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Development of Extraction Method and Characterization of TiO_2 Mineral from Ilmenite

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Abstract : Study on mineral characterization and TiO_2 extraction by leaching method using H_2SO_4 on iron sand of Tapunggaya–Southeast Sulawesi has been conducted. Results of initial testing using X-Ray Diffraction (XRD) and X-Ray Flouresence (XRF) on sample of Tapunggaya iron sand showed that there were 4 major compounds namely Fe_2O_3, TiO_2, MgO, and SiO_2 in the mineral. TiO_2 extraction was conducted using sulphate method, by reacting milled iron sand with sulfuric acid at high temperatures (>110°C) for ± 30 minutes. Titanium extract was then heated at a temperature of 90°C to precipitate TiO_2. The precipitate was obtained by centrifugation at 10,000 rpm for 10 minutes to solidify the precipitate and separate from the liquid phase of H_2O and the remaining sulfuric acid. Subsequently, the extract was calcined for ±7 hours at temperature of 500°C and 1000°C to obtain TiO_2 anatase and rutile then purified with HCl and HNO_3. XRD and XRF characterization results showed the obtained TiO_2 extract containing anatase of 8.97% (70.3°; 1.337 Å) and rutile of 19.78% (54.2°; 1.699 Å).
Keywords : Extraction, Ilmenite, H_2SO_4, TiO_2, Southeast Sulawesi.

Introduction

The mining sector in Southeast Sulawesi Province is quite potential and becomes the attention of national and foreign investors. There have been many companies that conduct mineral exploration, particularly in North Konawe, Buton, Konawe, South Konawe, Kolaka, North Kolaka and other regencies of Southeast Sulawesi. Southeast Sulawesi has reliable potential mining, but has not been utilized optimally. This is related to the lack of researches done to increase knowledge and raise the sale value of mine materials.

One type of Indonesia's Natural Resources, which has a high sales value is Titanium. Currently the Indonesia still imports a variety of titanium products, while in Indonesia itself there are many ilmenite minerals which are the main source of natural titanium. Titanium is a type of metal that has a lot of special qualities, i.e light, shiny, strong, heat resistant, non-toxic, corrosion resistant and has high biocompatibility to the body[1]. Titanium metal is always joined with other elements in nature. This metal is the ninth most abundant element in the earth's crust (approximately 0.63%) and consisted mostly in igneous rocks and sediment[2]. This metal widely exists especially in minerals anatase, brookite, ilmenite, perovskite, rutile, titanite and most of iron ores. Titanium compound is generally in combination with iron as ilmenite ($FeTiO_3$), which so-called iron-titanium sand[3,4]. Ilmenite contains nearly 53% of TiO_2 (rutile) and is an essential mineral for the processing of titanium with silica impurities of about 10%, iron oxide, vanadium, niobium, tantalum, tin, chromium, and molybdenum compounds. In the form of magmatic, titanium is shaped in titanite ($CaTi(O|SiO_4)$) containing silica[5-8]. Ilmenite and rutile are difficult to obtain in high concentrations[9,10]. Therefore, titanium is easily bonded to oxygen and carbon at a high temperatures[11,12].

Titanium is a metal which is resistant to corrosion and high temperatures, with a high melting point, and low weight. Titanium can be extracted by hydrometallurgy method which includes ilmenite leaching with hydrochloric acid and sulfuric acid[13-15]. This process is one of the effective separation method for separating minerals from other elements. Titanium extracts obtained can be processed further specifically depending on their intended use, one of them as a base material in the production of TiO_2[16].

Titanium dioxide (anatase and rutile) can also serve as photocatalyst in oxidation and reduction reactions[17,18]. TiO_2 compound can absorb ultra violet irradiation that can be used as TiO_2 photocatalyst[19-21]. TiO_2 photocatalytic is a photochemical reaction that is accelerated by the presence of a catalyst, and has the ability to absorb photons to produce hydroxyl radicals that would degrade an organic pollutants, bacteria and fungi into eco-friendly end products[22,23]. TiO_2 has been chosen for the photocatalytic process because it is a semiconductor catalyst that is both biologically and chemically inert, stable against corrosion and non-toxic[24,25]. Given the increasing needs of metallic titanium dioxide, the study aims to obtain a compound of titanium dioxide extracted from ilmenite (iron ore) using the leaching method, then characterization was conducted to determine the content of titanium dioxide grades obtained using XRD and XRF. This research will add sale value to titanium in Indonesia in producing functional useful materials in the application of photocatalytic systems.

Experimental

1. Materials

Materials used in this research were iron sand ore from Tapunggaya – Southeast Sulawesi, H_2SO_4 (p.a), distilled water, HNO_3 (p.a), HCl (p.a).

2. Location of Research Sample

The sample used in this study were iron sand ore taken from Tapunggaya Southeast Sulawesi-Indonesia, location map of sampling can be seen in Fig. 1.

Fig. 1. Location map of iron sand sampling in Tapunggaya village North Konawe - South East Sulawesi - Indonesia

3. Preparation and separation of mineral sand

Black pure mineral sand from Tapunggaya were dried in the sun for ± 4 hours. Further the sand was stirred and separated magnetically. Mineral sand separation magnetically and non-magnetically was performed using a bar magnet with magnet strength of 0:42 Tesla. Sand separation magnetically and non-magnetically was done by moving the magnet under a sheet of paper, thus magnetic sand will drew in the magnetic field provided by the magnet.

4. Extraction process

a. Preparation of mineral sand and TiO_2 extraction leaching process

Mineral sand were milled using *Laboratory Discmill* for 10 minutes and the resulting powder was filtered with a size of 53 µm. A total of 20 grams of magnetic iron sand was added with 70 mL of 30% H_2SO_4, then the acid solution containing TiO_2 was heated at a temperature of 110-120°C ± 30-40 minutes until almost in the form of slurry and added iron powder of 1.0 grams. The slurry is cooled and filtered with filter paper and vacuum pumps to draw filtrate from the slurry. Then the filtrate was heated by adding water until there were white precipitate and centrifuged to separate from the acid solution. The precipitate was washed and calcined at a temperature of 500°C and 1000°C for 7 hours. To determine the crystal formed of TiO_2, the samples were tested using XRD and XRF[26].

b. Purification of the TiO_2 extract with HCl and HNO_3

To increase the content of TiO_2 purification was done with HNO_3 and HCl. TiO_2 extract was washed repeatedly with HNO_3 while heated for one hour. For washing using HCl solvent, it was done in the same way, washing using HNO_3 solvent until TiO_2 extract became white in color.

Results and Discussion

1. Characterization of Mineral Compounds in Iron Sand

Sand are sediment mineral which have grain size from 0.074 to 0.075 mm with coarse size (3-5 mm) and fine (<1 mm). Based on sand sediment, it enables the physical character differences of mineral sand contents such as Fe, Ti, Mg, and Si. Magnetite (Fe_3O_4) compound is a magnetic mineral that is usually found in coastal or river areas. Naturally, this compound derived from the titanomagnetite (Fe_{3}-xTixO$_4$ ($0 \leq x \leq 1$)) compounds. Before conducting the extraction process characterization was performed using XRF and XRD to identify the components of iron sand forming elements and to determine concentration of the targeted compounds.

In this study, iron ore (mineral) was washed to remove impurities such as soil and other organic compounds contained in the sampling areas. Then the samples were dried then sand separation was performed magnetically. Sand separation process magnetically and non-magnetically used a magnet intended that the components of the magnetic minerals were separated from non-magnetic mineral components. Once separated, magnetic material milled and screened using a miller and filtered. Furthermore, magnetic iron sand were characterized using XRF and XRD.

Tab. 1. Data of XRF characterization of iron sand sample from Tapunggaya-Southeast Sulawesi, Indonesia

No.	Atoms	Content (%)
1	Fe	34.07
2	Ti	11.74
3	Mg	6.87
4	Si	5.94
5	Ca	2.76
6	Cr	1.39
7	Mn	1.03
8	K	0.177
9	Zr	0.0513
10	Zn	0.0514
11	Nb	0.0230
12	Sr	0.0140

In Tapunggaya iron sand, there were 12 elements with Fe, Ti, Mg and Si as the four highest elements. After magnetic separation, these four elements could still be detected in part with magnetic and non-magnetic.

Based on XRD data of rock minerals iron analysis, it was known that most compounds were in the form of Fe_2O_3, TiO_2, MgO and SiO_2. These data reinforced the results of research conducted by Wahyuningsih et al. which indicates that the fourth largest element in iron sand are $FeTiO_3$, Fe_2O_3, MgO, and SiO_2[27]. Full list of oxide compounds in the sample are presented in Tab 2 below.

Tab. 2. Data of Compound XRF Characterization on Iron Sand from Tapunggaya-Indonesia

No.	Compounds	Content (%)
1	Fe_2O_3	48.71
2	TiO_2	19.58
3	MgO	11.39
4	SiO_2	12.70
5	CaO	3.85
6	Cr_2O_3	2.03
7	MnO	1.33
8	K_2O	0.214
9	ZrO_2	0.0693
10	ZnO	0.0640
11	Nb_2O_3	0.0329
12	SrO	0.0165

Tab 2 shows the TiO_2 compound contained in the sample with the second largest concentration of Fe_2O_3 compound which is the dominant compound in the iron sand sample, so it can be ascertained that after leaching process, TiO_2 extract was obtained with quite large percentage (19:58%).

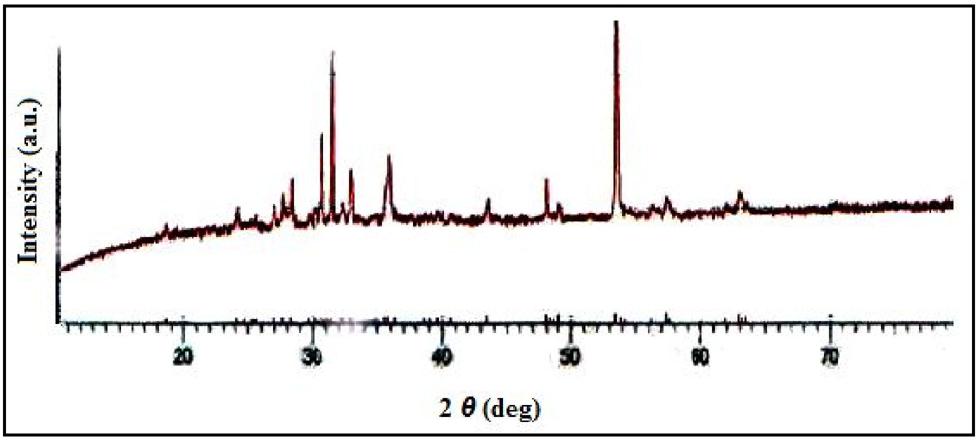

Fig. 2. Pattern of XRD of Tapunggaya Iron Sand

The main peak of Tapunggaya iron sand found on 53.43º (d = 1.71 Å); 31.41º (d = 2.84 Å); 30.65º (d = 2.91 Å); 35.75º (d = 2.50 Å); 48.27º (d = 1.88 Å) and 28.32º (d = 3.14 Å) (Fig. 2).

2. Leaching Process of TiO_2 Mineral Compound in Iron Sand

a. Extraction process

In this study TiO_2 extraction was conducted by reacting iron sand with H_2SO_4 using high temperature. Iron sand after physically separated, then was identified its compounds and elements using XRD and XRF. In theory, a chemical reaction occurs between the compounds of ilmenite and sulphate acid during the leaching process is as follows:

Digestion : $FeTiO_{3\,(s)} + 2H_2SO_{4\,(aq)} \rightarrow FeSO_{4\,(s)} + TiOSO_{4\,(aq)} + 2H_2O_{\,(l)}$ (1)

Presipitation : $TiOSO_{4\,(aq)} + 2H_2O_{\,(l)} \rightarrow TiO_2.H_2O_{\,(s)} + H_2SO_{4\,(aq)}$ (2)

Calcination : $TiO_2.H_2O_{\,(s)} \rightarrow TiO_{2\,(s)} + H_2O_{\,(g)}$ (3)

Ilmenite dissolved in hot concentrated sulfuric acid will decompose to form compound of $FeSO_4$ (insoluble phase) and $TiOSO_4$ (dissolved phase). After going through the process of leaching and calcination in $TiOSO_4$ dissolved phase then TiO_2 obtained in powder form. This is why the percentage of TiO_2 after leaching was increased.

In the leaching process, diffusion occurred from the sample into the solvent liquid phase which later form a balance. In this condition, the components in the sample can not be diffused into the solvent. Important parameters in the extraction of solid-liquid are mass transfer coefficient and the equilibrium constant. The equilibrium constant shows the minimum ratio between the solvent and extracted solids[28]. In the leaching process it was used by 20 grams of sand which were dissolved in 75 mL of 30% sulfuric acid at a temperature of heating >110°C. Chemical reactions in the leaching process is described as follows:

$FeTiO_{3\,(s)} + 2H_2SO_{4\,(aq)} \rightarrow FeSO_{4\,(s)} + TiOSO_{4\,(aq)} + 2H_2O_{\,(l)}$ (4)

Extract obtained from filtered leaching process (filtration) using a Buchner filter and drawn by using a vacuum pump. This process was done to separate the dissolved phase ($TiSO_4$) from its non-dissolved phase ($FeSO_4$). In this process, $TiOSO_4$ was dissolved in sulfuric acid solvent, while $FeSO_4$ is an insoluble substance, the results of this process produced viscous liquid (slurry). After the cooling process the viscous liquid was filtered to obtain filtrate which is rich in titanium then added with water and continued by heating it at temperature of 80°C. At this stage the chemical reaction occurred as follows:

$TiOSO_{4\,(aq)} + 2H_2O_{\,(l)} \rightarrow TiO_2.H_2O_{\,(s)} + H_2SO_{4\,(aq)}$ (5)

$TiO_2.H_2O$ sediment was cleaned by several times washing (until pH was neutral) using distilled water to remove the existing sulfuric acid content. $TiO_2.H_2O$ which has been free of sulfuric acid was then H_2O content was removed by heating process. The chemical reactions that occurred as follows:

$TiO_2.H_2O_{\,(s)} \rightarrow TiO_{2\,(s)} + H_2O_{\,(g)}$ (6)

White TiO_2 sediment was then separated from the solution consisting of water and H_2SO_4. H_2SO_4 aqueous phase was discarded and replaced with distilled water[29]. This was done in order to separate the acid solution from the TiO_2 sediment.

TiO_2 suspension was centrifuged for 10 minutes at a speed of 10,000 rpm for 10 times. $TiO_2.H_2O$ produced was furnaced at two different temperatures, i.e 500°C and 1000°C performed to remove H_2O and expected to produce two characteristics of the titanium dioxide i.e rutile and anatase[30].

b. Purification of TiO_2

To obtain TiO_2 content purification was done using HNO_3. Where TiO_2 extract was washed repeatedly using HNO_3 while heating it for one hour until TiO_2 extract almost in white.

c. Characteristics of TiO_2 Compound of Extraction Results

The characterization of the extract were obtained by using XRF and XRD to determine the components contained in the extract and obtained the data crystals formed. XRF data showed TiO_2 extracted by calcination process at temperature of 1000°C that showed greater concentration than at temperature of 500°C. XRF characterization results for 500°C TiO_2 (anatase) (Tab 3) and 1000°C (rutile) (Tab 4).

Tab. 3. XRF Characterization Results of TiO_2 at 500°C

Compounds	Level (%)	Unsur	Level (%)
SO_3	56.82	Sx	22.75
Fe_2O_3	32.67	Fe	22.85
TiO_2	8.97	Ti	5.38
P_2O_5	1.27	Px	0.554
CaO	0.176	Ca	0.126
ZnO	0.0523	Zn	0.0420
ZrO_2	0.0246	Zr	0.0182
Nb_2O_3	0.0175	Nb	0.0122

Tab. 4. XRF Characterization Results of TiO_2 at 1000°C

Compounds	levels (%)	Unsur	Levels (%)
Fe_2O_3	78.32	Fe	54.77
TiO_2	19.78	Ti	11.86
MnO	1.23	Mn	0.951
P_2O_5	0.239	Px	0.104
CaO	0.231	Ca	0.165
ZnO	0.114	Zn	0.0917
ZrO_2	0.0484	Zr	0.0358
Nb_2O_5	0.0284	Nb	0.0199
SnO_2	0.0070	Sn	0.0055

XRD data was used to determine the characteristics of formed TiO_2 crystalline anatase/ rutile. Fig. 3 shows the characterization data crystalline anatase by calcination at 500°C.

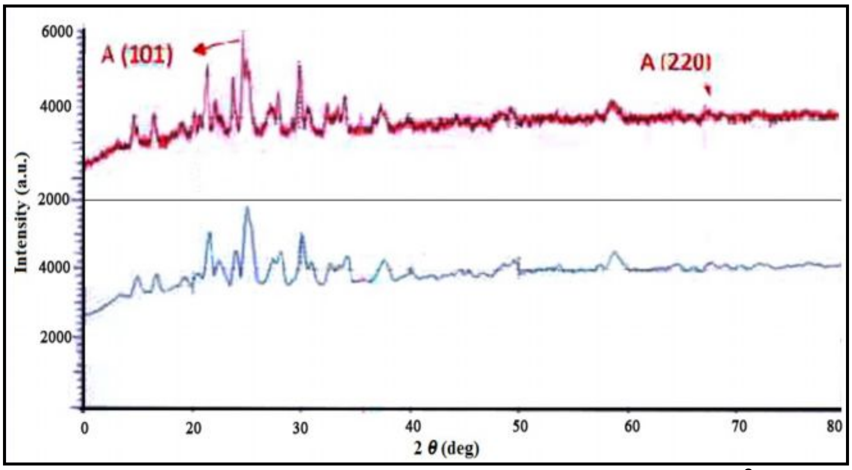

Fig. 3. Pattern of XRD on extract TiO_2 at calcination of 500^oC

Fig. 3 shows the extraction results of iron ore samples contained crystalline anatase TiO_2. It is indicated the specific peaks in the spectrum of XRD characterization results by the *Joint Committee on Powder Diffraction Standards* (JCPDS). The diffraction pattern showed a peak which is the spectrum produced by crystalline anatase TiO_2, which is the peak of 70.3 in the field of 220 and eight other peaks that are assumed as TiO_2 crystals. This eighth peaks respectively are at 25.07 (as the highest peak); 37.54; 53.55; 68.98; 74.62; 74.62; 75.54; 76.69; and 78.68.

Extract anatase was carried out at lower temperatures (500°C) and rutile at high temperatures (1000°C). Both rutile and anatase were extracted by leaching technique using H_2SO_4 solvent at high temperatures. The difference of the type of TiO_2 crystals was depended on the calcination process (anatase 500°C and rutile 1000°C). Rutile characterization test results with the XRD can be seen in Fig. 4.

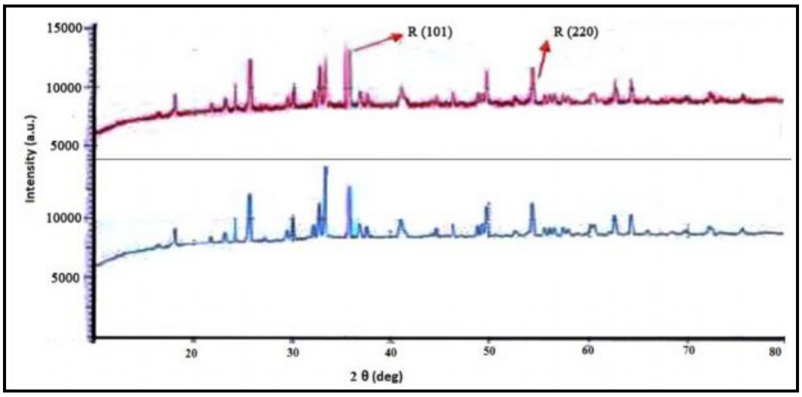

Fig. 4. Patterns of XRD on TiO_2 extract at temperature of 1000°C

Fig. 4 shows the best rutile TiO_2 extract is projected at peak of 54.2 R (220), while for other TiO_2 possible peaks is at the peak of 35.72 R (101). There are still few peaks that are identical to the characters of rutile TiO_2 at the above diffraction pattern which are 27.2; 39.2; 39.4; 41.2; 43.6; 54.25; 62.6; 63.7; and 69.7.

Conclusions

1. The mineral compound on Tapunggaya iron sand were Fe_2O_3 48.71%; TiO_2 19.58%; SiO_2 12.7%; MgO 11.39%; CaO 3.85%; Cr_2O_3 2.03%; MnO 1.33%; K_2O 0.214%; ZrO_2 0.093%; ZnO 0.0640%; Nb_2O_5 0.0329%; dan SrO 0.0165%.
2. TiO_2 extraction of iron sand can be done by leaching with solvent H_2SO_4 at temperature of > 110°C, oxidized at a temperature of 90°C, centrifuged, calcined and then washed with acid.
3. TiO_2 compound were obtained by extraction with concentration of (mass %) 19.78% (1000°C) and 8.97% (500°C).

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