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RESEARCH ON THE POSSIBILITY OF USING ELECTRICAL DISCHARGE IN A LIQUID FOR PROCESSING PRODUCTS AND WASTES OF TITANIUM PRODUCTION

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Abstract. The article is devoted to the issue of improving the technologies for processing titanium-containing raw materials. Today, this task is relevant, has scientific and practical significance for Ukraine, given the significant reserves of titanium ores on its territory and the strategic importance of the titanium industry. The article focuses on examining the main traditional methods of processing titanium ores and on analyzing their drawbacks, among which are high energy consumption, the need for a large amount of sulfuric acid, the formation of harmful sulfur-containing waste during processing, and the need for additional cleaning processes. As a promising alternative to existing methods, the use of an electric discharge in a liquid is proposed - a high-voltage breakdown of the liquid gap between the electrodes, as a result of which a high energy density occurs in the discharge channel for a short period of time, the pressure and temperature rise sharply, the discharge channel expands rapidly, and a pressure wave is emitted into the external environment. The advantages of using an electric discharge include: environmental safety, the absence of pollution, the possibility of adjusting processing parameters. Experimental studies of the effect of electric discharge on titanium dioxide paste and waste from titanium ore production were conducted. The results of the studies showed that under the influence of factors, the source of which is an electric discharge in a liquid, the sulfur concentration decreases: in the dioxide paste up to three times, and in waste from titanium ore production - up to 2 times. At the same time, during electric discharge processing, grinding of the primary material is observed, as evidenced by the results of the analysis of the particle size composition. The study of different modes of electric discharge action made it possible to determine the rational specific energy of impact - a further increase in the specific energy of impact does not lead to significant changes in the sulfur concentration and particle size of the products. The results obtained show the possibility and confirm the prospects of using electric discharge in a liquid for processing products of the sulfate method of obtaining titanium dioxide and waste from its production for the purpose of their desulphation.

Key words: titanium dioxide, electrical discharge technology, waste desulphation.

1. Introduction

The modern world is characterized by constant and rapid development of industry. This, in turn, leads to an increase in the needs of mankind in various natural minerals - hydrocarbons, ores, etc. One of the most important and necessary resources used in various fields of human activity is titanium and its compounds [1, 2].

Thus, titanium dioxide (TiO_2) is one of the most important inorganic materials in modern industry, which is the main commercial product of the titanium industry [2]. Titanium dioxide is used in construction as an additive to cement [3], in medicine as an additive in drugs that protects against ultraviolet radiation [4], in the food industry (additive E171), the cosmetic industry, for the production of plastics and paper [5, 6], in the manufacture of fire-resistant paints [7]. One of the promising areas of use of titanium dioxide is the treatment of wastewater [8, 9] and oil field waters [10].

Titanium industry is one of the most strategically important in Ukraine [1, 11]. It is of paramount importance for the defense capability and ensuring the economic growth of the country both in wartime and post-war times.

The titanium industry of Ukraine has a complete production cycle - from the extraction of titanium-containing ores, their enrichment, the production of sponge titanium and to the smelting of titanium ingots. The main source of supply for enterprises producing titanium products is Ukrainian mineral and raw material base (according to world experts, Ukrainian deposits of titanium ores rank tenth in the

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world in terms of volume). There are approximately 40 titanium deposits in Ukraine, among them one unique, 13 large and 10 medium deposits. At the same time, titanium ores are currently mined in Ukraine only from placer deposits, which account for about 10% of all explored reserves [11].

Taking into account all of the above, all works related to the development of the titanium industry of Ukraine and extraction of titanium-containing ores and their processing are relevant and have important scientific and practical significance.

2. Methods

The starting minerals for producing titanium products are ilmenite and rutile. Since the amount of rutile in nature is insignificant, the most important and main titanium ore is considered to be the fairly widespread ilmenite. Ilmenite is one of the most stable natural compounds of titanium dioxide and iron oxide $FeTiO_3$ ($TiO_2 \cdot FeO$).

Obtaining titanium and its compounds from ore material is a complex, labor-intensive and expensive production, which is characterized by multi-stage technological processes and significant energy and material costs. Currently, two main methods of processing titanium-containing ores are used - sulfate and chlorine [2, 5, 6]. The two above-mentioned methods can be considered the main ones for obtaining titanium products in spite of some alternative methods of processing, for example, titanium dioxide [5].

Each of these two methods is characterized by a complex technological process, high energy consumption and is environmentally hazardous [5]. Thus, the chlorine method is more expensive than the sulfate method, selective to the raw material, and due to its high activity requires the use of corrosion-resistant equipment and high safety requirements. In turn, the disadvantages of sulfate production of titanium dioxide are significant consumption of sulfuric acid and a large amount of sulfur waste, as well as the need to use additional technological processes to reduce the percentage of sulfur in the initial product. Various additional technological processes are used to solve this problem: vacuum evaporation, hydrolysis of titanium dioxide concentrate and additional heat treatment and purification processes to reduce the sulfur content to the level required for further use of titanium dioxide. At the same time, the search for alternative ways to solve the problem of reducing the sulfur concentration in titanium production products continues.

One of such alternative methods could be the use of an electric discharge in a liquid. An electric discharge in a liquid is a breakdown of the liquid gap between the electrodes (anode and cathode) as a result of applying a high voltage to the anode. In the process of an electric discharge, a high energy density arises in a certain volume (discharge channel) for a short period of time. As a result, the pressure and temperature increase sharply, the discharge channel rapidly expands, so a pressure wave is emitted into the external environment [12].

Electrical discharge methods for processing materials have a number of advantages:

- regulation of impact energy in a wide range and establishment of selective processing modes of raw materials;
- absence of contamination of the processed raw materials during the material processing;
- intensification of chemical reactions under the influence of high temperatures and pressures, which generates an electric discharge;
- environmental friendliness;
- relative ease of maintenance.

The electric discharge method can be effectively used for the desulphation of ore raw materials [13] and coal in the production of water-coal fuel [14], which gives grounds to assume the possibility and prospects of using such technology to solve problems in the titanium industry.

Currently, work is also underway on the processing of titanium production waste [15], in particular, on the extraction of titanium dioxide from polymetallic ore waste [16]. This may be another promising direction for the application of electric discharge in the titanium industry, given the experience of its use for the processing of tailings (waste) of various ores [17].

Taking into account all of the above, the purpose of this work was to investigate the possibility and prospects of using electric discharge for processing products of the sulfate method of obtaining titanium dioxide and waste from its production for the purpose of their desulphation.

3. Theoretical and experimental parts

Experimental installation, initial parameters and research methodology.

The assessment of the possibility of using electric discharge in the technology of production of pigment titanium dioxide on samples of titanium concentrate (titanium dioxide) and waste from enrichment of titanium concentrates was carried out on laboratory high-voltage equipment of the Institute of Pulse Processes and Technologies (Figure 1). The laboratory equipment consists of a pulse current generator (PCG) 1 and a discharge chamber 2. The PCG provides high voltage to the electrodes. The electrode - anode is installed in the upper flange of the discharge chamber 2, where the raw material is processed, the discharge is carried out on the bottom of the chamber.

The parameters of the laboratory equipment (Table 1) were selected in such a way that they provided power and specific energy characteristics as in similar experimental studies, for example [17].

Table 1 – Parameters of the laboratory equipment

Parameter	Variation range
Maximum voltage, kV	from 40 to 50
Capacitance, μF	from 0.5 to 2
Inductance, μH	from 5 to 6
Discharge frequency, Hz	from 2 to 4
Discharge energy, kJ	from 0.625 to 1.25
Discharge gap length, m	from 0.03 to 0.05

1 – PCG; 2 – discharge chamber

Figure 1 – Laboratory high-voltage equipment

The volume of the discharge chamber is 10 dm^3. The discharge chamber was made of steel grade 20X13 to ensure the purity of the experiments. The electrodes were also made of steel. The processing was controlled from the control panel (not shown in the photo), the number of pulses was recorded by the pulse counter A 440.

Raw material samples were loaded into the discharge chamber in a volume ratio with water of 1 to 4, respectively. Five treatments of each raw material sample were carried out in each mode.

The specific energy consumption W_s (1) was taken as a measure of impact, it was controlled by the processing time, discharge energy and pulse frequency. The dimension of W_s is given in (kW·h)/t to adapt the results of laboratory studies to the scale of production and to estimate the necessary costs:

$$W_s = 3.6 \frac{W_0 \cdot f \cdot t}{M} . \qquad (1)$$

In (1) $W_0 = C \cdot U^2 / 2$ – stored energy, J; C – capacitor bank capacity, F; U – charging voltage, V; f – pulse frequency, Hz; t – processing time, s; M – mass of the processed product, g.

The starting material was titanium dioxide in the form of a paste (titanium concentrate $10TiO_2 \cdot 10H_2O \cdot SO_3$) after hydrolysis and first flotation of the technological chain of sulfate production of titanium dioxide (Figure 2, a) and waste from enrichment of titanium concentrates (Figure 2, b).

a) b)

a) titanium dioxide (paste); b) tailings from the enrichment of titanium concentrates

Figure 2 – Raw materials for electrical discharge processing

Experimental studies were divided into two independent parts.

The first part set the task of purifying titanium dioxide paste from SO_2, SO_3 by converting them into acids as a result of chemical reactions and then removing them from the final product by washing.

In the second part the electric discharge treatment of waste from the enrichment of titanium concentrates was studied. Waste from the enrichment of titanium concentrates is mainly a coglomerate of finely dispersed particles with the presence of metal particles and large amounts of environmentally harmful elements (for example, sulfur), i.e. everything that is removed from titanium dioxide when obtaining a high-purity product (within 99.9%). To dispose of enrichment waste, it is necessary to disintegrate, reduce the concentration of harmful elements to an acceptable level and ensure the maximum yield of valuable components from rocks. The resulting residues can be used, for example, in construction.

It was expected that the reduction of sulfur in the processed product (titanium raw materials or titanium production waste) leads to an increase of sulfur in the discharge medium with a simultaneous increase in its acidity and, accordingly, is accompanied by a decrease in pH. Therefore, tracking the change in pH of the discharge medium was used as express method for assessing the efficiency of the treatment process. The pH meter Ezodo pH 5011 was used to measure the pH of the discharge medium.

Analysis of the chemical composition of the processed product was carried out by an external physicochemical laboratory.

Previous experimental studies showed that the specific energy consumption put main influence on the processing. Therefore, all experiments were conducted to obtain the dependence of the the specific energy consumption on final results (pH, chemical composition of the processed material). In this case, it was assumed that the main operating factor is the generated pressure wave. It is known that the amplitude of the pressure wave depends on the charging voltage - the higher is the charging voltage, the greater is the amplitude of the pressure wave [18]. Based on this, experimental studies were conducted at the maximum possible charging voltage – 50 kV. The length of the interelectrode gap was unchanged and was 0.05 m.

The treatment energy was varied depending on the processing time, the energy of one discharge (varied by the capacitance of the capacitors) and the pulse frequency. Debugging experiments did not reveal a significant effect of the energy of one discharge, which was varied by the capacitance of the capacitors, and the pulse frequency (in the range of parameters given in Table 1.) alone on the final result. However, in the future, the influence of these parameters is planned to be investigated in more detail and in a more extended range.

4. Results and discussion

Results of experimental studies on the treatment of titanium dioxide paste and titanium ore production waste by electric discharge in liquid

The results of experimental studies of the treatment of titanium dioxide paste by electric discharge in a liquid are presented in Figures 3 and 4, as well as in Tables 2 and 3.

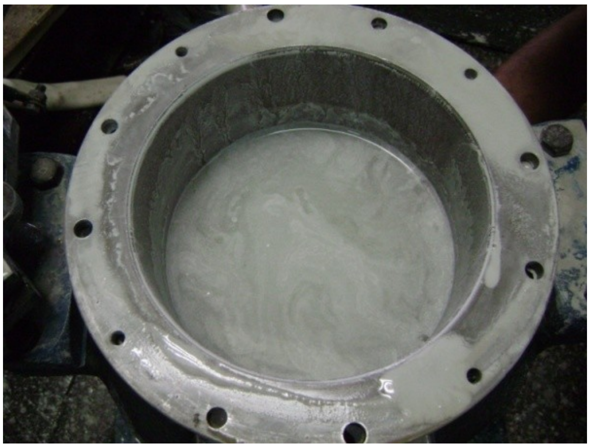
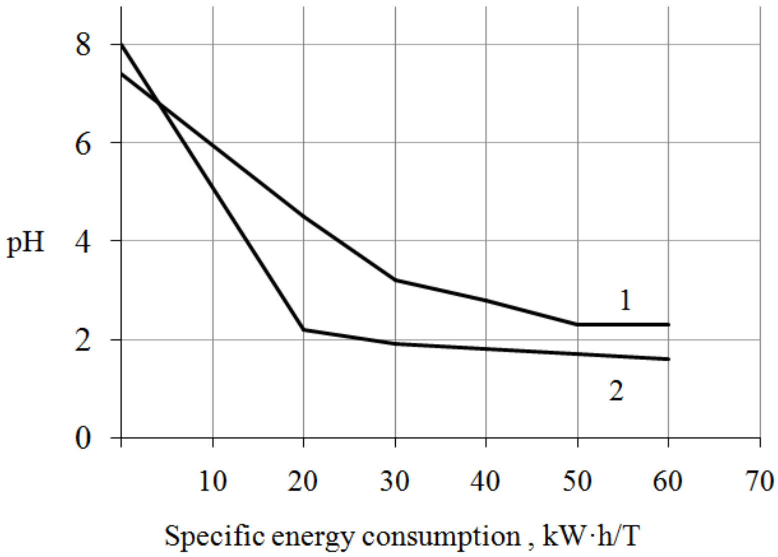


Figure 3 – Titanium dioxide (paste) after electrical discharge treatment



1 – titanium dioxide; 2 – waste from the production of titanium dioxide

Figure 4 – Change in pH in the discharge medium depending on the specific consumption of electric discharge energy during processing

Table 2 – Change in pH and sulfur content in titanium dioxide and titanium production waste depending on the specific energy consumption of electric discharges

Specific energy consumption, kW·h/t	Titanium dioxide		Titanium production waste	
	pH	Sulfur content, %	pH	Sulfur content, %
0	7.4	2.8	8.0	4.5
20.0	4.5	2.0	2.2	3.8
30.0	3.2	1.7	1.9	3.0
40.0	2.8	1.2	1.8	2.4
50.0	2.3	0.9	1.7	1.9
60.0	2.3	0.9	1.6	1.9

Table 3 – Change in the chemical composition of titanium dioxide as a result of electrical discharge treatment with a specific consumption of 50 kW·h/t

Element	Before processing, %	After processing, %
Titanium (TiO_2)	96.08	98.0
Vanadium	0.3	0.38
Iron	0.35	0.356
Sulfur	2.9	0.91
Manganese	0.37	0.35

Figure 3 shows the appearance of titanium dioxide paste after electric discharge treatment, and Figure 4 shows the dependence of the pH change on the specific electrical energy consumption. Table 2 shows the dependence of the pH and the amount of sulfur on the specific energy consumption.

Analysis of the results showed that increasing of specific energy consumption to 50 kW·h/t leads to decrease of the pH value. This indicates an increase in the amount of acid in the working medium and, accordingly, a decrease in the amount of sulfur in the processed materials (more than three times compared to the initial product). There is no point in further increasing the specific energy consumption, since it does not lead to a decrease in the amount of sulfur in the processed material. Therefore, the mode with a specific energy consumption of 50 kW·h/t can be recommended for the treatment of titanium dioxide paste and waste of the production of titanium ores. Table 3 shows the change in the chemical composition of titanium dioxide as a result of electric discharge treatment in the recommended mode.

Figure 3 and Table 4 show the results of the electric discharge treatment of titanium ore production waste. The treatment was carried out in a mode with a specific energy consumption of 50 kW·h/t. Analysis of the results shows that the sulfur concentration in the treated material decreased (more than 2 times) with a simultaneous increase in the concentration of titanium dioxide.

During the experimental studies, it was noted that the initial material is ground under the influence of electric discharge factors (generated pressure waves). Therefore, experimental studies on the influence of different processing modes on the particle size composition of titanium concentrates were conducted. The results in Table 5 show that under the influence of electric discharge, with an increase in the specific energy of action, the primary product is ground - the number of particles with a size of (1.0–0.63) mm decreases, while an increase in the number of particles with a size of (0.316–0.1) mm is observed. It is advisable to carry out processing with a specific impact energy of not more than 50 kW·h/t, since a further increase in the specific energy consumption does not lead to a significant change in the particle size.

Table 4 – Change in the chemical composition of waste from the enrichment of titanium concentrates as a result of electrical discharge treatment with a specific consumption of 50 kW·h/t

Element	Before processing, %	After processing, %
Titanium (TiO_2)	54.6	61.9
Vanadium	0.9	1.1
Iron	20.01	20.4
Sulfur	4.5	1.8
Manganese	0.4	0.62
Silicon	15.7	12.58
Zinc	1.4	1.6

Table 5 – Particle size distribution of titanium concentrates after electrical discharge treatment

Specific energy consumption, kW·h/t	Particle size distribution, %			
	(1.0–0.63) mm	(0.64–0.315) mm	(0.316–0.2) mm	(0.21–0.1) mm
20.0	48.0	30.0	19.0	3.0
30.0	38.0	32.0	22.0	8.0
40.0	29.0	30.0	31.0	10.0
50.0	10.0	32.0	43.0	15.0
60.0	9.0	28.5	44.5	16.0

Typically, redox reactions that lead to the purification of titanium products and titanium production waste from sulfur require reaction activators (catalysts). At the same time, there is a requirement that catalysts should not affect the chemical composition of the final product, i.e. do not contaminate it. The use of an electric discharge solves this requirement, since in this case there is no need to use chemical catalysts. Purification occurs due to the influence on the starting material of factors generated as a result of the electric discharge action. First of all, these are high temperatures and pressure, due to which changes in the chemical composition of titanium dioxide and waste from its production occur. This is another advantage of using an electric discharge.

6. Conclusions

The results obtained during experimental studies show the possibility and prospects of using electric discharge for processing products of the sulfate method of obtaining titanium dioxide and waste from its production for the purpose of their desulphation.

Under the influence of factors originating from an electric discharge in a liquid, the sulfur concentration was reduced: in the dioxide paste by up to three times; in the waste from the production of titanium ores - by up to two times. At the same time, during the electric discharge treatment, the primary material is crushed, as evidenced by the results of the analysis of the particle size distribution.

The study of different modes of electric discharge action showed that the treatment is rationally carried out with a specific impact energy of 50 kW·h/t. Further increase in the specific impact energy does not lead to significant changes in the sulfur concentration and particle size of the product.

The results obtained can be the basis for further work on the study of the electric discharge effect on products and waste from titanium production in order to select effective treatment modes, determine economic feasibility, and implement them in existing technological processes.

Conflict of interest

Authors state no conflict of interest.

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ДОСЛІДЖЕННЯ МОЖЛИВОСТІ ЗАСТОСУВАННЯ ЕЛЕКТРИЧНОГО РОЗРЯДУ В РІДИНІ ДЛЯ ОБРОБКИ ПРОДУКТІВ ТА ВІДХОДІВ ТИТАНОВОГО ВИРОБНИЦТВА

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Анотація. Стаття присвячена питанню вдосконалення технологій переробки титановмісної сировини. Наразі ця задача є актуальною, має наукову і практичну значимість для України, зважаючи на значні запаси титанових руд на її території та на стратегічну важливість титанової промисловості. У статті розглянуто основні традиційні способи переробки титанових руд, проаналізовано їх недоліки, серед яких слід відзначити високу енергоємність, потребу у великій кількості сірчаної кислоти, утворенню під час переробки шкідливих сірковмісних відходів та потребу в додаткових процесах очищення. Як перспективну альтернативу існуючим методам запропоновано застосування електричного розряду в рідині - високовольтного пробою рідкого проміжку між електродами, внаслідок чого в каналі розряду протягом малого проміжку часу виникає висока щільність енергії, різко підвищуються тиск і температура, відбувається швидке розширення каналу розряду, та випромінюється хвиля тиску у зовнішнє середовище. До переваг застосування електричного розряду слід віднести: екологічну безпечність, відсутність забруднень, можливість регулювання параметрів обробки. Було проведено експериментальні дослідження впливу електророзряду на пасту діоксиду титану та відходи виробництва титанових руд. Результати досліджень показали, що під впливом факторів, джерелом яких є електричний розряд у рідині, здійснюється зниження концентрації сірки: у пасті діоксиду до трьох разів, а у відходах виробництва титанових руд - до 2 разів. Одночасно з тим, під час електророзрядної обробки спостерігається подрібнення первинного матеріалу, про що свідчать результати аналізу гранулометричного складу. Дослідження різних режимів електророзрядної дії дозволило визначити раціональну питому енергію впливу - подальше підвищення питомої енергії впливу не призводить до суттєвих змін концентрації сірки і розмірності частинок продукції. Отримані результати показують можливість та підтверджують перспективність використання електричного розряду у рідині для обробки продуктів сульфатного способу отримання діоксиду титану та відходів його виробництва з метою їх десульфатизації.

Ключові слова: діоксид титану, електророзрядна технологія, десульфатизація відходів.