The Effect of Roasting Temperature at 400°C and Sulphuric Acid Concentration on TiO_2 Extraction Process from Zircon Sand Ilmenite

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Abstract

In this research, roasting temperature at 400°C and sulphuric acid concentration variations was conducted to obtain titanium dioxide from zircon sand. The research was done through extraction metallurgy method which involved physical, chemical, and mechanical treatments. Zircon was magnetically separated, mixed with NaOH, and roasted with temperature of 400°C. Characterization with XRD of the sample before and after magnetic separation was conducted. Peaks obtained from $NaFeO_2$, $NaTiO_2$, $Na_{0.75}Fe_{0.75}Ti_{0.25}O_2$, and TiO_2 compounds were observed. In this study, the leaching process lasted in two stages. For the first leaching samples of combustion products are leached using distilled water to remove water-soluble impurities. For the second stage, the deposition obtained from stage one is leached using 8, 10, and 12 M concentrated sulphuric acid at high temperature to dissolve iron. XRD was used to characterize TiO_2. The result showed that the sample at temperature of 400°C and concentrated sulphuric acid of 12 M is whiter in colour compared to other samples. Furthermore, at temperature 400°C and concentrated sulphuric acid of 12 M, the titanium dioxide produced is 32.3% and 11.5% for rutile and anatase, respectively.

KEYWORDS: alkaline leaching, roasting temperature, ilmenite, zircon sand

I. INTRODUCTION

Indonesia encompasses abundant natural resources. One of these natural products which have not been properly exploited is zircon sand. Zircon sand contains iron and titanium (ilmenite). Zircon sand is potentially known as the basic ingredient for ceramics and electronic components, and also used in the nuclear reactor electricity generator. At the moment Central Kalimantan contains a big reserve of zircon sand in Indonesia.

A result of marketing aspect study of zircon sand conducted by Dubbo Zircon Project (DZP) in New South Wales-Australia predicts that the world consumption of zirconium products from 2012 until 2020 will rise quite rapidly outnumbering the industrial production capacity of factories producing zirconium based materials which exist now or will be built in the future. Hence, the prospect of new factories that processes zircon sand into zirconium based products are certainly sensible to be built in Kalimantan regions [1].

Generally, zircon sand mineral ($ZrSiO_4$) contains valuable compounds such as titanium in rutile (TiO_2) and ilmenite ($FeTiO_2$) minerals, and rare earth metals (Y, Dy, Tb, Gd, La, Ce, Nd, Pr, and Sm) [2]. One compound that has a potentially high economic value is titanium dioxide (TiO_2). This compound is used frequently for white pigment production, filler for paper production factory, plastic factory, rubber factory, and paint factory. Furthermore, TiO_2 can also be applied for environment pollution purification, gas censor, and photoelectric cell because of its unique characteristics. TiO_2 is highly refracted, has low conductivity, and high melting point. TiO_2 or titania may be obtained by extraction [3, 4] from ilmenite oxide ($FeTiO_3$). Based on the purity level, this compound is divided into three forms, that is anatase, brookite, and rutile.

There are three methods in extracting titania from zircon sand, i.e.: pyrometallurgy, electrometallurgy, and hydrometallurgy. The most effective and efficient method is hydrometallurgy, which is why this method is the most often used method for extraction. In principle, hydrometallurgy comprises of three stages, that is i) leaching, ii) purifying, and iii) recovery (alkaline leaching). However, this method requires high concentration of acid. This method has been used by several researchers, such as in [5]. Lasheen [5] aims to optimize titania extraction from slag with caustic method. 325 mesh slag is reacted with soda with temperature variation of 400 to 1000°C. The titania obtained is 97% at roasting tempe-

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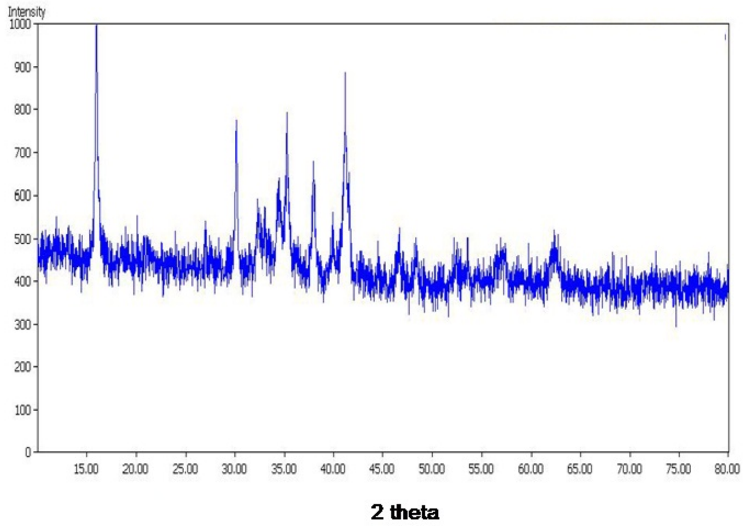


FIG. 1: XRD characterization result of the sample at 400°C.

rature of 850°C. Hence, there are a number of factors to increase the titania percentage in the hydrometallurgy method, such as roasting temperature and leaching. This is because the two factors aforementioned may affect ilmenite decomposition process.

Based on the above description, this research is conducted by varying the roasting temperature to find the optimum titania concentration. Hence, the objectives of this research are i) to determine the effect of roasting temperature towards the TiO_2 produced, and ii) to obtain a roasting temperature where TiO_2 is optimally produced. It is hoped that this research may give valuable insights about obtaining titanium dioxide from zircon sand.

II. EXPERIMENTAL PROCEDURES

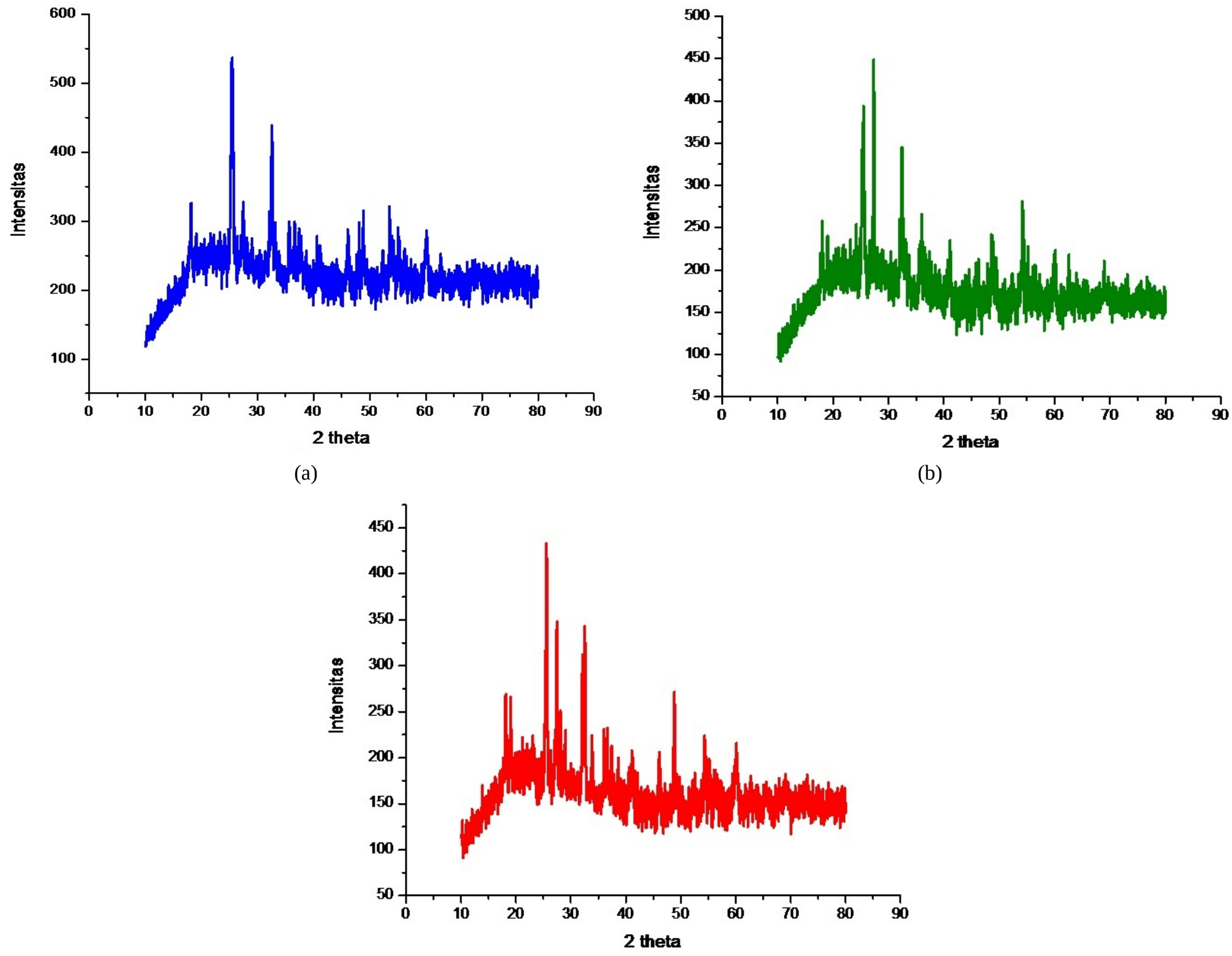
This is an experimental research to extract TiO_2 using ilmenit from zircon sand which contains TiO_2. The zircon sand sample is mixed with NaOH with ratio of 1:2 and a total mass of 200 grams. Then the sample is roasted inside a furnace with temperature of 400°C for one hour. The objective of NaOH addition and the roasting process is to decompose the ilmenite compound. After the sample is being roasted, the sample is given a number of leaching. The solution used in this process is distilled water and sulphuric acid with molarities variation. For the first leaching, the sample is mixed with 100 mL warm distilled water, and then processed using magnetic stirrer with temperature 150°C for 30 minutes. This is done to accelerate the reaction, and separate the sodium compound. Then filtration is done to separate the sodium compound and the sediment. The sediment obtained from distilled water leaching process is put into sulphuric acid solution with variation of 8, 10, and 12 M with ration of 1:1, and then processed using magnetic stirrer at temperature 200°C with angular speed 300 rpm for 2 hours. The acid leaching is conducted to separate Fe compound from impurities which may disrupt the purity of TiO_2. After the leaching process, the samples are left alone so that further sedimentation occurs. Stripping is done to separate between the solution and the sedimentation. The sediment obtained is washed using water to leave out the sulphuric acid excess. Then, the sample is dried at 200°C for 6 hours. This process is done in order to leave out water content in the sample. Furthermore, the sample is disc milled and continued by calcinations at 900°C for 2 hours.

III. RESULTS AND DISCUSSION

In this research, ilmenite is mixed with NaOH, and then mixing and roasting are conducted at temperature 400°C. The sample resulted from the roasting is characterized using XRD. The peaks obtained from the respective samples are analyzed using *Searchmatch* program which is equipped with *Crystallography Open Database* (COD). The initial characterization of ilmenite reaction with NaOH shows that the reaction is dominant. This is shown in the Figure 1.

Figure 1 shows that at temperature 400°C, the TiO_2 peak obtained is at angle 15.980 with COD number 96-400-8291. The compounds resulted from the roasting process are Fe_2TiO_5, Fe_3O_4, $Na_{0.75}Fe_{0.75}Ti_{0.25}O_2$, $FeTiO_3$, Ti_3O, Na_2TiO_3 and other small quantity compounds. At temperature 400°C, the compound with the highest intensity is $Na_{0.75}Fe_{0.75}Ti_{0.25}O_2$. This shows that at the aforementioned temperature, the compound is not separated into simpler component. Hence, leaching with water is conducted to separate and neutralize the pH of the solution such that Fe and Na may be dissolved in water. Furthermore, leaching on the sample is done using H_2SO_4 to bind the remainder of Fe element. In the final stage, concentration variation of 8, 10, and 12 M is done to find the optimum leaching process.

The XRD results for the 8, 10, and 12 M are shown in Figure 2. It may be observed from Figure 2(a) that some compounds are formed, such as $FeTi_2O_5$ with a peak at an-



(a) (b) (c)

FIG. 2: XRD results of the samples at 400°C for (a) 8 M, (b) 10 M, and (c) 12 M.

gle 52.59 with quantity 21.5%, TiO_2 (brookite) with peaks at 52.11 dan 60.20 with quantity 58.9%, and Fe_2TiO_5 with a peak at 56.63 and quantity of 19.7%. The compounds obtained from Figure 2(b) are TiO_2 (rutile) with peaks at 53.42 and 58.39 with quantity of 27.7%, $FeTi_2O_5$ with peak at 53.42 and quantity 22.1%, Fe_2TiO_5 with peaks at 53.57 and 54.95 with quantity of 40%, and TiO_2 (anatase) with peak at 52.48 and quantity of 10.1%. These results show that Fe element is still not perfectly dissolved so that leaching with distilled water is needed to dissolve Fe.

The alkaline leaching result in Figure 2(c) shows the compound produced are $Na_4Ti_5O_{12}$ with peak at 56.27 with quantity of 9.4%, TiO_2 (rutile) with peak at 52.14 dan 49.96 with quantity 32.2%, TiO_2 (anatase) with peak at 49.36 with quantity 11.5% and Fe_2TiO_5 with peaks at 51.71 and 52.72 with quantity of 46.9%. The presence of sodium titanate shows that there is still Na and Fe elements which are not perfectly dissolved in water, hence leaching with distilled water is needed to dissolve them. From the above analysis, the percentage of TiO_2 produced via leaching alkaline process at 400°C roasting temperature may be observed in Table I.

TABLE I: Percentage of TiO_2 produced by variation of concentration in the alkaline leaching process.

M	TiO_2(%)		
	brookite	anatase	rutile
8	58.9	-	-
10	-	10.1	27.7
12	-	11.5	32.2

IV. CONCLUSIONS

Sodium hydroxide has the ability to break ilmenite crystals because of its low melting temperature such that it becomes more reactive. The high roasting temperature, i.e. 400°C ensures that NaOH melts and reaction with zircon sand occurs. From the above data, it may be concluded that in the alkaline leaching process, the highest percentage of TiO_2 produced is using sulphuric acid with 12 M concentration and roasting temperature of 400°C.

Acknowledgments

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