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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

CHEMICAL ASPECTS OF THE VANADIUM 5-OXIDE EXTRACTION PROCESS FROM ALUNITE ORE

Speciality: 2303.01- Inorganic Chemistry

Field of science: Chemistry

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GENERAL DESCRIPTION OF THE WORK

Relevance of the topic and degree of development. Vanadium and its compounds are strategically important materials and play an important role in the development of steels, ion batteries, catalysts and many new products with high functional properties. The amount of vanadium produced worldwide is 116 thousand tons/year, which indicates a high demand for it. In particular, vanadium oxides are extensively used in energy storage systems, including as electrolytes in vanadium redox flow batteries (VRFBs) and as electrode materials in lithium-ion and sodium-ion batteries[1].

The expansion of vanadium's application areas and its high economic value in the international market make it urgent to explore new and alternative raw material sources for the production of the metal[2,3].

Vanadium is the 22nd most abundant element in the Earth's crust, with an average abundance of about $1.5 \cdot 10^{-2}$%. Although more than 65 minerals of this element are known, it is not found free in nature. Vanadium is mainly present in various minerals as isomorphic mixtures, which is one of the main factors that makes it difficult to extract it on an industrial scale.

At present, the industrial production of vanadium and its compounds is primarily based on the processing of vanadium-bearing titanomagnetite, uranium–vanadium sandstones, and high-grade vanadate ores. However, the progressive depletion of these conventional vanadium resources has intensified interest in the development of alternative feedstocks. Consequently, considerable research efforts have focused on the recovery of vanadium from secondary and

[1] Shin, J. New operating strategy for all-vanadium redox flow batteries to mitigate electrolyte imbalance / J.Shin, S. Jeongmin, K. Chanyoung [et al.] // Journal of Power Sources, -2022, -v. 526, -p. 231144.

[2] Kurniawan, K. A Review on the Metallurgical Recycling Process of Vanadium from Secondary Resources / K.Kurniawan, S.Kim, M.Bae [et al.] // Mineral Processing and Extractive Metallurgy Review, -2023, 45(1):-p..2243007

[3] Liu, T. Review on the separation and recovery technologies for vanadium and impurities in complex leaching solution / T. Liu, T. Liping, Z. Yimin [et al.] // Separation and Purification Technology, -2026, vol.387, -p.136709.

unconventional resources, including coal fly ash, petroleum coke, bauxite residues, industrial wastes, spent catalysts, and metallurgical slags, as sustainable and economically viable sources of this strategically important metal.

The content of vanadium (V) oxide in alunite ore varies between 0.04-0.05%. Since the production of clay-soil (Al_2O_3) belongs to the field of multi-tonnage processing, even the presence of a small amount of vanadium in the raw material can be considered a great potential source for its acquisition. The complex processing of alunite ore has been studied by a number of researchers, including G.V.Labutin, H.B.Shakhtakhtinski, E.A.Tagiyev, T.D.Israfilov and others[4]. However, since these approaches have a number of difficulties, such as the release of harmful gas-ash particles into the environment as a result of the combustion of alunite ore, contamination of the aluminate solution with foreign ions, its inability to be recycled, and the use of only rich ore in the studies, the processing of alunite ore in our studies was carried out directly with an alkaline solution without burning. Since the aluminate solutions obtained at this time are periodically returned to the process, the amount of vanadium in the solution gradually increases significantly. The extraction of vanadium during the complex processing of alunite leads to both the production of pure aluminum oxide and the recovery of the precious metal, as well as to an environmentally friendly waste-free technology.

The complex processing of alunite ore is a combined extraction method, which ensures the extraction of vanadium along with aluminum, potassium and other main components during processing. This, in addition to ensuring more efficient use of resources, creates conditions for reducing the volume of technogenic waste and minimizing harmful environmental impacts.

The main directions of the research are to study the chemistry of the existing forms of vanadium compounds in the products of alunite processing, to determine its location at each stage of the process and to find a new processing method that ensures the selective separation of V_2O_5. Today, the mining and metallurgical industry faces an

[4] Taghiyev, İ. Cost Effective Technology of Alunite Ore Processing / E.İ.Taghiyev, E.Taghiyev, L.Agayeva // International Journal of Chemistry , -2019, vol. 11,-p.36-41.

important task of obtaining valuable materials, including vanadium compounds with high physicochemical properties (vanadium (V) oxide, ferrovanadium, vanadium carbide, etc.) by creating innovative technologies. In this regard, the topic of the dissertation work solves an important scientific and technological problem, and is relevant by contributing to the development of efficient approaches for the recovery and use of vanadium from alternative raw material sources.

The object and subject of the research. The object of the research was alunite ore samples taken from the Zaylik alunite deposit located in the Dashkesan region of the Republic of Azerbaijan. The transformations occurring during the extraction of vanadium compounds from alunite ore, the mechanism of their formation, and the factors affecting the efficiency of the process are the subject of the research.

Aims and objectives of the research:

Purpose and objectives of the research: The aim of the present study is to develop an environmentally safe and efficient method for the production of high-purity V_2O_5 by investigating the chemical regularities governing the separation stages of vanadium-containing compounds in the form of selective concentrates during the complex processing of alunite ore.

To achieve the stated objective, the following tasks were formulated and accomplished:

- Investigation of the distribution of vanadium between aluminate solutions and alunite slurry during the dissolution of alunite ore in alkaline and acidic media according to the major components, as well as the study of the kinetics of vanadium transfer into solution upon adjustment of the aluminate solutions to the required alkali concentration;

- Study on the optimization of vanadium extraction from alunite ore into aluminate solutions through regression modeling;

- Study of phase transformations occurring in the V_2O_5-H_2SO_4-SO_3-H_2O system obtained during sulfation of alunite slurry and investigation of thermal decomposition of the obtained vanadyl sulfate compound ($VOSO_4 \cdot 3H_2O$) by physicochemical analysis methods;

- The study aimed to determine the optimal conditions for the precipitation of vanadium from aluminate solutions in the form of

ammonium metavanadate and ammonium polyvanadate, to investigate the phase composition of the obtained precipitates using physicochemical characterization methods, and to evaluate the kinetic parameters of the precipitation process;

- Synthesis of an iron-modified activated carbon (Fe–AC) nanocomposite, adsorption recovery of vanadium from alunite-processing solutions using the Fe–AC nanocomposite and determination of the thermodynamic parameters of the adsorption process based on the Langmuir and Freundlich isotherm models.

Research methods. During the course of the study, X-ray diffraction (XRD), X-ray fluorescence (XRF), infrared spectroscopy (IR), differential thermal analysis (DTA), thermogravimetric analysis (TGA), scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS) and photometric analysis methods were employed.

The main provisions of the defense.

- Investigation of the effect of various factors (temperature, time, ratio of solid:liquid phases, mixing speed, alkali concentration) on the release of vanadium into the solution during the alkaline processing of raw alunite ore and statistical analysis of the process using the mathematical regression method;

- Precipitation of vanadium from aluminate solutions in the form of ammonium metavanadate and ammonium polyvanadate compounds, followed by the production of high-purity V_2O_5.Finding the conditions for the precipitation of vanadium in the form of ammonium metavanadate, ammonium polyvanadate compounds and obtaining pure V_2O_5;

- Investigation of the conditions for the transfer of vanadium into the solution during the sulfation of alunite slurry, thermolysis of the $VOSO_4 \cdot 3H_2O$ compound;

- Adsorption of vanadate ions from aluminate solutions and solutions obtained from the processing of alunite slurry with iron activated carbon (Fe-AC) nanocomposite, study of the main factors affecting adsorption, selection of optimal conditions.

Scientific novelty.

-The optimum conditions for the precipitation of vanadium from aluminate solutions in the form of ammonium metavanadate

(NH_4VO_3), ammonium polyvanadate ($(NH_4)_2V_6O_{16}$) and calcium pyrovanadate ($Ca_2V_2O_7$) were determined. The kinetic parameters and mechanism of the precipitation process were investigated.

- New intermediate compounds formed during the thermal decomposition of vanadylsulfate ($VOSO_4 \cdot 3H_2O$), obtained in the V_2O_5–H_2SO_4–SO_3–H_2O system, were identified within the temperature range of 100–700°C. These compounds include $VOSO_4 \cdot 2H_2O$, $VOSO_4 \cdot H_2O$, $VOSO_4$, $(VO_2)_2SO_4$.

- The adsorption conditions for vanadium recovery from solutions obtained during alunite ore processing using the Fe–AC nanocomposite were investigated, and the main factors affecting the sorption process, including sorbent dosage, pH, initial vanadium concentration, temperature, and contact time, were determined. Langmuir and Freundlich adsorption isotherms were constructed, and the thermodynamic parameters of the sorption process were evaluated. The adsorption models demonstrated that both the Langmuir and Freundlich equations adequately describe the adsorption characteristics.

- A technological flowsheet for the production of V_2O_5 was proposed based on the investigation of the chemical regularities governing vanadium enrichment during the complex processing of alunite ore.

Theoretical and practical significance of the research: The theoretical significance of the research lies in the scientific substantiation of vanadium recovery from aluminate solutions and alunite sludge during the complex processing of alunite ore.

Kinetic and thermodynamic quantities of processes, constants characterizing isotherms, thermal decomposition products of vanadyl sulfate obtained from the V_2O_5-H_2SO_4-SO_3-H_2O system and adsorption results with Fe-AC nanocomposite can be used as material in international information systems.

The practical significance of the work is that, as a result of the conducted research, the recovery of gallium and vanadium alongside aluminum during the complex processing of aluminate solutions was achieved. Based on these results, a patent entitled “Method for the Extraction of Gallium and Vanadium from Aluminate Solutions” was granted by the Intellectual Property Agency of the Republic of Azerbaijan (Patent No. I 2023 0073).

The results of the research may be proposed for implementation in the technology of complex processing of alunite ore at the Ganja Alumina Plant. The regularities established in this study may also be applied to the processing of other vanadium-containing materials.

Approbation and implementation: A total of 17 scientific works have been published on the subject of the dissertation, including 6 journal articles (4 of which were published in journals indexed in the WOS and Scopus international databases), 1 patent and 10 conference proceedings.

The materials of the dissertation were presented and discussed at the following scientific conferences:

Proceedings of the 10th İnternational Scientific and Practical Conference Scientıfıc research in XXI century Ottawa, Canada (11-12 noyabr 2021); The 21st ICS International Chemistry Congress, Aserbaijan Shahid Madani University, Tabriz, Iran (26-28 iyul 2022); Azerbaıjan; VI İnternational scientıfıc Conference of Young Researchers, BMU. Bakı, Azərbaycan (29-30 aprel 2022; 28-29 april 2023; 26-27 aprel 2024); Ümummilli lider Heydər Əliyevin anadan olmasının 99-cu ildönümünə həsr olunmuş Tələbə və Gənc tədqiqatçıların III Beynəlxalq elmi konfransları, BANM, Bakı, Azərbaycan (18-19 aprel 2022; 12 aprel-3 may 2023; 23 aprel-1 may 2024; 6 dekabr 2024); BMU Azərbaycanda su ehtiyyatları: Problemlər və yeni çağırışlar AZTU, Bakı, Azərbaycan (29 oktyabr 2024).

Name of the organization where the dissertation has been performed: The dissertation work was carried out in the laboratory "Processing of non-ferrous and ferrous metal-containing mineral raw materials" of the ARETN Institute of Chemistry, Department of Chemistry (formerly the laboratory "Processing of non-ferrous metal-containing mineral raw materials" of the M. Naghiyev Institute of Catalysis and Inorganic Chemistry).

Structure and scope of the dissertation. The total volume of the dissertation is 171 pages. The dissertation consists of an introduction (12149), 5 chapters (Chapter I 51505, Chapter II 16424, Chapter III 38520, Chapter IV 16530, Chapter V 34129), conclusions (2525 characters), 59 figures, 15 tables, 2 schemes and a list of 176 references, and concludes with abbreviations, symbols and appendices.

Personal contribution of the author. The author independently carried out all laboratory experiments conducted within the framework of the dissertation research.

The processing and interpretation of the experimental results, their presentation at scientific conferences, the preparation of scientific articles for publication, and the writing of the dissertation were performed directly by the author.

Main Content of the Work

The introduction substantiates the relevance of the research topic, defines the aim of the study and the tasks required to achieve the stated objective, presents the main scientific propositions submitted for defense and discusses the scientific novelty and practical significance of the work.

The first chapter of the dissertation analyzes the main vanadium minerals, reserves and the current state of vanadium production. In addition, this chapter provides information on the properties of vanadium and the chemistry of its most important compounds, the ionic species present in solutions, as well as methods for its separation by precipitation, adsorption, and extraction techniques.

At the end of the chapter, information is provided on the extraction of vanadium from various types of raw materials, including industrial waste, titanomagnetites, coal, fuel oil combustion ash and bauxites.

Analysis of the reviewed literature showed that, despite the existence of many methods and technologies for the extraction of aluminum and other valuable components (fertilizers, alkalis, and acids) from aluminum-containing ores such as bauxite, alunite, and nepheline, the concentration and accumulation stages occurring during the extraction of vanadium from alunite ore have not yet been comprehensively investigated. In order to obtain vanadium-containing products with high recovery efficiency, it is necessary to study the chemistry, mechanism, and kinetic regularities of the transformations taking place during the processing stages of alunite ore.

The **second chapter**, this section is devoted to the materials and methods used in the study and provides information on the chemical and mineralogical composition of the investigated alunite

ore, the determination of vanadium by various analytical methods, and the adsorption of vanadium ions from solutions

The investigations were carried out using an average ore sample delivered to the Ganja Alumina Plant from the Zaglik alunite deposit for processing. According to the results of X-ray fluorescence analysis, the chemical composition of the ore sample was as follows (wt.%): Al_2O_3 – 26.44; SiO_2 – 33.45; Fe_2O_3 – 3.85; TiO_2 – 0.45; Cr_2O_3 – 0.41; K_2O – 5.21; Na_2O – 1.85; SO_3 – 27.15; V_2O_5 – 0.0487; Ga_2O_3 – 0.003; MnO – 0.005; CaO – 0.07; As_2O_3– 0.005; SrO – 0.3; P_2O_5 – 0.215; F – 0.29 and H_2O (moisture) – 0.25.

The results of X-ray phase analysis indicate that the ore contains the minerals alunite (55.1%), kaolinite (dickite) (12.3%), quartz (25.2%) and hematite (7.4%).

The **third chapter** of the dissertation is devoted to the investigation of the conditions for the extraction of vanadium into solution during the alkaline leaching of alunite ore.

In the present study, the processing was carried out by monitoring the dissolution dynamics of vanadium contained in alunite ore into the solution. It was established that, without a roasting stage, a 5% alkaline solution at 95°C decomposed up to 92.0% of the alunite mineral within 30 minutes, while the decomposition degree reached 96.6% after 60 minutes. Under the same conditions, a 10% alkaline solution was capable of decomposing up to 98% of the alunite mineral.

Among the minerals present in alunite ore, alunite is the one that dissolves most readily in alkaline solutions.

$$K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 2Al_2O_3 \cdot 6H_2O + 12NaOH = 3(Na_2O \cdot Al_2O_3) + 3Na_2SO_4 + K_2SO_4 + 12H_2O \quad (1)$$

The crystal structure of the minerals (alunite, kaolinite, hematite, quartz) in alunite ore cannot be completely decomposed with ordinary solvents (water and acids). It is impossible to extract vanadium from alunite by processing it in water, but it is possible to extract up to 10% of vanadium into the solution by dissolving it with HCl and HNO3, and up to 57.14% with sulfuric acid. The main difficulties encountered during the acid treatment of alunite ore are the contamination of the aluminate solution with iron ions, the inability to recycle the solution back into the process, and the achievement of more favorable results

only when processing higher-grade ores. Taking these factors into consideration, the processing of alunite ore was mainly carried out using alkaline solutions. In experiments, the dynamics of vanadium release into the solution was studied by changing various parameters (alkali concentration, temperature, solid: liquid phase ratio, time).

It was found that when the mixing speed during processing was increased above 700 rpm, the concentrations of Al and V in the solution did not change, therefore, mixing was mainly carried out at this speed in experiments. In the studies, the optimal solid:liquid phase ratio was taken as 1:6.

The dissolution dynamics of vanadium into solution were investigated by different parameters, including alkali concentration, temperature, solid-to-liquid ratio and leaching time.

The alkali concentration, temperature, and solid-liquid ratio significantly affect the extraction efficiency of vanadium into solution. It was found that treatment of alunite ore with a 10% NaOH solution at a solid-to-liquid ratio of 1:6 for 2 hours at 60°C resulted in the extraction of 50.2% of gallium and 49.23% of vanadium into solution. When the temperature was increased to 90°C, the extraction efficiencies increased to 90.1% for gallium and 71.3% for vanadium. The results of the experiments are presented in Table 1.

Table 1

Effect of temperature on the extraction of vanadium and gallium (C_{NaOH} = 10%, τ = 2 h, S:L = 1:6, $m_{alunite}$=50q) [9]

Temperature	50 ^{0}C	60 ^{0}C	70 ^{0}C	80^{0}C	90 ^{0}C
The amount of Ga in the solution is mg/l	2.2	2.68	3.16	3.7	4.1
Percentage of separation of Ga, %	48.6	50.2	70	82	90.1
The amount of V in the solution is mg/l	19.9	25.84	30.92	36.93	42.68
V separation, V, %	36.2	49.23	58.9	69.2	71.30

An analysis of the dissolution dynamics of aluminum, gallium, and vanadium from alunite ore at different leaching times shows that when alunite ore is leached in a 10% sodium hydroxide solution at 90°C for 1

hour, the extraction of aluminum reaches 98%, while the extraction efficiencies of gallium and vanadium are 65% and 52.4%, respectively.

Increasing the leaching time to 2 h causes no significant change in aluminum extraction, while gallium and vanadium extraction increase to 90.1% and 71.3%, respectively. Vanadium transfer into solution during alunite ore processing was also predicted using the **mathematical regression method.** This approach made it possible to determine the optimum alkali concentration, temperature, and leaching time for maximum vanadium extraction while reducing time and material consumption. Based on the regression equation, statistical analysis was performed, and the average approximation error and root mean square deviation were calculated.

A three-factor experimental design was employed in the study ($N = 2^k = 8$). The effects of three factors on vanadium extraction efficiency were investigated: Z_1 – NaOH concentration (5–10%), Z_2 – solution temperature (40–90°C), and Z_3 – leaching time (30–120 min).

After verifying the reproducibility of the experiments and calculating the variance, the regression coefficients were determined using the matrix method. The regression equation was expressed as follows:

$$Y = 15.9564 - 0.23X_1 + 0.231X_2 + 0.03429X_3 \qquad (1)$$

It was established that the coefficient X_1 indicates that, beyond an alkali concentration of 10%, each 1% increase in concentration decreases vanadium extraction by 0.23%. The coefficient X_2 shows that a 10°C increase in temperature increases vanadium extraction by 0.23%, whereas the coefficient X_3 indicates that each additional minute of leaching time increases vanadium extraction by 0.034%. At alkali concentrations above 10%, vanadium extraction decreases because undissolved silicon and iron oxides increase, promoting vanadium sorption onto their active sites. Several methods, including adsorption, solvent extraction, electrolysis, and precipitation, have been proposed for vanadium recovery from complex technological solutions. For aluminate liquors, the selected method must recover vanadium without affecting aluminum, the main product. The aluminate solution ($K_m = 1.88$; $Al_2O_3 = 74.5$ g/L; $Na_2O = 85.2$ g/L; $Ga_2O_3 = 0.124$ g/L; $V_2O_5 = 0.678$ g/l) contains aluminum together with valuable vanadium and gallium. Therefore, this study investigated the

simultaneous recovery of Al, V, and Ga based on the principle of integrated processing. The method entitled "Recovery of Ga and V from aluminate solutions," developed by the authors, has been patented by the Intellectual Property Agency (Patent No. I 2023 0073) [7]. According to this method, aluminum, then gallium and then vanadium are separated from the recirculated, concentrated aluminate solutions in the form of concentrates. By gradually adding a 1 N sulfuric acid solution to the aluminate liquor, aluminum hydroxide was first precipitated, followed subsequently by the precipitation of gallium hydroxide.

$$Na[Al(OH)_4] + H^+ \rightarrow Al(OH)_3\downarrow + H_2O + Na^+ \text{ (pH=12.8-9.5)} \quad (2)$$

$$Na[Ga(OH)_4] + H^+ \rightarrow Ga(OH)_3\downarrow + H_2O + Na^+ \text{(pH=9.5-7.0)} \quad (3)$$

$Al(OH)_3$ and $Ga(OH)_3$ precipitate at pH=9.5 and pH=7.0, respectively, at 90%. At this pH value, vanadates present in the solution practically do not precipitate and therefore accumulate and become concentrated in the aluminate liquor.

The mother liquor separated from aluminum and gallium compounds consists of sodium vanadate, sodium sulfate, and potassium sulfate salts. First, the solution containing these components is heated to boiling point (125^0C-130^0C) and cooled to 30-40^0C. At this time, Na_2SO_4 and K_2SO_4 salts crystallize together and are separated from the vanadate solutions. After evaporation, the concentration of vanadium in the solution varies between 1.5-2g/l (V_2O_5). By precipitating vanadium from these solutions with slaked lime, a concentrate in the form of calcium pyrovanadate is obtained.

The optimal process conditions were determined as follows: initial vanadium concentration, $C_v(V_2O_5)$ = 1.5 g/l; temperature, 80–95°C; reaction time, τ = 2 h; pH = 10–11 and molar ratio CaO/V_2O_5 = 10:1. Under these conditions, approximately 95% of vanadium precipitated in the form of calcium pyrovanadate.

The kinetics of the precipitation process were investigated and the activation energy was calculated to be E_a = 14.6 kJ·mol^{-1}. This relatively low value indicates that the process proceeds under diffusion-controlled conditions. The individuality and phase purity of the synthesized calcium pyrovanadate were confirmed by X-ray diffrac-

tion analysis, corresponding to PDF (01-070-0942)(Figure 1).

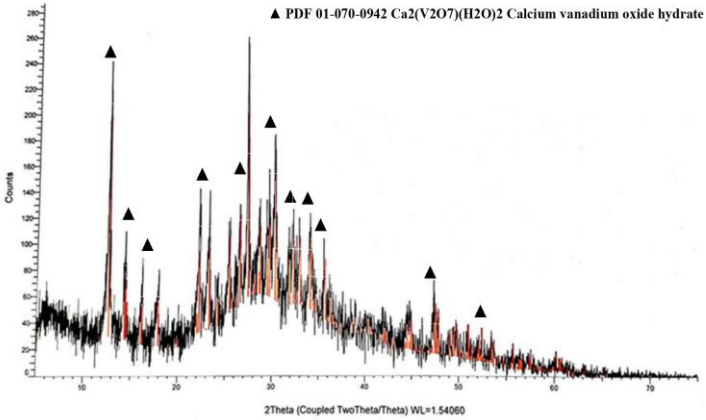

Figure 1. XRD pattern of calcium pyrovanadate [17]

The fourth chapter investigates the precipitation of vanadium as ammonium salts from aluminate solutions obtained during alunite ore processing. Precipitation with ammonium sulfate was studied in both alkaline and acidic media. Experimental results showed that a solution containing 6.95 g/L $NaVO_3$ forms white ammonium metavanadate (NH_4VO_3) at 25°C and pH 7–9, whereas red-brick ammonium polyvanadate ($(NH_4)_2V_6O_{16}$) precipitates at 90–95°C and pH 2. The precipitation isotherms of meta- and polyvanadates as a function of solution pH are presented in Figure 2.

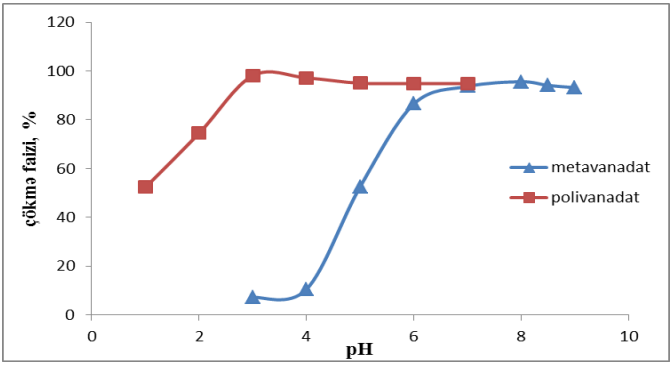

Figure 2. Effect of solution acidity (pH) on the degree of vanadium precipitation in the form of ammonium metavanadate and polyvana-

date (C_{NaVO_3} = 6.95g/l, $(NH_4)_2SO_4$:V_2O_5 = 3,0-4,0:1) [8]

Due to the presence of alkali metal sulfates in the solutions obtained from the processing of alunite ore, precipitation was carried out not with NH_4Cl (to avoid introducing extraneous Cl^- ions into the solution), but with a $(NH_4)_2SO_4$ solution. The precipitation reactions are expressed as follows:

$$2NaVO_3 + (NH_4)_2SO_4 = 2NH_4VO_3 + Na_2SO_4 \quad (4)$$

To determine the optimum precipitant dosage, different amounts of ammonium sulfate were added to sodium vanadate solutions. Under optimum conditions (12% $(NH_4)_2SO_4$, pH 8, 25°C, 2 h), 97% of vanadium precipitated as ammonium metavanadate (NH_4VO_3). Acidification of the resulting NH_4VO_3 suspension with sulfuric acid at 90°C and pH 2 converted ammonium metavanadate into ammonium polyvanadate through the formation of hexavanadates (polyvanadates):

$$6NH_4VO_3 + 2H_2SO_4 \rightarrow (NH_4)_2V_6O_{16} + 2(NH_4)_2SO_4 + 2H_2O \quad (5)$$

The diffractograms of the obtained ammonium metavanadate and ammonium polyvanadate compounds are given in Figure 3.

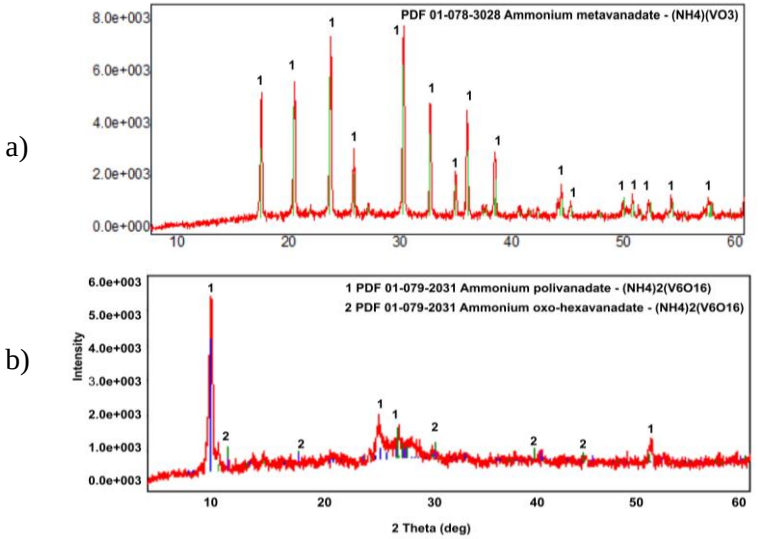

Figure 3. The X-ray diffraction patterns of the precipitates obtained at

different solution acidity: pH=8 (a) and pH=2 (b) [8]

The diffraction peaks of the precipitate obtained at pH=8 correspond to the orthorhombic structure of the ICPDC database [01-078-3028] card, and the diffraction peaks of the precipitates obtained at pH=2 correspond to the [010792031] card. The obtained precipitates were characterized by both SEM-EDS and IR spectroscopy analysis methods.

In subsequent studies, the effect of temperature on the precipitation of vanadium in the form of NH_4VO_3 was studied. The results of experiments conducted at different temperatures (20^0C, 30^0C, 40^0C) as a function of time are given in Figure 4.

Increasing the temperature from 20^0C to 40^0C causes a decrease in the percentage of vanadium precipitation in the form of ammonium metavanadate. If 91% of the vanadium in the solution precipitates in the form of NH_4VO_3 within 3 hours at 20^0C, then increasing the temperature to 40^0C under the same conditions reduces the percentage of vanadium precipitation to 70%. A comparison of the water solubility of both salts showed that ammonium polyvanadate hydrolyzes less in water than ammonium metavanadate. Thus, when studying the temperature dependence of the solubility of NH_4VO_3 in water, it was determined that 0.48 g at 20^0C, 0.84 g at 30^0C, and 1.32 g and 2.42 g at 40^0C and 60^0C, respectively, dissolve in 100 ml of water. Ammonium polyvanadates pass into the solution as a solution at a concentration of 0.17 g/l at 20^0C and 0.33 g/l at 95^0C. This increases the importance of precipitation in the form of ammonium polyvanadates.

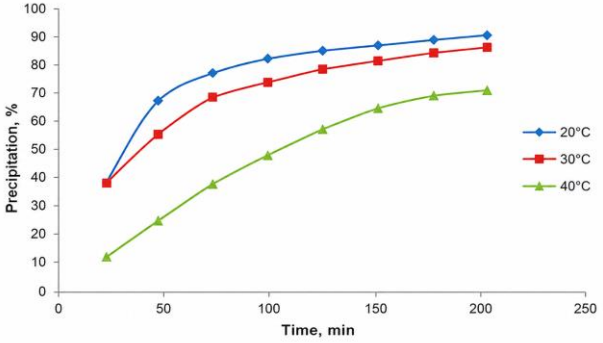

Figure 4. Effect of mixing time on the degree of vanadium precipitation at different precipitation temperatures:

20 ^{0}C, 30^{0}C, 40^{0}C (NaVO3 - 6.95 g/L; $(NH_4)_2SO_4/V_2O_5$ = 2) [8]

During the process, the kinetic parameters of precipitation were calculated using the Avram kinetic equation:

$$\alpha = 1 - e^{-k\tau^n} \quad (2)$$

Here α - is the percentage of vanadium deposition depending on time, k - is the reaction rate constant, τ - is the duration of the precipitation, n - is the kinetic parameter characterizing the reaction sequence. When n<0.5-1, the diffusion process; when n>1, the kinetic process is characterized.

The dependence of the reaction rate constant on temperature is expressed by the Arrhenius equation:

$$k = Ae^{-E_a / RT} \quad (3)$$

The activation energy value was calculated based on the tangent of the slope of the straight line obtained from the graph of the reaction rate constant versus temperature (Figure 5).

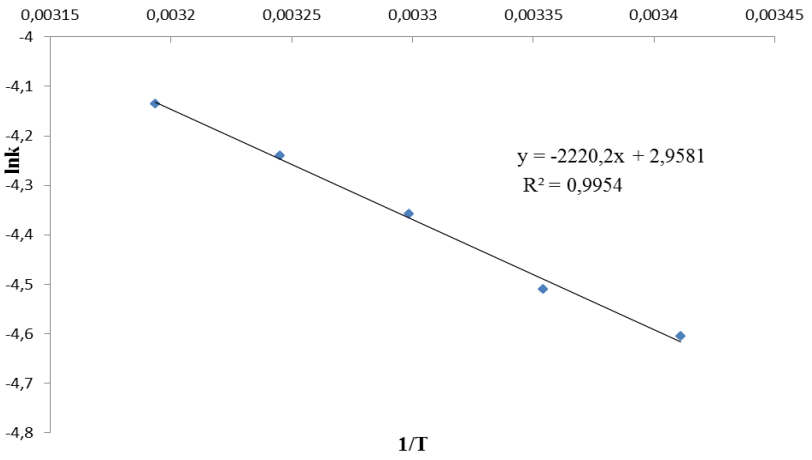

Figure 5. Relationship dependence of the reaction rate constant [12]

The calculated activation energy (18.4 kJ/mol) indicates that precipitation is diffusion-controlled. Such processes are characterized by low temperature dependence (activation energy <20 kJ/mol) and

are independent of mixing. The precipitate was separated by filtration, dried, and thermally decomposed in a muffle furnace at 550°C to obtain V_2O_5.

$$2NH_4VO_3 \xrightarrow{t} 2NH_3+V_2O_5+H_2O \quad (6)$$

The EDS analysis confirmed the high purity of the obtained V_2O_5 product, which contained 99.52 wt.% V_2O_5. The remaining 0.48 wt.% consisted of trace impurities, including Si (0.005 wt.%), Fe (0.08 wt.%), P (0.01 wt.%), S (0.007 wt.%), As (0.006 wt.%), and K_2O + Na_2O (0.15 wt.%).The individuality of the precipitate was confirmed by X-ray diffraction analysis (PDF 01-072-0433).

In the **fifth chapter**, the extraction of vanadium from alunite slurry was investigated. Alunite slurry has the following chemical composition, %: K_2O-0.07, Na_2O-0.23, CaO-0.28, Al_2O_3-4.53, SiO_2-82.2, Fe_2O_3-8.60, MgO-0.17, MnO-0.01, P_2O5 – 0.24, SO_3-0.15, V_2O_5-0.099, Ga – 0.005, TiO_2- 1.15, Rb-0.0012. During alunite ore processing, dickite and hematite remain undecomposed and concentrate in the slurry. As vanadium is isomorphously substituted for aluminum in the dickite lattice, it is also enriched in the slurry. To convert vanadium into a soluble form, the slurry is sulfated, causing dickite decomposition and transformation of vanadium into vanadyl sulfate.The sample was mixed with concentrated sulfuric acid (ρ = 1.84 g/cm³) at a sample-to-acid mass ratio of 2:1 and dried in a drying oven at a temperature of 130^0C for 12 hours.

At this time, the vanadium compound in the slurry is converted into vanadyl sulfate by the action of concentrated sulfuric acid and sulfur gases:

$$V_2O_5+SO_2+H_2SO_4 \rightarrow 2VOSO_4+H_2O \quad (7)$$

The sulfated mass is then burned in a muffle furnace at a temperature of 550^0C. During sulfation, sulfates of iron, aluminum, titanium and vanadium compounds contained in the alunite slurry are obtained. During 550^0C burning, iron, titanium and partly aluminum sulfates decompose from the oxides contained in that mass and accumulate in the residue in the form of oxides. When the sulfated

mass is dissolved in water, vanadium easily passes into solution.

The results of the X-ray analysis of the blue crystalline precipitate obtained in the V_2O_5-H_2SO_4-SO_3-H_2O system prove that it is $VOSO_4 \cdot 3H_2O$ (PDF 01-072-0912) (Figure 6).

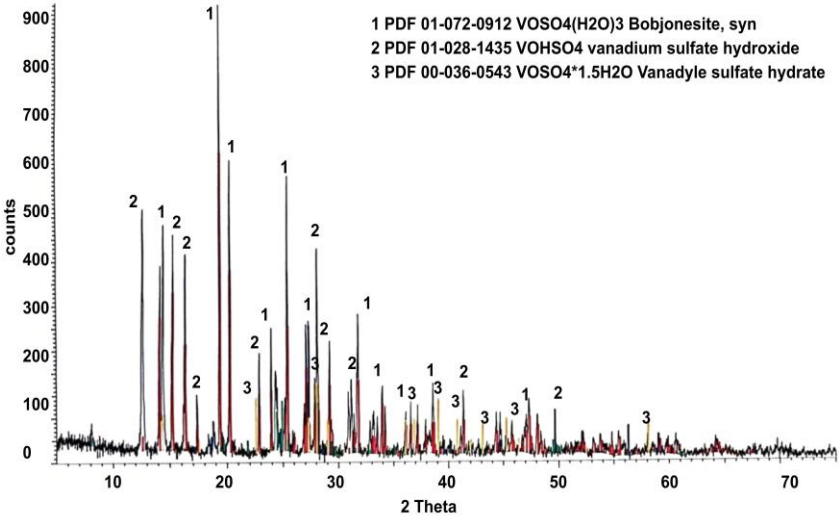

Figure 6. XRD pattern of $VOSO_4 \cdot 3H_2O$ freshly precipitated in the V_2O_5-H_2SO_4-SO_3-H_2O system [6]

The phase composition of the initial substance and its thermal decomposition products was confirmed by IR spectroscopy (Fig. 7, a). As can be seen from the IR spectrum, the precipitate consists of the H_2O molecule (valence absorption band at 3592 cm^{-1} and deformation absorption band at 1651 cm^{-1}) and valence and deformation absorption bands at 1154 cm^{-1} corresponding to the SO_4^{2-} ion and 428.9 cm^{-1} corresponding to the SO_4^{2-} ion (Fig. 7, b, c, d). The absorption band at 721 cm^{-1} observed in all samples corresponds to the valence vibration of the asymmetric bond of V-O-V.

The thermal decomposition of the $VOSO_4 \cdot 3H_2O$ compound is stepwise and is observed in DTA with endoeffects at t= 130; 217; 424; 520 and 600^0C with weight losses (Figure 8).

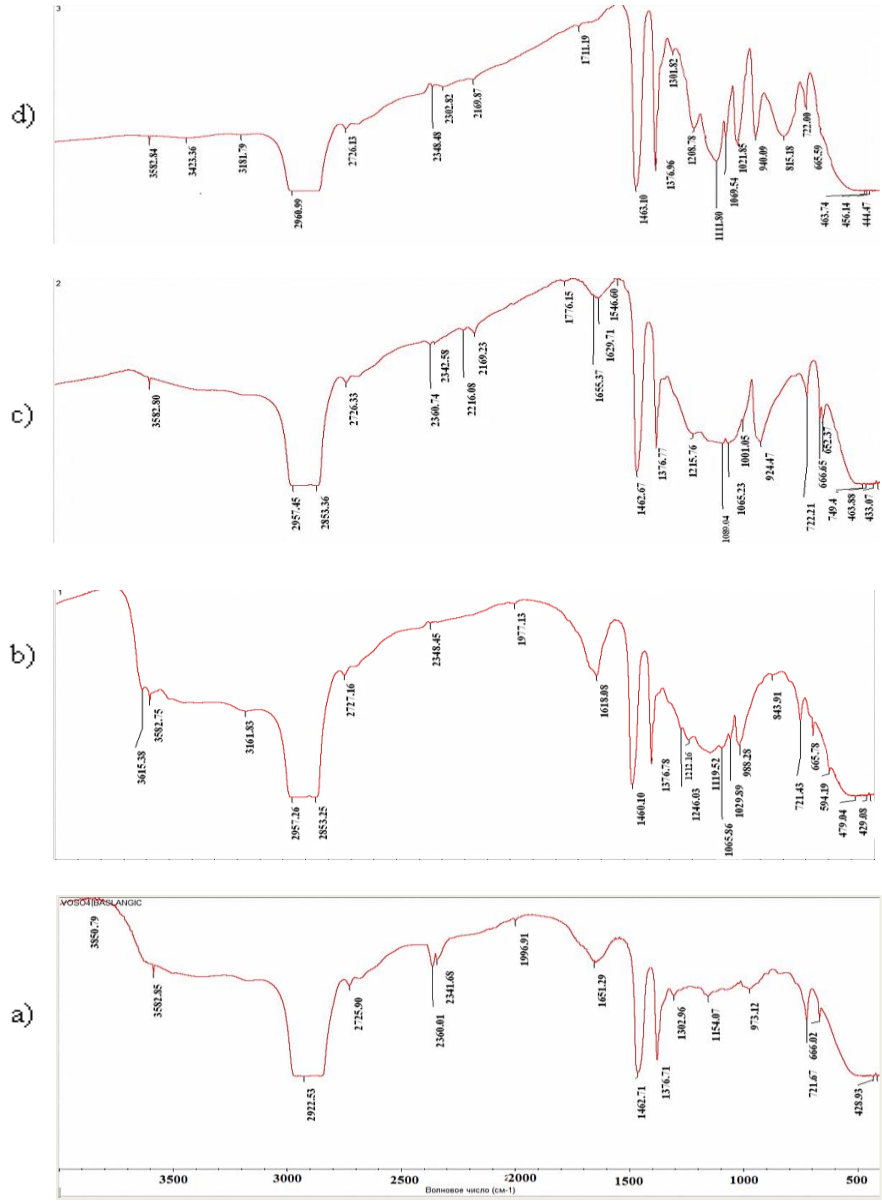

Figure 7. IRspectra of $VOSO_4 \cdot 3H_2O$ (a); products of its thermolysis at 220^0C (b); 400^0C (c); 600^0C (d) [5]

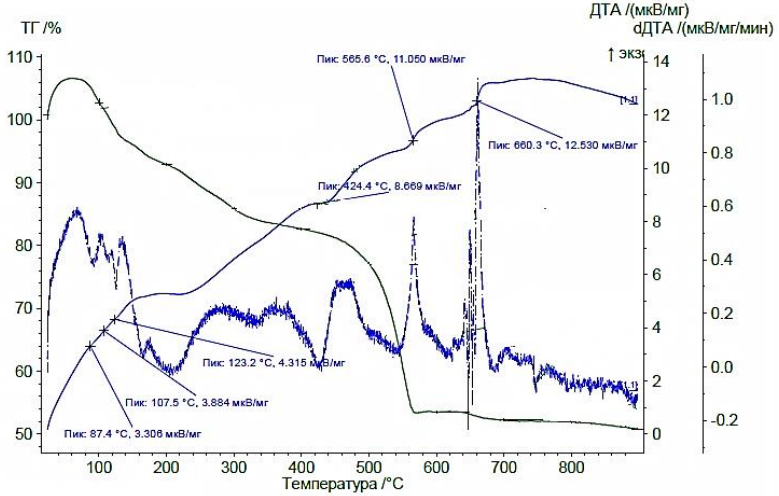

Figure 8. DTA and TG data for $VOSO_4 \cdot 3H_2O$ (m=5.6 mg) [5]

The loss of water of crystallization occurs in three stages: the first stage at 130^0C, the second at 217^0C, and the third stage at 424^0C. In the temperature range of 130-217^0C, 1 molecule of water is released from the $VOSO_4 \cdot 3H_2O$ compound. According to the TQ curve, the mass loss at this stage is 11.59% of the total mass (m=0.65mg):

$$VOSO_4 \cdot 3H_2O \xrightarrow{t} VOSO_4 \cdot 2H_2O + H_2O \ (t \leq 217^0C) \qquad (8)$$

In the second stage (217-400^0C), one more water molecule is separated from the $VOSO_4 \cdot 2H_2O$ compound. At this time, according to the TG curve, the total mass loss is 1.28 mg (0.63 mg in the second stage). This is a mass loss corresponding to 21.81% of the total mass (theoretical 22.08%).

$$VOSO_4 \cdot 2H_2O \xrightarrow{t} VOSO_4 \cdot H_2O + H_2O \ (t \leq 400\ ^0C) \qquad (9)$$

Below 500^0C, the water in the compound is completely separated (0.61 mg in the third stage). The total loss up to 500^0C (0.65+0.63+0.61=1.89 mg) is 1.89 mg or 33.17%, which corresponds to the separation of 1 more mole of water (theoretically 33.129%):

$$VOSO_4 \cdot H_2O \xrightarrow{t} VOSO_4 + H_2O \ (t \leq 500^0C) \qquad (10)$$

In the IR spectrum of this stage, the bands corresponding to the deformation vibrations of the water molecule (220^0C-1618cm^{-1}, 400^0C-1629cm^{-1}) are fully visible, but the formation of the $VOSO_4$ phase (924cm^{-1} band) also occurs. This band corresponds to the valence vibration of the V=O group. While a broad band characteristic of the SO_4^{2-} group with a frequency of 1111.8cm^{-1} is observed in the IR spectrum of the product obtained without thermal decomposition at 600^0C, the band corresponding to the water molecule disappears, which differs from the IR spectrum of the sample obtained without decomposition at 400^0C.

In the $VOSO_4 \cdot 3H_2O$ sample, a rapid weight loss is observed at 500^0C and vanadyl sulfate is converted to vanadium oxosulfate ($V_2O_4(SO_4)$).

$$2VOSO_4 \xrightarrow{t} (VO_2)_2SO_4 + SO_2 \ (>500^0C) \qquad (11)$$

The formation of vanadium oxosulfate is confirmed by the IR spectrum. In the compound formed at 600^0C, a broad-band valence vibration characteristic of the ($SO4^{2-}$) group (~1069cm^{-1}) and a band characteristic of the $(VO_2)_2SO_4$ compound (~815.18cm^{-1}) can be observed. In the thermolysis products obtained at 700^0C, the absorption band characteristic of the SO_4^{2-} group disappears, but a band characteristic of V_2O_5 is formed in the sample. The total mass loss at the end of the thermolysis is practically 50.73%.

$$(VO_2)_2SO_4 \xrightarrow{t} V_2O_5 + SO_2 + 1/2\ O_2 \ (>600^0C) \qquad (12)$$

Thus, it can be said that the decomposition of the compound $VOSO_4 \cdot 3H_2O$ is a multi-step process, which proceeds with the formation of the following intermediate products:

$$VOSO_4 \cdot 3H_2O \xrightarrow{130^0C} VOSO_4 \cdot 2H_2O \xrightarrow{217^0C} VOSO_4 \cdot H_2O \xrightarrow{424^0C} VOSO_4 \xrightarrow{500^0C} (VO_2)_2\,SO_4 \xrightarrow{700^0C} V_2O_5 \qquad (13)$$

As a result of the conducted studies, it was determined that the precipitate obtained in the V_2O_5-H_2SO_4-SO_3-H_2O system corresponds

to the formula $VOSO_4 \cdot 3H_2O$ and its thermal decomposition occurs in several stages. $VOSO_4 \cdot 3H_2O$ dehydration occurs in 3 stages, and $VOSO_4$ decomposition occurs in two stages.

Adsorption of vanadium by iron with activated carbon nanocomposite (Fe-AC). During the experimental studies, an Fe-AC nanocomposite synthesized from activated carbon and iron(II) sulfate was used as the adsorbent. Incorporation of FeOOH (goethite) into the activated carbon matrix enhanced its sorption capacity, while the combined effect of the FeOOH active sites and the porous carbon structure resulted in high vanadium adsorption. XRD analysis confirmed the presence of the goethite phase, with diffraction peaks matching the standard FeOOH pattern (PDF 01-074-3080) (Fig. 9).

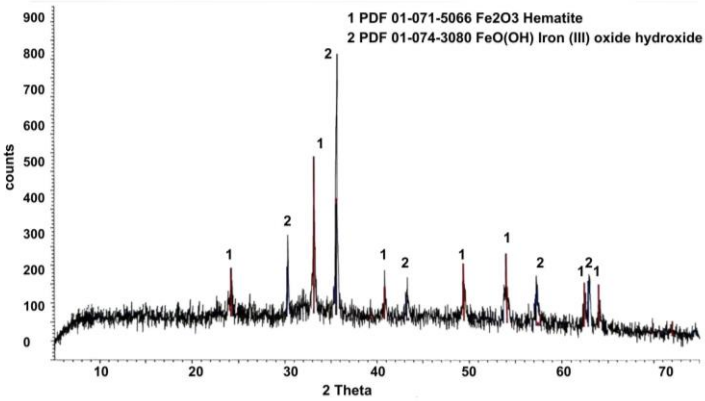

Figure 9. XRD pattern of Fe-AC nanocomposite [2]

Comparison of the obtained experimental results shows that the sorption capacity of the synthesized Fe-AC nanocomposite is higher than that of activated carbon. Therefore, the experiments were continued with this sorbent. Adsorption experiments were statistically carried out using the Fe–AC nanocomposite in vanadate solutions with different initial concentrations (C_0= 25–200 mg/L). The equilibrium sorption capacity (q_e) and sorption efficiency (R, %) were calculated using Eqs. (4) and (5), respectively:

$$q_e = (C_0 - C_e)\ \frac{V}{m} \qquad (4)$$

$$R = \frac{C_0 - C_e}{C_0} \cdot 100\% \quad (5)$$

where q_e is the equilibrium sorption capacity (g/g or mol/g); C_0 and C_e are the initial and equilibrium concentrations of vanadium in the solution (g/l), respectively; V is the volume of the solution (l); and m is the mass of the sorbent (g).

One of the main factors affecting vanadium adsorption is the pH of the solution. Figure 10 shows the dependence of vanadium adsorption by activated carbon (AC) and iron-activated carbon (Fe-AC) nanocomposite on the pH value of the medium.

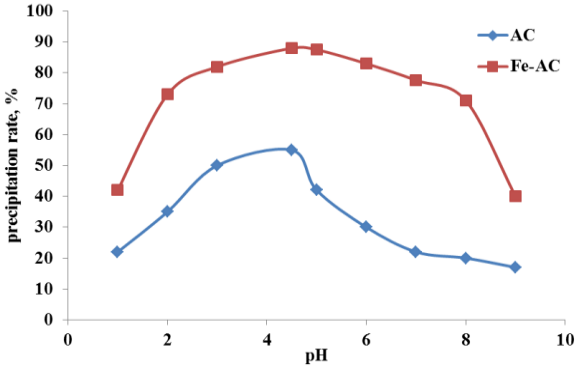

Figure 10. Effect of pH on vanadium adsorption results using AC and Fe-AC on the pH of the medium (C_v=100 mg/l, m_{ads}=0.25 g, T=298K, τ=180 min.) [2]

The Fe-AC nanocomposite exhibited a sorption capacity of 110 mg/g, significantly higher than that of activated carbon (39 mg/g), owing to the presence of FeOOH active sites. As shown in Figure 10, maximum vanadium adsorption occurred at pH 2.5–4.5, where vanadium predominantly exists as polyanions ($H_2V_{10}O_{28}^{4-}$ and $HV_{10}O_{28}^{5-}$). At pH 4.5, adsorption was enhanced by electrostatic attraction between negatively charged vanadium species and positively charged adsorbent sites, leading to Fe–O–V bond formation. At pH >6, adsorption decreased because of the lower affinity of vanadate anions and competition from OH^- ions. Under optimum conditions (50–100 mg/L V, pH 4.5), the adsorption efficiency reached 86%. The SEM–EDS analysis of the adsorbent after adsorption is presented in Figure 11.

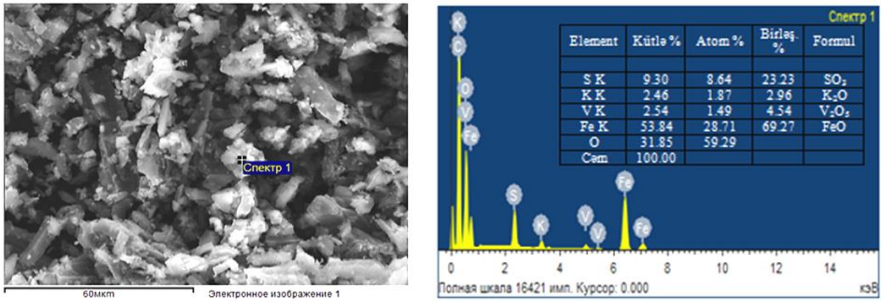

Figure 11. Result of SEM-EDS analysis of Fe-AC sorbent after sorbtion [2]

Vanadium adsorption increased rapidly during the first 15 min, gradually approached equilibrium within 60 min, and reached equilibrium after 180 min due to saturation of the adsorbent active sites. The adsorption behavior was evaluated using the Langmuir and Freundlich isotherm models. According to the Langmuir model, adsorption occurs as a monolayer on energetically uniform active sites.

$$\frac{C_e}{q_e} = \frac{C}{q_{max}} + \frac{1}{K_L \cdot q_{max}} \qquad (6)$$

-where q_e is the amount of adsorbed vanadium (V) ions at equilibrium (mg/g), q_{max} is the maximum adsorption capacity (mg/g), K_L is the Langmuir constant (l/g) and C_e is the equilibrium concentration of the sorbate in solution (g/L).

The adsorption process occurring at active centers with different energies of adsorbents with heterogeneous surfaces is characterized by the Freundlich model. The linear equation of the Freundlich model is expressed as follows:

$$\lg q_e = \lg K_F + n \lg C_e \qquad (7)$$

- where K_F is the Freundlich constant related to the sorption capacity of the adsorbent (mol/g) and n is the adsorption intensity parametr.

According to the results obtained, Langmuir and Freundlich isotherms were constructed (Fig. 12).

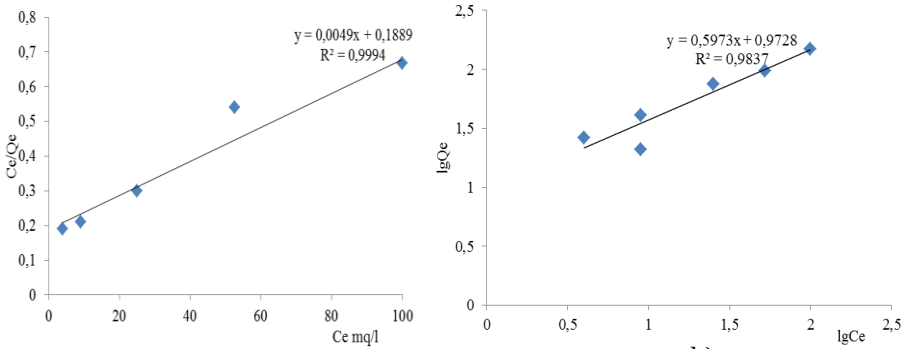

Figure 12. Adsorption isotherms of vanadium (V) ion with Fe-AC nanocomposite: Langmuir (a) and Freundlich (b) models [2]

Both isotherms describe the adsorption of vanadium on Fe-AC nanocomposite satisfactorily, which is characterized by high correction factors. The correction factor is close to unity (R^2=0.999), which indicates that the adsorption results are in full agreement with the Langmuir model at the selected temperature. The chemical interaction occurs between the vanadate ion and the active sites.

Since the correction factor found according to the Freundlich equation (R^2=0.983) at the studied temperatures is lower than the Langmuir correction factor, equation (6) fully describes the sorption.

Following the sulfation of alunite sludge with concentrated sulfuric acid and subsequent roasting of the sulfated mass at 550°C, water leaching of the calcined product resulted in the selective dissolution of vanadium and aluminum compounds into the aqueous phase. Meanwhile, iron was converted into stable iron oxide phases and remained in the solid residue together with silicon oxide. This behavior enabled the effective separation of vanadium and aluminum from iron-bearing and siliceous components of the sludge. The concentration of vanadium in the resulting solution reaches 244 mg/l. V_2O_5. In the next stage, vanadium is separated from the solution by adsorption.

Adsorption constants of vanadium ion adsorption at different temperatures were determined based on Langmuir and Freundlich

isotherms (Table 2).

Table 2

Equilibrium adsorption constants of vanadium(V) ions on the Fe–AC nanocomposite [1,10]

	Langmuir equation			Freundlich equation		
T	q_{max}; mg/g	K_L,l/mg	R^2	K_F, l/mg	n	R^2
298	72	0.16	0.999	21	2	0.983
308	110	0.12	0.992	15	1.5	0.982
318	152	0.1	0.992	10	0.5	0.980

As a continuation of the research, the thermodynamics of the process were studied. Thermodynamic studies proved that the sorption process is spontaneous. The enthalpy (ΔH), free energy (ΔG) and entropy (ΔS) during the adsorption of vanadium by Fe-AC nanocomposite were calculated using the following equations (9-11)

$$K_{ads}=q_{max} \cdot K_L \quad (8)$$

$$\Delta G= -RT\ln K_{ads} \quad (9)$$

$$\Delta S = (\Delta H-\Delta G)/T \quad (10)$$

$$\Delta H^0 = -\frac{RT_1T_2 lnK_1K_2}{T_2 - T_1} \quad (11)$$

First, the equilibrium adsorption constants at different temperatures were calculated using Equation (8). The maximum adsorption capacity (q_{max}) and the Langmuir equilibrium constant (K_L) were determined by the graphical method based on the linear dependence of C_{eq} on C_{eq}/q_{eq}. In the linearized form of the Langmuir equation, the slope of the straight line corresponds to $1/q_{max}$, while the intercept represents $1/(q_{max} \cdot K_L)$. The calculated thermodynamic parameters are presented in Table 3.

The positive value of ΔH indicates that the adsorption is endothermic (ΔH=10.94 kC/mol). Also, the positive value of entropy

(ΔS= 57.05 C/mol· K) indicates an increase in the chaotic state at the substance/solution boundary during adsorption. The negative value of free energy (in the range of -6.05 to -7.19 kC/mol) indicates the spontaneous (arbitrary) nature of adsorption.

Table 3

Thermodynamic parameters of adsorption of vanadium (V) ions by Fe-AC nanocomposite at different temperatures and pH=4.5 [1,10]

T (K)	ΔG (kC/mol)	ΔH (kC/mol)	ΔS (C /mol·K)
298	-6.05	10.94	57.05
308	-6.63		
318	-7.19		

The generalization of the experiments conducted during the research can be described by the following block diagram. Finally, a principle technological scheme for the concentration and extraction of vanadium during the processing of alunite ore was proposed (Fig. 13).

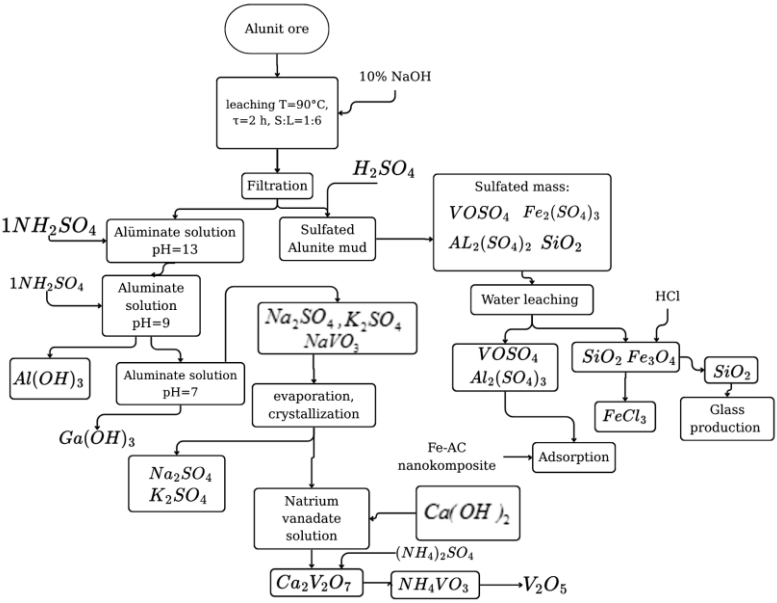

Figure 13. Principle technological scheme of obtaining V_2O_5 from complex processing of alunite ore [6]

Thus, by an environmentally friendly method, without energy consumption, without burning, by carrying out complex processing of alunite, pure V_2O_5 was finally obtained and its existence was confirmed by physicochemical analysis methods. As a result of the conducted studies, it was determined that during the complex processing of alunite ore, along with macrocomponents (Al_2O_3, H_2SO_4, K_2SO_4), it is possible to selectively obtain vanadium compounds.

CONCLUSIONS

1. The effects of various factors on the leaching of vanadium during the alkaline processing of raw alunite ore were investigated. It was determined that up to 98% of the alunite mineral dissolved and passed into solution when leaching was carried out with a 10% alkaline solution at 90°C for 2 h, a solid-to-liquid ratio of 1:6, and a stirring rate of 700 rpm. Under these conditions, 71.3% of the vanadium contained in the ore was transferred into the solution [7,9].
2. To refine the optimum conditions obtained, the experimental results were modelled using the mathematical planning method (regression analysis), and the following regression equation was established: $Y = 15.9564 - 0.23X_1 + 0.231X_2 + 0.03429X_3$. It was determined that the vanadium extraction increased with increasing alkali concentration up to 10%; however, beyond this value, each 1% increase in alkali concentration reduced vanadium extraction by 0.23%. An increase in temperature by 10°C increased the extraction by 0.23%, while an increase in leaching time by 1 min increased the extraction by 0.034% [16].
3. The optimum conditions for the recovery of aluminium and vanadium from aluminate solutions were determined. By neutralizing the solution with a 5% sulfuric acid solution, $Al(OH)_3$ (90%) was precipitated at pH 9.0–9.5, while $Ga(OH)_3$ (90%) was precipitated at pH 6.8–7.5. Alkali metal sulfate salts were subsequently separated from the remaining solution by crystallization, and finally, the vanadium concentrate was precipitated using a slaked lime solution [11].
4. Vanadium was precipitated from sodium vanadate solutions containing 1.5–2.0 g/L V_2O_5 by adding calcium hydroxide at a molar ratio of $CaO:V_2O_5 = 10:1$. Under the conditions of 85–90°C, 2 h, and pH 10–

11, up to 95% of the vanadium was precipitated in the form of calcium pyrovanadate salts. The precipitation kinetics were investigated, and the activation energy was calculated (E_a = 14.6 kJ·mol^{-1})[13, 17].

5. The conditions for the precipitation of vanadium in the form of ammonium metavanadate and ammonium polyvanadate compounds have been studied. It has been established that vanadium precipitates from a solution containing sodium vanadate (6.95 g/l $NaVO_3$) at pH=8 and 25°C as ammonium metavanadate (NH_4VO_3), and at pH=2 and 90-95°C as ammonium polyvanadate $(NH_4)_2V_6O_{16}$) salts. The kinetics of the process have been studied, and the kinetic parameters (n=1, Ea=18.4 kC/mol) have been determined. It is concluded that the precipitation process occurs in the diffusion region [8,12].
6. The thermal decomposition mechanism of vanadyl sulfate ($VOSO_4·3H_2O$) obtained in the V_2O_5–H_2SO_4–SO_3–H_2O system was investigated in the temperature range of 100–700°C. It was established that the thermolysis of $VOSO_4·3H_2O$ proceeds through five stages with the formation of intermediate products [4,5]:

$$VOSO_4·3H_2O \xrightarrow{130^0C} VOSO_4·2H_2O \xrightarrow{217^0C} VOSO_4·H_2O$$
$$\xrightarrow{424^0C} VOSO_4 \xrightarrow{500^0C} (VO_2)_2SO_4 \xrightarrow{700^0C} V_2O_5.$$

7. Optimal conditions for adsorption of vanadium from alunite processing solutions by iron-activated carbon (Fe-AC) nanocomposite have been found. It was found that at pH=4.5, adsorption of vanadium from the solution at a concentration of 100 mg/l reaches 86% within 3 hours [2,3].
8. The results of adsorption of vanadium from alunite slurry processing solutions with Fe-AC were investigated according to Langmuir and Freundlich isotherms. While the adsorption capacity of vanadium from solutions with activated carbon was 39 mg/g, the adsorption capacity with Fe-AC nanocomposite was 110 mg/g. The thermodynamic parameters of adsorption were determined as ΔH=10.94 kC/mol, ΔS=57.05, and ΔG=-6.63 kC/mol at 308K. This indicates that the sorption of vanadium ions occurs mainly due to active centers on the surface of the adsorbent [2,10].

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