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

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Abstract: This research aims to determine the content of TiO_2 from Lampung's iron sand by leaching method with temperature variation. The iron sand was mixed with sodium hydrogen carbonate ($NaHCO_3$), then roasted at 700˚C for an hour. This research used 5 samples with leaching temperature variation of 70, 80, 90, 100, and 110˚C for 2 hours. The acid leaching process used HCl 12 M with a ratio of iron sand, and HCl was 1:4, for the water leaching process used 50 ml of distilled water. Furthermore, samples were characterized using XRD and XRF to determine the content of Lampung's iron sand. The result shows that the highest amount of TiO_2 was obtained in a sample with a leaching temperature of 110˚C at 2 hours, i.e., 60,701%.

Keywords: Ilmenite; Leaching; TiO_2

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 its compounds contain a lot of magnetite or iron oxide compounds consisting of a combination of iron and oxygen, including 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].

Titanium is an abundant element found in the earth's crust, around 0.63%. It is always present in other mineral forms such as ilmenite (with its crystalline structure rutile, anatase, brookite), leucoxene, perovskite, and sphene which are found in titanate and some iron sands. Materials that contain titanium and are the most abundant on earth and most often used by humans are rutile and anatase. Rutile is the most stable form of titania and the most widely used source of titanium [2].

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. Titanium dioxide (TiO_2) is an inorganic chemical that can be applied, especially in the manufacture of the best quality white pigments, as a filler in paper mills, plastics factories, and rubber factories and as a flux in the glass industry. The largest consumption of TiO_2 is used by the pigment industry and only about 6% of TiO_2 is processed into titanium metal [3].

Titanium dioxide (TiO_2) is a white powder with opaque properties (transparent material). It is applied as a raw material for paint, paper, plastics, and gas sensors [4]. Titanium dioxide is a polymorphy compound with three crystal structures, namely rutile (a thermodynamically stable high temperature phase), anatase (a thermodynamically stable low-temperature phase), and brookite. Rutile is the type most commonly used in the pigment industry. Brookite has an orthorhombic structure and changes the crystal system to rutile at a temperature of 700ºC, while anatase has a tetragonal structure and changes its shape to rutile at a temperature of 915ºC [5]

The advantage of titanium is strong as steel, its weight is only 60% of the steel weight. The fatigue strength is higher than aluminum alloys. Titanium applications in various fields such as in the fields: military, industry, medicine, machinery [6]. Titanium dioxide is also used as an ingredient in memristors, a new electronic circuit element. It can be used for the conversion of solar energy based on dyes, polymers, TiO_2 nanocrystalline solar cells using conjugated polymers as solid electrolytes. Synthetic single crystals and TiO_2 films are used as semiconductors, and also in Bragg-stack force dielectric mirrors due to the high refractive index of TiO_2 (2.5 - 2.9) [7].

Several methods have been used in the extraction of titania from iron sands, including pyrometallurgy, pyrometallurgy, and hydrometallurgy [8]. The pyrometallurgical method is a method of burning iron sand with the help of carbon as a reducing agent at high temperatures so that the iron in ilmenite can be reduced and produce slag rich in TiO_2. However, this method has a disadvantage, i.e., not all iron can be separated from TiO_2. So that it requires heating conditions that are able to melt iron. Whereas in the hydrometallurgical process, the dissolving of iron sand uses a solution of hydrochloric acid or sulfuric acid. This method is carried out by dissolving iron sand using an acid solution followed by complex formation using a neutral or acidic organophosphorus solvent [9]. In this way, further processing is still needed because of the presence of iron dissolved in the acid solution [10].

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. This is so that the metal can be obtained which is then dissolved and separated. This method is often used in the refining process because of its effectiveness in purifying the metal.

Some of the researches which have been done such extraction of TiO_2 from mineral sand using Hydrometallurgy method and HCl, In Wu et al research [11] TiO_2 extraction from ilmenite using HCl hydrometallurgy obtained 98% TiO2, Zhang et al [12] 90.50% rutile TiO_2, and Tao et al [13] 98.5% Rutile TiO_2.

Based on the state of the arts, which have been explained above, this study aims to extract of TiO_2 from the iron sand of Lampung by leaching method using HCl with the variation of temperature leaching 70 ºC, 80 ºC, 90 ºC, 100 ºC, and 110 ºC.

II. METHOD

The iron sand was refined with a mortar, then sifted with a sieve 200 mesh and added the sodium hydrogen carbonate ($NaHCO_3$) with a ratio of 1:2. Next, it was roasted sand at 700°C for an hour [14 - 16]. The acid leaching process used 12 M HCl with a ratio of iron sand: HCl = 1 : 4. Then the sample was placed in the beaker glass which was on the hot plate and stirred with a magnetic stirrer [15]. After the HCI was boiled, then added iron sand slowly for 2 h with a leaching temperature variations temperature 70ºC, 80ºC, 90ºC, 100ºCand 110ºC [16]. The next process was water leaching using 50 ml distilled water at 80ºC for 30 minutes [15]. The sediment obtained is then washed using distilled water to remove acid residues in the sample. The next step is to filtered the obtained sediment with filter paper by adding distilled water until the yellow color of the sediment disappears [17]. Furthermore, the resulting sediment is dried in an oven at 100ºC for 30 minutes to remove the moisture content of the water leaching. After the drying process, calcination at 480°C with a total time of five hours. Afterward, characterized the sample using XRD (X-Ray Diffraction) and XRF (X-Ray Fluorescence).

III. RESULTS AND DISCUSSION

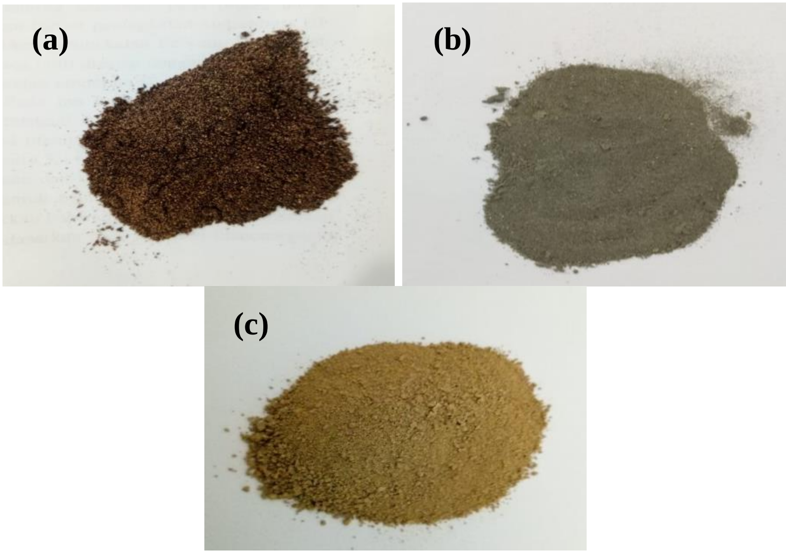

Figure 1. (a) Mix of iron sand and $NaHCO_3$ after roasted at 700°C for an hour, (b) Samples after acid leaching and drying at 100°C for 30 minutes, and (c) Samples after calcination at 480°C for five hours.

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 480°C for 5 hours.

Table 1. XRF test results on raw material and samples were leached with temperature variations of 70°C, 80°C, 90°C, 100°C, 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	-

The powder Colour changes after roasting, leaching process and calcinated at 480˚C. The color changes are observed in Figure 1. The result of XRF characterization can be seen in table 1. The sample with treated but without leaching HCl determine the amount of TiO_2 i.e., 12,849%. And the highest amount of TiO_2 obtained in the sample with leaching at 110˚C i.e 60,701%. From the result in table 1. There was an increase in the purity of TiO_2 obtained from the extraction of iron sand using leaching process.

Figure 2 shows XRD analysis from iron sand before the leaching process (a) and iron sand after the leaching process for 110 ^{O}C (b). From figure 2, 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. There are diffraction peaks TiO_2 Rutile (JCPDS No. 21-1276) of iron sand after the leaching process for 110 ^{O}C was at angle 2θ (27.962°; 36.076°, 39.152°, 41.258°, 54.243°, and 56.649°. And there are other phases in accordance with reference to 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.

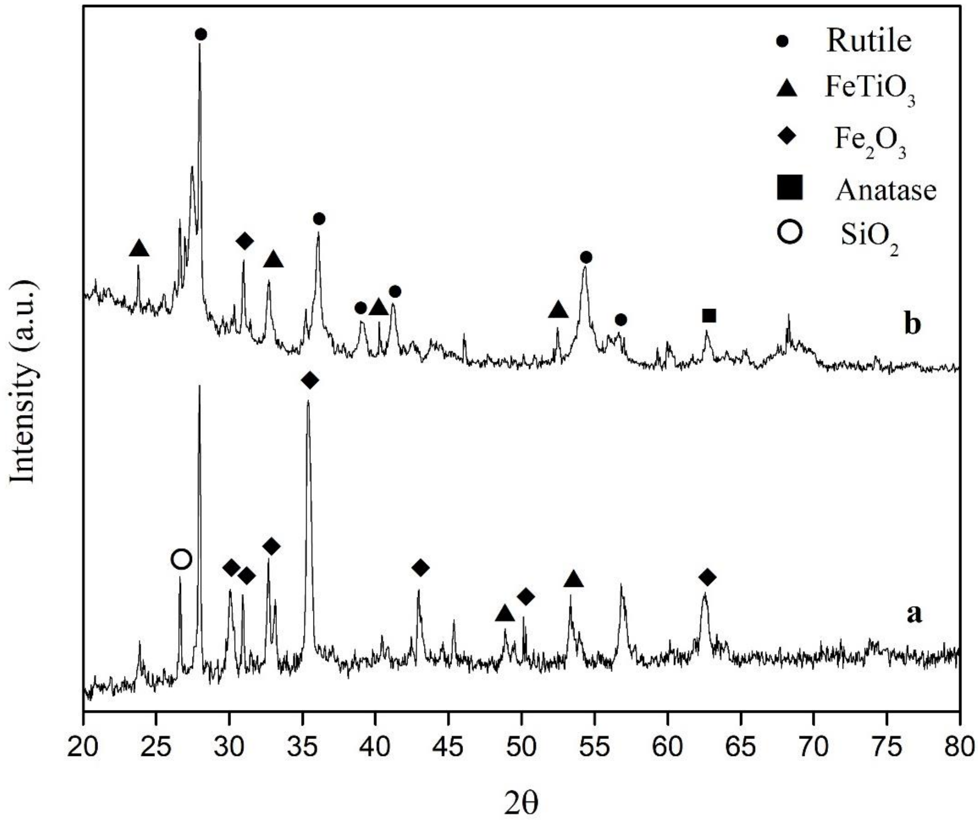

Figure 2. X-Ray Diffraction Patterns of the Samples, a) Iron sand before the leaching process, b) Iron sand after the leaching process for 110 ^{O}C

It seen that ilmenite sand gives the titania phase and other phases. It is seen that ilmenite sand gives titania and other phases. The rutile phase is actually already formed when the iron sand is activated by thermal treatment (Fig 2a), but after the leaching process of 110 ^{O}C and calcination of 480 ^{O}C there is an increase in the diffraction peak in the TiO_2 rutile phase. So from XRD analysis, in general it has succeeded in increasing the TiO_2 rutile, but impurity phases such as Fe_2O_3 and SiO_2 are still included in the leaching sample.

IV. CONCLUSION

The conclusion of this research indicating that Lampung’s ilmenite has the main compounds from Fe_2O_3 and TiO_2 with a percentage of 65.852% and 13.808%. The highest TiO_2 percentage in Lampung's ilmenite was obtained in samples with a leaching temperature at 110ºC for 2 hours, i.e., 60.701% from XRF analysis. The XRD analysis shows the increase of the rutile phase, but the impurity phases, such as iron oxide and silica, are still included in the sample.

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V. REFERENSI

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