Eksplorium
Volume 47 No. 1, May 2026: 39–48

p-ISSN 0854-1418
e-ISSN 2503-426X

Study on TiO_2 Extraction from Ilmenite Sand in Ketapang Regency Through Various NaOH Molar Ratios

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Article received: 30 October 2025, revised: 2 May 2026, accepted: 31 May 2026
DOI: 10.55981/eksplorium.2026.13619

ABSTRACT

Titanium Dioxide (TiO_2) is a high-value inorganic material widely used in various industrial sectors, including paints and coatings, cosmetics, and ceramics, as well as in photocatalytic energy and environmental applications. One of the main sources of TiO_2 is the mineral ilmenite ($FeTiO_3$), which is abundant in iron sand deposits in Ketapang Regency, West Kalimantan. However, its use has thus far been limited to raw mining activities without downstream processing, resulting in relatively low economic value added. This study aims to extract TiO_2 from Ketapang ilmenite sand through the alkali fusion method using NaOH with molar ratios of 1:1, 1:2, 1:3, and 1:4. The fusion product (frit) was subsequently washed with distilled water, leached with 37% HCl solution, and precipitated using NH_4OH to obtain Ti precipitates, which were then characterized using X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF), and Scanning Electron Microscopy coupled with Energy Dispersive X-ray (SEM-EDX). Initial characterization revealed that ilmenite sand contained 43.78% TiO_2, 27.84% Fe_2O_3, and 15.34% ZrO_2 along with minor amounts of other associated minerals. XRD analysis of the extracted product confirmed the presence of diffraction peaks matching the standard TiO_2 pattern, with relative intensity reaching 100% at molar ratios of 1:1 to 1:4. The dominant crystalline phase identified was anatase. Morphological observations by SEM revealed TiO_2 particles with nano–submicron crystal structures and rough surfaces characteristic of anatase, while EDX analysis confirmed titanium as the dominant surface element, with an atomic composition up to 90.21%.

Keywords: Ilmenite, titanium dioxide, alkali fusion, leaching, precipitation

INTRODUCTION

Titanium dioxide (TiO_2) is an inorganic material with high strategic and applicative value. It is widely used across various industrial sectors, including paints and coatings, cosmetics, ceramics, sensors, solar cells, and photocatalysis in energy and environmental applications. The global demand for TiO_2 continues to increase in line with technological advancements and industrial needs, making the availability of raw materials and efficient synthesis methods highly important [1], [2].

One of the main sources of TiO_2 is ilmenite ($FeTiO_3$), which is abundant in iron sand deposits. Indonesia, particularly Ketapang Regency in West Kalimantan, possesses considerable potential of iron sand mineral resources with significant ilmenite content [3], [4]. However, the use of ilmenite in this region has been limited to raw mining, with no downstream processing. This condition results in low economic value added, despite the substantial potential to develop downstream titanium-based industries to support the country's self-sufficiency in strategic materials [5], [6].

Initial characterization of Ketapang ilmenite sand shows 43.78% TiO_2, 27.84% Fe_2O_3, and 15.34% ZrO_2, along with minor associated minerals. This composition indicates that ilmenite sand has great potential as an alternative raw material for TiO_2 production. Therefore, an effective processing method is required to extract TiO_2 from ilmenite sand, thereby increasing its value and supporting downstream mineral processing.

Several studies have been conducted to enhance the utilization of ilmenite for TiO_2 production. Alkali fusion is an extractive metallurgical technique that decomposes refractory mineral structures into more reactive phases, thereby facilitating subsequent extraction processes [7]. Hydrometallurgical routes involving acid leaching are widely employed for the extraction and purification of valuable metals because they selectively dissolve target components under controlled conditions [8]. The extraction of TiO_2 from Bangka Belitung ilmenite via microwave-assisted roasting followed by hydrochloric acid leaching yielded highly crystalline anatase TiO_2 [9]. Variations in NaOH concentration significantly influenced the crystallinity and morphology of TiO_2 precursor nanobelts, indicating the important role of alkaline treatment in determining the final microstructure of titanium oxide products [10]. Furthermore, the production of TiO_2 pigment from ilmenite requires oxidation, reduction, aeration, and leaching stages to remove iron-bearing phases, underscoring the importance of preliminary purification to increase TiO_2 purity prior to further processing [11].

Based on these studies, the alkali fusion method, followed by leaching and precipitation, is a promising approach for producing high-quality TiO_2 [12]. The combination of characterization techniques, such as X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF), and Scanning Electron Microscopy with Energy-Dispersive X-ray (SEM-EDX), is essential for identifying the crystalline phases, chemical composition, and morphology of the TiO_2 products obtained. Therefore, this research is expected to provide scientific contributions and open opportunities for the development of downstream processing of Ketapang ilmenite sand as a raw material for titanium-based industries in Indonesia.

METHODOLOGY

This study focused on extracting TiO_2 from ilmenite sand collected from Ketapang, West Kalimantan. The process began by preparing the sample, drying, and grinding the ilmenite sand to achieve a fine particle size (Figure 1). The core extraction involved alkali fusion, in which ilmenite was mixed with sodium hydroxide (NaOH) at molar ratios of 1:1, 1:2, 1:3, and 1:4. The mixture was heated to 700 °C for 1 hour, forming a solid fusion product, known as frit (Figure 2). The frit was then washed with distilled water to eliminate any unreacted alkali residues. Next, the washed solid underwent leaching with a 37% HCl solution at 90 °C for 1 to 2 hours. This step aimed to dissolve impurities, mainly iron compounds, thus enabling the separation of titanium into the solution.

The leaching filtrate was treated with NH_4OH to precipitate a white titanium hydroxide. The precipitate was then filtered, washed to neutral pH, dried, and calcined to produce TiO_2 powder. The resulting TiO_2 was characterized using various analytical methods. The XRD identified the crystalline phases and crystal structures, and the XRF determined the elements present and their concentrations. Additionally, SEM-EDX analysis was used to examine the surface morphology and elemental composition of the TiO_2 product.

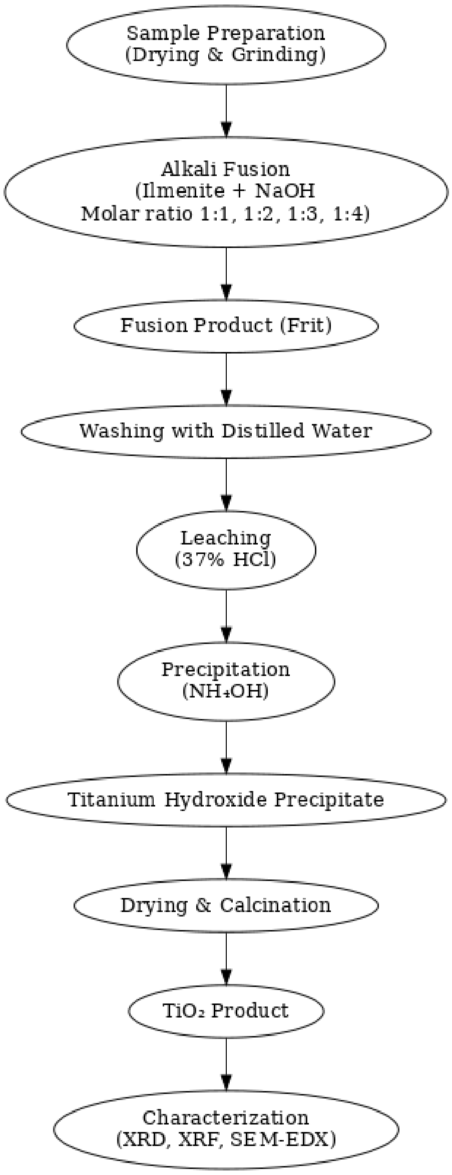

Figure 1. TiO_2 Extraction Process Flow Diagram

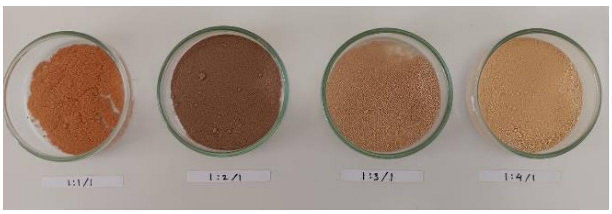

Figure 2. TiO_2 frit products

RESULTS AND DISCUSSION

The research began with XRD and XRF characterization of ilmenite sand samples from Ketapang Regency. This characterization was conducted to determine the crystalline phases and chemical composition of the raw material before alkali fusion, acid leaching, and precipitation.

The XRD result (Table 1) and the diffraction pattern (Figure 3) show dominant peaks at 2θ = 26.97° (d = 3.306 Å), indicating the presence of the main phases $FeTiO_3$

(ilmenite) and TiO_2. The sharp diffraction peaks suggest that the sample has a high degree of crystallinity.

The characterization of the TiO_2 product aims to identify its crystallographic properties, surface morphology, and microstructure. The results of this characterization are summarized below, including the XRD peak list and its correlation with the sample diffractogram, as shown in Figure 4 [13].

Table 1. XRD analysis results on ilmenite sand

2θ (°)	**d-spacing (Å)**	**Relative Intensity (%)**	**Phase Identification**
26.969	3.306	100.0	TiO_2 / $FeTiO_3$
32.815	2.729	16.3	$FeTiO_3$
35.612	2.521	23.9	$FeTiO_3$
41.149	2.194	12.9	TiO_2
53.309	1.715	17.3	TiO_2 / $FeTiO_3$
63.046	1.474	11.7	TiO_2
70.653	1.333	9.7	TiO_2 / $FeTiO_3$

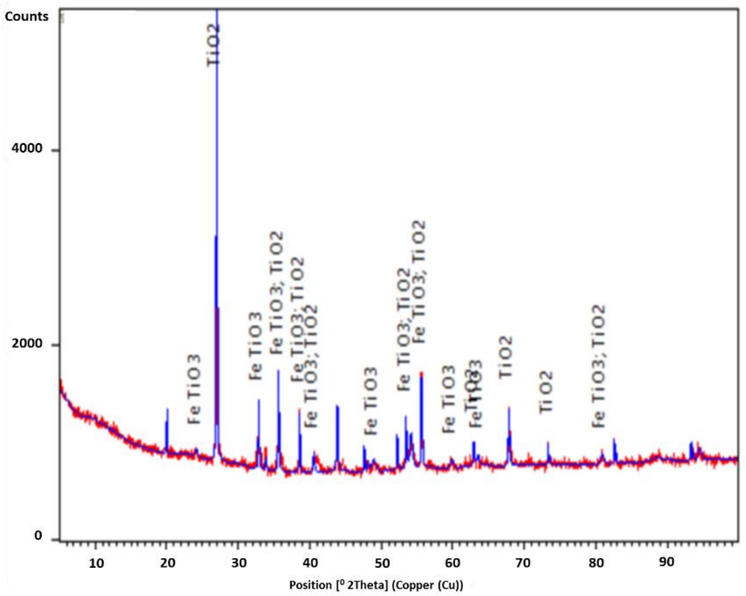

Figure 3. XRD analysis diffractogram on ilmenite sand

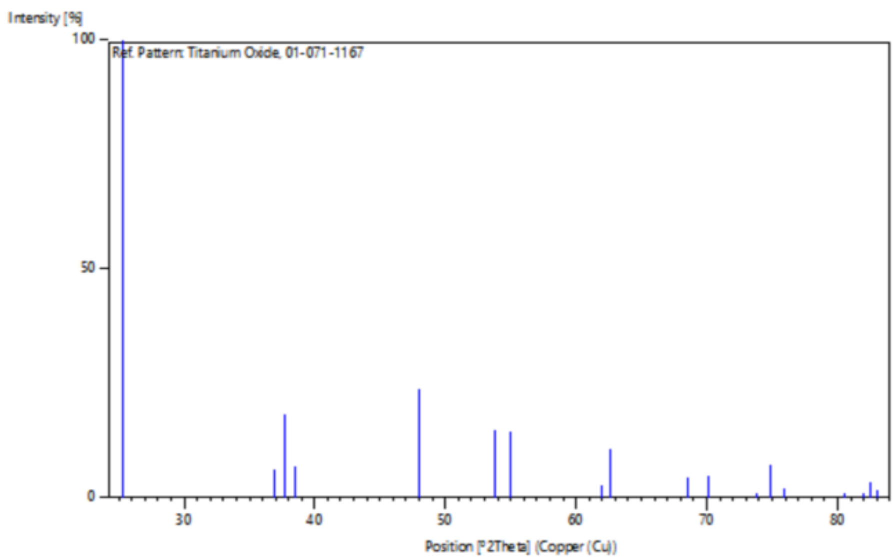

Figure 4. X-ray diffraction pattern of TiO_2 product

The XRD results indicate that variations in the ilmenite to NaOH molar ratio significantly affect the formation and crystallinity of the TiO_2 phase (Table 2). In Figure 5, corresponding to a 1:1; 1:2; 1:3 and 1:4 molar ratios, the XRD diffractogram demonstrates a strong agreement between the experimental pattern (red line) and the standard reference pattern (blue lines) of anatase TiO_2. The dominant peak at $2\theta \approx 25°$ corresponds to the (101) plane, confirming the presence of anatase phase. Additional peaks such as (004), (200), (105), and (211) are also observed and match well with the reference data.

Table 2. Peak list XRD pattern of anatase mineral

No	h	k	l	d (Å)	2θ (°)	I (%)
1	1	0	1	3.52143	25.271	100
2	1	0	3	2.43542	36.877	6.1
3	0	0	4	2.38425	37.698	18.4
4	1	1	2	2.33588	38.509	7.1
5	2	0	0	1.89460	47.980	23.7
6	1	0	5	1.70372	53.761	14.7
7	2	1	1	1.66845	54.992	14.4
8	2	1	3	1.49539	62.010	2.5
9	2	0	4	1.48331	62.572	10.5
10	1	1	6	1.36705	68.593	4.5
11	2	2	0	1.33968	70.198	4.8
12	1	0	7	1.28207	73.858	0.4
13	2	1	5	1.26684	74.897	7.2
14	3	0	1	1.25213	75.932	2.0
15	0	0	8	1.19212	80.505	0.3
16	3	0	3	1.17381	82.027	0.5
17	2	2	4	1.16794	82.529	3.5
18	3	1	2	1.16212	83.034	1.5

Meanwhile, the (101) peak intensity is the highest among all samples, indicating that the TiO_2 crystal structure is most perfectly formed under these conditions. This suggests that the 1:4 molar ratio is optimal for TiO_2 crystal growth, yielding the highest crystallinity, likely due to a more complete alkali fusion or calcination reaction at this composition.

Sharper, more intense diffraction peaks are associated with larger crystallites, while broader peaks indicate smaller crystallites or reduced crystallinity [14], [15]. Therefore, the product extracted from ilmenite in this study contains not only the TiO_2 phase but also exhibits relatively high crystal quality, particularly at the 1:4 molar ratio, which yields the most dominant and well-ordered anatase crystals.

The XRF analysis of the ilmenite sand sample was carried out to determine the major and minor oxide composition in the sample, as presented in the following Table 3. The ilmenite sand sample is dominated by the major oxides titanium dioxide (TiO_2) at 43.78% and iron oxide (Fe_2O_3) at 27.84%. In addition to these main oxides, zirconium dioxide (ZrO_2) was also detected at 15.34%, indicating the presence of important accessory minerals such as zircon ($ZrSiO_4$) or zirconia (ZrO_2). In addition to the dominant components, the analysis results revealed several minor oxides, including SiO_2 (4.42%), Al_2O_3 (0.72%), MgO (1.12%), and CaO (0.88%), which serve as impurities. Transition metal oxides, including Cr2O3 (1.34%), MnO (1.05%), and V2O5 (0.26%), were also detected in relatively small amounts. Nevertheless, the presence of these minor oxides is not highly significant to the main composition, but still provides additional information regarding the mineral heterogeneity in the sample.

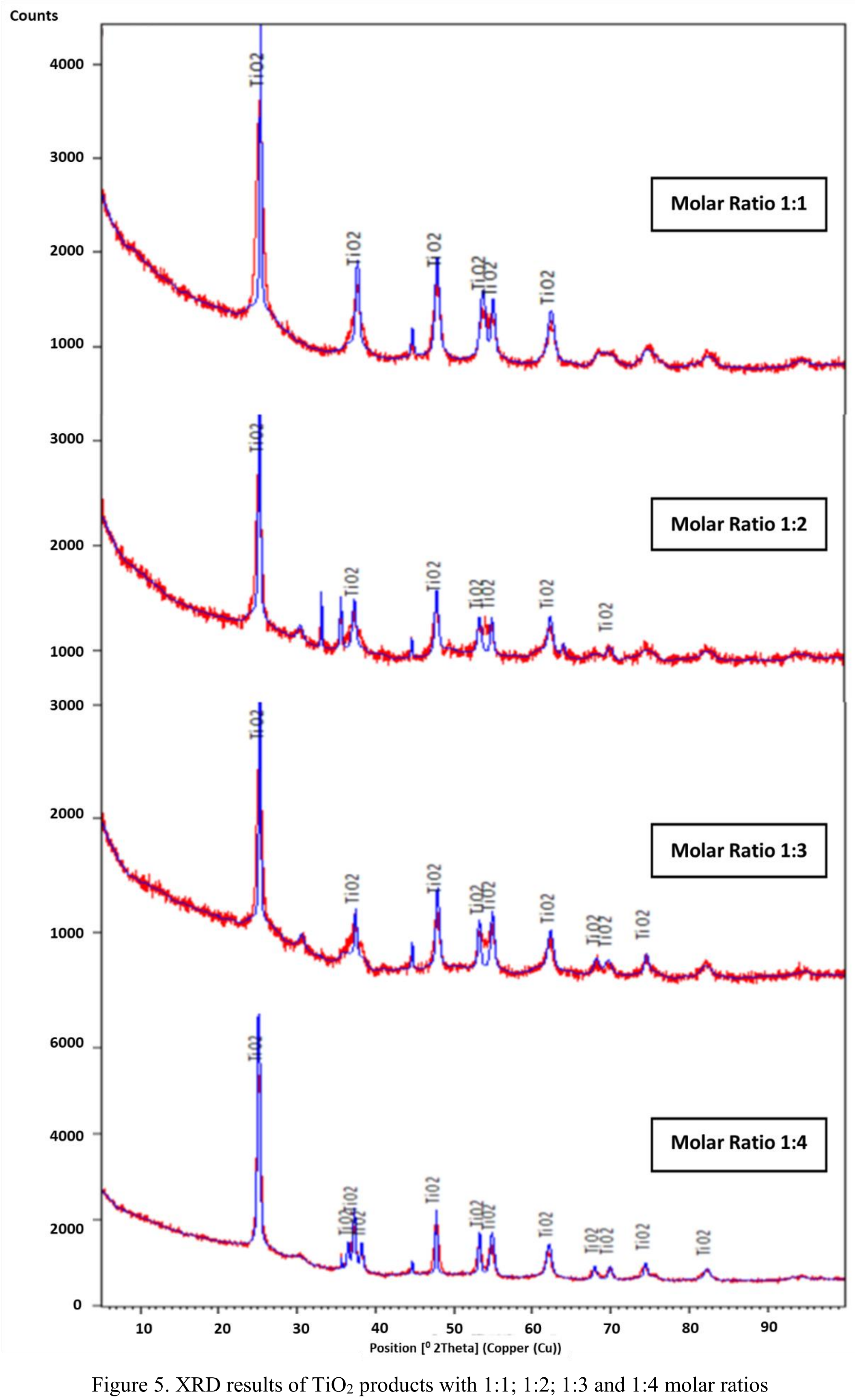

Figure 5. XRD results of TiO_2 products with 1:1; 1:2; 1:3 and 1:4 molar ratios

p-ISSN 0854-1418
e-ISSN 2503-426X

Table 3. Chemical composition analysis of ilmenite sand using XRF

Element/ Oxide	Concentration (%)	Explanation
TiO_2	43,78	Main content (rutile/ilmenite phase)
Fe_2O_3	27,84	Main content (ilmenite phase)
ZrO_2	15,34	Significant associated minerals (zircon/zirconia)
SiO_2	4,42	Main impurity (silica)
Al_2O_3	0,73	Minor impurities
MgO	1,12	Minor impurities
CaO	0,88	Minor impurities
Cr_2O_3	1,34	Minor transition oxides
MnO	1,05	Minor transition oxides
V_2O_5	0,26	Minor transition oxides
P_2O_5	1,44	Minor follow-up
K_2O	0,10	Minor follow-up

The synthesis of titanium dioxide (TiO_2) was confirmed by SEM-EDX, which showed that the extracted product had an agglomerated grain structure composed of micro- and submicron-sized particles, mostly TiO_2, with no notable impurities (Figure 6). Anatase was found to be the predominant crystalline phase. SEM morphological examinations revealed TiO_2 particles with rough surface textures and nano–submicron crystal structures, which are characteristic of the anatase phase.

The EDX analysis was conducted to determine the elemental composition of the extracted sample. Based on the EDX spectrum (Figure 7), two dominant elements were detected: Titanium (Ti) and Oxygen (O). The oxygen peak appeared at approximately 0.5 keV, while the titanium peaks were observed at high intensity in the 4.5–4.9 keV range. Quantitative results indicated that the titanium content was 90.205 at.% (96.5 wt.%), and the oxygen content was 9.795 at.% (3.5 wt.%). The EDX investigation verified titanium as the predominant surface element, with an atomic composition of 90.21% (Table 4).

This composition indicates that the sample is predominantly titanium dioxide (TiO_2), consistent with the stoichiometric Ti/O ratio. The absence of significant peaks from other elements indicates a high degree of purity, although minor elements may be present at trace levels below the EDX detection limit. These results are consistent with the XRD analysis, which also confirmed the formation of the crystalline TiO_2 phase.

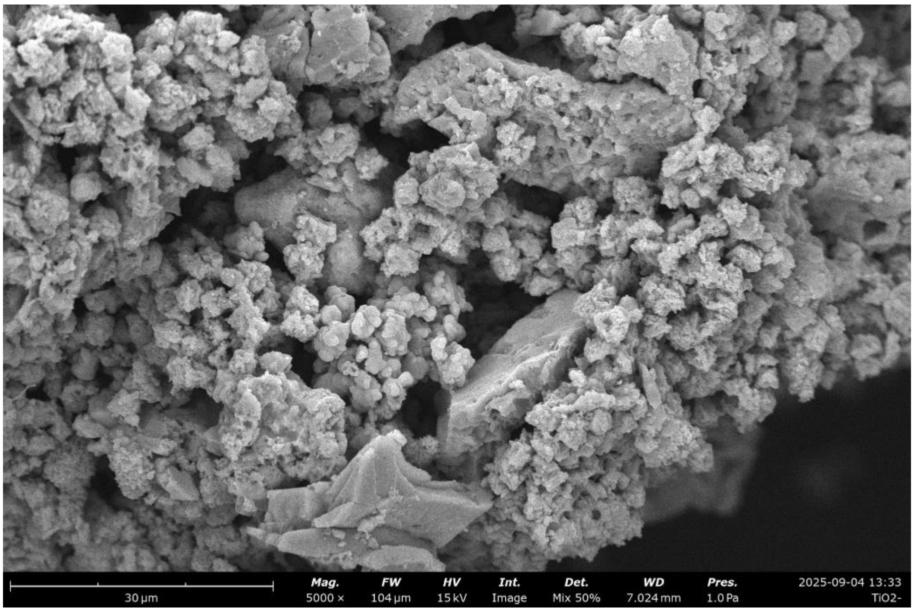

Figure 6. Morphology of TiO_2 products from SEM-EDX analysis using 5000 x magnification

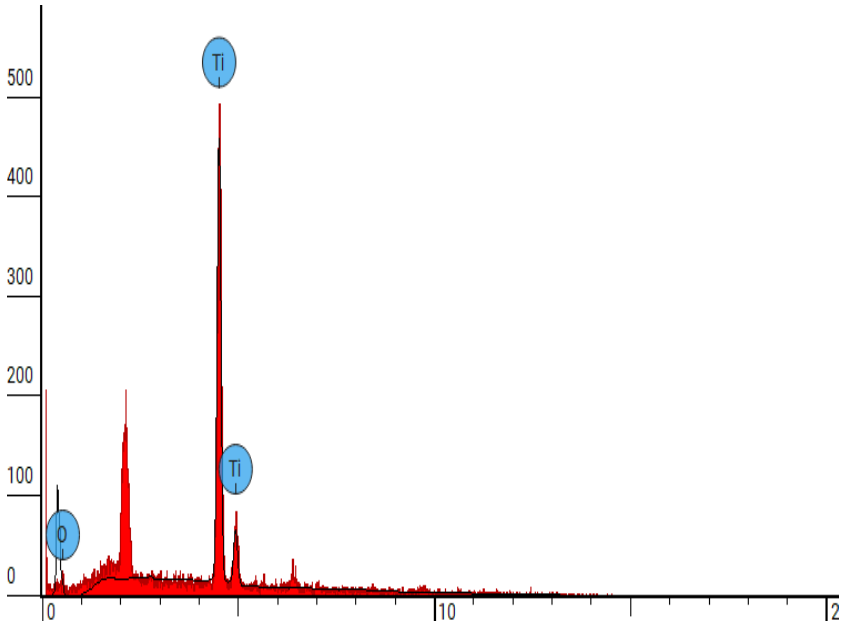

Figure 7. EDX spectrum showing two dominant elements, Ti and O

Table 4. Results of the EDX analysis

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	9.795	3.500
22	Ti	Titanium	90.205	96.500

CONCLUSION

This study demonstrated the extraction of titanium dioxide (TiO_2) from Ketapang ilmenite sand using the alkali fusion method. Initial characterization of the raw material confirmed 43.78% TiO_2, 27.84% Fe_2O_3, and 15.34% ZrO_2 as the major components. The alkali fusion process with NaOH, followed by washing, leaching, and precipitation, effectively produced TiO_2 with improved phase purity. XRD analysis confirmed the formation of TiO_2, with diffraction peaks matching the standard reference pattern. The highest relative intensities were observed at molar ratios of 1:1 and 1:4. The dominant crystalline phase in the product was anatase. SEM observations revealed nano–submicron TiO_2 particles with rough surfaces typical of anatase morphology, while EDX confirmed titanium as the predominant surface element, with an atomic composition of 90.21%. These findings indicate that Ketapang ilmenite sand is a promising local resource for TiO_2 production, offering potential for downstream processing and higher economic value in industrial applications.

ACKNOWLEDGMENT

The authors sincerely thank BIMA KEMENDIKTISAINTEK and P3KM POLITAP for their valuable support in ensuring the smooth implementation of this research, particularly through financial and institutional support.

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