The research object is the ilmenite concentrate from the Irshansky deposit. This study describes an elemental composition of the mineral raw material and confirms its structure using the X-ray diffraction and scanning electron microscopy. Experimental studies have shown that the ilmenite concentrate from the Irshansky deposit has a significant titanium content in terms of titanium dioxide (79 %). Mineral raw materials with such a chemical composition are unique, so there is a need to find alternative methods for its processing. The research demonstrates that the maximum degree of extraction in the process of alkaline leaching of the ilmenite concentrate is achieved under the condition that the average diameter of particles of the mineral raw material should be ≤71 μm. As a result of temperature studies, it has been found that a temperature of 453 K would suffice to obtain potassium titanate at atmospheric pressure. Further temperature increase does not provide for a significant increase in the degree of titanium extraction, and also contributes to the formation of polytitanates of various compositions. The study of the influence of the molar ratio of the starting reagents on the degree of extraction of titanium(IV) from the ilmenite concentrate has showed that the optimal molar ratio between the components corresponds to the stoichiometric one and is 1:2. Increasing the amount of potassium hydroxide in the reaction mixture is impractical as it reduces the yield of potassium titanate, and the final product will have high alkalinity due to excess alkali. The optimal time for alkaline leaching is three hours of continuous heating in a glycerin bath. A further increase in the duration of heating does not lead to an increase in the degree of extraction, which is associated with the diffusion of alkali from the surface of the nucleus into the volume of ilmenite particles due to the formed products of interaction and annihilation of the initial nuclei

Keywords: ilmenite concentrate, alkaline leaching, potassium hydroxide, potassium titanate, degree of extraction

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DETERMINING PATTERNS OF LEACHING TITANIUM(IV) FROM THE IRSHANSKY DEPOSIT ILMENITE

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1. Introduction

Titanates are compounds with the general chemical formula $Me_2O{\cdot}nTiO_2$, (n=1–8), in which the metal cations are Na^+, K^+ ions [1]. The structure and properties of titanates are influenced by the chemical composition of the initial components, the ratio between oxides in the molecule, and the conditions of synthesis. By adjusting the conditions for alkaline leaching of ilmenite, it is possible to obtain, for example, potassium titanates of amorphous or crystalline structure with different particle sizes and morphologies, which, in turn, expands the scope of their use.

Thus, the inclusion of potassium titanate fibers in the structure of an aluminum alloy composite improves its mechanical properties [2]. Adding fiber-like crystals of potassium titanate to the composition of carbon foam materials increases their strength and, accordingly, improves thermal insulation properties [3].

Potassium titanates and polytitanates of different chemical composition and structure are actively used as photocatalysts of water decomposition [4], dyes [5]. A catalyst for the process of transesterification of oils and production of biodiesel was designed on the basis of potassium titanate [6].

The principal method for obtaining titanates and polytitanates is the alkaline leaching method, the essence of which is the interaction of titanium-containing raw materials with a solution or molten alkali. Most often, titanium dioxide is used as a titanium-containing raw material, which requires preliminary preparation and purification, which increases the cost of producing the target product [7]. Ilmenite is also a promising raw material for obtaining titanates by the alkaline leaching method. A significant advantage of using such ore as a starting reagent is the reduction of the number of stages of preliminary processing of raw materials, which accordingly reduces the cost of the final product. Therefore, research aimed at finding suitable production conditions for the process of alkaline leaching using ilmenite is promising.

2. Literature review and problem statement

According to 2022 data from the United States Geological Survey, the global reserves of titanium ore are about 800 billion tons, which are located in 48 countries of the world. In Ukraine, titanium ores are concentrated at 27 deposits of various types, but the main titanium-containing raw materials of the country are ilmenite ores from loose deposits [8].

On an industrial scale, ilmenite concentrate is mostly processed by acid methods using chloride or sulfate acids [9]. When choosing a method for ilmenite processing, the phase composition of the raw material has a significant influence. Thus, the advantage of the sulfuric acid method is the possibility of using ore with a low content of ilmenite but one of the disadvantages is the low efficiency of processing altered ilmenite (with a high content of TiO_2), while for the chloride technique, the phase composition is not of significant importance. However, acid leaching technologies require long-term heating and the use of an excess amounts of starting acids.

However, there are more and more studies related to the modernization of methods of acid processing of ilmenite, in particular the use of fluoride activators, weak mineral acids, organic acids and their derivatives, etc. An alternative method for acid leaching, which is of interest to researchers, is the method for alkaline leaching of Ti(IV) from titanium-containing raw materials.

For the processing of ilmenite from the Shan Dong province (China) by the alkaline leaching method, the authors of [10, 11] determined that the maximum extraction of titanium(IV) is affected by the particle size of the mineral raw material. Equally important are the ratio of the initial components, the concentration of the aqueous alkali solution, the temperature and time regime. It was established that the optimal conditions for the leaching process are particle sizes of 58–75 μm, the ratio of alkali and ilmenite 7:1, the concentration of potassium hydroxide 84 %, the temperature 533 K, and the interaction time 60 minutes. However, the use of such an amount of alkali complicates further purification of the target product.

The authors of [12, 13] chose ilmenite from the Abu Ghalaga region (Egypt), in which the content of titanium dioxide is 40 %, as a research object. For leaching, a 70 % solution of potassium hydroxide was used, in a mass ratio between alkali and ilmenite of 5:1. The duration of interaction is 3 hours at a temperature of 423 K with constant stirring at 375 rpm. However, this procedure is effective for ilmenite with a low TiO_2 content.

The authors of [14] established that for the alkaline leaching of ilmenite concentrate produced by Kahnooj Titanium Complex, Kerman (Iran), the optimal conditions are a three-hour interaction of the components at a temperature of 493 K using a 70 % potassium hydroxide solution. However, this is only one of the stages of processing ilmenite concentrate into the target product and the authors propose to carry out further processing using the chloride method.

In works [15, 16], samples of ilmenite separated from the black sands of the Rosetta coast (Egypt) were used as the research object. The experiment was carried out in two stages: the first using aqueous solutions of potassium and sodium hydroxides (70 %), which were used to treat the original ore at a temperature of 393 K, the second – acid leaching of the obtained ilmenite paste. In addition, samples of ilmenite from the same coast were fused with crystalline alkalis at different mass (molar) ratios in the temperature range of 623–723 K. However, the drawback of the procedure is that polytitanates of different compositions are formed at high temperatures.

Due to the unique composition of ilmenite from the Irshansky deposit (~79 % TiO_2), the use of alternative processing methods, including alkaline leaching, is promising. All of this allows us to state that it is appropriate to determine proper production conditions for the process of alkaline leaching of ilmenite with a high content of titanium dioxide.

3. The aim and objectives of the study

The purpose of our research is to determine proper production conditions for processing ilmenite concentrate from the Irshansky deposit by the alkaline leaching method for potassium hydroxide, which will allow creating an economically feasible technology for obtaining potassium titanate.

To achieve the set goal, a number of tasks must be completed:

– to determine the elemental composition of mineral raw materials using a set of physical and chemical methods;

– to conduct a study on the dependence of the degree of titanium extraction on various parameters (dependence on the size of the particles of mineral raw materials, the molar ratio between the components, the temperature and time of the leaching process);

– to study the morphology of synthesized potassium titanate particles.

4. The study materials and methods

96 % ilmenite concentrate from the Irshansky deposit and crystalline potassium hydroxide (Merck KGaA) were used as the research object.

Since titanium dioxide exhibits acidic properties, its interaction with alkalis is more effective in comparison with acids. According to the results from studying the elemental composition of the ore, ilmenite from the Irshansky deposit has a high TiO_2 content. Therefore, it is more appropriate to use solutions or melts of compounds with well-defined alkaline properties as leaching agents.

Samples of ilmenite and the obtained potassium titanate were examined by scanning electron microscopy with X-ray spectral elemental microanalysis on an electron microscope "JSM-6490 LV" (Japan) with energy dispersive and wave dispersive spectrometers "Energy Plus" ("Oxford Instruments"). To confirm the structure of the samples, the X-ray diffraction method was used (DRON 3M diffractometer (Kα (Cu), λ=0.1540 nm).

In order to establish the influence of particle size of the ilmenite concentrate on the degree of Ti(IV) extraction, the mineral raw material was previously crushed in an agate mortar. Fractions in the range from 71 to 630 μm were obtained using a set of appropriate sieves. Leaching was carried out for 3 hours at a temperature of 453 K with a molar ratio of $FeTiO_3$: KOH (1:2) for each fraction of the concentrate.

The optimal molar ratio between the initial components was determined using the ilmenite fraction with an average particle diameter ≤71 μm. Leaching reactions were carried out for 3 hours at a temperature of 453 K with a molar ratio of $FeTiO_3$: KOH (1:2; 1:3; 1:4; 1:5; 1: 6).

Determination of the effect of temperature on the degree of titanium(IV) extraction was carried out in the temperature range of 453–573 K. To this end, the ilmenite fraction with an average particle diameter of ≤71 μm was used. The duration of the leaching process is 3 hours at the molar ratio of $FeTiO_3$: KOH components (1:2).

The study of the dependence of the degree of titanium(IV) extraction on the contact time of the reagents was carried out in the range of 30–300 minutes. An ilmenite fraction with an average particle diameter of ≤71 μm, a molar ratio of $FeTiO_3$: KOH components (1:2), and a temperature of 453 K were used.

To explore the degree of extraction of Ti(IV) during leaching from ilmenite, the peroxide method for quantitative determination of titanium in solution using a UV-1200 spectrophotometer was used [17].

The degree of extraction (X, %) of titanium(IV) from ilmenite is calculated according to the formula:

$$X = \frac{m\left(\mathrm{Ti}\left(\mathrm{IV}\right)_e\right)}{m\left(\mathrm{Ti}\left(\mathrm{IV}\right)_o\right)} \cdot 100\ \%,$$

where $m(\mathrm{Ti(IV)})_e$ is the mass of extracted titanium, mg;

$m(\mathrm{Ti(IV)})_o$ is the mass of titanium in the original melt, mg.

5. Results of the study of alkaline leaching of ilmenite from the Irshansky deposit

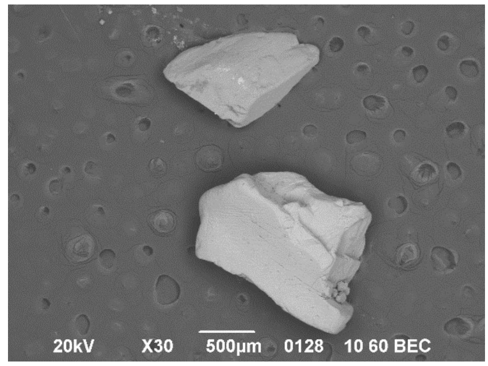
5. 1. Determining the elemental composition of mineral raw materials by a set of physical and chemical methods

The initial analysis of the raw mineral raw materials was carried out using the methods of scanning electron microscopy with X-ray spectral elemental microanalysis (SEM).

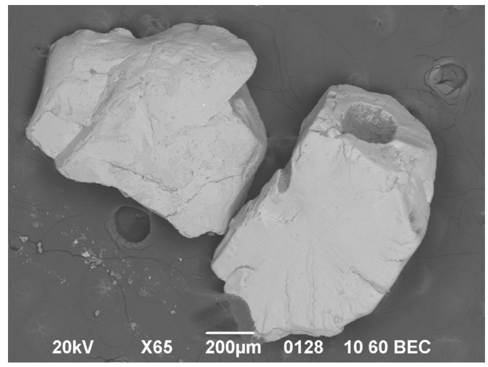
Fig. 1 shows a SEM photograph of a sample of the original ilmenite.

Fig. 2 shows the diffraction pattern of a sample of ilmenite concentrate from the Irshansky deposit.

It is shown that the total content of titanium in the ore (in terms of TiO_2) is more than 70 %, which indicates that the ilmenite from the Irshansky deposit belongs to the group of altered ilmenites.



a



b

Fig. 1. Scanning electron photograph of a sample of ilmenite from the Irshansky deposit: *a* – 30 times magnification; *b* – 65 times magnification

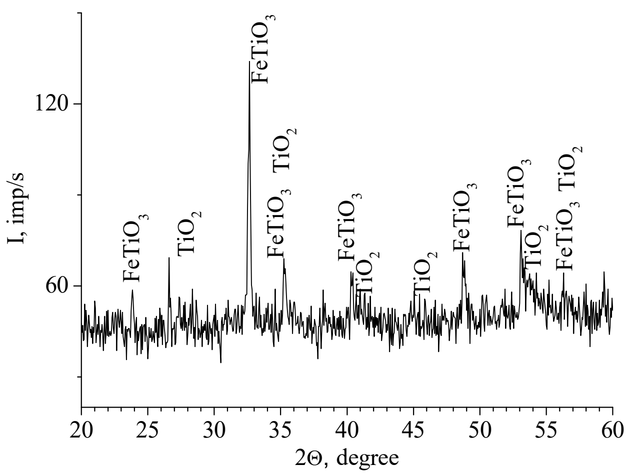


Fig. 2. Diffraction pattern of a sample of ilmenite concentrate from the Irshansky deposit

5. 2. Studying the dependence of the degree of titanium extraction on various parameters

To study the effect of particle size on the leaching process, the ilmenite concentrate (96 %) from the Irshansky GZK was previously ground in an agate mortar. As a result, fractions with the following particle sizes were obtained: ≤71 μm; 71–140 μm; 140–315 μm; 315–630 μm. The reaction mixture in the minimum molar ratio of 1:2 ($FeTiO_3$:KOH) was kept in a glycerol bath at a temperature of 453 K for 3 hours.

Leaching of ilmenite with potassium hydroxide under conditions of atmospheric pressure and at a temperature of 453 K can be described by the following equation [18]:

$$4FeTiO_3 + 8KOH + O_2 \rightarrow 4K_2TiO_3 + 2Fe_2O_3 + 4H_2O.$$

A qualitative sign of the completion of the reaction is a change in the color of the reaction mixture from gray to green-brown (Fig. 3).

Fig. 3. General view of the melt after the alkaline leaching process

Fig. 4 shows the dependence of the degree of titanium(IV) extraction on particle size of the original ilmenite concentrate.

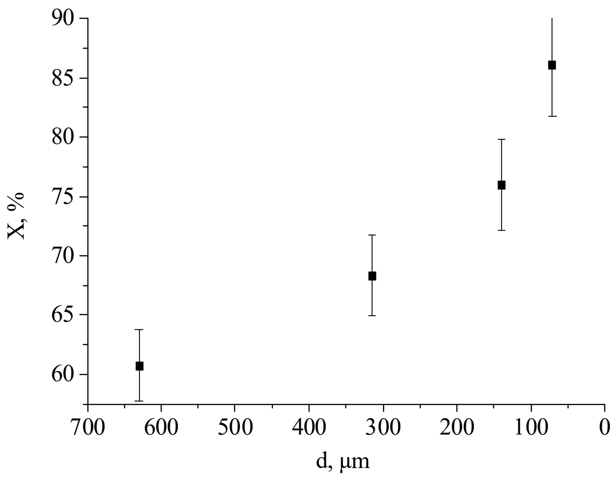


Fig. 4. Dependence of the degree of extraction of titanium (IV) on the particle size of ilmenite concentrate during alloying with potassium hydroxide in the molar ratio of ore to alkali 1:2 for 3 hours at a temperature of 453 K

Fig. 4 demonstrates that the reduction of the particle size of mineral raw materials during fusion under the above conditions increases the degree of extraction of the target element.

To study the influence of the molar ratio between the components on the degree of extraction, the fraction of ilmenite with particle sizes ≤ 71 μm was chosen. The molar ratio of the initial components of the concentrate and alkali was used, respectively 1:2; 1:3; 1:4; 1:5 at a temperature of 453 K for 3 hours.

The quantitative influence of the molar ratio of reagents in the initial mixture on the degree of extraction of titanium(IV) was determined (Fig. 5).

To establish the optimal temperature for leaching ilmenite with potassium hydroxide, the initial ilmenite fraction ≤71 μm, a mixture of ilmenite and alkali (1:2), and a temperature range of 453–573 K were used, with a heating time of 3 hours.

Fig. 6 shows the dependence of the degree of titanium(IV) extraction on the temperature regime of the alkaline leaching process.

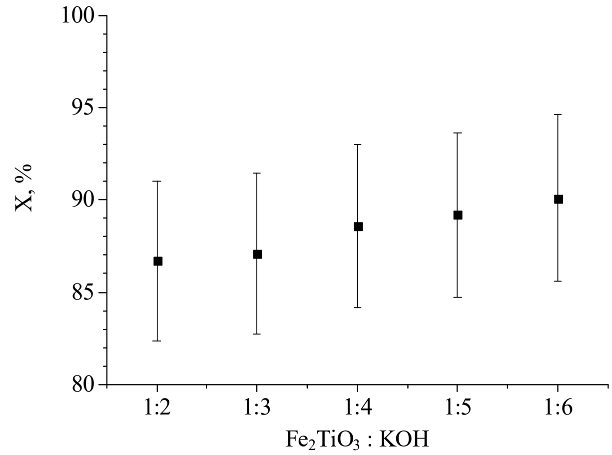


Fig. 5. Dependence of the degree of extraction of titanium(IV) on the quantitative ratio of starting substances with an average diameter of particles ≤71 μm for 3 hours at a temperature of 453 K

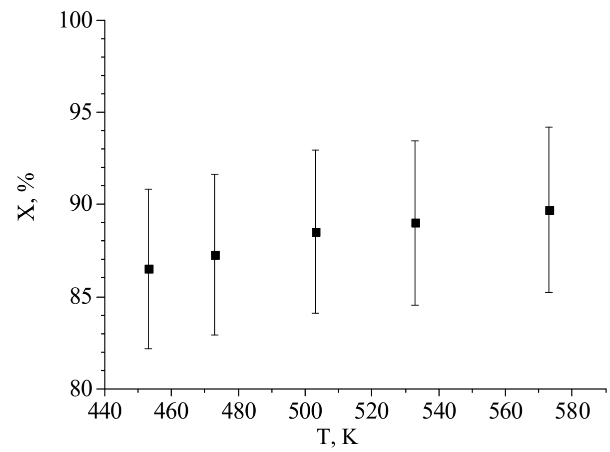


Fig. 6. Dependence of the degree of extraction of titanium(IV) on temperature during fusion of a mixture of ilmenite and alkali (1:2) for 3 hours using the fraction of the original ilmenite concentrate ≤71 μm

It has been shown that the temperature above 453 K does not significantly affect the degree of titanium(IV) leaching.

To study the degree of titanium(IV) extraction, an ilmenite concentrate with an average particle diameter of ≤71 μm, an interaction temperature of 453 K, and a quantitative ratio between components of 1:2 was used. The time range of the leaching process is 30–300 minutes.

Fig. 7 shows the dependence of the degree of titanium(IV) extraction on contact time between ilmenite concentrate and potassium hydroxide.

The nature of the curve of dependence of the degree of extraction on leaching time (Fig. 7) takes the form of a curve approaching saturation.

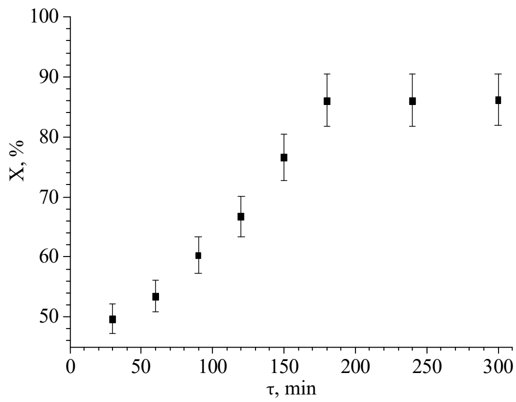


Fig. 7. Dependence of the degree of titanium(IV) extraction on the contact time of $FeTiO_3$ and KOH with an average particle diameter of ≤71 μm, an interaction temperature of 453 K, and a quantitative ratio between the components of 1:2, respectively

5. 3. Studying the morphology of synthesized potassium titanate particles

In order to further purify the synthesized potassium titanate, the obtained alloy was dissolved in distilled water, filtered from unreacted impurities and by-products of the reaction. Precipitation of potassium titanate from the filtrate was carried out with 96 % ethanol; the resulting crystals were dried in air.

Fig. 8 shows a diffractogram of the obtained potassium titanate. "Match!3" software was used to identify the structure of potassium titanate.

The morphology of the particles of synthetic potassium titanate was investigated using the SEM-microscopy method (Fig. 9).

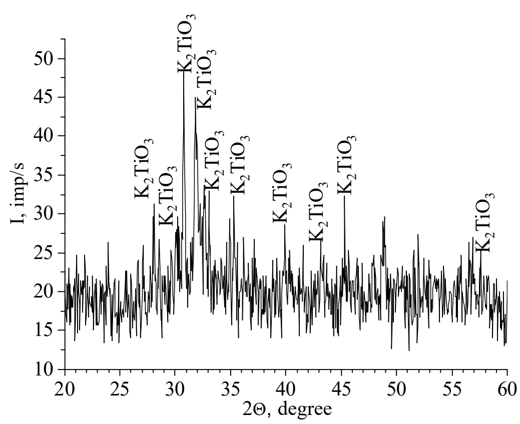


Fig. 8. Diffractogram of a purified sample of potassium titanate

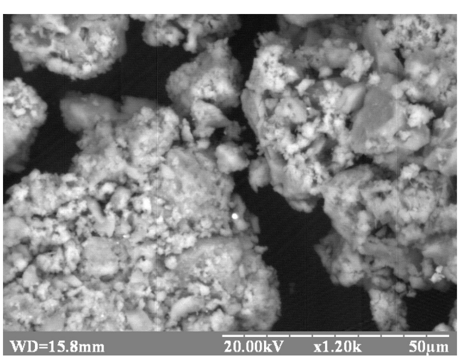


Fig. 9. Scanning electron photograph of purified potassium titanate

It was determined that individual crystallites of potassium titanate have a shape close to orthorhombic, which was also confirmed by X-ray diffraction.

6. Discussion of results of investigating the alkaline leaching of ilmenite from the Irshansky deposit

At the first stage of the research, the phase and chemical composition of the original mineral raw materials was determined by a set of physicochemical methods. The study of the chemical and phase composition of the ilmenite concentrate could reveal the factors determining the reactivity of the ilmenite samples from the Irshansky group of deposits, as well as determine possible techniques of processing this mineral raw material.

In accordance with the data from scanning electron microscopy and X-ray spectral elemental microanalysis (Fig. 1), it has been shown that the selected samples of raw materials belong to altered ilmenite based on their chemical composition. This means that this mineral has a high content of titanium (79 % in terms of TiO_2), which makes it difficult to process it using classic acid methods.

The phase composition and structure of the original ilmenite was also investigated by X-ray diffraction methods; the corresponding diffractogram was constructed (Fig. 2). The main reflexes of the $FeTiO_3$ phase [96–900–0907] at 23.80°; 32.52°; 35.25°; 48.71°; and 53.03° 2θ angles were identified using the "Match!3" software and cards of typical X-ray patterns of substances. In addition, it was determined that at 26.52°; 35.25°; 41.20°; 54.30°; and 56.60° 2θ angles contain the TiO_2 phase [96–153–0151]. The presence of a separate phase of titanium dioxide is proof that the ilmenite samples from the Irshansky deposit have a high content of titanium in the ore, which is 79.2 % (in terms of TiO_2). The reason for the increased content of titanium dioxide is the presence of microcracks that arose in the process of long-term weathering [17].

At the next stage of our research, the production and appropriate conditions for the processing of the ilmenite concentrate from the Irshansky deposit by the method of alkaline leaching with potassium hydroxide were determined. Among the main parameters that directly affect the production process, the following can be distinguished: the size of the particles of mineral raw materials, the molar ratio between the components, the temperature and the time of the leaching process. The degree of extraction was selected as a quantitative indicator of effectiveness of the leaching process, which was calculated according to the formula given in chapter 4.

It should be noted that the size of the ore particles affects the leaching process, which was also shown by the authors of works [10, 11]. When studying the influence of the size of ilmenite particles on the degree of extraction of titanium(IV), it was found that the maximum value of the degree of extraction of 86.1 % is achieved using raw materials with an average particle diameter of ≤71 μm (Fig. 4). This dependence can be explained by the fact that particles with a smaller size of mineral raw materials have a larger contact area with alkali. The degree of extraction does not reach 100 % due to the presence of impurities in the raw material and the effect of the formed ferrum(III) oxide, the layer of which hinders the further diffusion of alkali to ilmenite. This deficiency can be corrected by removing ferrum(III) oxide from the reaction mixture. Solving this problem is one of the promising tasks for further research.

The degree of extraction of titanium(IV) in the leaching process depends on the molar ratio between the components [12]. It was determined that the maximum degree of extraction of 90.1 % is achieved at a quantitative ratio of ilmenite and alkali of 1:6, but the difference between the minimum and maximum degree of extraction is less than 5 % (Fig. 5). The leaching process is also possible using alkali solutions [12–14] but is more effective for ilmenites with a low content of titanium dioxide. In addition, the use of saturated solutions increases the heating time of the system, complicates the general scheme of obtaining potassium titanate, which is not economically feasible. The results obtained during alkaline leaching showed that in the case of using a larger amount of alkali in the initial mixture, polytitanates are formed [19–21], which are not the target product of the study. Also, an excess amount of alkali leads to its accumulation in the form of an inactive reagent, which complicates further purification of the obtained potassium titanate. One of the possible ways to overcome this problem is to use a mixture of potassium hydroxide and potassium carbonate, which is the next stage of experimental research.

Studies of the effect of temperature on the alkaline leaching process (Fig. 6) showed that at a temperature of 453 K, the degree of titanium(IV) extraction is 86.5 %. A further increase in the melt heating temperature leads to a slight increase in product yield (89.8 %), which is insignificant compared to the energy costs for maintaining a high process temperature. The research results allow us to state that the 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. We note that, as a rule, the temperature of the leaching process is in the range of 623–723 K [15, 16]. However, as a result, polytitanates of different chemical composition can be obtained. In order to obtain potassium meta titanate, it is advisable to use minimum temperatures at a molar ratio that corresponds to the stoichiometry of the chemical reaction.

At the next stage of the study, it was found that in the first 30 minutes of interaction of the starting substances, the degree of extraction is 50 %. The maximum degree of titanium(IV) extraction of 86.13 % was achieved after three hours from the beginning of contact of the starting reagents (Fig. 7). According to the kinetics of heterogeneous processes, the fusion time should be sufficient for the passage of diffusion processes at the boundary of phase separation and a more complete course of the chemical reaction with unreacted ilmenite particles. The calculation of kinetic regularities showed that the leaching process is most successfully described by the kinetic model of the "compressed sphere" with the limiting stage of the chemical reaction, which was determined as a result of earlier studies [22]. It was determined that a further increase in the heating time does not lead to an increase in the degree of extraction of titanium (IV) from the ilmenite concentrate from the Irshansky deposit.

Our results provide for the leaching of altered ilmenite with solid potassium hydroxide in order to obtain potassium titanate with an extraction rate of up to 89 %. The production-appropriate conditions for obtaining potassium titanate using ilmenite from the Irshansky deposit are as follows: ore fraction less than 71 μm, quantitative ratio of ilmenite and alkali 1:2, temperature 453 K, and contact time 3 hours. If the specified conditions are met, a qualitative sign of the leaching reaction is a change in the color of the mixture from gray to green-brown (Fig. 3).

At the stage of extracting potassium titanate, the alloy was dissolved in a minimum amount of distilled water, filtered from unreacted impurities and by-products of the reaction. Next, K_2TiO_3 was precipitated from the filtrate with 96 % ethanol, and the resulting powder was air-dried. In order to identify the potassium titanate obtained by alkaline leaching of ilmenite from the Irshansky deposit, the X-ray diffraction method was used (Fig. 8). It was established that the intensity of the main reflections of K_2TiO_3 are in the range of 29–34° angles of 2θ, which indicates the orthorhombic structure of the crystals and is in good agreement with literature data [23]. SEM-microscopy methods show that potassium titanate particles are orthorhombic in shape and tend to form aggregates (Fig. 9).

The potassium titanate obtained by the proposed procedure of alkaline leaching was tested as a catalyst for the photo destruction of methylene blue dye from aqueous solutions. The results of the research showed the prospects of using the synthesized potassium titanate as a photocatalyst in water purification processes from this dye. A limiting factor in the use of potassium titanate as a photocatalytic agent is that its effectiveness has been shown for cationic dyes such as methylene blue. Regarding dyes of the anionic type, for example, Congo red, the photocatalytic activity of potassium titanate was not detected. The study of the processes of modification of the surface of potassium titanate and the investigation of photocatalytic activity is a prospect for further research.

7. Conclusions

1. The elemental composition of the raw material was determined by the methods of scanning electron microscopy with X-ray spectral elemental microanalysis and X-ray diffraction. It was established that the content of titanium in terms of titanium dioxide is more than 79 %, which allows processing of mineral raw materials by alkaline leaching.

2. The effect of particle size, molar ratio of reagents, temperature, and interaction time on the degree of extraction of titanium(IV) from ilmenite of the Irshansky deposit was investigated. It was established that the molar ratio between the components is 1:2 ($FeTiO_3$: KOH) as appropriate production conditions. The melting process is proposed to be carried out during three hours of heating the mixture at a temperature of 453 K. The degree of extraction of titanium(IV) under these conditions is more than 86 %, when using raw mineral raw materials with an average particle diameter of ≤71 μm.

3. The obtained sample of purified potassium titanate was examined by SEM and X-ray diffraction methods; it was established that it has an orthorhombic crystal structure, and the particles are prone to the formation of ag-

gregates. The method of alkaline leaching of ilmenite with potassium hydroxide allows obtaining potassium titanate with an impurity content of less than 5 %. Our data allow us to state that water-alcohol purification of potassium titanate is promising for use in the technological process of processing ilmenite ore by the alkaline leaching method.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study and the results reported in this paper.

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Data availability

All data are available in the main text of the manuscript.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

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