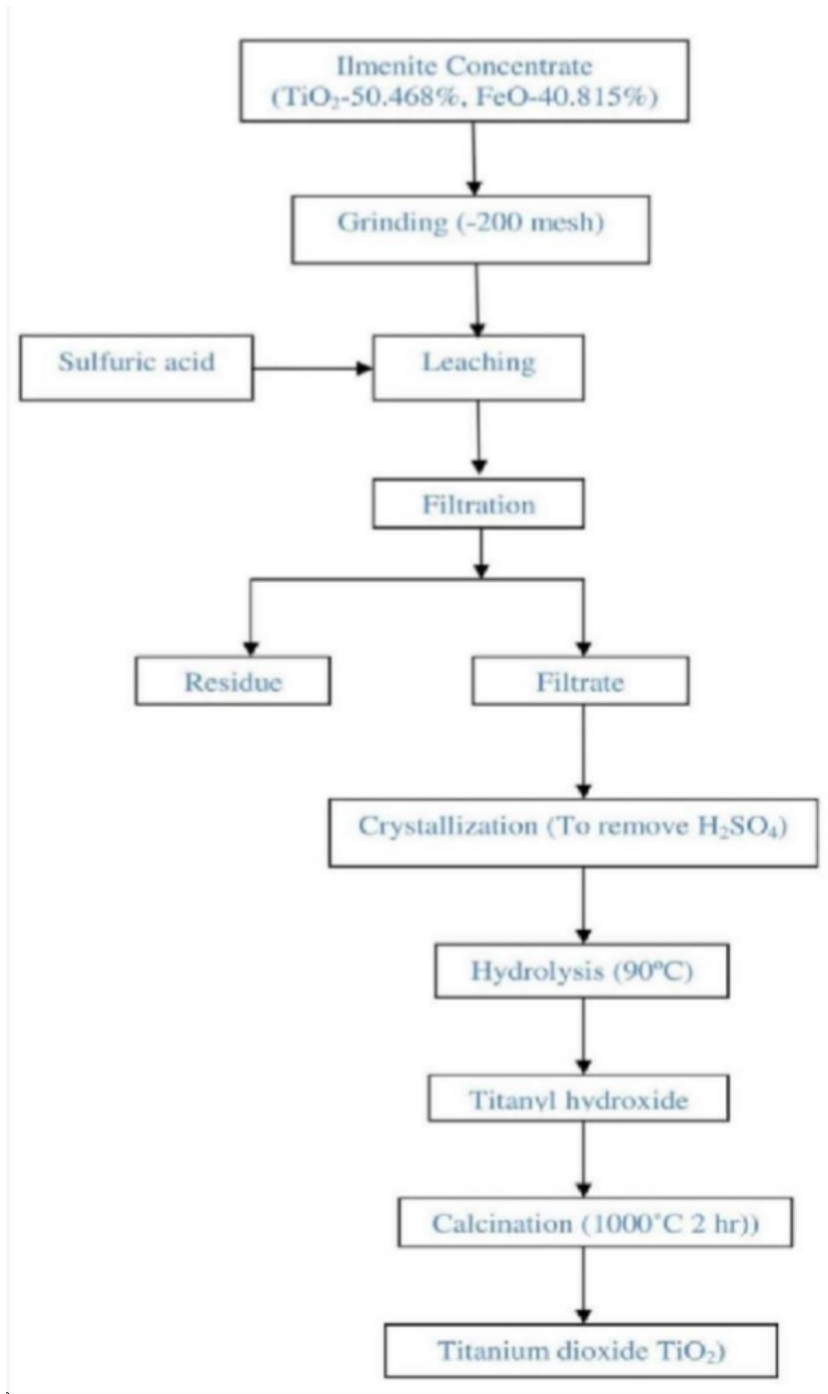


Fig. 4.1: Methodology

## 4.2 MATERIAL AND METHODS

Titanium Dioxide, H<sub>2</sub>SO<sub>4</sub>, Ilmenite, HCL.

### Methods

Experimental Procedure A. Maters The extraction process of titanium dioxide from ilmenite concentrate involves sample preparation, leaching with sulfuric acid, filtration, hydrolysis and calcination. The process of extraction of Titanium dioxide (TiO<sub>2</sub>) from ilmenite concentrate shows in figure-1. The ilmenite concentrate used in this study was obtained from Myitsone area in Moemeik Town ship in Myanmar. This concentrate was ground to (-200) mesh size by pulveriser. The concentrate was weight and samples were prepared for chemical analysis. Leaching was carried out with sulfuric acid at various concentrations. In all cases 25 g of ilmenite concentrate were mixed with 50 mL, 60 mL, 70 mL, 80 mL and 90mL of acid concentration in a beaker. Also, amount of distilled water were added to the beaker. The tests were performed, using sand bed hot plate temperature at >150° C for (6 hr). The following reaction is dissolution of ilmenite concentrate. Then filtration was carried out.

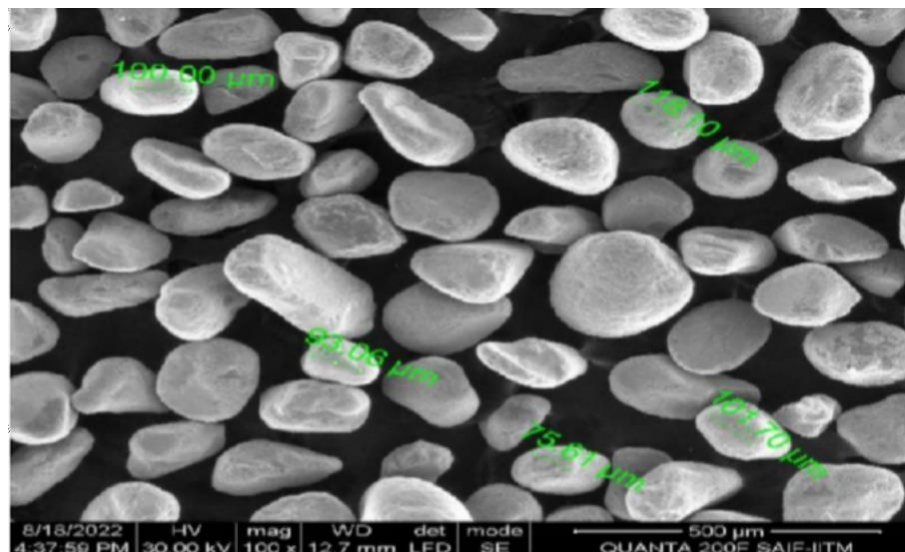
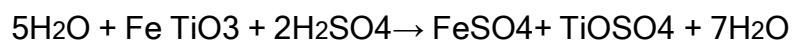
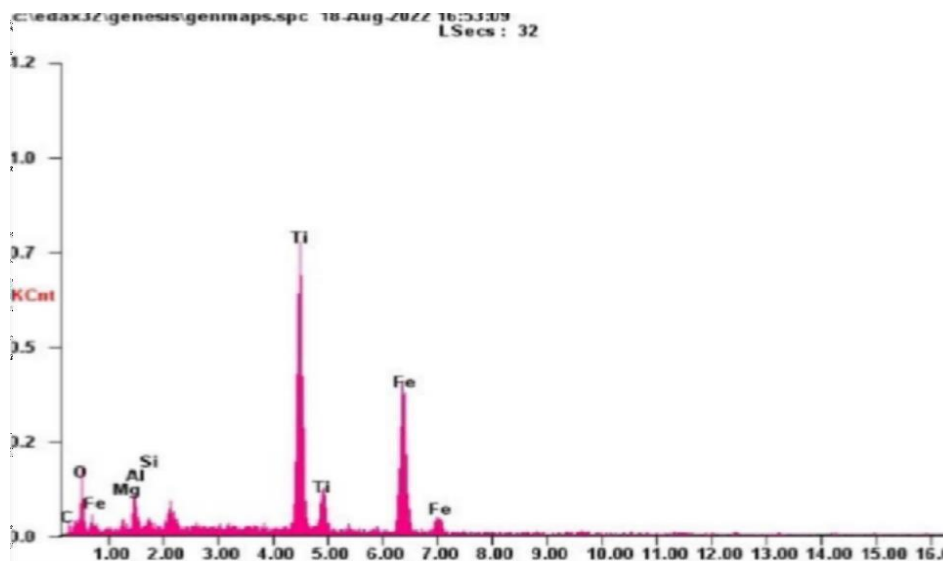


Fig.4.2: Sample test

After leaching process, the products were separated by filtration. Solids were washed with distilled water. The residue was determined by XRF. The filtrate was heated at 90°C. The slurry is cooled and filtered with filter paper.  $\text{TiOSO}_4 + 2\text{H}_2\text{O} \rightarrow \text{TiO}(\text{OH})_2 + \text{H}_2\text{SO}_4$  [2] Then the filtrate was heated by adding water until there were white precipitates and centrifuged to separate from the acid solution. The precipitate was washed and calcined at a temperature of 1000°C for 2 hours. To determine the crystal formed of  $\text{TiO}_2$ , the samples were analyzed using XRF and XRD.

Ilmenite concentrates contain 50.47% from Myintsone were leached with sulfuric acid, hydrolyzed and calcined to produce high grade titanium dioxide. The dependence of  $\text{TiO}_2$  extraction on sulphuric acid concentration is shown in Table-2 and Figs.2. The effect of sulfuric acid concentration has a strong influence on the dissolution of titanium dioxide. In the range of sulfuric acid concentration 50 to 90% test. After performing a number of tests, it was found that the  $\text{TiO}_2$  extraction 77.75% with sulphuric concentration 60 ml. The percent extraction reduces 56% with increasing sulfuric acid concentration to 70 ml. From the results plotted in fig. 2 it is evident that with increasing sulphuric acid concentration, percent extraction increases, but after acid concentration 60 ml it decelerates. Therefore sulphuric acid concentration 60 ml is selected as optimum condition for the experiments. The XRD result of the filtrate sample



**Fig. 5.1: SEM readings**

TiO<sub>2</sub> powder can be synthesized from ilmenite concentrate by combining caustic fusion process with sulfuric acid leaching followed by sol-gel method with the aid of calcination to convert TiO(OH)<sub>2</sub> to TiO<sub>2</sub> powder. TiO<sub>2</sub> powder with purity of 91.47% in anatase phase was obtained and used for photocatalytic evaluation, resulting in 80.51% decomposition rate of rhodamin B solution after 2 hours of irradiation. The measured band-gap energy of the synthesized TiO<sub>2</sub> was 2.467 eV. High photocatalytic activity of the synthesized TiO<sub>2</sub> was due to its low band gap energy which was causing TiO<sub>2</sub> to absorb visible light in high quantity rather than UV light. The presence of Fe and Si impurities in the synthesized TiO<sub>2</sub> powder were the main reason for the high photocatalytic activity of the synthesized TiO<sub>2</sub>

| <i><b>Element</b></i> | <i><b>Wt%</b></i> | <i><b>At%</b></i> |
|-----------------------|-------------------|-------------------|
| <i><b>CK</b></i>      | 09.20             | 19.70             |
| <i><b>OK</b></i>      | 27.82             | 44.72             |
| <i><b>MgK</b></i>     | 01.99             | 02.11             |
| <i><b>AlK</b></i>     | 05.52             | 05.26             |
| <i><b>SiK</b></i>     | 01.28             | 01.18             |
| <i><b>TiK</b></i>     | 27.20             | 14.60             |
| <i><b>FeK</b></i>     | 26.99             | 12.43             |
| <i><b>Matrix</b></i>  | Correction        | ZAF               |

Fig. 5.2: EDAX reading

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