Extraction of Titanium Dioxide from Ilmenite Concentrate by Sulfuric Acid Leaching

San Yu
Lecturer, Department of Metallurgical Engineering and Materials Science
Technological University (Mandalay) Mandalay City, Myanmar
Email - sanyu.mdy@gmail.com

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 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%). This ilmenite concentrate was taken from heavy sands of Myitsone area in Moemeik Township by magnetic separation. There are two processe to treat ilmenite concentrate, sulphate and chloride. The sulphate process was chocsen because this process treats low grade titanium ore. 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 sulfuric acid and hydrochloric acid. Sulfate process and chloride process produces TiO_2 intermediates and ferrous sulfate. 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 percentage extraction of titanium dioxide was determined.

2. MATERIAL AND METHOD:

2.1 The raw materials for experiment

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 sulphuric acid and distilled water. In this research, the sulphate process was chosen.

2.2 Experimental Procedure

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_2) 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 >150º C for (6 hr). The following reaction is dissolution of ilmenite concentrate. Then filtration was carried out.

$$5H_2O + Fe\,TiO_3 + 2H_2SO_4 \rightarrow FeSO_4 + TiOSO_4 + 7H_2O \quad [1]$$

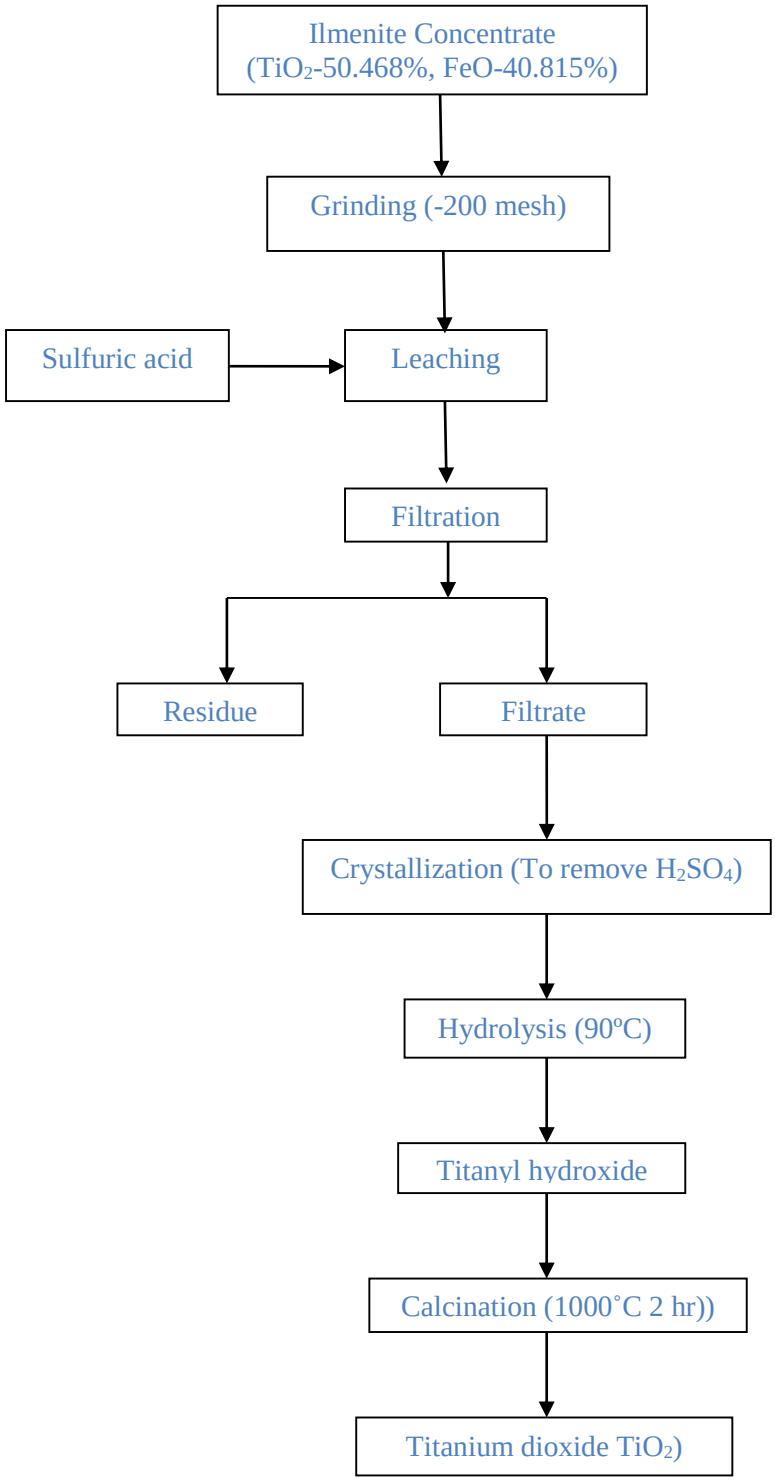

Figure-1 Flow sheet of extraction of Titanium dioxide (TiO_2) from ilmenite concentrate.

After leaching process, the products were separated by filtration. Solids were washed with distilled water. The residue was determinate by XRF. The filtrate was heated at 90℃. The slurry is cooled and filtered with filter paper.

$$TiOSO_4 + 2H_2O \rightarrow TiO(OH)_2 + H_2SO_4 \quad [2]$$

Then the filtrate was heated by adding water until there were white precipitates and centrifuged to separate from the acid solution. The precipitate was washed and calcine 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.

3. RESULT AND DISCUSSION :

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

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 sulphuric acid concentration is shown in Table-2 and Figs.2. The effect of sulfuric acid concentration has a strong influence 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 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 sulphur acid concentration, percent extraction increases, but after acid concentration 60 ml it decelerates. Therefore sulphur acid concentration 60 ml is selected as optimum condition for the experiments. The XRD result of the filtrate sample was shown in fig.3.

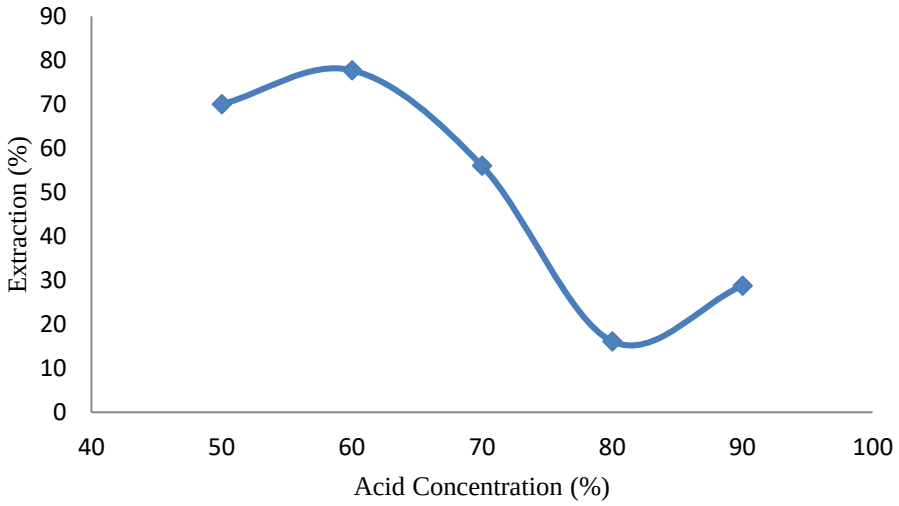

Figure-2 The effect of Acid concentration on the percentage extraction of Titanium dioxide

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

Sr No	Sulphuric acid conc; (mL)	Time (hrs)	Temp (°C)	Filtrate		residue		Extraction (%) $\frac{TiO_2 \text{ in filtrate} \times 100}{TiO_2 \text{ in sample}}$
				TiO_2%	Fe_2O_3%	TiO_2%	Fe_2O_3%	
1	50	6	150	86.93	8.56	69.21	21.37	70
2	60	6	150	89.18	8.03	67.67	21.95	77.75
3	70	6	150	91.62	5.62	68.41	13.54	56
4	80	6	150	88.18	6.53	71.56	12.83	16.1
5	90	6	150	93.2	6.41	74.65	13.08	28.75

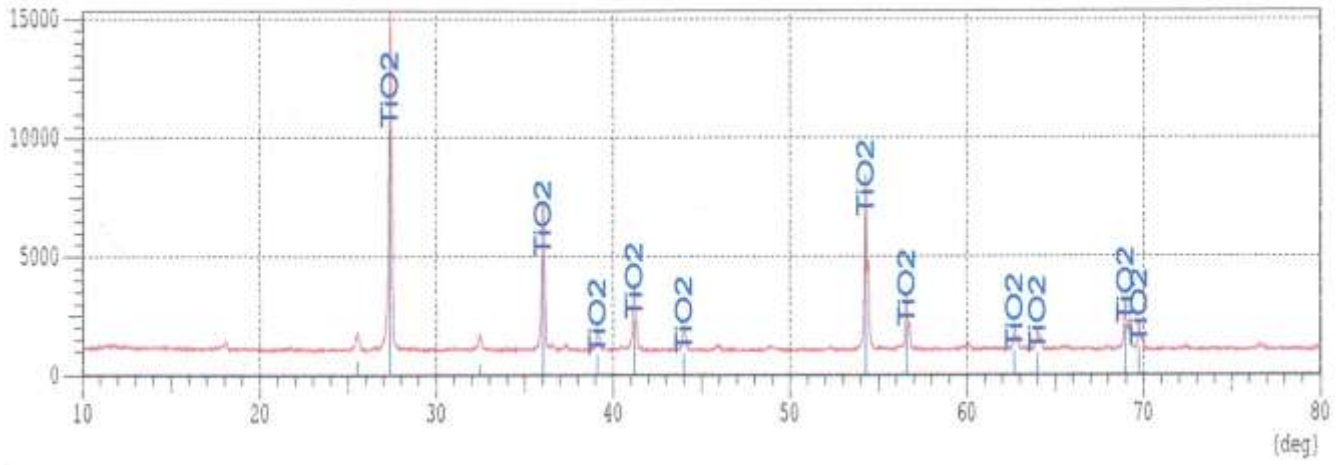

Figure-3 XRD Patterns of Calcine Product of TiO_2

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. After filtration and recrystallization of iron sulphate, 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.

ACKNOWLEDGEMENT

The authors would like to express appreciation to the Department of Metallurgical Research Division, Ela, Ministry of Education for providing the samples and others supporting.

REFERENCES:

1. Sayekti Wahyuningsih, Edi Pramonoi, Florentinus Firdiyono, Eko Sulistiyono (2013), Decomposition of Ilmenite in Hydrochloric Acid to Obtain High Grade Titanium Dioxide, Asian Journal of Chemistry; Vol. 25, No. 12 (2013), 6791-6794.
2. Sayekti Wahyuningsih, Ari Handono Ramelan, Edi Pramono, Ariantama Djatisulistya (2014), Titanium dioxide production by hydrochloric acid leaching of roasting Ilmenite sand, International Journal of scientific and research publications, Volume 4, Issue 11, November 2014.
3. Rudi subagja, Lia Andriyah, Latifa Hanum Lalasari , Titanium Dissoluation from Indonesian Ilmenite, International Journal of Basis and Applied Sciences; Vol:13 No.04
4. K.N. Han, T. Rubcumintara, M.C. Fuerstenau (1987), Leaching behaviour of Ilmenite with sulphuric acid, Metallurgical Transactions B, Volume 18B, June 1987-325.
5. Khin Swe Oo, Extraction of TiO_2 from Ilmenite Ore, Master Thesis, Yangon Technological University, Myanmar.