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Synthesis and characterization of TiO_2 from Lampung's ilmenite using leaching method with variation of time duration

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Abstract. Ilmenite is one of the natural materials that are abundant in Lampung province**.** This research was conducted to determine the content of TiO_2 from ilmenite Lampung by the leaching method. Previously, iron sand was mixed with sodium hydrogen carbonate ($NaHCO_3$) and roasted at 700ºC for an hour. In this research, five samples were used to determine the TiO_2 content of ilmenite Lampung, with variations in leaching time of 2, 3, 4, 5, and 6 hours at 110ºC. The acid leaching process was carried out using 12 M HCl with a ratio of 1: 4. Then the water leaching process was carried out using 50 ml distilled water. Furthermore, all samples were characterized using XRD and XRF to determine the content in Lampung ilmenite sand. In general, XRD analysis results have succeeded in increasing the rutile phase which is influenced by the heating temperature. The highest TiO_2 content of Lampung ilmenite sand was obtained in samples with a leaching time variation of 2 hours at a temperature of 110ºC i.e. 60.701%.

1. Introduction

Iron sand is a mineral resource that can be found along the southern coast of Java, Sumatra, and West Nusa Tenggara [1]. Some of the main minerals can be extracted in iron sand i.e. magnetite (Fe_3O_4), ilmenite ($FeTiO_3$), rutile (TiO_2) and hematite (Fe_2O_3) [2]. 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. The presence of titanium in ilmenite can increase the added value of iron sand significantly [1]. Nearly 96% of the world's demand for titanium is used as raw material for making TiO_2 pigments due to several advantages, such as the ability of TiO_2 to protect surfaces (in paint) better than ordinary pigments (ZnO) and its anti-microbial properties. Much researches have been done to improve the ilmenite product from its compounds [3]. Wu has extracted TiO_2 from ilmenite using HCl with temperature of 100 ^oC and obtained the purity of TiO_2 of 98% [4].

In Lampung Province, iron sand can be found in West Lampung, South Lampung and Bandar Lampung. The TiO_2 content in iron sand must reach about 80% in order to be sold on the market [5].

Therefore, it is necessary to separate titanium from iron sand and other components so that both titanium and iron can be utilized. Several methods have been used to extract titanium from iron sand, including magnetic separation, pyrometallurgy, and hydrometallurgy. The separation of titanium can be carried out by the magnetic method which means by utilizing the magnetic properties of the minerals in iron sand [6]. Unfortunately, titanium is difficult to separate from iron physically because of the presence ofbonds interlock. It needs to be separated by other methods, for example by pyrometallurgy [7] and hydrometallurgy [8]. Pyrometallurgy is a method of burning iron sand with carbon as a reducing agent at high temperatures to produce TiO_2 riched-slag as a result of iron reduction in ilmenite. This method has a disadvantage i.e.,not all of the iron can be separated from TiO_2, so it requires heating conditions that are needed to melt the iron. The second method, namely hydrometallurgy. 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].

Along with development of technology, the extracting method of titanium dioxide from ilmenite has been developed, where the most widely used process is the hydrometallurgical process. The hydrometallurgical process can be divided into the sulfate process and the chloride process. In the sulfate process, a solution of sulfuric acid is used to dissolve ilmenite and then will form a solution of titanium sulfate. Whereas in the chloride process, a solution of hydrochloric acid is used to dissolve ilmenite and then form a solution of titanium chloride [11]. In general, the chloride process is more widely used, although the sulfate process was developed earlier [12]. This is because chloride is a strong acid which in high concentrations, has the ability to dissolve most of the various impurities, but relatively does not dissolve titanium [13] Several studies have been conducted to obtain TiO_2 from ilmenite, Setiawati et al. reported that acid washing using HCl with a concentration of 32% can achieve TiO_2 purity up to 91.46% [14], besides that Zhang has extracted TiO_2 using chloride from ilmenite which is preheated at 105 ^oC and the result is a TiO_2 of 90.5 % [15]. 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 12 M HCl with the variation of leaching duration i.e. 2, 3, 4, 5, and 6 hours. The leaching process result is then calcined at 480 ^oC for 2 hours. To determine the yield of TiO_2, the X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF) characterization was carried out.

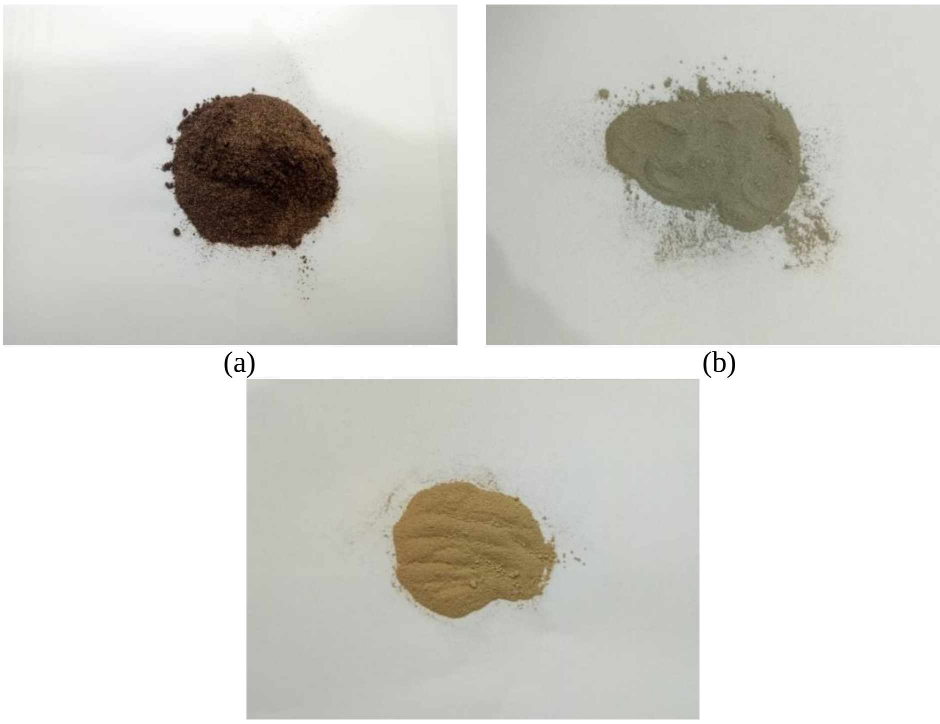
2. Method

This experiment begins with a preparation sample. The iron sand was refined using a mortar, then sifted using a sieve 200 mesh and finally added with sodium hydrogen carbonate ($NaHCO_3$) with the ratio of iron sand : $NaHCO_3$ = 1:2. The next process, the iron sand was roasted at 700°C for an hour [1] [16].

After that, the acid leaching process was carried out using 12 M HCl with a ratio of iron sand : HCl = 1 g : 4 ml (e.g., 20 gram iron and 80 ml HCl). Then the beaker glass was placed on the hot plate and boiled at 110ºC and stirred with a magnetic stirrer [17]. After the HCI was boiled, then it was added iron sand slowly and waited for some time duration i.e., 2, 3, 4, 5, and 6 h. When the volume of solution in the beaker glass was reduced until half of the initial volume, it was added 50 ml HCl. Then, the beaker glass was taken out from the hot plate and waited until it forms the sediment. Afterward, the liquid part of the solution was removed from the beaker glass.

The next process was water leaching. Aquadest (50 ml) was heated until 80°C in another beaker glass [18]. Afterward, the sediment was added into the aquadest and waited for 30 minutes without stirring. Then, the beaker glass was moved from the hot plate. In the next step, the liquid and its sediment were filtered with filter paper by adding aquadest until the yellow color of the sediment disappears [16]. Furthermore, the filtered sediment was heated in an oven at 100°C for 30 minutes. After heating, the sediment/sample was weighed and calcined at 480°C with a total time of five hours. Afterward, the sample was characterized using XRD (X-Ray Diffraction) and XRF (X-Ray Fluorescence).

3. Results and Discussion



(a) (b)

(c)

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 calcined at 480° C for five hours.

According to Figure 1, there is a revolution on the sample physically, from roasting at 700 ° C for an hour until calcined at 480° C for five hours. This change can be seen from the different colors in the samples. With the color change, it is assumed that the iron sand has dissolved so that TiO_2 has been formed.

Table 1. XRF test results on raw material and samples were leached with time variations of 2, 3, 4, 5, and 6 h

Compound	Unit	Iron sand	Without leaching	Leaching duration (h)				
				2	3	4	5	6
MgO	%	2,049	1,637	0,617	0,459	0,700	0,544	0,719
Al_2O_3	%	2,706	2,560	0,812	0,690	0,868	0,557	0,416
SiO_2	%	11,876	8,165	15,106	11,603	13,541	9,014	6,669
Cl	%	-	-	-	-	32,480	24,185	18,629
P_2O_5	%	0,502	0,4272	0,687	0,494	-	-	-
CaO	%	1,389	1,151	1,071	1,373	1,569	1,243	1,373
TiO_2	%	13,808	12,849	60,701	52,398	30,340	46,442	53,837
V_2O_5	%	0,498	0,475	0,574	0,543	0,181	0,258	0,415
MnO	%	0,570	0,573	0,221	0,463	0.275	0,218	0,246
Fe_2O_3	%	65,852	71,361	19,273	31,049	19,265	16,876	17,010
ZrO_2	%	937,4	0,113	0,641	0,559	0,410	0,428	0,509
ZrO	%	-	-	-	-	-	0,428	-
Eu_2O_3	%	0,206	0,208	-	0,123	722,7	591,7	545,8

If the amount of TiO_2 in iron sand is compared with TiO_2 in treated iron sand, but without HCl leaching in Table 1, it appears that the percentage of TiO_2 is almost the same i.e., 13.808% and 12.849%. This proves that HCl leaching plays a major role in increasing the purity of TiO_2. Then, based on Table 1, it shows that the purity (P_i) of TiO_2 changes as duration leaching (t) varies with 3rd order polynomial i.e. $P_i = at^3 + bt^2 + ct + d$, where a = 2.1 %/hour^3; b = -20.4 %/hour^2; c = 54.6 %/hour dan d = 14.3 %, as presented in Figure 2. According to Figure 2, it appears the highest purity of TiO_2 (60,701%) is obtained in leaching duration for 2 hours.

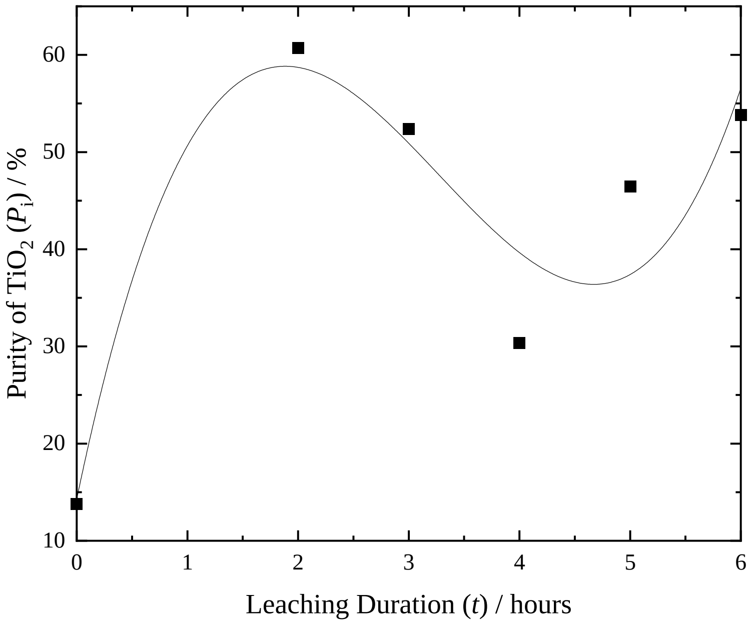


Figure 2. The dependency of TiO_2 purity (P_i) to the leaching duration (t)

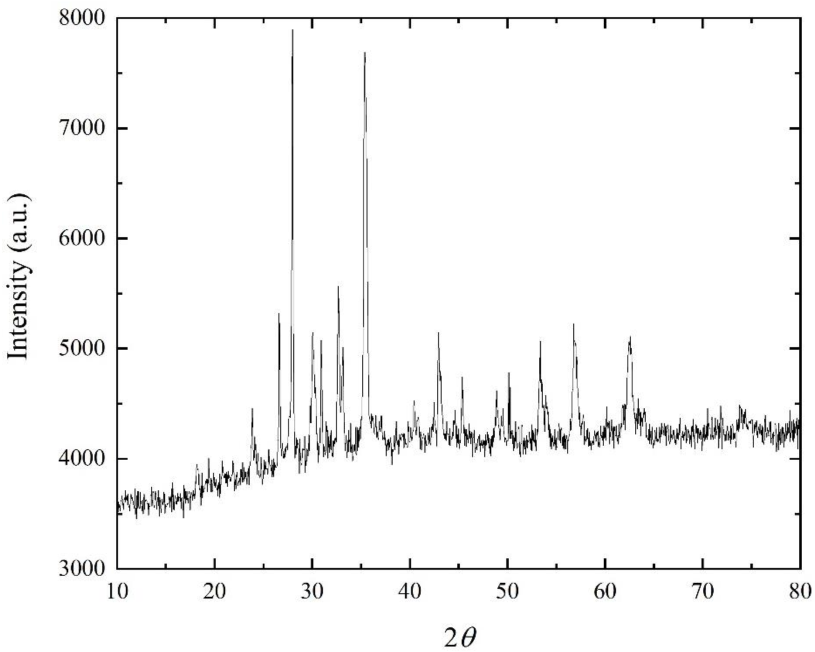


Figure 3. Iron sand diffractogram before the leaching process.

The diffractogram of iron sand before the leaching process is presented in Figure 3. Based on Figure 2, it can be seen that there are many mixed phases such as alumina, iron dioxide, and titanium dioxide. The presence of the rutile phase can be seen at the diffraction peak of 27.446°; 36.085°, and 54.322°. The presence of an iron oxide phase can be seen at 36.820°, 59.303°, and 65.185°. Apart from these three phases, there are still many peaks of minor phases, which sometimes only exist as a single peak. It is difficult to identify it accurately. From Figure 2, it is also clear that ilmenite sand basically already forms the titania phase, which is mixed with other phases. It can be understood because the material has been roasted at 700°C where the rutile phase has started to be formed.

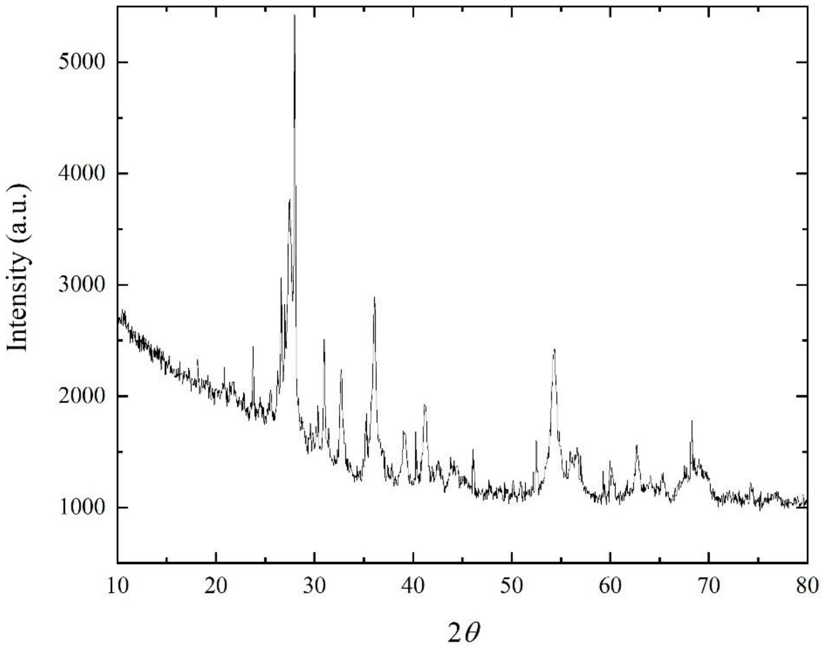


Figure 4. The diffractogram of the best sample which was obtained from the leaching process for 2 h.

Additionally, from Figure 4, the three highest diffraction peaks were obtained at the scattering angles of 2θ i.e., 27.446°, 36.085°, and 54.322°. This is in accordance with reference to titanium dioxide or rutile PDF Number 21-1276 (Natural Bureau Standard (US) Monograph 25, 7, 83 (1969). So in terms of the presence of rutile crystals in the sample is real. Besides titanium dioxide, there are still many peaks. At 2θ angle of 36.085° and 36.820°, there are overlapping between the Fe_2O_3 and TiO_2 peaks. In addition to this phase, there are also alumina phases at 2θ angle of 43.340°; 35.150°, and 57.498°. It seems this phase already exists in the sample before it was carried out by the leaching process. Finally, based on XRD data, this research has succeeded in increasing the rutile phase, but impurity phases such as iron oxide and alumina are still included in the sample. This result is in agreement with other literature [15].

4. Conclusion

Based on the results of this research indicating that Lampung's iron sand has the main compounds in the form of Fe_2O_3 and TiO_2 with a percentage of 65.852% and 13.808%. The XRF analysis results show that the highest TiO_2 percentage in Lampung's iron sand was obtained in samples with a leaching time duration for 2 hours at temperature 110ºC i.e., 60.701%. In general, XRD analysis shows the increase of the rutile phase, but the impurity phases, such as iron oxide and alumina, are still included in the sample. With this increase of the rutile phase, it can be used as a material for making thin films.

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