Titanium dissolution from decomposed Ilmenite using NaOH into the aqueous sulphuric acid solutions

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Titanium dissolution from decomposed Ilmenite using NaOH into the aqueous sulphuric acid solutions

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Abstract. At present work, the experiment to dissolve titanium from the Indonesian Ilmenite into the aqueous sulphuric acid solutions has been done through the following process: Ilmenite decomposition using NaOH at temperature 800°C for 5 hours to produced decomposed Ilmenite, followed with titanium dissolution from the decomposed Ilmenite into the aqueous sulphuric acid solutions in the glass reactor. The variables used for dissolution process were covering: dissolution temperature from 30°C to 70°C, sulphuric acid concentrations from 5% to 15%, and dissolution time from 30 minutes up to 180 minutes. The influence of those all variables were observed on the titanium dissolution from the decomposed Ilmenite. Result of the dissolution experiment show that the dissolved titanium in the aqueous sulphuric acid solutions increased when the dissolution temperature was increased from 30°C to 60°C, sulphuric acid concentration were increased from 5% to 15% and dissolution time were extended from 30 minutes to 180 minutes.

1. Introduction

Ilmenite is a mineral with chemical formula $FeTiO_3$. It is one of the primary raw materials for titanium containing material and iron productions. In the world, almost 95% of titanium containing in titanium feedstock was for TiO_2 production and only 5% in feedstock were converted to titanium metal [1-2]. TiO_2 is widely used in many applications such as a pigment for paint production, additive for paper manufacturing, ceramic industries, pharmaceutical industries, environment purification, gas sensor, and in photovoltaic for photo catalyst application due to its unique characteristics [3-7].

Indonesia has a potential resource of Ilmenite scattered at some areas, however those Ilmenite are not well utilized due the appropriate processing technology for Indonesian Ilmenite is un available. The problem face to utilize the Indonesian Ilmenite is the mineral structure of Indonesian Ilmenite is sometimes very complex that lead to the processing of Indonesian Ilmenite becomes difficult. Therefore, in order to developed an appropriate processing technology for Indonesian Ilmenite, the Research centre for Metallurgy and Material – the Indonesian Institute of sciences (RCMM-LIPI), conducts some experiment on the processing of the Indonesian Ilmenite [8-10] and at present research activity, the titanium dissolution into aqueous sulphuric acid solutions was carried out through the following steps of processes : the decomposition of Ilmenite at elevated temperature with NaOH followed with the titanium dissolution from the decomposed Ilmenite into the aqueous sulphuric acid solutions. The experiment was carried out at laboratory scale experiments. The purpose of experiments is to investigate the influence of some parameters such as sulphuric acid concentration, dissolution temperatures and dissolution time on the titanium dissolution from the decomposed Ilmenite.

2. Experiment

2.1. The raw materials for experiment

The raw material used for experiment were Ilmenite, analytical grade sodium hydroxide (NaOH), and aqua demineral water. Table 1 shows the chemical composition of Ilmenite obtained by X-Ray Fluorescence and figure 1 shows the X-Ray Diffraction pattern of Ilmenite used in the experiment. The chemical composition data at table 1 shows the raw material Ilmenite has 49,44% Fe_2O_3 and 38,30% TiO_2. Other compounds exist at the Ilmenite sample are SiO_2, Al_2O_3, MnO, MgO, CaO, K_2O, P_2O_5, Cr_2O_3, SnO_2, but their quantities are relatively lower, and the X-Ray diffraction pattern at Figure 1 shows Ilmenite ($FeTiO_3$) as the main mineral exist at the raw material for experiment.

Table 1. Chemical composition of Ilmenite.

COMPOUND	Fe_2O_3	TiO_2	SiO_2	Al_2O_3	MnO	MgO	CaO	K_2O	P_2O_5	Cr_2O_3	SnO_2
%	49,44	38,30	1,76	1,78	2,00	1,44	0,08	0,03	0,17	2,66	1,16

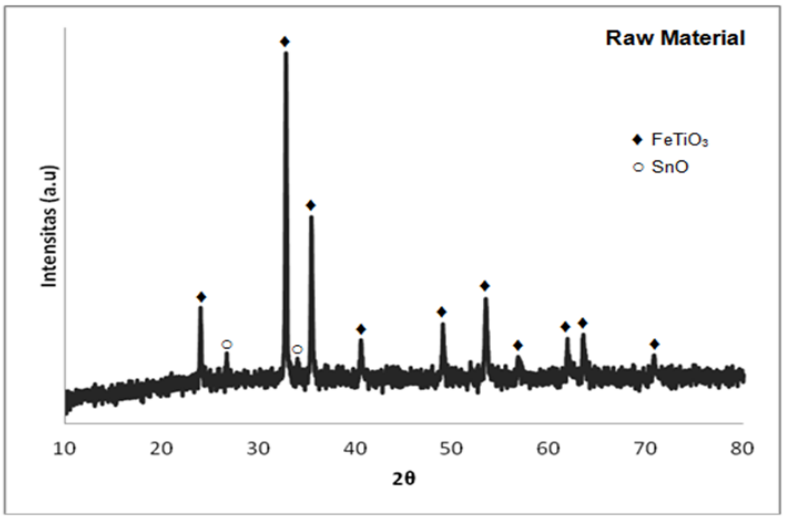

Figure 1. X-Ray Diffraction pattern of Ilmenite.

2.2. The Equipment for experiment

There were two main equipments were used in experiment. The 1st equipment was Programmable Muffle Furnace for decomposition of Ilmenite with NaOH at elevated temperature to produce decomposed Ilmenite and 2nd equipment was 1-liter capacities of glass reactor for the titanium dissolution from the decomposed Ilmenite into the aqueous sulphuric acid solutions. The glass reactor was mantled with controllable heater to control the reaction temperatures during dissolution process.

2.3. Procedure of experiment

The procedure of experiment was covering the procedure for decomposition of Ilmenite with NaOH at elevated temperature and the procedure for titanium dissolution from the decomposed Ilmenite.

2.3.1. The procedure for Ilmenite decomposition process

The Ilmenite decomposition process was carried through the following steps: Firstly, Ilmenite was mixed with NaOH in a mass ratio of 2:1 in Stainless steel crucible. The mixed Ilmenite-NaOH were then roasted in the programmable Muffle Furnace, in air atmosphere at temperature 800°C for 5 hours. That roasting temperature was choose based on our previous experiment for the decomposition of Ilmenite with NaOH which show that the best temperature for the decomposition of Ilmenite to sodium titanate and sodium ferrite was at temperature 800°C [11]. At elevated temperature, Ilmenite

will be decomposed by sodium hydroxide to produce sodium ferrite ($NaFeO_2$) and sodium titanate (Na_2TiO_3) according to the reaction (1). The calculated Gibbs free energy reaction using HSC 5.1 software for reaction 1 gives a negative value at temperature lower than 1000°C [12], therefore the formation of sodium titanate and sodium ferrite through the reaction 1 will be thermodynamically favorable at temperature 800°C:

$$2\ NaOH + 2/3\ FeTiO_3 + 1/6\ O_2\ (g) = 2/3\ NaFeO_2 + 2/3\ Na_2TiO_3 + H_2O \quad (1)$$

The generated water at reaction 1 creates trans granular cracks throughout Ilmenite structure which promote the diffusion oh Na^+ ions throughout the structure, leading to the formation of sodium titanate [12].

The decomposed Ilmenite from reaction 1 was used further as the raw material for titanium dissolution experiment.

2.3.2. The procedure for dissolution process

The procedure for titanium dissolution from decomposed Ilmenite was carried out through the following steps: the washing process of decomposed Ilmenite with water and the titanium dissolution process from the washed decomposed Ilmenite in to the aqueous sulphuric acid solutions.

In the washing process, the decomposed Ilmenite samples from decomposition experiment were washed by demineralized water until the washed solution pH reached 7. After washing process, the washed decomposed Ilmenite samples were added into the aqueous sulphuric acid solutions in 1-liter glass reactor. The dissolution process was carried out at certainties period of time and temperature, under agitation speed 300 rpm. At this dissolution process, the sulphuric acid concentration were varied from 5% up to 15%, dissolution temperature was varied from 30°C up to 70°C, and dissolution time were from 30 minutes to 300 minutes. After the dissolution experiment finished, the mixed solution and solid were discharged out from the glass reactor and it was filtered to separate solid from solution. The solid cake was then washed by water and dried in oven at 100°C for 24 hours prior to X-Ray Diffraction (XRD) analysis, and the titanium content in solution was analyzed by using atomic absorption spectrophotometer.

3. Results and Discussion

3.1. X Ray diffraction pattern of the decomposed Ilmenite

The decomposed Ilmenite produced by roasting process of the mixed Ilmenite-NaOH was analyzed using X-Ray diffractometer. Sample was analyzed over an angle 2θ with a maximum range of 10 to 80°. The result of analysis by X Ray diffractometer at figure 2 shows that the formed phases during the roasting process of the mixed Ilmenite-NaOH at 800°C were sodium titanate (Na_2TiO_3) and sodium Ferrite ($NaFeO_2$). This result of experiment agrees with findings by Stephen Parirenyatwa et al which conducted roasting experiments on the mixed Ilmenite and NaOH [12]. The formation of sodium titanate and sodium ferrite are taking place through the reaction 1 which is thermodynamically favorable at temperature 800°C.

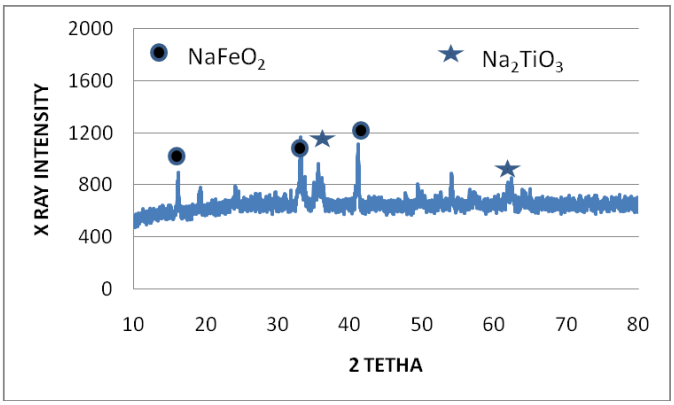

Figure 2. X-Ray Diffraction pattern of the decomposed Ilmenite

3.2. Effect of dissolution time on Ti dissolution

During the titanium dissolution process from the decomposed Ilmenite into the sulphuric acid solutions, the sodium titanate containing in the decomposed Ilmenite reacts with sulphuric acid to produce titanium sulphate and sodium sulphate according to reactions 2 [13]:

$$Na_2TiO_3 + 3\ H_2SO_4 \rightarrow Ti(SO_4)_2 + Na_2SO_4 + 3\ H_2O \quad (2)$$

Reaction 2 is influenced by dissolution time, temperature and sulphuric acid concentrations. The effect of dissolution time on titanium dissolution from the decomposed Ilmenite in 10% aqueous sulphuric acid solutions, at dissolution temperature 60°C is presented at figure 3. The figure shows that at the beginning of the dissolution process, fast titanium dissolution process happened, however when the dissolution time were extended from 0.5 hours to 3 hours, the dissolved titanium in 10% aqueous sulphuric acid solutions increased from 58% at 0.5 hours dissolution time to 82% at 3 hours dissolution time and slowly titanium dissolution process happened, that probably due to the formation of undissolved iron material at the decomposed Ilmenite. Referring to Stephen Parirenyatwa finding the presence of iron in the sample roasted with NaOH may be due to the presence of unreacted Ilmenite [12].

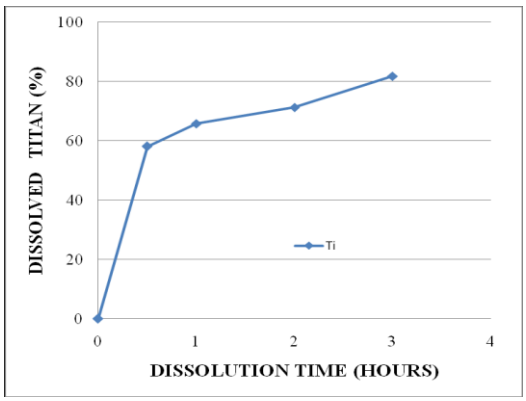

Figure 3. The effect of dissolution time on Ti dissolution at 10% H_2SO_4

3.3. The effect of temperature on Ti dissolution

The influence of temperatures on titanium dissolution from the decomposed Ilmenite were investigated at the temperature range 30°C – 70°C, in 10% aqueous sulphuric acid solutions. The

result of experiment at figure 4 shows that temperature gives a significant effect on the titanium dissolution from the decomposed Ilmenite. Almost at all dissolution times, the dissolved titanium increased with the increased of the dissolution temperatures from 30°C up to 60°C. However, when the dissolution temperature was increased from 60°C to 70°C, the dissolved titanium from the decomposed Ilmenite decreased that probably due to the precipitation of some of the dissolved titanium from the solutions at higher temperature than 60°C.

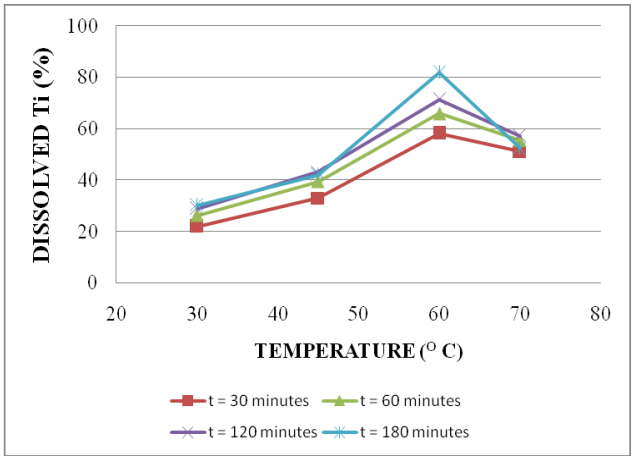

Figure 4. The effect of temperature on titanium dissolution.

3.4. The effect of H_2SO_4 on Ti dissolution

The influence of sulphuric acid concentration on titanium dissolution from the decomposed Ilmenite in aqueous sulphuric acid solutions were investigated at temperature 60°C, the sulphuric acid concentration were varied from 5% to 15%, and the dissolution time were from 30 minutes to 180 minutes. The result of experiment at figure 5 shows that at temperature 60°C and 3 hours dissolution time, the dissolved titanium from decomposed Ilmenite increased from 30% in 5% sulphuric acid concentration to 88% in 15% sulphuric acid concentration. This result of experiment shows that the sulphuric acid concentration has a great effect on titanium dissolution.

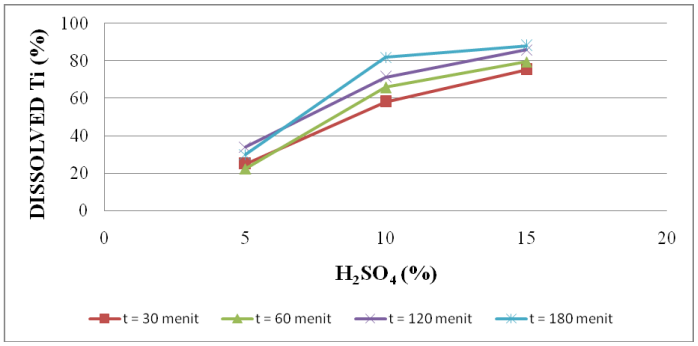

Figure 5. Effect of Sulfuric acid concentration on titanium dissolution.

4. Conclusion

The dissolution experiment to dissolve titanium from Indonesian Ilmenite has been investigated through the decomposition process of Ilmenite with NaOH at temperature 800°C for 5 hours, followed with titanium dissolution from the decomposed Ilmenite in the aqueous sulfuric acid solutions.

The result of experiment shows the titanium dissolution from the decomposed Ilmenite in aqueous sulfuric acid solution were influenced by dissolution time, dissolution temperature and sulphuric acid concentration. The titanium dissolution increased when dissolution time, dissolution temperature or sulphuric acid concentration were increased.

When the dissolution time were extended from 0,5 hours to 3 hours, the dissolved titanium into 10% aqueous sulphuric acid solutions increased from 58% at 0,5 hours dissolution time to 82% at 3 hours dissolution time.

In 10% aqueous sulphuric acid solutions at 3 hours dissolution times, when the dissolution temperatures were increased from 30°C up to 60°C, the dissolved titanium increased from 30% at dissolution temperature 30°C to 82% at dissolution temperature 60°C.

At temperature 60°C and 3 hours dissolution time, the dissolved titanium from decomposed Ilmenite increased from 30% in 5% sulphuric acid concentration to 88% in 15% sulphuric acid concentration.

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