Synthesis and characterization of TiO_2 from Lampung's Iron Sand using leaching method with temperature variation

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Abstract: Ilmenite is a natural material in the form of sand which contains heavy metal minerals that are very abundant in Lampung province. This research aims to determine the content of TiO_2 from Lampungs iron sand by leaching method with temperature variation. The iron sand are mixed with sodium hydrogen carbonate ($NaHCO_3$), then heated at 700°C for an hour. This research uses 5 samples with leaching temperature variation of 70, 80, 90, 100, and 110°C for 2 hours. The acid leaching process uses HCl 12 M with a ratio of iron sand and HCl of 1:4, while the water leaching process uses 50 ml of distilled water. Furthermore, the samples are characterized using XRD and XRF to determine TiO_2 content in the iron sand. The result shows that the highest amount of TiO_2 is obtained in the sample with the leaching temperature of 110°C for 2 hours, which is approximately 61%.

Keywords: Ilmenite; Iron oxide; Leaching; TiO_2.

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I. INTRODUCTION

Indonesia has abundant natural resources. One of the products of natural resources in the mining sector is iron sand. Iron sand is sand which contains a lot of magnetite or iron oxide compounds, consisting of a combination of iron and oxygen. It includes hematite (Fe_2O_3), magnetite (Fe_3O_4), and titanium oxide (TiO_2). The existence of iron sand as a mining material can be found in several parts of Indonesia, among others, on the southern coast of Java and in several areas on Kalimantan, Sulawesi, and Papua [1].

On the island of Sumatra, the location of resources and reserves of iron sand can be found in several provinces, namely Aceh, Bengkulu, and Lampung. The sand can be formed in Lampung province forming ilmenite and magnetite phases with a total resource of 945,429 tons based on data from the Indonesian Ministry of Energy and Human Resources. With such potential, however, iron sand in Lampung province has not been fully explored.

Titanium is an abundant element found in the Earth's crust with the content of about 0.63%. It always presents in other mineral forms such as ilmenite with rutile, anatase, and brookite crystalline structure, leucoxene, perovskite, and sphene, which are found in titanate and some iron sands [2]. Titanium is as strong as steel but weighted only 60% of that of the steel. The fatigue strength of titanium is higher than that of aluminum alloys. Titanium has potential applications in various fields such as in the fields of military, industry, medicine, and machinery [3]. Moreover, titanium dioxide is also used as an ingredient in a memristor, a new electronic-circuit element. Synthetic single crystals and TiO_2 films are used as semiconductors, and also in Bragg-stack force dielectric mirrors due to its high refractive index, reaching 2.5 - 2.9 [4].

One of the ingredients of iron sand is titanium dioxide. In general, titanium is rarely found in pure metal form. Most titanium is found in the form of rutile, which contains about 95% TiO_2. TiO_2 is an inorganic chemical compound which is used in the production of high-quality white pigments, such as a filler in paper mills, plastics/rubber, and glass factories. The largest consumption of TiO_2 is used by the pigment industry and only about 6% of TiO_2 is processed into titanium metal [5].

Several methods have been used in the extraction of titania from iron sands, including pyrometallurgy, pyrometallurgy, and hydrometallurgy [6]. The pyrometallurgical process involves burning iron sand at high temperatures with carbon as a reducing agent in order to reduce the iron in ilmenite and forming TiO_2-rich slag. However, there is a downside of this method, that not all iron can be removed from TiO_2. As a result, it necessitates heating conditions capable of melting iron. Whereas in the hydrometallurgical process, the dissolving of iron sand uses a solution of hydrochloric acid or sulfuric acid. The process involves dissolving iron sand in an acid solution, then forming a complex with a neutral or acidic organophosphorus solvent [7]. Because of the presence of iron dissolved in the acid solution, additional processing is needed in this procedure [8].

To obtain TiO_2 from iron sand, it can be done through the extraction process. One of the most commonly used methods is hydrometallurgy. Hydrometallurgy uses chemicals to dissolve certain particles, so that the metal can be obtained, which is then dissolved and separated. This method is of-

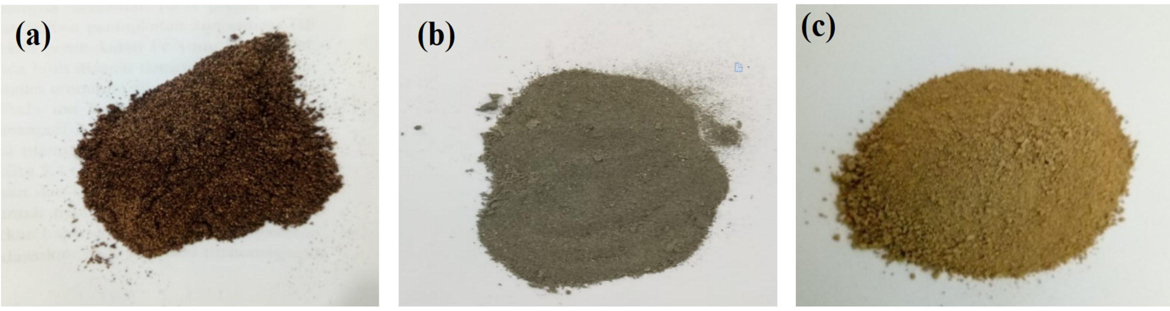

FIG. 1: (a) Result of roasting process of iron sand and $NaHCO_3$ at 700°C for an hour, (b) samples after acid leaching and drying at 100°C for 30 minutes, and (c) samples after calcination at 480°C for 5 hours.

TABLE I: XRF analysis results of iron sand and samples which were leached at different temperatures of 70, 80, 90, 100, and 110°C.

Compound	Unit	Iron sand	Without leaching	Leaching temperature (°C) 70	80	90	100	110
MgO	%	2.049	1.637	1.458	1.358	1.222	1.537	0.617
Al_2O_3	%	2.706	2.560	1.595	1.609	1.716	1.524	0.812
SiO_2	%	11.876	8.165	13.169	15.597	15.437	17.454	15.106
P_2O_5	%	0.502	0.4272	0.577	0.543	0.605	0.609	0.687
CaO	%	1.389	1.151	1.453	1.342	1.338	1.486	1.071
TiO_2	%	13.808	12.849	25.639	25.396	24.994	26.908	60.701
V_2O_5	%	0.498	0.475	0.524	0.538	0.525	0.505	0.574
MnO	%	0.570	0.573	0.504	0.496	0.493	0.501	0.221
Fe_2O_3	%	65.852	71.361	54.302	52.291	52.848	48.777	19.273
ZrO_2	%	937.4	0.113	0.237	0.172	0.163	0.206	0.641
Eu_2O_3	%	0.206	0.208	0.181	0.167	0.160	0.174	-

ten used in the refining process because of its effectiveness in purifying the metal. Some researches have been done such extraction of TiO_2 from mineral sand using hydrometallurgy method with HCl. Wu *et al.* [9] reported that TiO_2 extraction from ilmenite using HCl hydrometallurgy resulted 98% of TiO_2. Report by Zhang *et al.* [10] obtained 90.50% rutile TiO_2, and that by Tao *et al.* [11] produced 98.5% Rutile TiO_2.

According to the previous explanations, the goal of this research is extracting TiO_2 from the iron sand Lampung by HCl leaching at 70, 80, 90, 100, and 110°C.

II. METHOD

Iron sands were finely ground in a mortar, then sieved through a 200 mesh sieve before mixed with $NaHCO_3$ in a 1:2 ratio. Afterward, it was roasted for an hour at 700°C [12–14]. 2 M chloride acid (HCl) was used as a leaching medium, and the iron sand:HCl ratio was 1:4. Then, the sample was put in a beaker glass on a hot plate and stirred with a magnetic stirrer [13]. When HCl was boiling, iron sand was slowly added for 2 hours at varied temperature of 70, 80, 90, 100, and 110°C [14]. These variations are chosen by considering HCl boiling temperature at 110°C. The next step was water leaching using 50 ml distilled water at 80°C for 30 minutes [13].The obtained sediment was then washed using distilled water to remove acid residues in the sample. Subsequently, the sediment was then filtered with filter paper and distilled water until the yellow color of the sediment vanished [15]. In addition, the resulting sediment was dried in an oven at 100°C for 30 minutes to eliminate the water's moisture content. The calcination procedure was carried out at 480°C for 5 hours after the drying process.

Characterizations the samples were done using XRD (X-Ray Diffraction) and ED-XRF (Energy Dispersive X-Ray Fluorescence). XRD was used to analyze the formed phase with 2θ range of 10.02-80.01° with Cu K_α radiation (λ = 0.154 nm), where the voltage and the current were set at 40 kV and 30 mA. ED-XRF (PANanalytic Epsilon 3 XLE) was employed to examine the content of iron sand.

III. RESULTS AND DISCUSSION

Shown in Fig. 1, there is a physical color change in the powder resulting from the leaching process, from roasting at 700°C for 1 hour to calcination at 480°C for 5 hours. The color change occurs in the sample because the iron sand has dissolved into TiO_2 [15]. The result of XRF characterization can be seen in Table I.

TiO_2 of about 12.849% is obtained in the treated sample without HCl leaching. The highest amount of TiO_2 is found in the sample with leaching at 110°C, which reaches 60.701%. Referring to the result in Table I, there is an increase in the

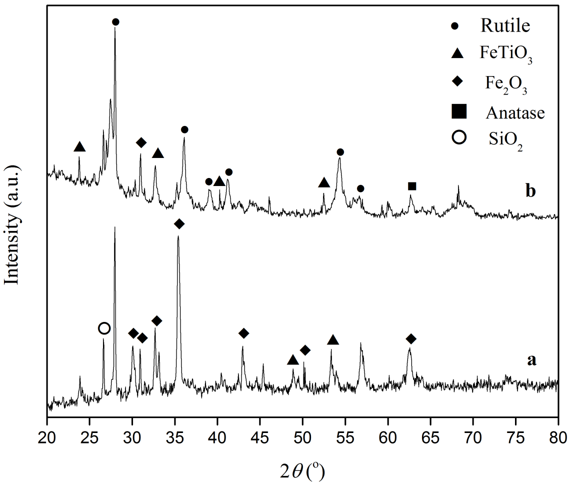

FIG. 2: X-Ray diffraction patterns of the samples: a) iron sand before the leaching process and b) iron sand after the leaching process at 110°C.

purity of TiO_2 acquired from the extraction of iron sand using leaching process. This is indeed consistent with the result reported by Purwani *et al.* (2016) [14], in which the leaching process of 50 g ilmenite was done in 11 M HCl. The resulted titanium compounds increase with increasing temperature applied during the extraction process.

Fig. 2 shows XRD analysis from iron sand before and after the leaching process at 110°C . It can be seen that there are many phases such as TiO_2 rutile, TiO_2 anatase, $FeTiO_3$, Fe_2O_3, and SiO_2. Diffraction peaks of TiO_2 Rutile (JCPDS No. 21-1276) after the leaching process at 110°C was observed at 2θ of 27.962, 36.076, 39.152, 41.258, 54.243, and 56.649°. Other observed phases are TiO_2 anatase (JCPDS No. 21-1272), $FeTiO_3$ (JCPDS No. 41-1432), Fe_2O_3 (JCPDS No. 33-0664), and SiO_2 (JCPDS No. 33-116). In addition to these phases, there are still many minor peaks that are only present in the form of single peak make it difficult to be identified accurately.

Our result suggests that ilmenite sand exhibits multiple phases, including Titania. The rutile phase is actually already formed when the iron sand is activated by thermal treatment [Fig. 2 (a)]. After the leaching process at 110°C and calcination at 480°C, there is an increase in the diffraction peak intensity of TiO_2 rutile phase. Based on XRD analysis, the employed method, in general, has successfully improved TiO_2 rutile content. However, impurity phases, such as Fe_2O_3 and SiO_2, still remain in the obtained sample.

IV. SUMMARY

This research shows that Lampungs ilmenite has the main compounds of Fe_2O_3 and TiO_2 with the relative percentage of 65.852% and 13.808%, respectively. The highest TiO_2 percentage in Lampung's ilmenite is obtained in the sample with the leaching temperature at 110°C for 2 hours, which from XRF analysis is found to be 60.701%. Analysis result of XRD shows an increment of the rutile phase content by leaching process. However, the impurity phases, such as iron oxide and silica, still presents in the sample.

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