UPGRADING OF TITANIUM DIOXIDE FROM ILMENITE CONCENTRATE

San Yu[1], Aung Myint Wai[2]

Abstract—Ilmenite is an economically important and interesting mineral. This mineral can be used as the source of making titanium metal, titanium dioxide (TiO_2) pigment and in sunblock, toothpaste, cosmetics and material for dye sensitized solar cell. The aim of this research is to obtain high grade of titanium dioxide (TiO_2) from ilmenite concentrate (TiO_2-50.468%-FeO-40.815%). Firstly, This mineral was taken from heavy sands of Myitsone area in Moemeik Township. And then seperate it to obtain concentrate by magnetic seperation. This concentrate was ground to (-200) mesh size by using pulveriser. Secondly, Leaching was carried out with sulfuric acid at various concentrations. After leaching process, the products were separated by filtration. Then the filtrate was heated at a temperature of 90°C to precipitate TiO_2. finally, It was washed and calcined at a temperature of 1000°C for 2 hours to obtain TiO_2.To determine the crystal form of TiO_2, the sample was analyzed using XRF and XRD. .In this research, an attempt to investigate the effect of sulfuric acid concentration upon leaching process, hydrolysis of the pregnant solution and calcination of titanyl hydroxide [$TiO(OH_2$] to produce high grade titanium dioxide. Result of the sulfuric acid leaching of ilmenite concentrate was obtained with 77.75% TiO_2.

Key Words: Ilmenite Concentrate, Sulfuric Acid, Leaching, Hydrolysis, Calcination, Titanium Dioxide

1 INTRODUCTION

Titanium dioxide (TiO_2) is the most important white pigment used in the coating industry.It is widely used because it efficiently scatters visible light, thereby imparting whiteness, brightness, opacity when incorporated into a coating. Titanium dioxide occurs in polymorphic forms as rutile, anatase and brookite. Titanium dioxide is commercially available in two crystal structure, anatase and rutile. Rutile TiO_2 pigments are preferred because they scatter light more efficiently, are more stable and are more durable than anatase pigment [1,2].

In the world, there are many research activities to utilize ilmenite for TiO_2 production. Those processes are categories into pyrometallurgical process, hydrometallurgical process or combination of both those processes. In the pyro metallurgical process, ilmenite is reduced by reducing agent such as anthracite, then it was melt to produce molten iron and slag containing TiO_2 [3,4]. Another alternative process is through a hydrometallurgical ways which uses sulphuric acid and hydrochloric acid. Sulfate process and chloride process produces TiO_2 intermediates and ferrous sulfate.

Titanium is a type of metal that has a lot of special qualities, i.e light, shiny, strong, heat resistant, non-toxic, corrosion resistant and has high biocompatibility to the body.Titanium metal is always joined with other elements in nature. This metal is the ninth most abundant element in the earth's crust (approximately 0.63%) and consisted mostly in igneous rocks and sediment. This metal widely exists especially in minerals anatase, brookite, ilmenite, perovskite, rutile, titanite and most of iron ores. Titanium compound is generally in combination with iron as ilmenite ($FeTiO_3$), which so-called iron-titanium sand [6].

The sulfate process was chosen in this research. Ilmenite concentrate from Myitsone area in Moemeik Township were used as the raw material for the production of high grade titanium dioxide. To study the effect of the variables that has an influence on the percentage extraction of titanium dioxide. In the series of treat performed, the influence of sulfuric acid strength on the percentag extraction of titanium dioxide was determined.

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2. MATERIAL AND EXPERIMENTAL PROCEDURE

2.1 The raw materials

Ilmenite concentrate (TiO_2-50.468%-FeO-40.815%) from Myitsone area in Moemeik Township was used as the raw material for the production of high grade titanium dioxide [5]. Other raw materials for experiment were analytical grade sulfuric acid and distilled water.

2.2 Location of Research Sample

The sample used in this study were ilmenite ore taken from Momeik Township, Myanmar, location map of sampling can be seen in Fig. 1.Momeik, also known as Mong Mit in Shan, is a town situated on the Shweli River in northern Shan State of Myanmar.

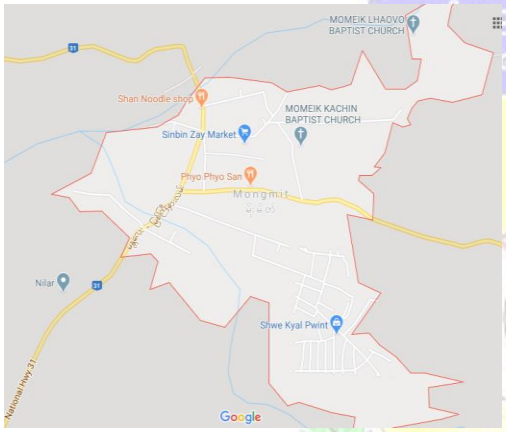

Figure-1Location Map of Illeminate Ore Sampling in Momeik

2.3 Experimental Procedure

The extraction process of titanium dioxide from ilmenite concentrate involves sample preparation and extraction of TiO_2 which involves leaching with sulfuric acid, filtration, hydrolysis and calcination. The process of extraction of Titanium dioxide (TiO_2) from ilmenite concentrate shows in figure-1.

2.3.1. Sample preparation

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 using pulveriser. The concentrate was weight and samples were prepared for chemical analysis.

2.3.2. Extraction Process

All cases of 25 grams of ilmenite concentrate was added with 50 mL, 60 mL, 70 mL, 80 mL and 90mL of H_2SO_4,then the acid solution containing TiO_2 was heated at a temperature of 150°C 6 hours until almost in the form of slurry. The slurry is cooled and filtered with filter paper and vacuum pumps to draw filtrate from the slurry. Then the filtrate was heated by adding water until there were white precipitated and centrifuged to separate from the acid solution. The precipitate was washed and calcination at a temperature of 1000°C for 2 hours. To determine the crystal formed of TiO_2, the samples were tested using XRD and XRF.

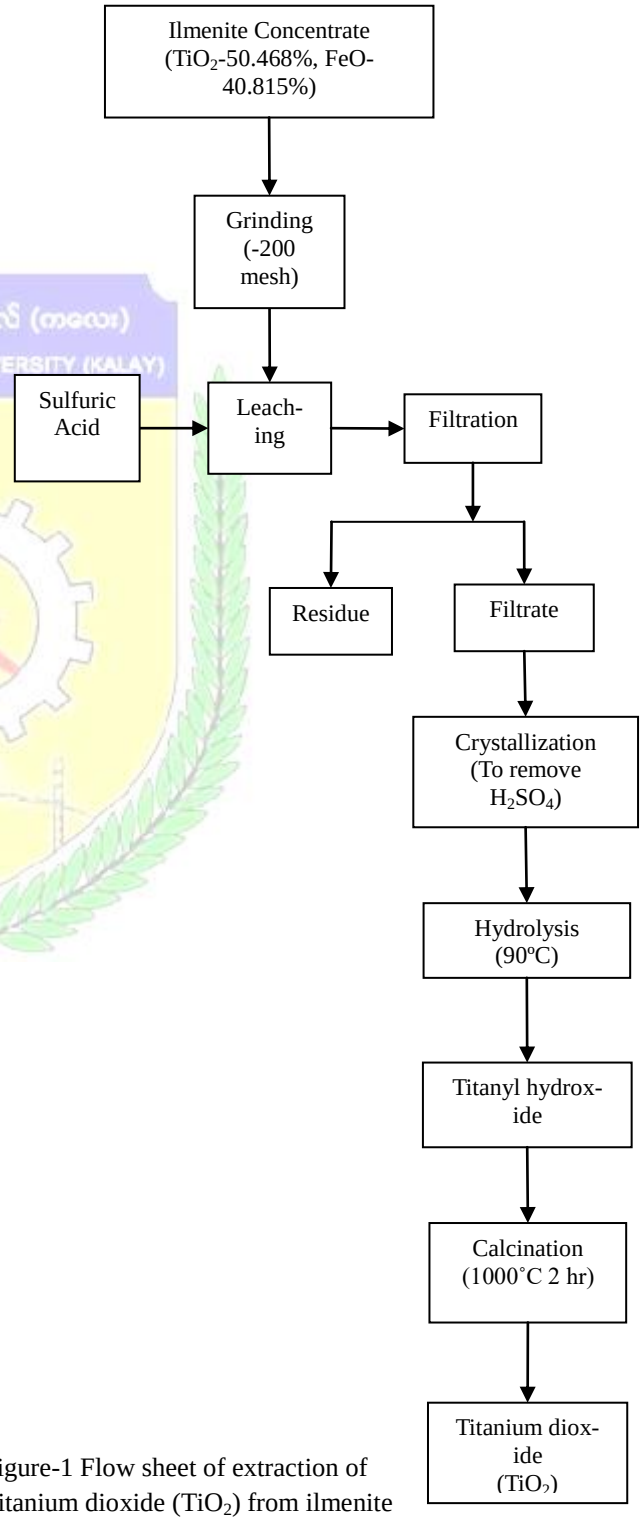

Figure-1 Flow sheet of extraction of Titanium dioxide (TiO_2) from ilmenite concentrate

3. RESULT AND DISCUSSION

3.1. Sample preparation

In this study, heavy sand was washed to remove impurities such as soil and other organic compounds contained in the sampling areas. Then the samples were dried then sand separation was performed magnetically.This concentrate was ground to (-200) mesh size by using pulveriser.It was weight and sample were prepared for chemical analysis using XRF. Based on XRD data of r analysis, it was known that most compounds were in the form of Fe_2O_3, TiO_2, MgO and SiO_2.

The XRF data of ilmenite concentrate from Myitsone area in Moemeik Township shown in table-1.

Table-1 Chemical compositions of ilmenite concentrate by XRF

Compound	TiO_2 Weight (%)
TiO_2	50.468
Fe_2O_3	40.815
SiO_2	6.089
MnO	1.394
CaO	0.443
SnO_2	0.189
Cr_2O_3	0.149
NbO	0.146
ZnO	0.131
ZrO_2	0.115
CuO	0.033
Y_2O_3	0.028

3.2. .Extraction Process

In this study, Extraction of TiO_2 was sulfuric acid leaching process. 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 >150º C for (6 hr). The chemical reaction in the leaching process is described as follows.Then filtration was carried out.

$$Fe\ TiO_{3(s)} + 2H_2SO_{4\ (aq)} \rightarrow FeSO_{4(s)} + TiOSO_{4\ (aq)} + 2H_2O_{\ (l)} \quad [1]$$

After leaching process, the products were separated by filtration. Solids were washed with distilled water. In this process, $TiOSO_4$ was dissolved in sulfuric acid solvent, while $FeSO_4$ is an insoluble substance,the results of this process produced slurry.This slurry was filtrated to obtain filtrate added with water The filtrate was heated at a temperature of 90℃.The following reaction is occurred.

$$TiOSO_{4\ (aq)} + 2H_2O_{\ (l)} \rightarrow TiO(OH)_{2\ (s)} + H_2SO_{4\ (aq)} \quad [2]$$

Then the filtrate was heated by adding water until there were white precipitates and centrifuged to separate from the acid solution. The precipitate was washing with distilled water several times to remove exiting acid .TiO_2 product was furnace to remove water at a temperature of 1000°C for 2 hours. To determine the crystal formed of TiO_2, the samples were analyzed using XRF and XRD.

$$TiO_2.H_2O_{(s)} \rightarrow TiO_{2(s)} + H_2O_{\ (g)} \quad [3]$$

Ilmenite concentrates contain 50.47% from Myintsone were leached with sulfuric acid, hydrolyzed and calcined to produce high grade titanium dioxide. The dependence of TiO_2 extraction on sulfuric acid concentration is shown in Table-2 and Figs.2. The effect of sulfuric acid concentration has a strong influnence on the disolution 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 TiO_2 extraction 77.75% with sulfuric concentration 60 ml. The percent extraction reduces 56% with increasing sulfuric acid concentration to 70 ml.

From the results plotted in figure 2 it is evident that with increasing sulfur acid concentration, percent extraction increases, but after acid concentration 60 ml it decelerates. Therefore sulfuric acid concentration 60 ml is selected as optimum condition for the experiments.

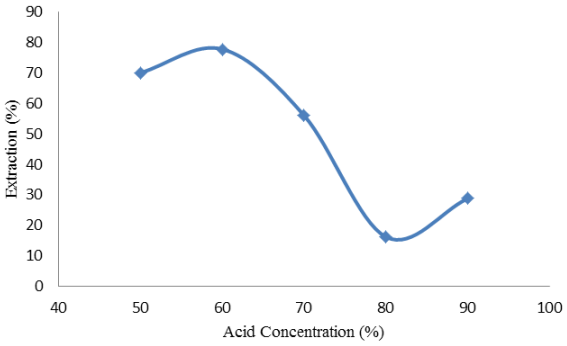

Figure-2 The effect of Acid concentration on the percentage extraction of Titanium dioxide

Table-2 The percentage extraction of Titanium dioxide

Sr No.	Sulfuric acid conc; (mL)	Time (hrs)	Temp (ºC)	Filtrate		Extraction (%) TiO_2
				TiO_2 %	Fe_2O_3 %	
1	50	6	150	86.93	8.56	70
2	60	6	150	89.18	8.03	77.75
3	70	6	150	91.62	5.62	56
4	80	6	150	88.18	6.53	16.1
5	90	6	150	93.2	6.41	28.75

According to the above table, the percentage extraction of Titanium dioxide can be seen.

Time duration and temperature of all saturations is same as 6 hours and 150°C. Among them, the percentage extraction of Titanium dioxide of sulfuric acid concentration (60 ml) is the largest amount and then the percentage extraction of Titanium dioxide of sulfuric acid concentration (80 ml) is the smallest amount.

Therefore sulfuric acid concentration (60 ml) is selected as optimum condition for the experiments. The XRF and XRD result of the filtrate sample and the XRD patterns of Calcite product of TiO_2 is expressed in the following figures.

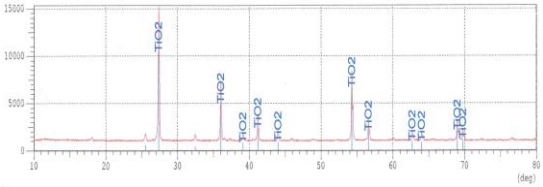

Figure-3 XRD Patterns of Calcine Product of TiO_2

Table-3 Chemical compositions of product of TiO_2 by XRF

Compound	TiO_2 Weight (%)
TiO_2	89.179
Fe_2O_3	8.032
SiO_2	2.461
CaO	0.081
NbO	0.214
ZrO_2	0.011
CuO	0.022

4. CONCLUSION

From the series of leaching tests performe the following condition gave an maximum extraction of titanium dioxide acid concentration 78%, leaching time (6) hr and temperature of 150°C. After filtration and recrystallization of iron sulfate, the concentrated filtrate was heat to 90ºC whereupon titanyl sulphate hydrolysed to insoluble titanyl hydroxide $TiO(OH)_2$. Calcinations of the titanyl hydroxide at 1000 ºC for (2 hr) gave a product containing TiO_2 78%.

The following recommendations are draw based on the experimental results.

i. All leaching and calcinations tests were performed only once. For more reliable results each point on the respective curves should be the average obtained from a least three repeated tests.
ii. Leaching of ilmenite was manual. Better results will surely be obtained if mechanical agitator was used for dissolution.
iii. Calcinations of titanyl hydroxide was tested only at 1000ºC and (2) hours calcinations time. Different calcinations temperature and time should be investigated.
iv. Because of concentration of acid and high temperature required during leaching, corrosion resistant material must be carefully chosen in the mass production of titanyl sulphate.
v. Calcinations product obtained is 77.75% TiO_2 grade. Refining of the calcinations product need to be studied for higher grade.

vi. Future studies should include at least: effect of other variables, such as particle size, leaching time and temperature.

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