

Original article

## Technology for obtaining potassium titanate by ilmenite alkaline leaching with potassium hydroxide

### Tecnología de obtención de titanato de potasio mediante lixiviación alcalina de ilmenita con hidróxido de potasio

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#### Abstract

Among other uses, potassium titanate enhances materials' heat resistance in the ceramic industry, acts as a dielectric and component of piezoelectric devices in electronics, and facilitates the production of catalytic materials. Here, we used ilmenite concentrate (96%) from the Irshansky deposit in Ukraine's Zhytomyr region, characterized by a high  $\text{TiO}_2$  content, as a promising raw material for synthesizing potassium titanate. We aimed to synthesize potassium titanate ( $\text{K}_2\text{TiO}_3$ ) via alkaline leaching of ilmenite and to determine the optimal process conditions for achieving a high degree of titanium(IV) extraction. The methodology included alkaline leaching under controlled temperature, time, particle size, and  $\text{FeTiO}_3\text{:KOH}$  molar ratio, followed by phase and morphological characterization of the product obtained. The experimental results showed that the optimal leaching conditions include an ilmenite particle size of  $\leq 71 \mu\text{m}$ , a  $\text{FeTiO}_3\text{:KOH}$  molar ratio of 1:2, a temperature of 453 K, and a leaching duration of 3 h, providing a titanium(IV) extraction degree of 86.7%. Further increases in temperature or alkali content resulted in only a slight increase in extraction efficiency (up to 89.7%), which was deemed economically unfeasible. The high efficiency of the process was attributed to an increased reaction surface area, optimal thermal conditions, and favorable thermodynamic parameters. A technological scheme for the potential industrial production of potassium titanate is proposed based on these results, with prospects for reduced energy consumption, improved environmental safety, and minimal raw material losses.

**Keywords:** Mineral raw materials; Ilmenite; Leaching; Titanium-containing compounds; Potassium titanate; Titanium dioxide.

#### Resumen

Entre otros usos, el titanato de potasio mejora la resistencia térmica de los materiales en la industria cerámica, actúa como dieléctrico y componente de dispositivos piezoeléctricos en electrónica, y facilita la producción de materiales catalíticos. En este estudio, utilizamos concentrado de ilmenita (96%) del yacimiento de Irshansky en la región de Zhytomyr, Ucrania, caracterizado por un alto contenido de  $\text{TiO}_2$ , como materia prima prometedora para la síntesis de titanato de potasio. Nuestro objetivo fue sintetizar titanato de potasio ( $\text{K}_2\text{TiO}_3$ ) mediante la lixiviación alcalina de ilmenita y determinar las condiciones óptimas del proceso para lograr una alta extracción de titanio(IV). La metodología incluyó la lixiviación alcalina en condiciones controladas de temperatura, tiempo, tamaño de partícula y relación molar  $\text{FeTiO}_3\text{:KOH}$ , seguida de la caracterización morfológica y de fase del producto obtenido. Los resultados experimentales mostraron que las condiciones óptimas de lixiviación incluyen un tamaño de partícula de ilmenita  $\leq 71 \mu\text{m}$ , una relación molar  $\text{FeTiO}_3\text{:KOH}$  de 1:2, una temperatura de 453 K y una duración de lixiviación de 3 h, lo que resulta en un grado de extracción de titanio(IV) del 86,7 %. Un aumento adicional de la temperatura o del contenido de álcali solo produjo un ligero incremento en la eficiencia de extracción (hasta un 89,7%), lo que se consideró económicamente inviable. La gran eficiencia del proceso se atribuyó a una mayor superficie de reacción, condiciones térmicas óptimas y parámetros termodinámicos favorables. Con

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base en estos resultados, se propone un esquema tecnológico para la posible producción industrial de titanato de potasio, con perspectivas de reducción del consumo energético, mejora de la seguridad ambiental y mínimas pérdidas de materia prima.

**Palabras clave:** Materias primas minerales; Ilmenita; Lixiviación; Compuestos que contienen titanio; Titanato de potasio; Dióxido de titanio.

## Introduction

Ilmenite ore is a primary source of industrial titanium-containing materials used in titanium metallurgy, ceramic production, electronics, pigment manufacturing, catalytic systems, and advanced functional materials (Thambiliyagodage, 2021; Nguyen, 2018; Gázquez, 2014). The continuously growing global demand for titanium dioxide and reactive titanate compounds requires the development of efficient, economically viable, and environmentally sustainable technologies for processing titanium-bearing minerals.

Conventional methods for processing titanium-containing ores include pyrometallurgical and hydrometallurgical processes. Acid leaching is the most extensively studied approach (Mostafa, 2013; Jayaweera, 2011). However, these technologies are often characterized by high energy consumption, multistage processing schemes, significant reagent costs, equipment corrosion, and environmental concerns related to acidic waste streams. Additionally, ilmenites with high titanium(IV) oxide content may exhibit limited reactivity in acidic environments, reducing extraction efficiency. These limitations underscore the necessity of alternative processing strategies.

A competitive and increasingly researched alternative is the alkaline leaching of titanium from natural compounds. This approach usually involves the single-stage fusion of finely ground ilmenite with either sodium hydroxide or potassium hydroxide. Another method is the treatment of ilmenite with concentrated alkaline solutions at elevated temperatures. Although alkaline leaching has not yet been widely adopted in industry, it offers several advantages over acid processes. These advantages include rapid conversion of titanium into soluble titanate forms, high overall recovery rates, reduced toxicity and environmental impact, and no need for expensive, corrosion-resistant equipment (Fouda, 2010; Kordzadeh-Kermani, 2020; Subagja, 2013; Yousef, 2017; Parirenyatwa & Escudero-Castejon, 2016).

Numerous studies have demonstrated the effectiveness of alkaline treatment in converting ilmenite into reactive titanate phases. However, most of them focus on ilmenite samples from particular geographic regions (e.g., Shandong Province, China, Abu Ghalaga, Egypt, Rosetta black sands, Egypt, and Bangka Island, Indonesia), whose mineralogical compositions, impurity contents, and phase associations differ significantly from those of ilmenite concentrates in other deposits. This compositional variability affects dissolution behavior, reaction pathways, and product distribution during alkaline leaching. Thus, it is difficult to directly transfer optimized conditions from one deposit to another without adjustment.

In this context, ilmenite from Shandong Province, China, was treated with a concentrated KOH solution, resulting in the formation of potassium titanate ( $K_4Ti_3O_8$ ) in works (Liu, 2006; Liu, 2012). The authors investigated the effects of temperature, KOH concentration, reaction time, the alkali-to-ore mass ratio, and particle size. They identified optimal leaching conditions as follows: a particle size of 58–75  $\mu\text{m}$ ; an alkali-to-ilmenite ratio of 7:1; a KOH concentration of 84%; a temperature of 260°C, and a reaction time of 60 minutes. Under these conditions, 98.2% pure anatase  $TiO_2$  has been obtained after purification. It was also determined that the leaching rate increased with stirring speed up to 100 rpm and remained nearly constant between 500 and 1,200 rpm.

The production of nanoscale  $TiO_2$  particles via alkaline treatment was reported in Kordzadeh-Kermani (2020) using ilmenite concentrate from the Kahnooj Titanium Complex in Iran. The leaching process involved using 70% KOH at 220°C for three hours, followed by an acid treatment with 9 M HCl at 80 °C. Iron impurities were then removed

via EDTA complexation and ammonia precipitation. The particles were subsequently filtered and calcined at 500°C. Scanning electron microscopy (SEM) and field emission scanning electron microscopy (FESEM) analyses showed an average  $\text{TiO}_2$  particle size of approximately 70 nm in 90% of the particle population. The filtrate, which contains Fe-EDTA complexes and alkali metals, has been proposed for use in fertilizer production, demonstrating the potential for integrated waste utilization.

The influence of technological parameters on alkaline processing has also been investigated in the ilmenite from Abu Ghalaga, Egypt (Nayl, 2009). The optimal alkaline treatment conditions have been determined to be 70% KOH, 150 °C for 3 hours, a stirring speed of 375 rpm, and an alkali-to-ilmenite ratio of 5:1. Subsequent leaching with sulfuric or oxalic acids enabled the formation of anatase  $\text{TiO}_2$ . The reaction temperature and acid concentration were found to significantly influence titanium extraction efficiency.

Ilmenite concentrates from the Rosetta black sands in Egypt have been studied by Yousef (2017) and Amer (2002). The two-stage process involving alkaline treatment (70% NaOH or KOH at 393 K) followed by acid leaching produced  $\text{TiO}_2$ -containing materials suitable for uranium (VI) adsorption. Under optimized conditions, approximately 90% titanium extraction was achieved within the first 30 minutes (Amer, 2002). Mechanistic studies confirmed that the reaction kinetics followed the shrinking-core model, as evidenced by the calculated activation energies. Mechanical activation using a ball mill further enhanced titanium recovery.

Autoclave alkaline leaching of ilmenite from Bangka Island, Indonesia, was reported in Subagja (2013). Concentrated KOH (2–20 mol/L) at 473 K facilitated the formation of  $\text{K}_2\text{TiO}_3$ , as confirmed by XRD analysis. Particle size (40–200  $\mu\text{m}$ ) and temperature significantly affected phase transformation efficiency.

Alkaline fusion has also been applied to titanium-bearing slags. In Chen's study (2013), sodium hydroxide fusion of titanomagnetite slag from China at 500 °C with a NaOH/slag ratio of 1:1 resulted in 96.3% titanium conversion.  $\text{MgTi}_2\text{O}_5$  transformed into  $\text{Na}_2\text{TiO}_3$ , and iron oxidized to  $\text{Fe}_2\text{O}_3$ . Similar high conversion efficiencies (>98%) were achieved using molten NaOH-KOH mixtures for ilmenite slag from Shandong Province, China, with diffusion through the product layer controlling the reaction (activation energy: 43.1 kJ/mol). Regeneration efficiencies of 98% KOH and 86% NaOH were reported. The processing of titanium slag from Hebei Province, China, using NaOH at atmospheric pressure, yielded 95–98% titanium extraction and  $\text{TiO}_2$  purity up to 99.3%, with an activation energy of 40.8 kJ/mol and kinetics described by the shrinking-core model (Xue, 2009).

These studies provide valuable insights; however, variations in the mineralogical and chemical composition of ilmenite samples from different deposits often result in significant differences in reaction behavior, dissolution kinetics, and product formation. For instance, ilmenite from the Irshansk deposit, the focus of this study, exhibits a unique compositional profile, impurity distribution, and phase association compared to samples previously investigated. These differences can affect the effectiveness of alkaline leaching, reaction mechanisms, and optimal process conditions. Therefore, there is a scientific and practical need to develop an improved alkaline processing method tailored to the characteristics of Irshansk ilmenite.

Scientific literature indicates that alkaline processing of ilmenite and titanium-containing slags can achieve titanium conversion rates of 90–99% under optimized conditions. The efficiency of the process depends heavily on particle size, alkali concentration, temperature, reaction time, the mass ratio of reagents, and the hydrodynamic conditions. Despite the potential of alkaline methods, the comprehensive optimization of process parameters and the systematic study of thermodynamic and kinetic regularities are necessary for industrial implementation, particularly for ilmenite ores with specific compositional characteristics.

The purpose of this study was threefold: to synthesize potassium titanate ( $\text{K}_2\text{TiO}_3$ ) via alkaline leaching of ilmenite; to determine and optimize the synthesis conditions, and to substantiate the feasibility of developing an efficient technological scheme specifically adapted to producing potassium titanate from Irshansk ilmenite concentrate. Our research

objectives included evaluating how particle size, the  $\text{FeTiO}_3$ :KOH molar ratio, temperature, and the reaction time influenced titanium(IV) extraction efficiency and establishing the thermodynamic and kinetic characteristics of the alkaline leaching process for Irshansk ilmenite. We also aimed to develop an optimized technological approach for synthesizing potassium titanate with high titanium recovery, reduced energy consumption, improved environmental safety, and the ability to be implemented on an industrial scale, taking into account the unique properties of the Irshansk ore.

## Materials and methods

The following substances were used for the experimental study of the 96% ilmenite concentrate of the Irshansky deposit (TUU 14-10-009-97), Zhytomyr region: potassium hydroxide (p.a.), ammonium sulfate (p.a.), methylene blue (p.a.), sulfuric acid (p.a.), orthophosphoric acid (p.a.), 35% hydrogen peroxide solution (p.a.), and 96% ethyl alcohol (p.a.).

Ilmenite ore samples were examined by scanning electron microscopy with X-ray spectral elemental microanalysis on a JSM - 6490 LV electron microscope with energy dispersive (EDS) and wave dispersive (WDS) spectrometers (EnergyPlus, Oxford Instruments). Electron microscopic studies were also conducted on a Selmi scanning electron microscope at a 50-200  $\mu\text{m}$  resolution.

Diffraction patterns of ilmenite samples and synthesized potassium titanate were recorded using a DRON-3M X-ray diffractometer ( $K\alpha$  (Cu),  $\lambda = 0.1540$  nm). The XRD diffractograms obtained were analyzed using the MATCH3 software, and the average crystallite size was calculated using the Scherrer method.

For the synthesis of potassium titanate ( $\text{K}_2\text{TiO}_3$ ), we used the solid-phase method with an ilmenite fraction  $\leq 71$   $\mu\text{m}$ , and an ilmenite-to-alkali molar ratio of 1:2 at 453 K for 3 hours.

The main thermodynamic state functions of the system ( $\Delta G$ ,  $\Delta H$ ,  $\Delta S$ , etc.) were determined under standard conditions following electronic databases for the ilmenite alkaline leaching process with potassium hydroxide. We calculated the change in the Gibbs free energy of the leaching processes over the temperature range 468-698 K using the Tammann-Schwartzman method.

The standard heat to form potassium titanate was estimated with the empirical method of **Le Van** (1972):

$$\Delta H_{298}^0 = n \cdot A + m \cdot C + (2m)^2 + n^2, \quad (1)$$

where  $n$  and  $m$  are the number of anions ( $\text{TiO}_3^{2-}$ ) and cations ( $\text{K}^+$ ) in the molecule, and  $A$  and  $C$  are constants for the anions (795.45 kJ/mol) and cations (447.96 kJ/mol), respectively.

For the quantitative titanium(IV) content, the dye concentration before and after adsorption, and the photocatalytic studies we used a UV-1200 spectrophotometer.

## Results and discussion

### *Morphology of the original ilmenite sample*

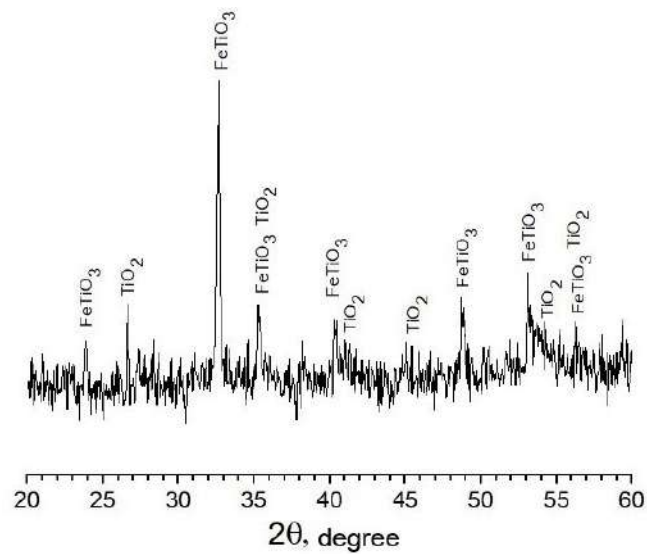
Using MATCH3 with the PCPDFWIN database on the diffractogram (**Figure 1**), we identified the main  $\text{FeTiO}_3$  reflexes at 23.80, 32.52, 35.25, 48.71, and 53.03°  $2\theta$  angles. We also detected the  $\text{TiO}_2$  phase at 26.52, 35.25, 41.20, 54.30, and 56.60°  $2\theta$  angles.

We calculated that in the ilmenite concentrate sample, the ilmenite content was 70±5%, the rutile was 20±5%, and the rest were the pyrite, pseudorutile, and impurity phases.

To calculate the average particle size of the ilmenite phase for the given  $2\theta$  angles, we used the Scherrer formula (**Drapak**, 2022):

$$D = \frac{K\lambda}{\beta \cos\theta}, \quad (2)$$

where  $D$  is the average particle size in nm,  $K$  is the Scherrer constant, with an average value of 0.92 for crystalline systems,  $\lambda$  is the wavelength of the X-ray radiation

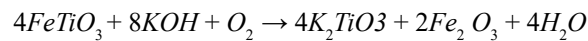


**Figure 1.** Ilmenite concentrate diffractogram

( $K\alpha$  line = 0.1540 nm),  $\beta$  is the angular half-width of the diffraction maximum at the characteristic Miller indices (hkl) for the crystal lattice, and  $\theta$  is the X-ray diffraction angle. We found that the XRD data corresponded to 26.62 nm.

**Thermodynamics of the ilmenite alkaline leaching process**

The ilmenite alkaline leaching processes with crystalline potassium hydroxide at a temperature of 453 K can be described by the reaction:



To establish the possibility of a spontaneous reaction, we calculated the main thermodynamic functions of the state of the system ( $\Delta G$ ,  $\Delta H$ ,  $\Delta S$ , etc.). The Le Van method shows that the average deviation between tabulated and calculated values is 3.87%.

To calculate the change in the Gibbs energy, we used the Tiomkin-Schwartzman method, as in the following formula:

$$\Delta G^0 = \Delta H_{298}^0 - T\Delta S_{298}^0 - T \int_{298}^T \frac{dT}{T^2} \int_{298}^T \Delta C_p dT \tag{3}$$

The temperature range for the ilmenite leaching process with the alkali used for the calculations was 468-698 K.

The last term on the right-hand side of equation (3) was estimated using the formula:

$$T \int_{298}^T \frac{dT}{T^2} \int_{298}^T \Delta C_p dT = \Delta aM_0 + \Delta bM_1 + \Delta cM_{-2},$$

where  $M_0$ ,  $M_1$ , and  $M_{-2}$  are coefficients depending on temperature, which in turn were calculated using formulas (5-7):

$$M_0 = \ln \frac{T}{298} + \frac{298}{T} - 1$$

$$M_1 = \frac{1}{2T} (T - 298)^2$$

$$M_{-2} = \frac{1}{2} \left( \frac{1}{298} - \frac{1}{T} \right)^2$$

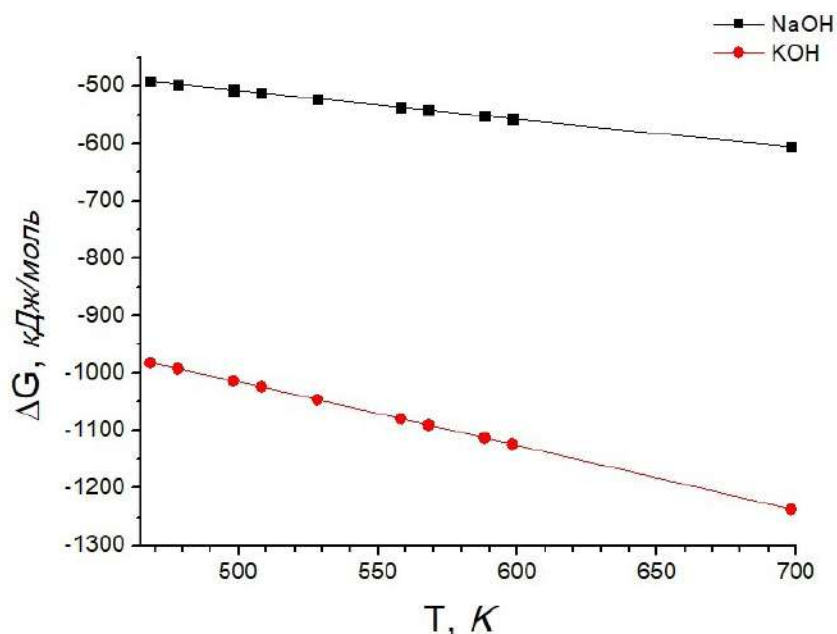
The data obtained are shown in **Table 1**.

**Figure 2** shows the dependence of the Gibbs energy values on temperature.

We confirmed that as the temperature increases, Gibbs energy values decrease linearly, which indicates the possibility of a spontaneous course of the leaching process.

**Table 1.** Thermodynamic characteristics of the leaching reaction of ilmenite with potassium hydroxide under standard conditions (298 K)

$\Delta H_{298}^0$ , kJ/mol	$\Delta S_{298}^0$ , J/mol·K	$\Delta G_{298}^0$ , kJ/mol	$\Delta a$	$\Delta b \cdot 10^{-3}$	$\Delta c' \cdot 10^{-5}$
-713.96	425.29	-840.76	656.38	480.99	-62.73

**Figure 2.** Gibbs energy values at different temperatures for the reactions of ilmenite leaching with potassium hydroxide**Determination of optimal conditions for the ilmenite alkaline leaching process**

To establish the ilmenite optimal particle size, we used ore fractions of  $\leq 71 \mu\text{m}$ , 71-140  $\mu\text{m}$ , 140-315  $\mu\text{m}$ , and 315-630  $\mu\text{m}$  particle sizes, which were separated by sieving and fused with potassium hydroxide in a molar ratio of ore to alkali of 1:2 for 3 hours at a temperature of 453 K.

To establish the optimal molar ratio between ilmenite and potassium hydroxide, we subjected a series of samples to alkaline melting with the following  $\text{FeTiO}_3$  molar ratios: KOH 1:2, 1:3, 1:4, 1:5, and 1:6, for 3 hours at 453 K and  $\leq 71 \mu\text{m}$  ilmenite fraction.

We analyzed the kinetic characteristics of the ilmenite leaching process ( $\leq 71 \mu\text{m}$  fraction) with potassium hydroxide in a molar ratio of 1:2 at 453 K during a 30 to 300 minute interval.

As for the dependence of the degree of extraction on temperature, the leaching process was done under the following conditions: ilmenite fraction:  $\leq 71 \mu\text{m}$ ; a 1:2  $\text{FeTiO}_3$ -KOH molar ratio, a 3-hour fusion time, and a temperature range of 453-573 K.

The Ti(IV) degree of extraction in the alkaline leaching process was calculated with the formula:

$$X = \frac{m(\text{Ti(IV)})_e}{m(\text{Ti(IV)})_p} \cdot 100\% \quad (8)$$

$m(\text{Ti(IV)})_e$  mass of extracted titanium (mg); mass of titanium in the initial melt (mg).

**Table 2** presents the experimental data for the dependence of the Ti(IV) degree of extraction on various factors of the leaching process.

**Table 2.** Dependence of the Ti(IV) degree of extraction on the size of the initial ilmenite particles

Effect of ilmenite particle size	
Particle size range, $\mu\text{m}$	Extraction rate (X), %
315-630	60.78
140-315	68.37
71-140	75.97
$\leq 71$	86.10
Effect of the components' molar ratio	
Molar ratio FeTiO <sub>3</sub> : KOH	Extraction rate (X), %
1:2	86.7
1:3	87.1
1:4	88.6
1:5	89.2
1:6	90.1
Effect of time on the leaching process	
Interaction time, min.	Extraction rate (X), %
30	49.69
60	53.48
90	60.31
120	66.76
150	76.62
180	86.13
240	86.10
300	86.10
Effect of temperature on the leaching process	
Temperature, K	Extraction rate (X), %
453	86.5
473	87.3
503	88.5
533	89.0
573	89.7

As shown in **Table 2**, the potassium titanate (K<sub>2</sub>TiO<sub>3</sub>) degree of extraction over 85% indicated that the optimal conditions for synthesis are the following:  $\leq 71 \mu\text{m}$  ilmenite fraction; ilmenite to alkali molar ratio 1:2, and heating at 453 K for 3 hours. The minimum particle size allowed for an optimization of the leaching process rate by increasing the contact area between the components.

The 90.1% titanium(IV) maximum degree of extraction was achieved at a FeTiO<sub>3</sub>:KOH molar ratio of 1:6; however, for a 1:2 molar ratio between components, this value was 86.7%. It should also be taken into account that when using a larger amount of alkali in the initial mixture, titanates with a different composition may be formed (Liu, 2006; Liu, 2012; Kordzadeh-Kermani, 2020; Nayl, 2009), and these were not the target product of the study. Also, an excess amount of alkali leads to its accumulation in the form of unused reagents, which further complicates the purification of the final product.

It has been established that during the first 30 minutes of interaction of the starting substances, the degree of extraction reaches almost 50%. The Ti(IV) maximum degree of extraction is 86.13%. Heating the melt for more than 3 hours at a given temperature does not increase the yield of the soluble form of Ti(IV), possibly due to the presence of impurities in the starting concentrate and the formation of iron (III) oxide, which prevents further penetration of alkali into the ilmenite reaction centers.

At 453 K, the Ti(IV) degree of extraction is 86.5%. Further increase in the ilmenite melt with alkali heating temperature led to a slight increase in the product yield (89.7%), which is insignificant compared to the energy costs of maintaining the high temperature of the process. Based on our results, we concluded that a heating temperature up to 453 K is technologically and economically justified, since the traditional processing temperature regime used in industrial titanium metallurgy is more than 623 K (Parirenyatwa, 2016; Mostafa, 2013).

### *Kinetics of the ilmenite alkaline leaching process*

To study the mechanism of the ilmenite alkaline leaching process with potassium hydroxide using a solid-phase method, we verified the kinetic dependences of the titanium(IV) degree of extraction on the leaching time and temperature with the following kinetic models for heterogeneous processes: pseudo-first and pseudo-second orders, Yander, Ginstling-Brownstein, Zhuravlev-Lesokhin-Tempelmann, Yerofeyev-Avraham, and compressed sphere (Pysarenko, 2023).

The pseudo-first-order kinetic equation is described by the formula:

$$k\tau = \ln X, \quad (9)$$

where  $k$  is the reaction rate constant,  $X$  is the degree of leaching of Ti(IV), and  $\tau$  is time.

The limiting pseudo-first-order stage is characterized by the rate of formation of active reactive centers on the ilmenite surface, which can form in any part of the crystal.

The pseudo-second-order kinetic equation in linear form takes the form:

$$k\tau = X \quad (10)$$

In this case, the rate of reaction center formation occurs on both the surfaces of ilmenite and alkali, and the limiting stage is the diffusion of alkali particles onto the surface of ilmenite with subsequent penetration into the volume of crystals.

The Yander equation describes the one-way diffusion process of alkali penetration into a spherical ilmenite grain covered with a layer of reaction products and takes the form:

$$k\tau = (1 - (1 - X)^{1/3})^2 \quad (11)$$

The equation describes heterogeneous solid-phase processes where the recovery degree of the final product does not exceed 40%.

The Ginstling-Brownstein kinetic equation also describes the one-way diffusion of alkali through the surface of ilmenite, whose active centers are located in two-dimensional planes. In its linear version, the equation takes the form:

$$k\tau = 1 - \frac{2}{3}X - (1 - X)^{2/3}. \quad (12)$$

The Zhuravlev-Lesokhin-Tempelmann equation describes solid-phase processes that occur at relatively low temperatures (200-300 °C):

$$k\tau = ((1 - X)^{-1/3} - 1)^2. \quad (13)$$

The reaction rate changes proportionally to the value  $(1 - X)$ .

The Yerofeyev-Avraham equation describes the growth rate of product crystals in one of the planes of the reactant (ilmenite) due to the multi-stage formation of reactive centers. This equation is described as:

$$k\tau = (-\ln(1 - X))^{1/n}, \quad (14)$$

where  $n$  depends on the geometric shape of the reagent particles and the mechanism of the heterogeneous process.

The compressed sphere model is described by the equation:

$$k\tau = 1 - (1 - X)^{1/3} \quad (15)$$

This squeezed sphere model indicates that at a certain point in time, active interaction centers are formed on the surface of ilmenite crystals due to surface defects in the crystal lattice and microcracks. Solid-phase interaction is limited by the diffusion of alkali from the surface of the nucleus into the volume of ilmenite particles through the interaction products formed, which leads to the annihilation of the initial nuclei.

Statistical processing of the kinetic dependences between leaching degree and isothermal interaction time using Fisher's dispersion relation at a significance level of 0.05 showed that the linearity hypothesis can be accepted for all equations; however, the correlation coefficients when approximating the experimentally established dependences  $X(\tau)$  differed significantly.

**Figure 3** shows the dependence of the titanium(IV) degree of extraction on the contact time between the substances at different temperatures (K).

As can be seen from the figure, the nature of the curve shapes at different temperatures is quite similar, and the difference in the values of the Ti(IV) degree of extraction is insignificant (these values at different temperatures belong to the same order and differ from each other by no more than 1.5%).

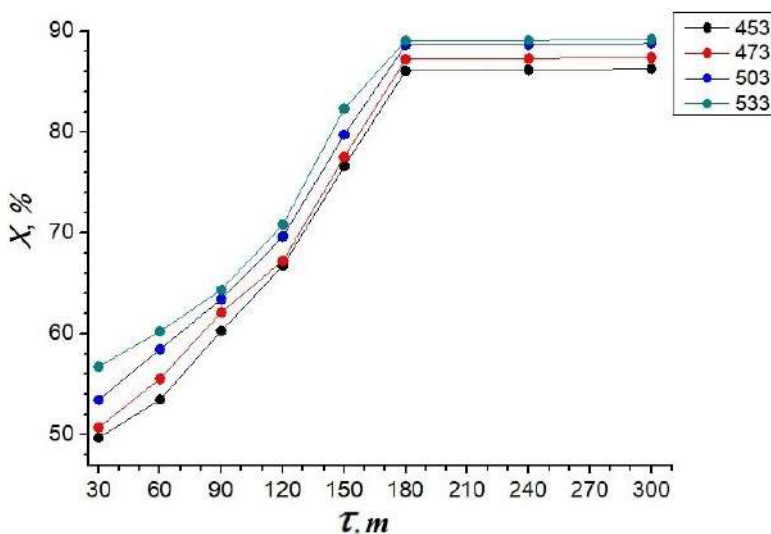
**Figure 4** shows the kinetic dependence of the ilmenite alkaline leaching process with potassium hydroxide according to the squeezed sphere model.

The figure shows how the process of ilmenite alkaline leaching with potassium hydroxide is most successfully described by the rate of interaction of the components according to the compressed sphere model with the limiting stage of the chemical reaction ( $R^2 = 0.9728$ ).

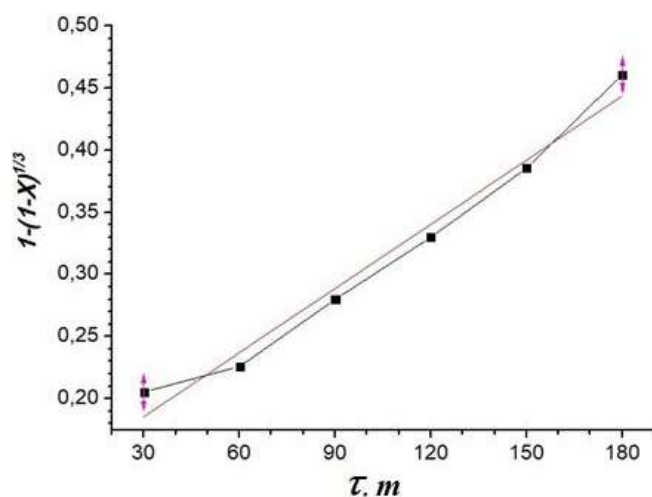
To calculate the activation energy of the ilmenite alkaline leaching process with potassium hydroxide, the dependence of the reaction rate constant on temperature was constructed using the Arrhenius equation (**Figure 5**).

We calculated that the apparent activation energy of alkaline leaching was 22 kJ/mol, which indicates a kinetic regime of the process with low activation energy.

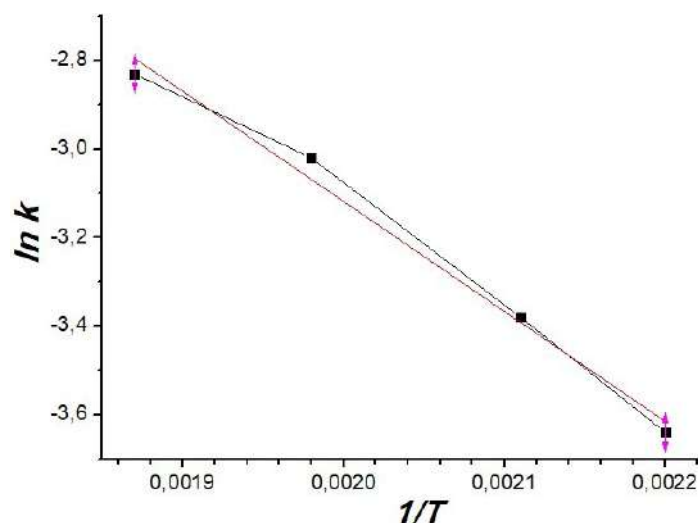
These calculations allowed us to develop the optimal technology for obtaining potassium titanate from the Irshan ilmenite deposit by alkaline leaching. The calculated apparent activation energy and the described process kinetic mechanism indicated that the



**Figure 3.** Dependence of the titanium(IV) degree of extraction on the contact time between substances at different temperatures (K)



**Figure 4.** Kinetics of ilmenite leaching by potassium hydroxide according to the compressed sphere model ( $T = 453 \text{ K}$ )



**Figure 5.** Graph of the rate constant of the ilmenite leaching process with potassium hydroxide as a function of temperature

specified conditions of alkaline leaching were practically optimal. In this case, the overall process speed, the increase in the speed of the chemical interaction limiting stages, and the energy costs for this process were taken into account.

#### ***Physicochemical studies of the obtained potassium titanate***

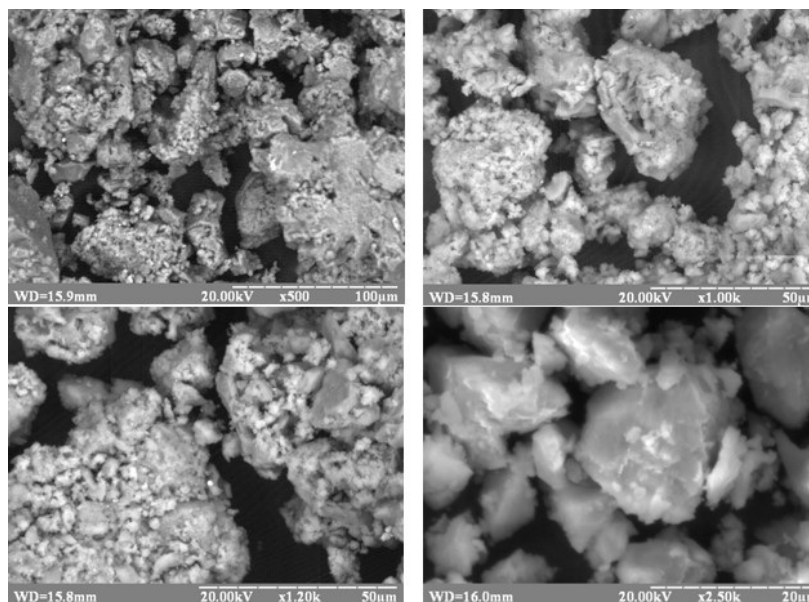
To purify potassium titanate from unreacted ilmenite residues and the formed iron (III) oxide, the melt was dissolved in distilled water, thoroughly mixed, and filtered by vacuum filtration using a water jet pump.

The resulting saturated alkaline solution of potassium titanate was precipitated with 96% ethanol taken in a fivefold excess. Potassium titanate was formed from the solution by coagulation and sedimentation of flesh-colored particles (**Figure 6**).

We examined the morphology of purified  $\text{K}_2\text{TiO}_3$  particles using SEM-microscopic studies (**Figure 7**). We found that the average particle size of potassium titanate was less than 200 nm, and the particles were prone to the formation of aggregates.



**Figure 6.** Appearance of potassium titanate



**Figure 7.** SEM photographs of purified  $K_2TiO_3$

In the SEM photographs, individual potassium titanate crystallites can be seen with a close-to-orthorhombic shape, as also confirmed by X-ray diffraction.

**Figure 8** shows the diffractogram of the obtained potassium titanate. We used the MATCH3 software to identify the structure of potassium titanate.

As can be seen from the figure, the intense reflexes in the range of  $29$  to  $45^\circ 2\theta$  angles indicated that  $K_2TiO_3$  was formed as a result of the leaching process, as revealed by the orthorhombic structure of crystals (Pysarenko, 2024).

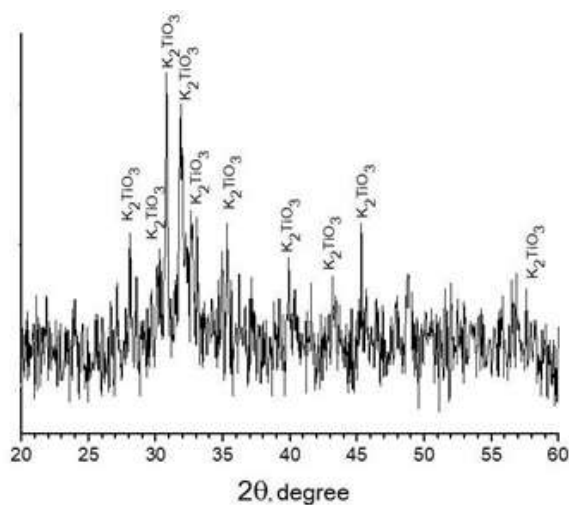
**Table 3** shows the main calculated data of the unit cell characteristics and the average particle size of potassium titanate.

We found that the average particle size of potassium titanate according to the X-ray diffraction data is 31.53 nm, which was also confirmed by electron microscopy data.

#### *Potassium titanate from ilmenite by alkaline leaching*

Based on the experimental data and the calculations of thermodynamic and kinetic parameters, we propose here a technological scheme for the production of potassium titanate, which minimizes harmful emissions into the environment and reduces the costs by recycling some substances in the process of producing potassium titanate (**Figure 9**).

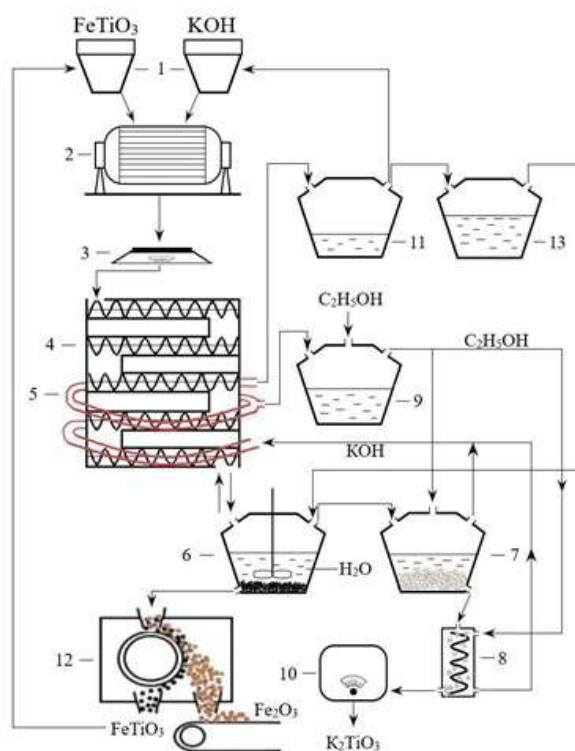
At the preparatory stage, if necessary, the raw material was dried and fed into the hoppers for the starting raw material (1). In the next stage, the raw material was ground in a ball mill (2). Next, the raw material was fed to the scales (3), where it was weighed in the ilmenite-potassium hydroxide molar ratio (1:2). Then the mixture entered the shelf furnace (4), where the leaching process took place at 180 °C with constant moderate stirring by screws for 3 hours. Air was additionally supplied to the furnace to provoke the chemical reaction between the starting components. The cooled melt was fed into the reactor containing distilled water and dissolved by stirring (6). After stirring for twenty minutes, the resulting solution was settled for one hour and then pumped into the reactor for the precipitation of potassium titanate (7); ethyl alcohol was added from the container for its condensation and accumulation (9). In the reactor (7), the precipitation of potassium titanate occurred for 30 minutes without stirring. At the next stage, the precipitated potassium titanate was fed together with ethyl alcohol from tank (9) to the tank for washing and purifying the potassium titanate (8), which was then fed from the tank (8)



**Figure 8.** Diffraction pattern of potassium titanate

**Table 3.** Basic calculated data of unit cell characteristics and average particle size of potassium titanate

2 $\theta$	$d_{(hkl)}$	hkl	$\beta$	D, nm
24.25	3.6778	120	0.1946	43.45
29.80	2.9942	023	0.3036	28.05
31.00	2.8812	032	0.3334	26.73
34.10	2.6304	202	0.2278	39.82
40.70	2.2817	140	0.4236	19.58



**Figure 9.** Technological scheme of the ilmenite alkaline leaching process with subsequent alcohol purification of potassium titanate: 1: hoppers for raw materials; 2: ball mill; 3: scales for raw materials; 4: shelf furnace for leaching; 5: reactor for recycling alcohol and potassium hydroxide; 6: reactor for dissolving the melt with stirring; 7: reactor for the precipitation of potassium titanate; 8: tank for washing and cleaning the potassium titanate; 9: container for condensation and accumulation of ethyl alcohol; 10: drying cabinet; 11: container for the accumulation and concentration of potassium hydroxide; 12: electromagnetic separator; 13: container for collecting water

to the drying cabinet (10). From the reactor (7) and the tank (8), the alcoholic solution of potassium hydroxide was fed to the reactor for recycling alcohol and potassium hydroxide (5). Then, the ethyl alcohol was fed and condensed into tank (9), and the aqueous alkali solution into tank (11). The sediment from the melt dissolution reactor (6) was fed into the electromagnetic separator (12), where the separation of ilmenite took place. The ilmenite was then returned to the bunker (1), and the iron (III) oxide was shipped for further use in other industries.

## Conclusion

This study investigated the ilmenite of the Irshan deposit using scanning electron microscopy with X-ray spectral microanalysis. The ore samples had a unique chemical composition, which distinguishes them from other known deposits. We calculated that the sample under study had an ilmenite concentrate content of  $70 \pm 5\%$ ; a  $20 \pm 5\%$  of rutile, and that the rest included pyrite, pseudorutile, and impurity phases. Using XRD methods, we calculated the average size of ilmenite particles as 26.62 nm.

Based on thermodynamic calculations using the Tiomkin-Schwartzman method, we showed that the alkaline leaching of ilmenite with potassium hydroxide is an energetically advantageous spontaneous process.

We determined the optimal technological parameters of the ilmenite alkaline leaching process: average particle size  $\leq 71 \mu\text{m}$ , molar ratio of the starting components 1:2, interaction time: 3 hours, and temperature – 453 K.

We found that the best titanium(IV) leaching mechanism corresponded to the compressed sphere kinetic model, with a chemical reaction at the limiting stage ( $R^2=0.9728$ ), i.e., the solid-phase interaction was limited by the diffusion of alkali from the surface of the nucleus into the volume of ilmenite particles through the formed interaction products, which led to the annihilation of the initial nuclei. We calculated the apparent activation energy to be 22 kJ/mol, indicating a kinetic regime of low activation energy.

We developed an effective method for purifying potassium titanate, ensuring an impurity content of less than 5%. We established that the average particle size of potassium titanate according to the X-ray diffraction data was 31.53 nm, which was also confirmed by electron microscopy data.

We propose here a technological scheme for the production of potassium titanate by alkaline leaching of ilmenite. Our results can become the basis for the creation of more efficient and economically advantageous technologies for ilmenite processing.

### Conflicts of interest

We declare no conflicts of interest, including financial, personal, authorship, or other, which could affect the study and its results.

### Financing

The study received no financial support.

### Data availability

The manuscript has no associated data.

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