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EXTRACTION OF TITANIUM DIOXIDE (TiO_2) FROM IRON SAND OF TEMBAKAK BEACH WEST COAST AS NANOPARTICLES USING HYDROMETALLURGY METHOD

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ABSTRAK

Sampel pasir besi berasal dari Pantai Tembakak Kabupaten Pesisir Barat, dipreparasi kemudian dianalisis dengan XRF, diperoleh kandungan Fe 58,294%; Si 18,525%; Ti 8,775%; Al 6,785%; Ca 3,885%; K 1,624%, serta unsur-unsur minor dengan kandungan di bawah 0,5%. Titanium dioksida (TiO_2) dapat diperoleh dari ilmenit ($FeTiO_3$), dengan metode ekstraksi hidrometalurgi. Hasil ekstraksi dianalisis dengan XRF, diperoleh TiO_2 pada variasi konsentrasi HCl 7 M sebesar 15,033%, HCl 9 M sebesar 16,367%, dan HCl 12 M sebesar 17,421%. Karakterisasi XRD pada hasil ekstraksi TiO_2 variasi konsentrasi HCl 12 M menunjukkan TiO_2 memiliki fasa kristal rutil dengan struktur kristal tetragonal, serta memiliki ukuran partikel sebesar 33,92 nm sehingga partikel TiO_2 yang diperoleh merupakan nanopartikel yang berperan penting dalam perkembangan teknologi dan industri.

Kata kunci: Ekstraksi, Hidrometalurgi, Konsentrasi, Nanopartikel, Pasir Besi.

ABSTRACT

The iron sand sample came from Tembakak Beach, Pesisir Barat Regency, was prepared and then analyzed using XRF, obtained an Fe content of 58.294%; Si 18.525%; Ti 8.775%; Al 6.785%; Ca 3.885%; K 1.624%, as well as minor elements with a content below 0.5%. Titanium dioxide (TiO_2) can be obtained from ilmenite ($FeTiO_3$), by hydrometallurgical extraction methods. The extraction results were analyzed using XRF, obtained TiO_2 at varying concentrations of HCl 7 M of 15.033%, HCl 9 M of 16.367%, and HCl 12 M of 17.421%. XRD characterization of TiO_2 extraction results with variations in HCl concentration of 12 M shows that TiO_2 has a rutile crystal phase with a tetragonal crystal structure, and has a particle size of 33.92 nm so that the TiO_2 particles obtained are nanoparticles that play an important role in technological and industrial development.

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Keywords: Extraction, Hydrometallurgy, Concentration, Nanoparticles, Iron Sand.

INTRODUCTION

Generally, iron sand is found along beaches with a characteristic blackish gray color. One of the areas in Lampung that has many beaches with predominantly black sand is Pesisir Barat Regency (Jalaludin *et al.*, 2021). In general, iron sand contains magnetite (Fe_3O_4), ilmenite ($FeTiO_3$), and hematite (Fe_2O_3), which have the potential to be nanoparticles (Bahfie *et al.*, 2022). Nanoparticles have great potential for technological development because they have several properties that are superior to bulk materials. Based on this, the West Coast has quite a large potential for iron sand nanoparticles, but until now, research in this area has been minimal. This creates a problem because this region's potential still needs to be discovered and has yet to be developed optimally.

According to Ermawati *et al.*, (2011), Titanium dioxide (TiO_2) can be obtained from ilmenite ($FeTiO_3$), which is one of the minerals found in iron sand. The advantages of TiO_2 include having good optical properties, good photocatalytic properties, dielectric properties, good biocompatibility as a semiconductor, low cytotoxicity, and good chemical stability (Akakuru *et al*., 2020). So, TiO_2 nanoparticles have a major contribution in technological and industrial applications, including as an agent for attenuating ultraviolet (UV) radiation in sunscreen (Ko *et al*., 2012), as a hybrid supercapacitor (Kim *et al*., 2013), as a semiconductor material in Dye Sensitized Solar Cell (DSSC) photoelectrode components (Komalasari *et al*., 2014), and so forth.

Ermawati *et al.* (2011) have conducted research on TiO_2 extraction from Pandeglang iron sand, West Java using the leaching method using HCl, and obtained a TiO_2 content of 64.62%. TiO_2 extraction from iron sand can be carried out using pyrometallurgical, electrometallurgical and hydrometallurgical methods (Priharyono dan Gusmarwani, 2022). The disadvantages of the pyrometallurgical method are that it requires high energy and temperature, high energy consumption, and produces lower product quality (Zheng *et al*., 2018). Meanwhile, the hydrometallurgical method has the advantage of using relatively low temperatures, low energy consumption, and producing TiO_2 products of sufficient quality for various applications (Zhang *et al*., 2011). Therefore, the extraction method used in this research is the hydrometallurgical method using HCl solvent.

In this research, extraction of iron sand from Tembakak Beach, Pesisir Barat Regency was carried out using the hydrometallurgical method using HCl to determine the mineral content found in iron sand in Pesisir Barat, determine the effect of HCl concentration on the extraction of TiO_2 from iron sand, determine the phase and crystal structure of TiO_2, and obtain TiO_2 nanoparticles which play an important role in technological development. TiO_2 characterization is carried out using X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD), where XRF is carried out to determine the elemental components of the sample and XRD to determine the phase, crystal structure and size of the particles formed.

METHOD

Tools and Materials

The tools used in this research were a bar magnet, PQ-N2 planetary ball mill, AS200 Tap sieve shaker, 325 mesh sieve, Nabertherm muffle furnace, spatula, porcelain cup, stirring rod, crucible, beaker, Erlenmeyer, filter paper, measuring cup. , measuring flask, glass funnel, oven, analytical balance, aluminum foil, magnetic stirrer, hot plate, X-Ray Fluorescence (XRF) Brand Panalytical Epsilon 3 XLE and X-Ray Diffraction (XRD) Brand Panalytical Xpert 3 Powder. The materials used are iron sand originating from the sand of Tembakak Beach (West Pesisir Regency, Lampung Province, Indonesia), HCl p.a Merck, NaOH, and distilled water.

Procedure

Sample Preparation

The samples were subjected to magnetic separation using a bar magnet. Samples containing magnetic minerals were ground using a planetary ball mill with a speed of 300 rpm for 30 minutes, then sieved using a sieve shaker with a 325 mesh sieve. An analysis of the elemental components of iron sand was carried out using an XRF instrument.

TiO_2 Extraction from Iron Sand

The prepared iron sand samples were then decomposed by adding NaOH. 20 grams of iron sand is added with 40 grams of NaOH in a ratio of 1:2, then heated in a muffle furnace at a temperature of 450 °C for 2 hours (Xue *et al*., 2009). The solids formed were leached with distilled water with a weight ratio of sample and distilled water of 1:5, at a temperature of 80 °C with a speed of 400 rpm for 30 minutes. The distilled water leaching results are filtered, and the precipitate is dried in an oven at a temperature of ±105 °C. Next, acid leaching was carried out using HCl with varying concentrations of 7 M; 9M; and 12 M at a temperature of 100 °C with a speed of 600 rpm for 2 hours, the weight ratio of the precipitate and HCl is 1:5. The acid leaching results were washed using distilled water, then the precipitate was dried in an oven at a temperature of ±105 °C (Firdaus *et al*., 2021). The dried precipitate was calcined at 600 °C for 2 hours (Rohmawati *et al*., 2020).

RESULTS AND DISCUSSION

Iron Sand Sample Preparation

Iron sand samples from Tembakak Beach, Pesisir Barat Regency were subjected to magnetic separation using a bar magnet to separate the samples from impurities. Magnetic separation produces magnetic minerals (concentrate) and nonmagnetic minerals (tailings). The mineral content of iron sand, especially the element titanium, has magnetic properties (Bahfie *et al*., 2022), so in this study samples were used that contain magnetic minerals with a shiny blackish gray physical condition after being ground and sifted (Figure 1).

Figure 1. Results of iron sand sample preparation

Characterization of Iron Sand Samples

The results of the iron sand sample preparation were then characterized using an X-Ray Fluorescence (XRF) instrument Merk Panalytical Epsilon 3 XLE brand to determine the components of Tembakak Beach iron sand elements (Table 1).

Table 1. Data on elemental components of Tembakak beach iron sand after sample preparation

No.	Element	Concentration (%)
1	Fe	58.294
2	Si	18.525
3	Ti	8.775
4	Al	6.785
5	Ca	3.885
6	K	1.624
7	Mn	0.433
8	V	0.343
9	P	0.289
10	Eu	0.283

Table 1 shows that the Ti element is one of the components with the largest concentration, which is smaller than Fe and Si, but much larger than Al, Ca, K, and other minor elements with a content below 0.5%. Priharyono and Gusmarwani (2022) also conducted research on iron sand from South Beach Kulon Progo Yogyakarta, obtained Ti elements of 4.618% after the Fe, Si, Ca, Al content, while at Tembakak Beach the Ti element content was 8.775% after the Fe and Si content. so it is greater than the Al and Ca content. Therefore, the Ti element content in Tembakak Beach iron sand is greater than in South Kulon Progo Beach iron sand, so it is the basis for using Tembakak Beach iron sand in this research.

TiO_2 Extraction from Iron Sand

The extraction process in this research was carried out by varying the HCl concentration during acid leaching. The extraction process begins with a decomposition process using NaOH to remove impurities and minor minerals in the sample such as the elements Nb, Bi, Ga, and others, so as to maximize the TiO_2 extraction results (Lalasari *et al*., 2012).

$$2FeTiO_{3(s)} + 4NaOH_{(s)} \xrightarrow[\text{calcination } 450°C]{} 2Na_2TiO_{3(s)} + 2FeO_{(s)} + 2H_2O_{(aq)} \quad (1)$$

The distilled water leaching process aims to dissolve iron, impurities and other minor minerals, where NaOH will be bound to impurities or other minor minerals that are easily dissolved (Middlemas *et al*., 2013). The results of distilled water leaching were filtered and an orange filtrate and reddish brown precipitate were obtained. The solubility of Fe in distilled water leaching is not perfect, so acid leaching is carried out using HCl with varying concentrations of 7 M; 9M; and 12 M.

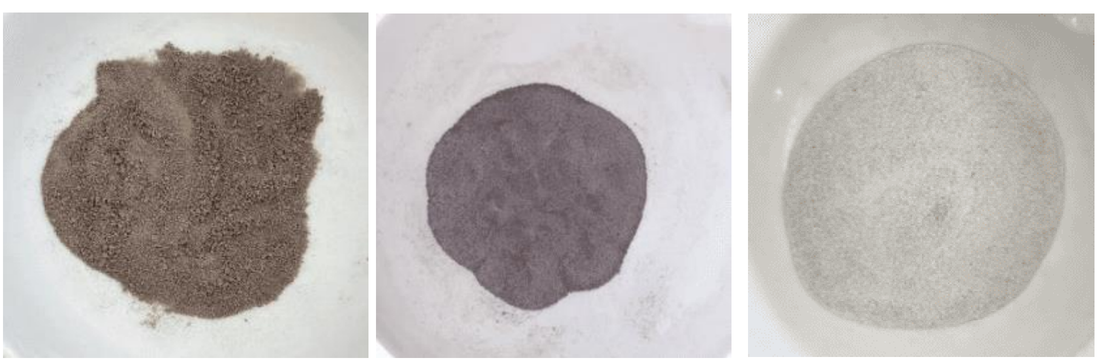
$$2Na_2TiO_{3(s)} + Fe(OH)_{3(aq)} + 11HCl_{(aq)} \rightarrow 2TiOCl_{2(s)} + FeCl_{3(aq)} + 4NaCl_{(aq)} + 7H_2O_{(aq)} \quad (2)$$

The results of the acid leaching were washed using distilled water (Reaction 3), the results obtained from the first washing at HCl 7 M were green, at HCl 9 M were orange, and at HCl 12 M were reddish orange. Li *et al.* (2008) stated that the acid concentration affects the leaching process, where the solubility of Fe can increase as the acid concentration increases. The acid leaching results are washed several times until a clear colored filtrate is obtained to remove acid residues and so that the precipitate obtained is free of Fe^{3+} ions.

$$2TiOCl_{2(s)} + 2H_2O_{(aq)} \rightarrow 2TiO_2{\cdot}H_2O_{(s)} + 4HCl_{(aq)} \quad (3)$$

$$2TiO_2{\cdot}H_2O_{(s)} \xrightarrow[\text{calcination } 600°C]{} 2TiO_{2(s)} + H_2O_{(g)} \quad (4)$$

The precipitate was dried in an oven at a temperature of ±105 °C, and calcined to form the crystallinity of TiO_2 particles (Reaction 4). The yield obtained at varying concentrations of HCl 7 M was 11.3% purplish gray, HCl 9 M was 9.6% gray, and HCl 12 M was 13.20% white (Figure 2).



(a) (b) (c)

Figure 2. Results of TiO_2 Extraction with Varying HCl Concentrations in Acid Leaching (a) HCl 7 M (b) HCl 9 M (c) HCl 12 M

The difference in color obtained is due to an increase in HCl concentration during acid leaching. It is known that acid concentration influences the leaching process, namely the solubility of Fe can increase along with increasing acid concentration (Li *et al*., 2008). In this research, the more concentrated the HCl concentration used, the higher the Ti content, while the Fe content and other minor elements will be lower.

Characterization of TiO_2

X-*Ray Fluorescence* (XRF)

The extraction results were then characterized using XRF to determine the iron sand components and the effect of HCl concentration on TiO_2 extraction.

Table 2. The results of XRF of TiO_2 Extraction with Varying HCl Concentrations

No	Compound	HCl Concentration (%)		
		7 M	9 M	12 M
1	SiO_2	60.488	63.940	62.105
2	Fe_2O_3	22.032	17.160	18.044
3	TiO_2	15.033	16.367	17.421
4	Al_2O_3	0.863	0.863	1.108
5	P_2O_5	0.430	0.445	0.418
6	V_2O_5	0.260	0.224	0.250
7	CaO	0.226	0.178	0.150
8	MnO	0.153	0.124	0.139

Table 2 shows that in this research we have succeeded in extracting TiO_2, the results of which are still smaller than SiO_2 and Fe_2O_3, but much larger than other oxides with a content below 1%. Based on the results obtained, it is known that the TiO_2 results in this study were less than optimal, although the Fe_2O_3 content was successfully reduced, there was an increase in the SiO_2 content. The TiO_2 obtained in this research is also in accordance with research conducted by Supriyatna *et al* (2020) which used iron sand samples from the coast of South Lampung. After leaching using HCl, the TiO_2 content was obtained at 21.21%. Based on this, it is known that the TiO_2 content resulting from iron sand extraction from a beach has less than optimal results.

Based on the characterization results of the three variations in HCl concentration above, the maximum TiO_2 content results were obtained, namely at a HCl concentration of 12 M, so that the influence of HCl concentration on the TiO_2

extraction process from iron sand is that the more concentrated the HCl concentration, the greater the TiO_2 content obtained.

X-*Ray Diffraction* (XRD)

The TiO_2 obtained from the extraction results was characterized using X-Ray Diffraction to determine the phase, structure and size of the TiO_2 particles formed. The diffractogram was analyzed using High-Score Plus (HSP) software which refers to standard Crystallography Open Database (COD) data (Figure 3).

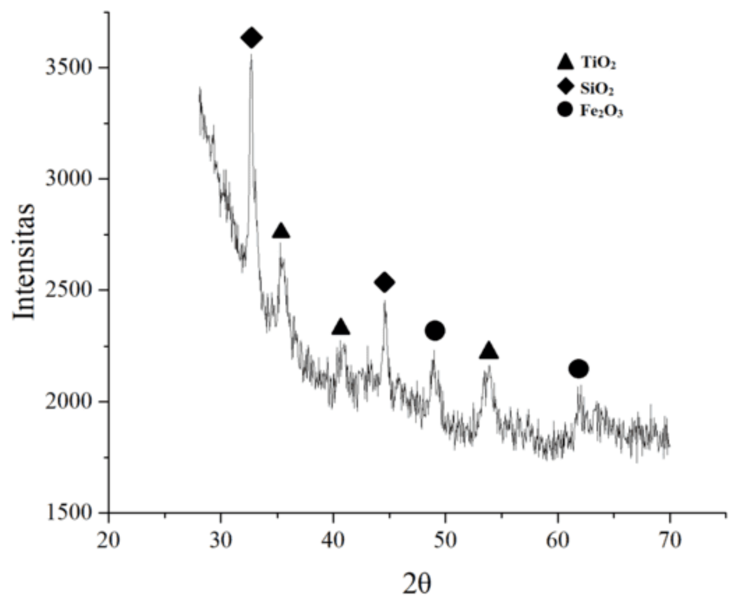


Figure 3. XRD Diffractogram of TiO_2 Extraction Results of Varying 12 M HCl Concentrations

Figure 3 shows the highest peak obtained including 32.6593°; 35.4074°; 40.7569°; 44.6239°; 49.0498°; 53.6867°; and 61.8818° which correspond to the crystal orientations in the hkl planes (002), (101), (111), (400), ($20\bar{4}$), (211), (214), respectively. The dominant compounds formed are SiO_2 with standard COD data 96-412-4048, Fe_2O_3 with standard COD data 96-900-9783, and TiO_2 with standard COD data 96-153-2820. The results of the diffractogram analysis are in accordance with the XRF results, namely that compounds with the largest concentrations were obtained, including SiO_2, Fe_2O_3, and TiO_2.

The diffractogram above shows that the TiO_2 obtained in this study is not a single compound, this is due to the presence of diffraction peaks which indicate the presence of other oxide compounds besides TiO_2. Therefore, the diffraction patterns of SiO_2 and Fe_2O_3 compounds as compounds with high content were identified in this study. Sumari *et al* (2023) reported their research on silica extraction from Bajul Mati beach sand, East Java, and the diffraction peaks obtained showed that a cristobalite crystal phase was formed. Xu *and* Deng (2018) reported their research on the extraction of Fe_2O_3 from iron ore tailings.

After comparing the SiO_2 diffractogram from the research of Sumari *et al* (2023) and Fe_2O_3 from the research of Xu *and* Deng (2018), it shows that the peak 40.7569° which is the peak of TiO_2 corresponds to the peak of Fe_2O_3 in the research of Xu *and* Deng (2018), while the peak of 53.6867° corresponds to the peak of SiO_2 in the research of Sumari *et al* (2023), therefore peaks of 40.7569° and 53.6867° are not peaks of pure TiO_2.

Apart from that, in this research there is rutile TiO_2 which is in accordance with the COD 96-900-4148 standard data so that the TiO_2 particles obtained have a rutile crystal phase. The calcination temperature influences the TiO_2 crystal phase that will be formed, where at a temperature of 100 ℃-400 ℃ a transformation of the amorphous phase to anatase crystal phase occurs, then at a temperature of 400 ℃-600 ℃ a transformation of anatase crystal phase to rutile occurs and the rutile crystal phase is stable at this temperature. 600 ℃-900 ℃ (Mahshid *et al*., 2007). The TiO_2 obtained has lattice parameters a = b = 4.6260 Å and c = 2.9810 Å with a crystallographic angle $\alpha = \beta = \gamma = 90°$, thus showing that the rutile crystal phase formed has a tetragonal crystal structure. The size of the TiO_2 particles is calculated using the Scherrer equation, the value used is the maximum peak value of the TiO_2 particles, namely at the peak of 35.4074°. Quantitative calculations are carried out using the Scherrer equation, namely:

$$D = \frac{0.9\lambda}{\cos\theta.\beta} = \frac{0.9\ (0.15406)}{\cos\frac{35.4074}{2}\ \text{x}\ 0.0042913} = 33.92\ \text{nm} \quad (5)$$

The information β is the Full Width Half Maximum (FWHM), λ is the X-ray wavelength, and θ is the angle of the Bragg plane diffraction peak. The particle size obtained shows that TiO_2 has a nanoscale size, because material with a size of 1-100 nm is classified as a nanoparticle (Rao *et al*., 2002), so that the TiO_2 particles obtained are nanoparticles.

Nanoparticles play an important role in technological development because they have a large surface area and volume so they are more reactive than bulk materials. TiO_2 with nanoparticle size has several advantages including optical properties, photocatalytic properties, good chemical stability, dielectric properties, biocompatibility, semiconductor and low cytotoxicity (Akakuru *et al*., 2020). Thus, TiO_2 nanoparticles are widely applied as semiconductor materials in Dye Sensitized Solar Cell (DSSC) photoelectrode components (Komalasari *et al*., 2014), as a hybrid supercapacitor (Kim *et al*., 2013), as an agent attenuating ultraviolet (UV) radiation in sunscreen (Ko *et al*., 2012), and so forth.

CONCLUSION

Research on TiO_2 extraction from Tembakak Beach iron sand has been carried out using the hydrometallurgical method. The results of this research can be concluded that after preparation, the Tembakak Beach iron sand contains 58.294% Fe elements; Si 18.525%; Ti 8.775%; Al 6.785%; Ca 3.885%; K 1.624%, as well as other minor elements with a content <0.5%. The results of TiO_2 extraction in variations of HCl 7 M were 15.033%%, HCl 9 M was 16.367%, and HCl 12 M was 17.421%, so the effect of HCl concentration on TiO_2 extraction shows that the more concentrated the HCl concentration, the greater the TiO_2 content obtained. The results of characterization using XRD show that TiO_2 has a rutile crystal phase with a tetragonal crystal structure. The TiO_2 particle size was obtained at 33.92 nm, which is a nanoparticle. TiO_2 nanoparticles have great potential in applications in the industrial world, including the cosmetics industry as an agent for attenuating ultraviolet (UV) radiation in sunscreen, in addition as a semiconductor material in Dye Sensitized Solar photoelectrode components Cell (DSSC), as a hybrid supercapacitor, and so on.

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