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Sorption Concentration of Uranium and Vanadium from Productive Solutions of Black Shale Ores

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ABSTRACT

This study examines the sorption and desorption processes of uranium and vanadium from acidic solutions produced during the processing of black shale ores in Southern Kazakhstan. Such ores are considered unconventional sources of strategic metals and are of particular interest under the conditions of limited traditional mineral resources. To evaluate efficiency, several anion-exchange resins (AMP, AV-17, A-140, and Amberlite) were tested, allowing a comparative analysis of their sorption capacity and selectivity. The AMP resin demonstrated the most favorable performance, providing high uranium uptake and satisfactory vanadium recovery in sulfuric acid media. Desorption experiments confirmed the possibility of efficient uranium transfer into the eluate, which is of great importance for subsequent concentration and purification stages. Vanadium recovery was limited due to the coexistence of different ionic forms of the element. The obtained results confirm the potential of sorption technology as a reliable stage for uranium concentration and indicate the need to apply additional methods, such as solvent extraction or selective precipitation, to enhance the completeness of separation and recovery of the target components.

Keywords: uranium, vanadium, sorption, desorption, anion-exchange resin.

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Introduction

The modern development of science and industry highlights the urgent need for sustainable access to strategic mineral resources, primarily uranium, vanadium, and rare-earth elements. These elements are key components in nuclear energy, high-temperature alloys, catalysts, rechargeable batteries, and other advanced technologies [[1], [2]]. In light of limited traditional deposits and increasing geopolitical risks, growing attention has been directed toward unconventional sources of raw materials, among which black shales occupy a special place.

Black shale formations are fine-layered sedimentary rocks enriched in carbon, organic matter, and finely dispersed mineral phases. Their characteristic features include high sorption capacity and the presence of complex element associations such as V, Mo, U, Ni, Zn, and REEs [[3], [4], [5]]. However, the complex matrix structure, the occurrence of uranium in sparingly soluble forms, and the incorporation of vanadium into phyllosilicates significantly complicate their processing [6]. Consequently, the treatment of black shales requires combined technological schemes that include beneficiation, activation, and subsequent hydrometallurgical extraction stages.

One of the most promising approaches involves the use of sorption technologies, which allow effective concentration of uranium and associated elements from productive solutions. A number of international studies have demonstrated that uranium in sulfuric acid media forms stable anionic complexes, which can be effectively recovered by anion-exchange resins with high selectivity [7]. The sorption capacity of modern resins may reach tens of milligrams per gram of material [[8], [9]], while desorption efficiency can exceed 90% [10].

Recent research emphasizes the application of sorption for uranium recovery from complex solutions, including those derived from black shales, through the use of various types of anion exchangers and optimization of process parameters [11]. To ensure the reliability of results, sorption has been studied under both static and dynamic conditions, including column experiments and mass-transfer modeling [12]. Theoretical approaches, such as modeling the selectivity of U and V, have also been applied to justify sorbent selection and to predict performance [[13], [14]].

Thus, a review of the literature confirms that sorption methods represent an efficient tool for concentrating uranium and separating it from other elements, including vanadium. In combination with acid leaching, sorption forms the basis of promising technologies for processing unconventional mineral resources.

In our previous studies [15], it was established that the combination of reverse coal flotation and sulfuric acid leaching with the use of trichloroisocyanuric acid ensured high uranium recovery (up to 94–95%) and resulted in productive solutions containing uranium and vanadium. These findings highlighted the importance of preliminary carbon removal and oxidation of uranium-bearing phases to enhance metal accessibility and improve subsequent hydrometallurgical operations. The present study focuses on further investigation of these productive solutions, specifically the sorption recovery of uranium and vanadium, in order to evaluate the effectiveness of different anion-exchange resins and to determine the optimal process parameters for maximizing metal recovery. The scientific novelty of the study lies in the mass balance analysis of sorption and desorption processes for both uranium and vanadium, as well as in identifying the limitations of vanadium recovery caused by its mixed ionic forms. The obtained results broaden the understanding of sorption behavior in complex acidic systems and provide a basis for improving technological flowsheets aimed at processing black shale ores and unconventional mineral raw materials.

Materials and methods

Materials

The object of the study was productive solutions obtained after sulfuric acid leaching of uranium-bearing black shale ore that had been preliminarily beneficiated by reverse coal flotation. The ore was mined in Southern Kazakhstan. The uranium concentration in the productive solutions ranged from 45 to 48 mg/L, while vanadium concentrations were 155 to 168 mg/L.

As sorbents, the following anion-exchange resins were used: AMP, AV-17, A-140, and Amberlite. For desorption of the saturated anion exchangers, a 1 M ammonium carbonate solution $(NH_4)_2CO_3$ was applied. Laboratory glassware, beakers, a sorption column, and vessels of various volumes were employed in the experimental work.

Methods

Sorption and desorption experiments were carried out in batch-operated columns. Productive solutions were passed through a layer of anion-exchange resin until equilibrium was achieved, after which the saturated resin was subjected to desorption using ammonium carbonate solution. The process was performed under controlled hydrodynamic and temperature conditions, ensuring reproducibility of results.

The efficiency of sorption and desorption of uranium and vanadium was calculated according to the following relationships:

Mass of element in solution (m, mg):

$$m=C\times V_m \qquad (1)$$

Sorption capacity of the resin (q, g/kg):

$$q=m/m_{resin} \qquad (2)$$

Recovery degree (E, %):

$$E=m_{sorb}m_{init}\times100 \qquad (3)$$

where C is the concentration of the element (mg/L); V is the volume of solution (L); m_{resin} is the mass of the sorbent (kg); m_{sorb} is the mass of the element fixed on the resin or transferred to the eluate (mg); and m_{init} is the mass of the element in the initial productive solution (mg).

Analytical Methods and Equipment

The elemental composition of the initial and treated samples was determined using inductively coupled plasma optical emission spectrometry (ICP-OES) and X-ray fluorescence (XRF) analysis. Sorption–desorption experiments were carried out under controlled hydrodynamic and temperature conditions with the use of a BT20-21 liquid thermostat (Russia) and an LS-301 peristaltic pump (China).

Results and discussion

The productive sulfuric acid solutions obtained as a result of leaching were used in experiments on the sorption recovery of target components-uranium and vanadium. Uranium sorption on anion exchangers occurs mainly due to ion exchange and complexation with uranyl sulfate anions. The process can be represented by the following reaction [11]:

$$(RN)_2SO_4 + [UO_2(SO_4)_2]^{2-} = (RN)_2(UO_2(SO_4)_2) + (SO_4)^{2-} \quad (4)$$

where RN is the matrix of the anion exchanger.

The kinetics of uranium sorption is governed by mass transfer across the diffusion layer surrounding the resin grains and strongly depends on the acidity of the medium. As the pH increases, the sorption rate decreases, thus prolonging the time required to reach equilibrium. Under industrial conditions, effective uranium recovery requires extended contact of the sorbent with the solution [16].

Unlike uranium, vanadium exists in solutions in various ionic forms: both as cations (in oxidation state IV) and as anionic complexes (in oxidation state V) [17]. Consequently, two mechanisms-cation exchange and anion exchange-are simultaneously involved in its recovery. This particularity complicates the selective extraction of vanadium and reduces efficiency compared with uranium, which predominantly exists in a single form.

The efficiency of uranium and vanadium sorption depends on the concentration of metals, the type of ion-exchange resin, the acidity of the medium, and the presence of interfering ions such as SO_4^{2-}, NO_3^-, Cl^-, and Fe^{3+}. At low uranium concentrations (1–25 mg/L), distribution coefficients are considerably higher than at elevated concentrations (100–1000 mg/L), regardless of the type of anion exchanger. Increased acidity, especially when using strongly basic resins, significantly reduces sorption capacity. Depressor anions also diminish recovery, with their effect intensifying as their affinity for the functional groups of the resin increases (Figure 1) [18].

Figure 1 – Sequence of increasing depressive effects of anions

At the stage of uranium and vanadium sorption studies, four types of anion exchangers were tested: AMP, AV-17, A-140, and Amberlite. The dependence of recovery efficiency on temperature (25, 35, and 45 °C) was also examined. The initial concentrations of uranium and vanadium in the solution were 48 mg/L and 168 mg/L, respectively. Each sorbent was loaded in a single-stage mode, and the residual metal concentrations in the raffinate were determined to calculate recovery degrees. The efficiency of uranium and vanadium recovery by different sorbents at various temperatures is shown in Figures 2 and 3.

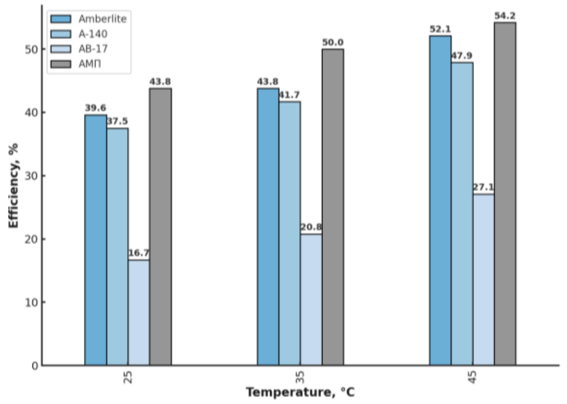

Figure 2 – Uranium recovery as a function of temperature

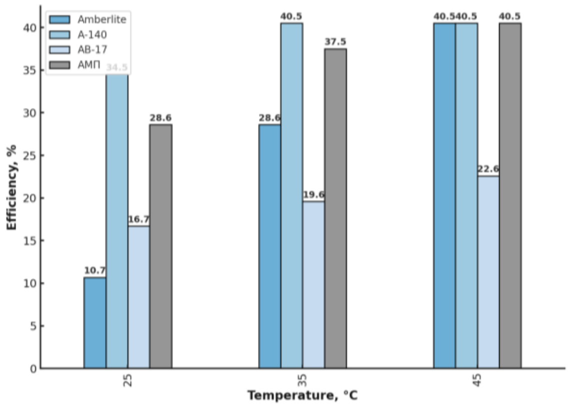

Figure 3 – Vanadium recovery as a function of temperature

For laboratory experiments, AMP resin was selected as the sorbent due to its high sorption capacity in acidic media (up to 80.0 g/kg at pH ≥ 2.0). The initial productive solution volume of 50.0 L contained uranium and vanadium at concentrations of 45.0 and 155.0 mg/L, respectively, corresponding to total masses of 2250.0 mg U and 7750.0 mg V. A total of 100 g of AMP resin was loaded into the column, which under the given conditions excluded reaching the maximum sorption capacity. Solution delivery was provided by a peristaltic pump at a flow rate of 0.5 L/h, while heating to 45 °C was maintained in a thermostatic water bath (Figure 4).

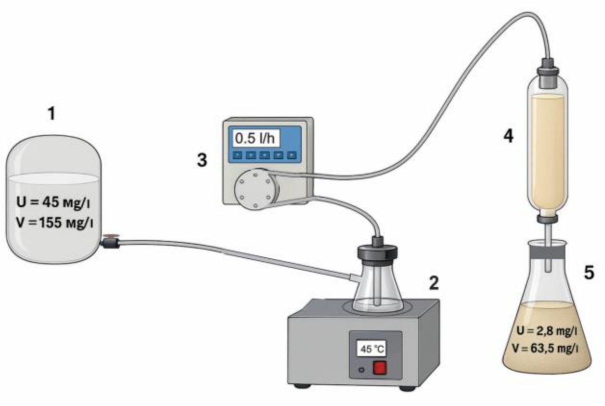

Figure 4 – Laboratory setup scheme for sorption experiments: 1 – tank with productive solution; 2 – flask with solution in thermostatic bath; 3 – peristaltic pump; 4 – column filled with resin; 5 – raffinate container

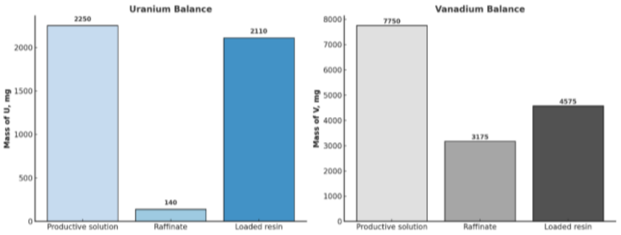

Figure 5 – Mass balance of uranium and vanadium during sorption

Figure 6 – Visual changes in AMP resin appearance

Sorption loading of the resin was conducted over three consecutive days, 10 hours each day. As the process progressed, a systematic decrease in the concentrations of target elements in the solution was observed: in the final raffinate, the residual uranium concentration was 2.8 mg/L, and vanadium was 63.5 mg/L. Mass balance calculations showed that 2110.0 mg U (21.1 g/kg) and 4575.0 mg V (45.75 g/kg) were sorbed onto 100 g of resin, corresponding to recovery efficiencies of 93.78% and 59.0%, respectively. The total resin loading with target components reached 66.85 g/kg. The distribution of uranium and vanadium masses between raffinate and resin is presented in Figure 5.

In addition to recovery of the target components, partial uptake of impurity elements, mainly iron and copper, was observed. After completion of the process, changes in the physical characteristics of the resin were noted: its mass increased by 15 g, granules swelled, and their color darkened, reflecting intensive loading of active sites with uranium and vanadium ions (Figure 6).

Comparative X-ray fluorescence analysis of the original and saturated AMP resin samples revealed pronounced structural and chemical modifications. In the saturated resin, oxygen content increased from 11.49% to 15.88% and sulfur from 4.09% to 10.41%, reflecting the accumulation of sulfate ions from the sulfuric acid solution and a higher oxidation degree of the organic matrix. Target elements—vanadium (4.5%) and uranium (2.1%)—were detected in the resin, with amounts consistent with the calculated sorption balance.

The chlorine content decreased nearly threefold (from 20.7% to 7.18%), indicating chloride substitution as a result of ion exchange, characteristic of chloride-type resins. The organic backbone, represented by CnH$_2$n spectra below oxygen, decreased by 2.7%, indicating partial involvement of functional groups in sorption. At the same time, the experiments demonstrated that the chlorine-active components of TCCA, used for preliminary oxidation of black shale ores, do not exert a depressive effect on the sorption capacity of the resin.

At the next stage of the technological process, desorption of uranium and vanadium from the saturated AMP resin was carried out. A 1 M ammonium carbonate solution heated to 60 °C was used in the experiments. The column was loaded with 100 g of resin previously washed of acid residues. The eluate volume was 0.2 L, and the process duration was 8 hours; the apparatus remained identical to the sorption stage.

According to mass balance analysis (Figure 7), out of the 2110 mg of uranium accumulated in the resin (21.1 g/kg), 1996 mg was transferred to the eluate, while the residual mass was only 120 mg (1.2 g/kg). Thus, desorption efficiency reached 94–95%, and the overall uranium recovery into the eluate amounted to 88–89%. The uranium concentration in the final eluate was 9.98 g/L, confirming a high degree of enrichment.

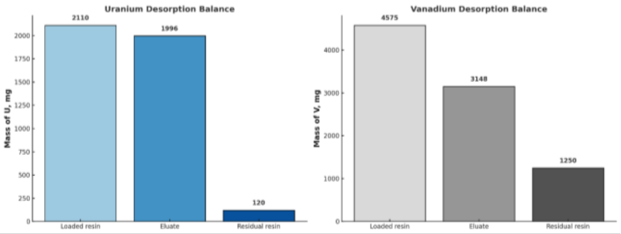

Figure 7 – Mass balance of uranium and vanadium during desorption

The behavior of vanadium differed substantially. With an initial loading of 4575 mg (45.8 g/kg) in the resin, only 3148 mg transferred to the eluate, while about 1250 mg remained in the sorbent. Desorption efficiency was approximately 69%, while the overall recovery of vanadium did not exceed 41–43%. Nevertheless, the concentration of vanadium in the eluate was relatively high, 15.74 g/L, g/L-confirming the feasibility of its further processing.

The difference in recoverability between uranium and vanadium is determined by their chemical nature. Uranium in sulfuric acid solutions forms stable anionic uranyl sulfate complexes, which are effectively desorbed by carbonate solutions. Vanadium, in contrast, occurs in mixed forms – both anionic and cationic – limiting its transfer into the carbonate eluate and lowering desorption efficiency. These observations are consistent with the literature, which emphasizes the need for additional steps, including pH-controlled precipitation, to enhance extraction completeness [[19], [20]]. The presence of vanadium in productive solutions also exerts a competitive influence on uranium sorption and desorption, reducing their selectivity and efficiency, which makes the problem of separation particularly relevant [21].

The obtained results convincingly confirm the high efficiency of uranium sorption on AMP resin: high recovery rates were achieved while forming a saturated sorbent with predictable loading parameters. This allows sorption to be considered a reliable stage of preliminary concentration. To achieve residual uranium and vanadium concentrations of less than 1–2 mg/L in solutions, it is advisable to use multi-stage sorption. In industrial practice, this approach is implemented through cascade passage of solution through multiple columns, ensuring complete resin saturation at the point of “breakthrough” of target ions past the last column. Optimal parameters, including the sorption layer length and the number of columns, are determined by solution acidity and salt composition.

At the same time, the limitations observed in vanadium recovery highlight the need to improve the technological scheme. To increase selectivity and ensure comprehensive separation of target components, supplementing the sorption stage with extraction methods is recommended, which can serve as an effective tool for the subsequent processing of productive solutions.

Conclusions

The conducted research confirmed the high efficiency of the sorption method for concentrating uranium from productive solutions of black shale ores. Several types of anion-exchange resins (AMP, AV-17, A-140, and Amberlite) were tested, enabling a comparative evaluation of their performance. Among them, the AMP resin demonstrated the best results, achieving the highest sorption efficiency (93.8%) and stable desorption parameters (94–95%), which ensured an overall uranium recovery into the eluate of 88–89%.

For vanadium, sorption efficiency was 59.0%, while desorption efficiency was approximately 41–43%. This is attributed to the complex chemical nature of the element and the coexistence of both anionic and cationic forms. Thus, AMP resin can be considered the most promising sorbent for uranium recovery, providing selective separation and enrichment compared with the other tested resins.

At the same time, the limited recoverability of vanadium and its relatively low desorption efficiency indicate the need for further improvement of processing schemes. To enhance separation efficiency and selectivity, it is recommended to supplement the sorption stage with extraction methods, which would enable more complete recovery and separation of uranium and vanadium within complex technological flowsheets. Black shale ores of Southern Kazakhstan are large-tonnage, unconventional sources of strategic metals; therefore, the proposed approaches may be applied in industrial processing of this type of raw material, contributing to a more comprehensive and efficient use of the mineral resource base.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

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Қара тақтатас кендерінің өнімді ерітінділерінен уран мен ванадийді сорбциялық концентрациялау

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ТҮЙІНДЕМЕ

Бұл жұмыста Оңтүстік Қазақстанның қара тақтатас кендерін өңдеу кезінде алынған өнімді қышқыл ерітінділерінен уран мен ванадийді сорбциялау және десорбциялау процестері зерттелді. Мұндай кендер стратегиялық металдардың дәстүрлі емес көздері болып табылады және дәстүрлі минералды-шикізат базасының шектеулі жағдайында ерекше қызығушылық тудырады. Тиімділікті бағалау үшін аниониттердің бірнеше түрі (AMP, AV-17, A-140 және Amberlite) сыналған, бұл олардың сорбциялық қабілеті мен селективтілігін салыстырмалы талдауға мүмкіндік берді. Ең жақсы нәтижелерді AMP шайыры көрсетті, ол күкірт қышқылды ортада уранды жоғары дәрежеде сіңіруді және ванадий бойынша қанағаттанарлық көрсеткіштерді қамтамасыз етті. Десорбциялық тәжірибелер уранды элюатқа тиімді көшіру мүмкіндігін растады, бұл концентрация мен тазартудың кейінгі кезеңдері үшін үлкен маңызға ие. Ванадийді бөліп алу элементтің әртүрлі иондық формаларда болуына байланысты шектеулі болды. Алынған нәтижелер уранды концентрациялаудың сенімді сатысы ретінде сорбциялық технологияның болашағын растайды және мақсатты компоненттерді бөлу мен қалпына келтірудің толықтығын жақсарту үшін экстракция немесе селективті тұндыру сияқты қосымша әдістерді қолдану қажеттілігін көрсетеді.

Түйін сөздер: уран, ванадий, сорбция, десорбция, анионит.

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Сорбционное концентрирование урана и ванадия из продуктивных растворов черносланцевых руд

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АННОТАЦИЯ

В данной работе исследованы процессы сорбции и десорбции урана и ванадия из продуктивных кислотных растворов, полученных при переработке черносланцевых руд Южного Казахстана. Такие руды относятся к нетрадиционным источникам стратегических металлов и представляют особый интерес в условиях ограниченности традиционной минерально-сырьевой базы. Для оценки эффективности были испытаны несколько типов анионитов (АМП, АВ-17, А-140 и Amberlite), что позволило провести сравнительный анализ их сорбционной ёмкости и селективности. Наилучшие результаты показала смола АМП, обеспечившая высокую степень извлечения урана и удовлетворительные показатели по ванадию в сернокислой среде. Десорбционные эксперименты подтвердили возможность эффективного переноса урана в элюат, что имеет важное значение для последующих стадий концентрирования и очистки. Извлечение ванадия оказалось ограниченным вследствие существования элемента в различных ионных формах. Полученные результаты подтверждают перспективность сорбционной технологии как надёжного этапа концентрирования урана и указывают на необходимость применения дополнительных методов – таких как экстракция или селективное осаждение – для повышения полноты разделения и извлечения целевых компонентов.

Ключевые слова: уран, ванадий, сорбция; десорбция; анионит.

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