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Optimization H_2so_4 Concentration on the Leaching Process of Extracting Titanium from Zircon Sand

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Abstract

Zircon sand was obtained from Kalimantan Province, Indonesia. Zircon sand contain ilmenite compound that it can be processed to extract TiO_2. To extract TiO_2 can be used Becher Process which is combined Pirometallurgy and Hydrometallurgy. Crystal Ilmenite separated from another purities through smelting process above 1200^oC for 6 hours. Ilmenite is reduced through aeration process with oxygen for 6 hours to enable the rust which is signed brownish colour, it is formed Fe_2O_3. The leaching residue was using H_2SO_4 solution with concentration variation of 6M, 8M, 10M, and 12M. Titanium bonded SO_4^- which is formed $TiSO_4$. Result powder of leaching was calcined by using arc furnace at 900^oC with the result that SO_4 evaporated from the powder and formed TiO_2 powder. Based on the XRD pattern showed that the recovery rutile TiO_2 obtained in concentration 10 M H_2SO_4 solution. XRD pattern showed high purities of $ZrSiO_4$. In this report did not include a quantitative

percent recovery of TiO_2. The analysis was performed by comparison of the intensities between raw material and after leaching residue, particularly on changes in the intensity of ilmenite and TiO_2.

1. Introduction

Indonesia has abundant mineral resources, one of that is the zircon sand. In kalimantan province is an area that has a very high zircon sand. Zircon sand is mineral that is extremely valuable since It has the essential elements or compounds, one of that titanium compound. Titanium is an important raw material in various industries. Its ability as an intermediate material in the manufacture of paint, paper, printing ink, rubber, floor coverings, ceramic, pharmaceutical and other chemical industries make titanium has a high value.

The use of TiO_2 is widely used in the form of rutile but very rarely found in nature. TiO_2 present in large amounts of ilmenite. Therefore, some of research efforts are converted titanium from ilmenite.

There are two processes used to produce titanium, namely sulfate process and chlorination (Kamala,2006). In the sulfate process, ilmenite grains dissolved in sulfuric acid solution to prepare a titanium sulfate. This solution was further purified and hydrolyzed to produce pure TiO_2. Sulfate process has long time, produce unmarketable copperas, spent sulfuric acid and produce acidic waste water for every tone of TiO_2 production that can lead to environmental problem (Liang, 2005).

Currently about 60% titanium dioxide produced by dry chlorination process in the world (Kamala, 2006). Shortage of natural rutile has prompted research efforts to convert ilmenite to synthetic rutile for dry chlorination process . It can be summarized into two categories that pyrometallurgy and hydrometallurgy process. Production of synthetic rutile pyrometallurgy include smelting process (Natziger, 1987), the Becher (Becher, 1963), the process MURSO (Sinha, 1979), the ERMS (Walpole, 1997).

In this study was using the becher process, a process was introduced by Australian scientists, Dr.Robert Becher. This method is used for the extraction of titania from zircon sand in Kalimantan Province, Indonesia.

2. Material and Analysis

Material used is derived from zircon sand in Kalimantan Province. This sand was burned using melting furnance with temperatures above 1200^{o}C for 6 hours. Dissolution was carried out for separating ilmenit from the impurities. Furthermore, zircon sand is reduced in the process of aeration with 1% NH_4Cl, water and oxygen from the compressor. This process lasted for 6 hours at a temperature of 150^{o}C. The function of aeration is forming Fe_2O_3 in the water. It can be separated from TiO_2. After sand was reduced, zircon sand was dried in the oven.

In leaching process, 50 g of titanium residue stirred with concentration variations of H_2SO_4, 6M, 8M, 10M, 12M respectively. Leaching was treated at around 30^{o}C (room temperature) for 2 hours. The filtrate was separated from residual titanium. Titanium residue calcined at 900^{o}C. Each titanium residue was characterized by X-ray Diffraction (XRD).

3. Discussion

Components of zircon sand as a raw material analyzed using XRD to be visible difference in the content of titanium to recovery.

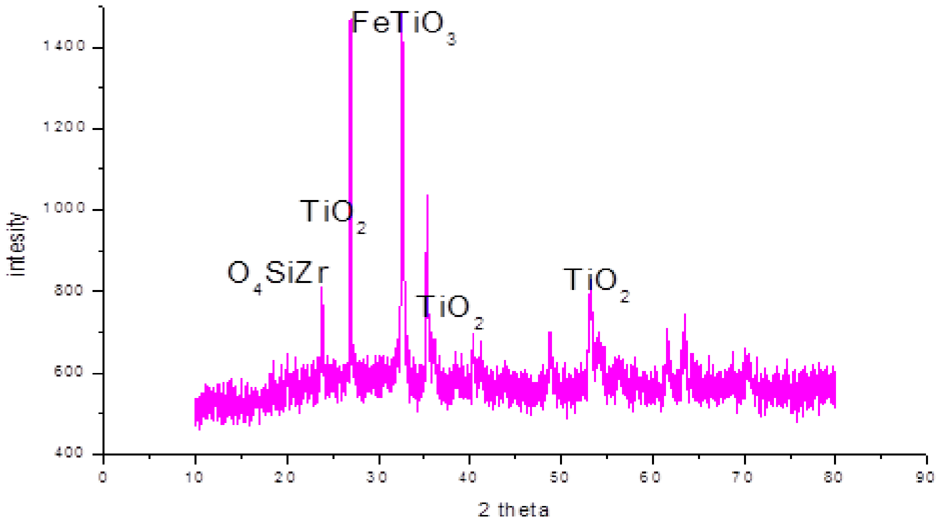
Figure 1 shows the XRD pattern of the raw material zircon sand. Ilmenite intensity is very high, reaching 1400. Raw material contained TiO_2 compound. Although it appears a low intensity. Then a very high impurity is $ZrSiO_4$. This shows that the zircon sand has the potential for high TiO_2 recovery.

To obtain the necessary separation of TiO_2 on the impurity. The method used in this study is the method of becher. Process in a way that combines pyrometallurgy in smelting and hydrometallurgy. In pyrometallurgy process, sample were burned to break $FeTiO_3$ crystal, so that the bond between Fe and Ti can break to be simpel compounds. Then, $ZrSiO_4$ impurity can be separated from TiO_2 and $FeTiO_2$.

In the hydrometallurgy process, samples of zircon sand is reduced using oxygen so that corrosion would be occurred which marked with brown color in the water. Fe contained in the material content can be reduced with oxygen, it would be Fe_2O_3.

$4Fe + 3O_2 \rightarrow 2Fe_2O_3$

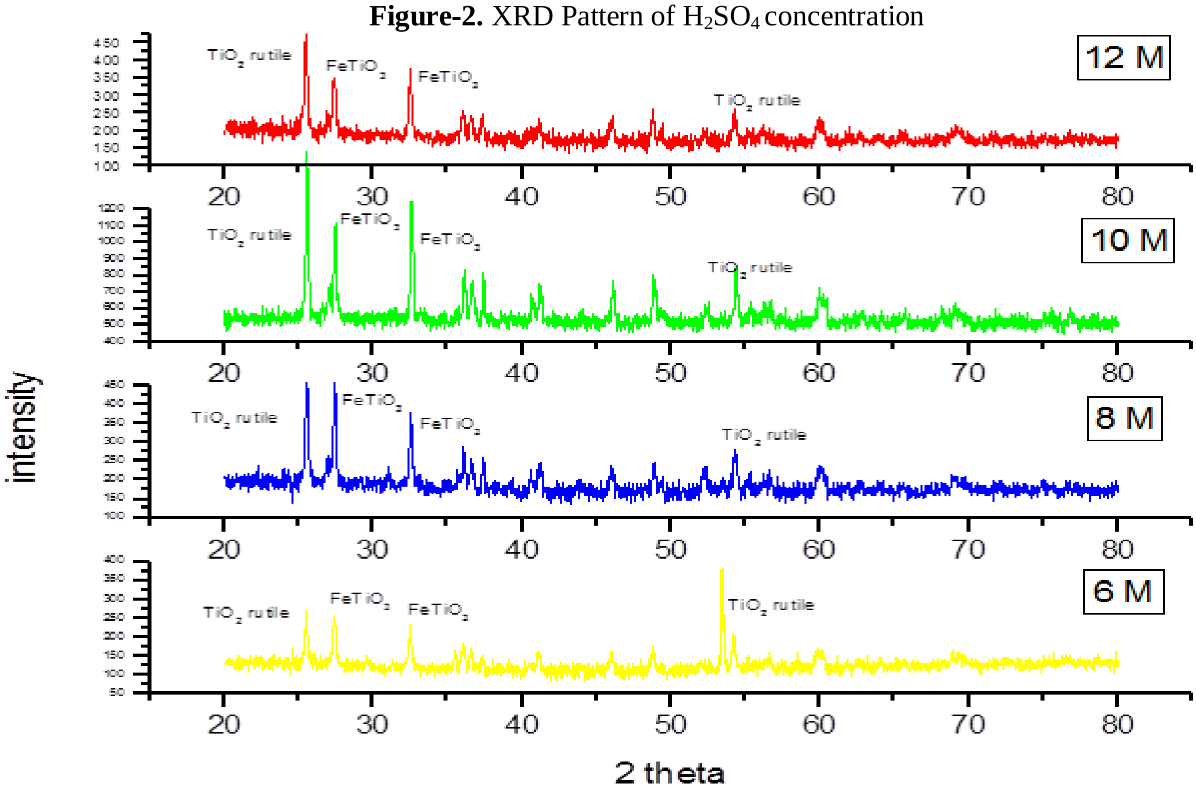
Figure-1. XRD Pattern of raw material



This process serves to reduce Fe in the samples through the process of oxidation. Fe_2O_3 bound in the form of liquid while Ti bound in a solid form so easily separated.

The next process is leaching with H_2SO_4 solution. The acid decomposition of Fe can tied with TiO_2. To view the optimum concentration in the sampe variation There are four variations of H_2SO_4 concentration on this study. Variations in the concentration of H_2SO_4 leaching indicated by the XRD pattern in figure 2.

Figure-2. XRD Pattern of H_2SO_4 concentration



The XRD pattern in figure 2 using radiation source of Cu-Ka (1.541874 A) and diffraction angle in the range of 20^o-80^o, characterization of XRD is used to identify compounds qualitatively and quantitatively but this study reported qualitatively only. Identify samples qualitatively using a program ‘search match’ with COD database. Through this software, the analysis results of XRD peak pattern can be identified by comparing peak existing in the database with the peak pattern obtained from the sample characterization.

Peak rutile TiO_2 was identified in a prominent peak in the angle (2θ=27,42 dan 2θ=54,14). The optimum intensity was obtained at a concentration of 10 M, reached 1200. This suggest that the appropriate concentration of H_2SO_4 leaching is used for the concentration of 10 M. In this study still found Ilmenite and impurities $ZrSiO_4$. This study also showed that the decrease in intensity is very high $FeTiO_3$ and increased intensity of TiO_2 increased when compared with the intensity of both the raw material XRD results. Percent recovery of TiO_2 in the study had not been identified but the results can be observed by the intensity of the two compounds.

Reaction that occur in the leaching process can be predicted as follows:

$Ti_2O_3 + 2H_2SO_4 \rightarrow TiSO_4 + TiO_2 + H_2O$

Then, the solution was stripped and dried to remove water in the sample. Sample were washed with water to remove water removal while SO_4^{2-}. SO_4^{2-} were left to be done by calcination.

$TiOSO_4 + H_2O \rightarrow \quad TiOSO_4.2H_2O$

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

$TiO(OH)_2 \quad \rightarrow TiO + H_2O$

4. Conclusion

Comparison between the XRD pattern of raw material to the results of the leaching process showed that the recovery of TiO_2 using the Becher process is very high, although this report has not been shown quantitatively percent recovery of TiO_2. TiO_2 recovery of zircon sand obtained at the optimum concentration of 10 M H_2SO_4 solution.

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