Indones. J. Chem., 2024, 24 (5), 1563 - 1572

Short Communication:

Preparation of Titanium Dioxide (TiO_2) from Waste of Polymetallic Ore Processing via Sulfurization Treatment

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Received: October 21, 2023
Accepted: April 26, 2024

DOI: 10.22146/ijc.89992

Abstract: *Various experimental conditions were applied to extract titanium oxide (TiO_2) from the waste of polymetallic ore. X-ray diffraction (XRD) analysis confirms that the waste of polymetallic ore contains including various minerals such as almandine ($(Fe,Mg,Ca)_3Al_2Si_3O_{12}$), brownmillerite ($FeAlO_3(CaO)_2$), quartz ($SiO_2$), and magnetite ($Fe_3O_4$). Using H_2SO_4 with a concentration of 93% in the S/L mass ratio of 1:1.5 at 140 °C is suggested as an optimum reaction condition. Its hydrolysis subsequently leads to the formation of the titanyl sulfate ($TiOSO_4$), and relatively pure TiO_2 is obtained through precipitation and calcination at 600 °C. The observed band gap value of 3.2 eV for the obtained TiO_2 corresponds to the typical band gap value of anatase-type TiO_2. We calculated the crystallite size of extracted anatase-type TiO_2 according to the Debye-Scherrer equation it was determined to be 96.35 nm. Fully reflecting X-ray fluorescence (XRF) determined that the purity of extracted TiO_2 is 93.18%. This report presents a newly developed process that enables the production of relatively high-purity TiO_2 (93.18%) from the waste of polymetallic ore (TiO_2 5.39%) by a simple sulfurization process.*

Keywords: *anatase-titanium dioxide; minerals leaching; low-grade ore; X-ray diffraction (XRD) analysis; secondary resource*

■ INTRODUCTION

As high-grade ore reserves continue to diminish, the utilization of low-grade ores becomes increasingly urgent. However, it poses a significant challenge as low-grade ores typically contain lower concentrations of desired minerals, making their processing more complex and economically challenging. Nonetheless, advancements in mining technologies and extraction techniques make it possible to overcome these challenges and extract valuable minerals from low-grade ores. Researchers and industry professionals are actively exploring innovative methods and technologies to optimize the extraction and processing of low-grade ores [1], and ensure the continued supply of essential resources in a sustainable and economically viable manner.

Titanium dioxide (TiO_2), a non-toxic and environmentally friendly material [2], has garnered considerable interest due to its wide range of applications in various fields [3]. The unique properties and versatility of TiO_2 make it an industrially important material [4]. While the global focus has traditionally centered on extracting and utilizing 5% of titanium raw materials for titanium metal production [5], it is crucial to recognize the significance of the remaining 95% for the extraction and production of TiO_2 [6-7]. This approach identifies the potential of using secondary sources to obtain valuable titanium products. Hydrometallurgy and pyrometallurgy are commonly employed methods for extracting valuable minerals from ores, TiO_2 is produced through both the sulphate and chloride processes to manufacture the two primary titanium products [7-9].

The sulfurization process involves converting the minerals into the highly soluble M_xSO_4 form using concentrated sulfuric acid, thereby enriching the low-grade minerals. The production of TiO_2 from raw materials, including natural, uses main extraction methods, such as alkaline treatment, hydrothermal, and acid leaching [10-11]. Sulfuric acid leaching is advantageous due to its low corrosiveness, cost-effectiveness, and low energy consumption, enabling the extraction of titanium from low-grade sources.

TiO_2, being non-toxic, finds wide applications such as a white pigment in paints and coatings as photocatalytic activity in the degradation of persistent organic pollutants and water splitting for lithium-sulfur batteries [10], degradation of persistent organic pollutants (POP) [11-13]. TiO_2 has three polymorph phases: anatase, rutile, and brookite. The anatase phase of TiO_2 exhibits higher photocatalytic activity compared to another phase. The photocatalytic activity of anatase [14-15] can be involved in the band gap energy, which is the gap between the valence and conduction band, and bulk properties of the surface, which may play a role in photon absorption. The band gap of anatase ≈3.2 eV is larger than rutile and brookite ≈3.0 eV, primarily active for UV light. If anatase contains impurities like Fe_2O_3 and SiO_2 [16] it contributes to enhancing the activity of TiO_2, especially in photocatalysis.

Given the increasing demand for TiO_2 and the need to explore alternative sources, researchers in Mongolia have focused on extracting TiO_2 from low-grade ores. Highlights the potential for producing titanium products from secondary sources across various fields [17]. As part of ongoing research on extracting final products from various sources, a particular objective has been demonstrating the feasibility of extracting TiO_2 from polymetallic ore. This work presents an efficient technology method for extracting TiO_2 from the waste of polymetallic ore with low titanium content. Indeed, the waste of polymetallic ore often contains various elements, including silica, iron, aluminum, and others. These elements can potentially serve as valuable raw materials for extracting high-purity trace metal oxides during the sulfurization process used in preparing TiO_2. It supports waste utilization and sustainable resource extraction by exploring high-purity trace metal oxide extraction from polymetallic ore. The sulfurization process is the simple, economical, and most frequently used method of preparing TiO_2.

■ EXPERIMENTAL SECTION

Materials

The raw material used for the experiment was received from the "Anand" ore deposit located in the Orkhontuul sum, Selenge province, approximately 250 km north of Ulaanbaatar, Mongolia. The sample belongs to a heavy fraction of the polymetallic ore after magnetic separation and is released, such as waste of polymetallic ore. The sample analysis demonstrated that the particle size was distributed within the range of 100–200 μm. Other reagents for the experiment were an analytical grade hydrochloric acid (HCl, 33%), and analytical grade sulfuric acid (H_2SO_4, 98% purity from Union, China).

Instrumentation

The chemical composition of the sample was studied using the full reflecting X-ray fluorescence (XRF) method using Axios Max, PW4400/40 device (Japan). The mineral compositions of the sample were studied using X-ray diffraction (XRD) analysis with an "Enraf Nonius Delft" XRD instrument. CuKα radiation (λ = 0.154056 nm), a voltage of 40 kV, and a current of 300 mA were used, and the recorded range was from 15° to 80° with a step size of 0.02°. The mineral morphology of the TiO_2 structure was examined using a Hitachi TM-1000 scanning electron microscope with energy-dispersive

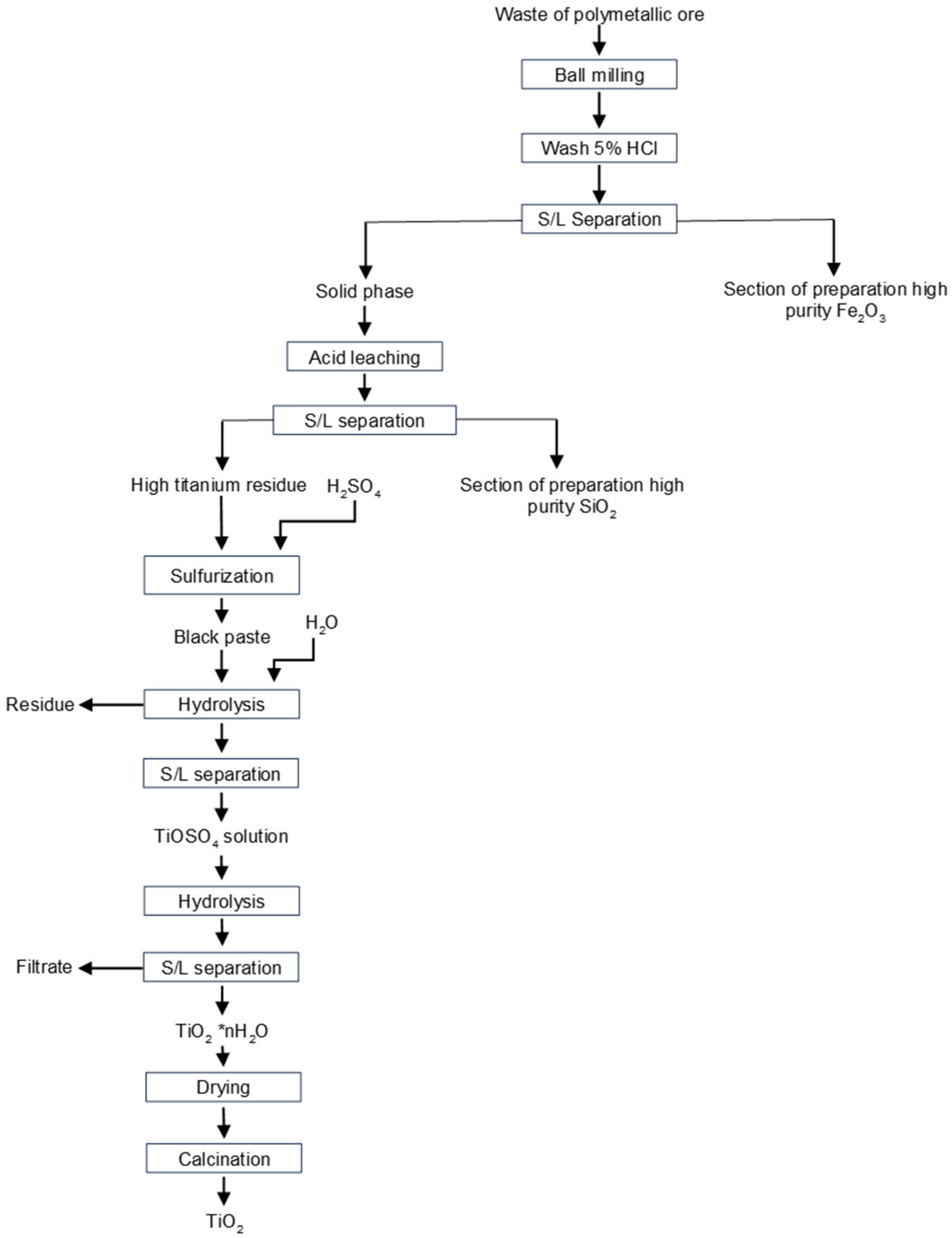


Fig 1. Experimental procedure of preparation TiO_2 from the waste of polymetallic ore

X-ray spectroscopy (SEM-EDS). Furthermore, the band gap of TiO_2 was measured using UV-2250 spectroscopy.

Procedure

Based on preliminary laboratory tests, a schematic flow sheet for the extraction of TiO_2 from the polymetallic ore has been proposed. The main process includes milling, washing acid, concentrated H_2SO_4 decomposition, water leaching, and the separation of companion elements with EDTA during hydrolysis. The experimental procedure is schemed in Fig. 1.

Sulfurization *process*: Before the sulfurization process, a 500 g sample was milled with the ball milling (100–200 μm) and 5% diluted HCl washing in which Mg, Fe, and Ca components could be removed [18-19]. The sulfurization process was conducted as batch experiments using 200 mL ceramic dishes. The 20 g sample was mixed with 20 mL of concentrated sulfuric acid to optimize various parameters while maintaining control over the weight of the process.

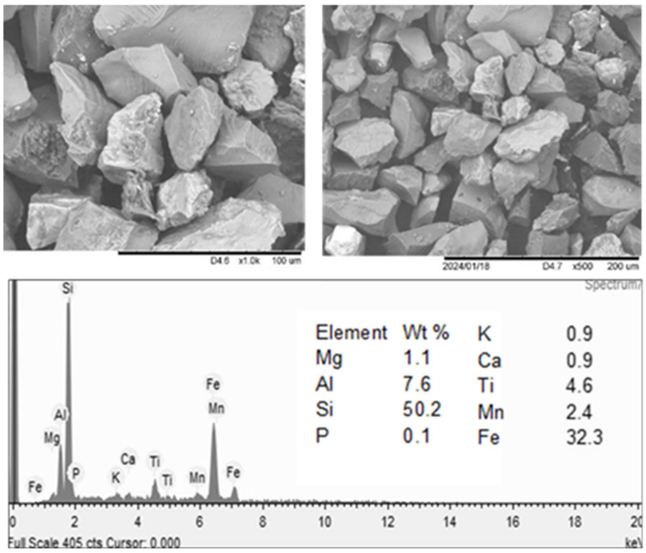
After the sulfurization process, the solid and liquid phases are separated through filtration. The solid part was washed to a continuous 30 mL-(20 wt.%) H_2SO_4 leaching at 90 °C for 1.5 h for the aluminum component's removal into the liquid phase [20]. During washing, metal ions such as Ca, Mg, Al, and Fe are removed to increase titanium components in the sample, and titanium is an enriched product. The decomposition process took place by heating a specific amount of the hydrolyzed high titanium solid residue with concentrated H_2SO_4 to 140 °C. Following this, 300 mL EDTA solution (0.1 mol/L) was added to the titanium sulfate solution for the hydrolysis process. The solution was heated to 90 °C for 90 min. The precipitate was filtered, washed with 300 mL of deionized water, and air-dried at room temperature for 24 h. Finally, the resulting powder was calcined at 600 °C for 2 h to synthesize anatase-type TiO_2.

■ RESULTS AND DISCUSSION

Characterization of the Waste of Polymetallic Ore

The chemical composition of the waste of polymetallic ore sample is measured by XRF and SEM-EDS analysis is used to observe the grains' morphology and are shown in Table 1 and Fig. 2. The SEM-EDS results indicate that the content of the TiO_2 is around 4.6 wt.%, while it is 5.39% for the XRF-measured sample. Generally, the SEM-EDS and XRF results closely match each other.

The XRD pattern of the raw waste of polymetallic ore is shown in Fig. 3. XRD analysis confirms that the waste of polymetallic ore contains various minerals, including almandine ($(Fe,Mg,Ca)_3Al_2Si_3O_{12}$), brownmillerite ($FeAlO_3(CaO)_2$), quartz ($SiO_2$), magnetite ($Fe_3O_4$). XRD diagrams of the untreated sample exhibited the peaks at 2θ = 26.70°, 38.35°, 48.70°, and 57.75° for quartz, almandine at 2θ°= 12.25°, 20.90°, and magnetite was observed at 2θ°= 30.25°, 31.05°, 35.25°, 36.60°, 38.35° and the broad peaks of brownmillerite appeared at 2θ = 34.85°.



Element	Wt %		
Mg	1.1	K	0.9
Al	7.6	Ca	0.9
Si	50.2	Ti	4.6
P	0.1	Mn	2.4
		Fe	32.3

Fig 2. SEM images and EDX spectrum of waste of polymetallic ore

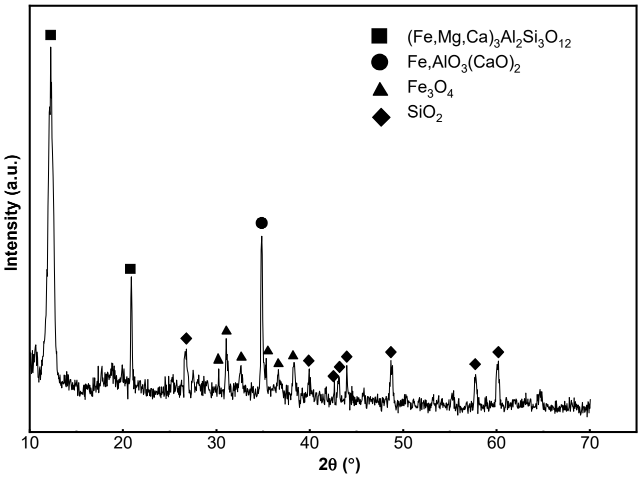


Fig 3. XRD pattern of the waste of polymetallic ore

Table 1. The chemical composition of the waste of polymetallic ore sample

Oxides (wt.%)	Fe_2O_3	Al_2O_3	SiO_2	TiO_2	Mn_3O_4	MgO	CaO	Others
Sample	27.85	16.47	41.11	5.39	1.28	1.74	0.79	5.37

Result of Experiments for Defining an Optimum Condition for the Sulfurization Process

Effect of H_2SO_4 concentration

Several experiments for the preparation of TiO_2 were performed using H_2SO_4. It was determined that the acid concentrations, solid-liquid ratio, and reaction time are crucial factors in the sulfurization process. The sulfurization process takes advantage of the removal capability of metal sulfate to remove impurities and facilitates the removal of trace elements. In the study, the sulfurization process underweight (sample) change control was investigated by examining various concentrations of H_2SO_4, including 81, 84, 87, 90, 93, and 96%.

Fig. 4 shows the change in sample weight treated with the various acid concentrations. The weighted sample reached maximum values in H_2SO_4 concentration of 93.00% (93% > 90% > 96% > 87% > 84% > 81%), increasing the weighted sample by 53.72%. The sulfurization process can be explained by the following reaction equation (Eq. (1–6)):

$$Me_2SiO_{4(s)} + 2H_2SO_{4(aq)} \rightarrow 2MeSO_{4(aq)} + H_2O + H_2SiO_{4(aq)} \quad (1)$$

$$\left[Fe^{2+}\,Fe_2^{\;3+}\right]O_{4(s)} + 2H^+_{(aq)} \rightarrow \left[Fe_2^{\;3+}\right]O_3 + Fe^{2+}_{(aq)} + H_2O \quad (2)$$

$$3\left[Fe^{2+}\,Ti\right]O_{3(s)} + 6H^+_{(aq)} \rightarrow 3\left[Ti\right]O_2 + 3Fe^{2+}_{(aq)} + 3H_2O \quad (3)$$

$$Fe_2O_{3(s)} + 3H_2SO_{4(aq)} \rightarrow Fe_2\left(SO_4\right)_{3(s)} + 3H_2O \quad (4)$$

$$MnO_{2(sol)} + 4Fe^{2+}_{(aq)} + 4H^+_{(aq)} \rightarrow 4Fe^{3+}_{(aq)} + Mn^{2+}_{(aq)} + 2H_2O \quad (5)$$

$$Fe_3O_{4(s)} + FeSO_{4(s)} \rightarrow 2Fe_2O_{3(s)} + SO_{2(g)} \quad (6)$$

However, as the concentration of H_2SO_4 further increased to 96.0%, the sample weight decreased to 50.5%. This decrease in weight change rate can be attributed to the strong electrolytic property of H_2SO_4 and the reduction of proton activity. Using an excessively high acid concentration (96.0%) will not improve the sulfurization process. The results are in agreement with the description of all of the research [21]. An acid concentration of 93% is suggested as an optimum reaction condition.

Fig. 5 displays the correlation between the weight alteration of the sample and the reaction time. It indicates that the weight of the sample grows as the reaction time varies from 2 to 10 h. However, if the reaction time increases too much, it can negatively impact the reaction process of weight alteration. Therefore, a reaction time of 6 h (6 h > 8 h > 10 h > 4 h > 2 h) would be the most effective sulfurization process period for maximizing the weight alteration of the sample.

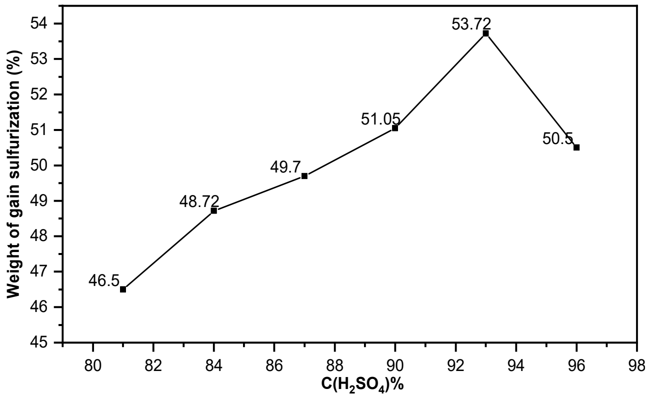


Fig 4. Effect of H_2SO_4 concentration on weight gain

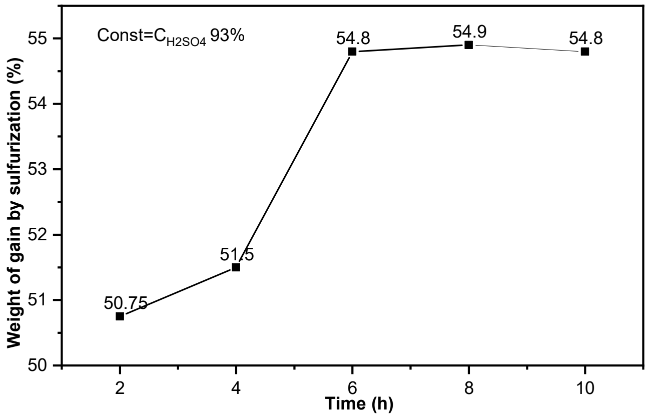


Fig 5. Effect of reaction time on weight gain by sulfurization

Effect of liquid-solid ratio in reaction

Sulfurization experiments were performed in H_2SO_4 (93%) with a S/L mass ratio varying from 1 to 3 at 140 °C for 6 h. Waste of polymetallic ore (sample)-to-H_2SO_4 mass ratios of 1:1.0, 1:1.5, 1:2.0, 1:2.5, and 1:3.0 was chosen to investigate the influence of the S/L ratio in the sulfurization process. An effect of varying the mass ratio is shown in Fig. 6. The results in Fig. 6 show that the sulfurization process is improved with the increase of maximum value at an S/L mass ratio of 1:1.5 and an increase of weight sample of 70%. However, extending the S/L mass ratio to 1:2.5 decreases the weight sample slightly. When the polymetallic ore sample waste was reacted with H_2SO_4 acid with a concentration of 93%, and the S/L mass ratio of 1:1.5 at 140 °C was suggested as

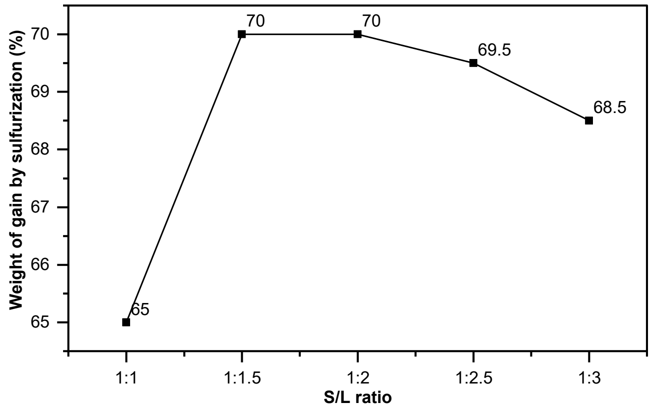


Fig 6. Effect of solid-to-liquid ratio in sulfurization process

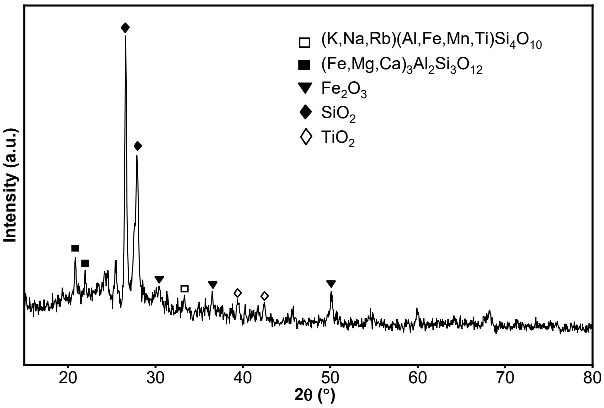


Fig 7. XRD pattern of the product after the sulfurization process

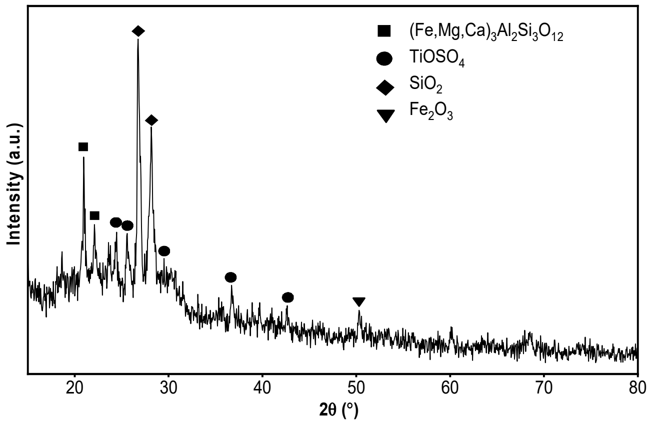


Fig 8. XRD pattern of titanium-bearing minerals product

an optimum reaction condition. These results of an optimum reaction condition were used for the following investigation.

Result of an Effect of the Sulfurization Process

The characterization of the product obtained after the sulfurization process was performed using XRD analysis. The results of the XRD analyses are shown in Fig. 7. The main minerals identified were almandine, exhibiting peaks at 2θ = 20.80°, 21.90°, quartz that was detected at 2θ = 26.60°, 27.90°, hematite at 2θ = 30.50°, 36.50° and 50.10°. Additionally, muscovite ($(K,Na,Rb)(Al,Fe,Mn,Ti)Si_4O_{10}$) was detected at 2θ = 33.30° and rutile at 2θ = 39.40° and 42.50°. This is because the H^+ ionized from H_2SO_4 plays a major role in the sulfuric process. At the same time, a new mineral phase, particularly muscovite at 2θ = 33.30°, was detected. The generation of the new phase indicated that the titanium-bearing minerals, including muscovite and rutile, had been converted into water-soluble sulfates after the sulfurization process. The mineral muscovite, known for its titanium content, was consistently utilized in experimental studies.

Notably, titanium-bearing minerals, including muscovite and rutile, exhibited enrichment following treatment with H_2SO_4 as the main constituent for extracting TiO_2 from the waste of polymetallic ore. The XRD pattern of sulfurization processed high titanium, resulting in the formation of $TiOSO_4$, is shown in Fig. 8. The effect of water leaching on the extraction of titanium, the water-soluble titanium sulfates solution, was utilized for hydrolysis, resulting in thick and inhomogeneous precipitation. Hydrolysis resulted in the Eq. (7–9) such as:

$$TiO_{2(s)} + H_2SO_{4(aq)} \rightarrow TiOSO_{4(s)} + H_2O \quad (7)$$

$$TiOSO_{4(s)} + 2H_2O \rightarrow TiO(OH)_{2(s)} + H_2SO_{4(aq)} \quad (8)$$

$$TiO(OH)_{2(s)} \rightarrow TiO_{2(s)} + H_2O \quad (9)$$

The process was repeated several times to achieve a high titanium extraction. The XRD analysis results indicate the presence of purified titanium-bearing minerals, resulting in the effectiveness of the sulfurization process.

Hydrolysis of Anatase TiO₂

Furthermore, using EDTA during hydrolysis, forming a complex with iron and other accompanying elements was feasible. EDTA plays a crucial role in enhancing the purity of TiO_2 particles. As a result of the hydrolysis process, it is observed that the stability constants of the complex compound between Fe^{3+} and EDTA are greater than (stability constant of Fe^{3+} 24.23 > TiO^{2+} 17.3) those of TiO^{2+} ions following the report of Zhang [21]. This is involved in the formation of Fe^{3+} ions that first form a complex and subsequently precipitate with titanium hydroxide. XRD patterns show that in Fig. 9, TiO_2 exhibits diffraction peaks at 2θ values of 25.35°, 37.80°, 48.10°, 54.00°, and 55.00°, confirming the presence of anatase phase in the prepared TiO_2.

The crystallite size determined using Scherrer's equation (Eq. (10)), reveals that the medium crystal size is 96.35 nm (Table 2).

$$D_c = \frac{k\lambda}{\beta \cos\theta} \tag{10}$$

The SEM-EDS image of TiO_2 (Fig. 10) reveals the presence of TiO_2 as the main crystalline phase with 91.7% in weight and agrees with the XRD pattern Fig. 9. Additionally, when TiO_2 was studied by XRF spectrometry after the heat treatment at 600 °C, the TiO_2 content was measured to be 93.18%, while the content of aluminum oxide was found to be about 0.01%, and the iron oxide content was measured at 6.06%. The results of XRF measurements are presented in Table 3. This confirmed

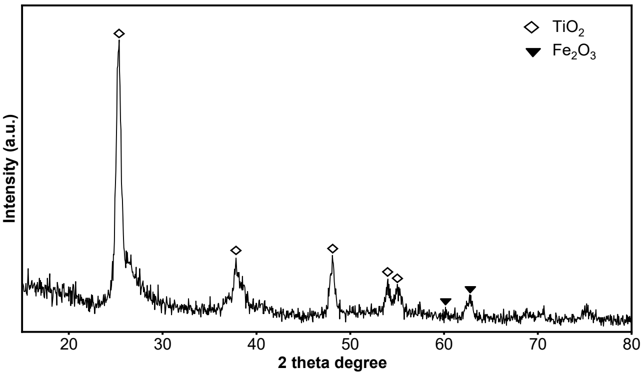


Fig 9. XRD pattern of obtained anatase type TiO_2

Table 2. The crystallite size of extracted anatase type TiO_2

2θ	FWHM (β)	D (nm)	D_{medium} (nm)
25.35	0.68	119.00	96.35
37.80	1.15	73.00	

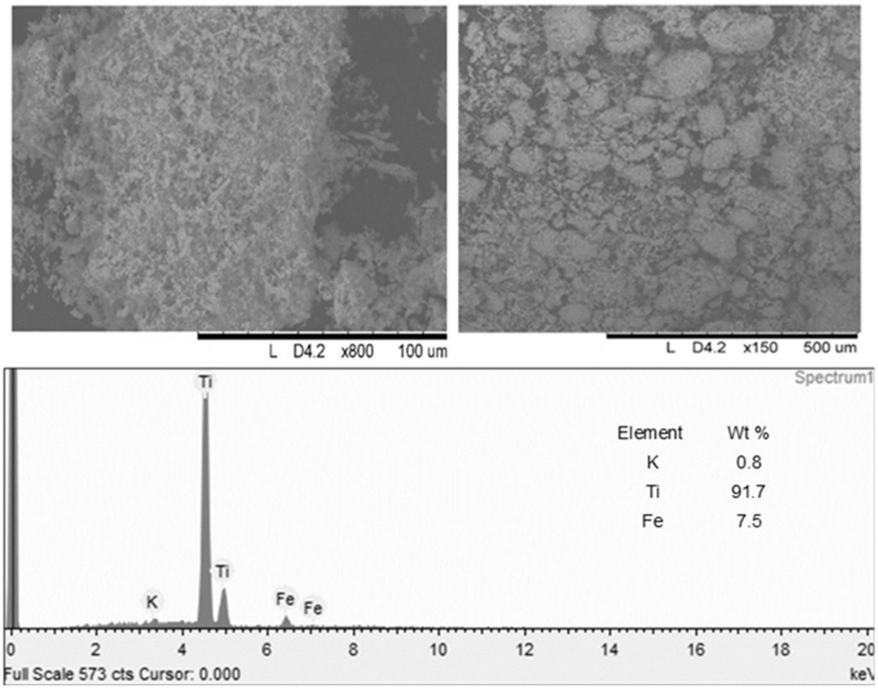


Fig 10. SEM-EDS images of anatase type TiO_2

Table 1. Chemical composition of prepared TiO_2/XRF

Oxides	Al_2O_3	BaO	CaO	Fe_2O_3	K_2O	MnO	P_2O_5	SiO_2	TiO_2	Total
Content (%)	0.01	0.05	0.01	6.06	0.02	0.01	0.34	0.08	93.18	99.76

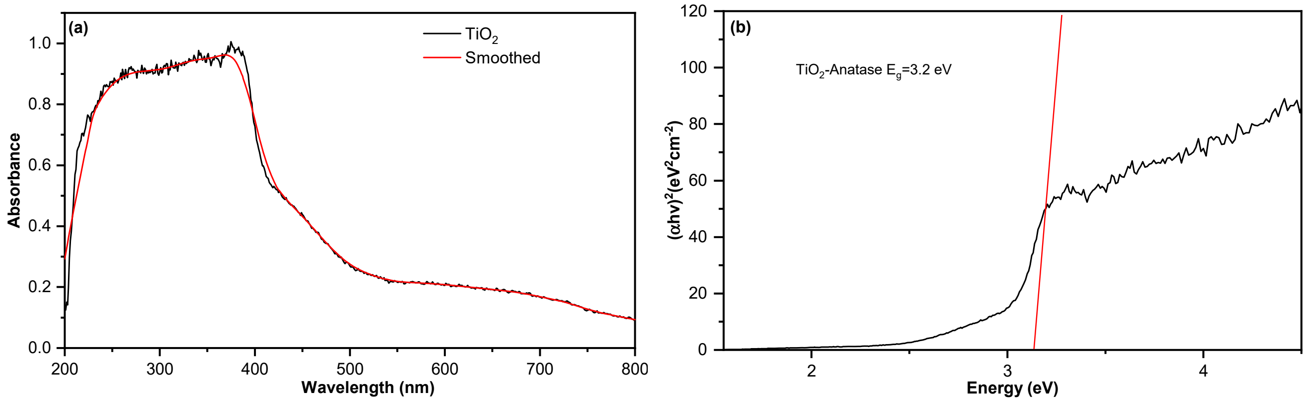


Fig 11. The (a) optical absorbance spectrum and (b) Tauc's plot for direct band gap energy of anatase type TiO_2

the potential for extracting relatively high-purity titanium from the waste of the polymetallic ore. The content of titanium oxide determined by the SEM-EDS analysis closely agrees with the result of XRF.

We also measured the band gap of anatase TiO_2, which was extracted from polymetallic ore waste. In Fig. 11, we can see that the sample's optical absorbance decreases throughout the UV region, which indicates that the extracted TiO_2 shows improved visible light absorption properties. The calculated value of 3.2 eV aligns with the typical band gap of anatase TiO_2. The optical band gap is an essential parameter that determines the semiconductor's ability to absorb photon energy [22] and generate photoexcited charge carriers. Although the anatase content wasn't as high as > 99%, the optical band gap was the same as that of pure TiO_2. The increase in visible absorbance was concomitant to the decrease in UV absorbance. The calculated value of the optical band gap indicates the potential of the extracted TiO_2 to be utilized as a photocatalyst. It has been confirmed that our previous research has reported on utilizing anatase TiO_2 extracted from polymetallic ore for the wastewater treatment process [23].

■ CONCLUSION

An important aspect of a circular economy is maximizing the use of waste of raw materials, including mineral processing wastes. In the present work, simple sulfurization can be used for the titanium extraction from a secondary resource. The optimal conditions for processing polymetallic ore-secondary resources and extracting titanium oxide through the sulfurization process were determined to be a solid-to-liquid phase ratio of 1:1.5, operating at 140 °C with 93% concentrated H_2SO_4 for 6 h. Under these conditions, the purification process resulted in 93.18% of TiO_2 chemical composition after calcination at 600 °C as the anatase polymorph mineral. The extracted TiO_2 crystal size was estimated using the Debye-Scherrer theoretical equation and was calculated to be 96.35 nm. The band gap value of 3.2 eV aligns with the typical band gap value of anatase TiO_2, indicating its potential for use as a photocatalyst.

■ ACKNOWLEDGMENTS

The research funding was provided by the "L2766-MON: Higher Education Reform" project financed by the Asian Development Bank and executed by the Ministry of Education, Culture, Science and Sports of Mongolia.

■ CONFLICT OF INTEREST

We have no conflicts of interest to disclose. All authors declare that they have no conflicts of interest.

■ AUTHOR CONTRIBUTIONS

Enkh-Uyanga Otgon-Uul: research analysis, methodology, formal analysis, writing - original draft, visualization, software, and data curation. Munkhtsetseg Baatar: software, data curation, writing - original draft, review, and editing, project administration. Ulziijargal Nanzad: wrote paper, designed, and analysed. Jargalsaikhan Lkhamsuren: validation, resources, review, and editing.

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