

Mineralogical composition of colmatants in uranium in situ leach mining: XRD characterization and SEM evaluation of decolmatation efficiency

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Abstract

Purpose. To determine the mineralogical composition of colmatants formed during uranium in situ leaching at the North Kharasan, North Karamurun, and Irkol deposits of the Syrdarya uranium province and to evaluate their structural response to chemical decolmatation treatments to substantiate deposit-specific approaches to well treatment.

Methods. Colmatant samples collected from the inner surfaces of submersible pump assemblies in production wells were examined using X-ray diffraction (XRD) and scanning electron microscopy (SEM). Quantitative phase composition was determined using a PANalytical X'Pert MPD PRO diffractometer. The samples were treated with two decolmatation solutions: Solution 1 containing 10% HF and Solution 2 containing 5% HF, 10% H₂SO₄, and 1% surfactant. SEM observations were used to compare changes in surface morphology, crystal fragmentation, and formation of cavities and voids before and after treatment.

Findings. Carbonate minerals constitute a major fraction of the studied colmatants, accounting for approximately 57% at North Kharasan, 29% at North Karamurun, and 45% at Irkol. North Kharasan is characterized by an ankerite-dominated assemblage, North Karamurun by a mixed carbonate-phosphate composition with a 51% phosphate fraction, and Irkol by a carbonate-rich assemblage containing 18% gypsum. Both decolmatation solutions substantially disrupted the colmatant structure. The 10% HF solution generally caused more intensive local destruction, whereas the composite solution produced a comparable overall structural effect while reducing the HF concentration by half. For gypsum-rich colmatants, HF treatment may promote CaF₂ precipitation, requiring sufficient pumping and subsequent flushing.

Originality. The study integrates quantitative XRD characterization of operational colmatants with SEM-based assessment of their responses to two chemical treatments. It establishes a direct relationship between deposit-specific mineralogical composition and the selection of decolmatation approaches.

Practical implications. The results provide a mineralogy-based framework for selecting effective decolmatation treatments at uranium ISL deposits. The composite 5% HF + 10% H₂SO₄ + 1% surfactant formulation can be considered for further field validation, while gypsum-rich systems require additional measures to prevent secondary colmatation caused by CaF₂ precipitation.

Keywords: *in situ leaching; uranium mining; colmatation; colmatants; mineralogical composition; X-ray diffraction; decolmatation; gypsum precipitation; well treatment*

1. Introduction

In situ leach (ISL) mining, also referred to as in situ recovery (ISR), has become the dominant technology for uranium extraction worldwide, accounting for more than 56% of global uranium production in recent years [1], [2]. In contrast to conventional open-pit and underground mining, ISL enables uranium recovery directly within the ore-bearing formation without large-scale excavation of the rock mass. This approach offers important advantages, including reduced surface disturbance, lower capital and operating costs, and

the ability to exploit low-grade sandstone-hosted uranium deposits that may be economically unattractive for conventional mining [3]-[5]. Kazakhstan remains the world's leading uranium producer, accounting for approximately 39-43% of global output. In 2024, uranium production by NAC Kazatomprom JSC increased by 10% to 23270 t U₃O₈ [6].

The technological cycle of uranium ISL includes injection of a leaching solution into the productive horizon, dissolution and mobilization of uranium-bearing compounds, recovery of pregnant leach solution (PLS) through production wells, sorption extraction of uranium, clarification of the

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barren solution, and its subsequent re-acidification and reinjection [7], [8]. In addition to uranium, ISL process solutions may contain a range of associated dissolved elements, making their chemical composition inherently multicomponent. Investigations of productive wells at uranium ISR sites have identified rare-earth and other associated metals. At the same time, recent studies have also considered the recovery of scandium from solutions generated during uranium extraction [9], [10]. Sulfuric acid is widely used as the principal lixiviant at uranium deposits in Kazakhstan because of its high leaching efficiency, availability, and relatively low cost. Nevertheless, reagent consumption represents a substantial component of operating expenditure, while fluctuations in sulfuric acid prices directly affect the economic performance of ISL operations [6], [11], [12]. The efficiency of acid leaching is also strongly dependent on the mineralogical composition of the host rocks. Elevated carbonate contents increase acid consumption through neutralization reactions and may significantly alter the geochemical conditions within the productive horizon [13], [14]. For deposits with high carbonate contents, alkaline or neutral carbonate-based leaching systems may be considered, although such systems generally exhibit lower extraction kinetics [15], [16].

More broadly, studies of hydrometallurgical and underground leaching systems demonstrate that mineralogical phase transformations, pretreatment conditions, reagent composition, and solution transport strongly control extraction efficiency. Such relationships have been demonstrated for microwave-assisted leaching of technogenic zinc-bearing materials; alkaline treatment and subsequent leaching of wolframite concentrates; sulfuric acid leaching of copper smelting slags; and underground leaching of polymetallic ores. Related studies also emphasize the potential of combined mining and leaching approaches for improving the utilization of mineral resources [17]-[21].

One of the major operational problems affecting the long-term efficiency of acid ISL is progressive well colmatation, or clogging, caused by the accumulation of mineral precipitates and fine particles within the near-wellbore zone and filtration pathways [22], [23]. Interaction between sulfuric acid solutions and carbonate, aluminosilicate, and iron-bearing minerals of the host rocks may result in the formation of secondary mineral phases, including gypsum, iron oxyhydroxides, and aluminosilicate compounds [24], [25]. Their precipitation reduces effective porosity and permeability, thereby decreasing the injectivity and productivity of technological wells. Mineral alteration and pore plugging associated with acid ISL have been experimentally documented for sandstone-hosted uranium deposits, where secondary gypsum and iron-bearing precipitates were shown to substantially modify the pore structure of the leached formation [26]. These processes are closely related to oxidation-reduction conditions within the reservoir and therefore influence uranium recovery, specific reagent consumption, and the overall techno-economic performance of ISL operations [27]-[32].

Biological approaches have also been investigated to control redox conditions and intensify uranium ISR. Studies of bacterial iron oxidation and uranium-bacteria interactions have examined the application of microbial processes to uranium leaching, including field-oriented and industrial implementations. Similar bioleaching approaches have also been investigated for other metal-bearing materials, demon-

strating the broader potential of microbial pretreatment under acidic leaching conditions [33]-[37].

Recent developments in reactive transport modeling have considerably improved the understanding of mineral dissolution and secondary precipitation during uranium ISL. Three-dimensional simulations have shown that mineral dissolution may increase porosity in the vicinity of injection wells. In contrast, secondary precipitation near production wells can produce a substantial decrease in both porosity and permeability [38], [39]. Kinetic reactive transport modeling of column experiments has further demonstrated the sequential development of gypsum and iron-bearing secondary phases along the solution flow path, confirming the importance of mineral precipitation in the evolution of filtration properties during uranium ISR [12]. Industrial-scale reactive transport simulations have also demonstrated the potential of coupled geochemical-hydrodynamic models to reproduce the evolution of mineral dissolution and precipitation fronts over prolonged periods of ISL operation [40]. At the pore and mineral scales, the spatial distribution of clay and carbonate phases has been identified as an important factor controlling solution flow paths, reagent-rock interaction, and the subsequent development of permeability restrictions [41]. Detailed petrological and mineralogical investigations of sandstone-hosted uranium deposits have also demonstrated that pre-leaching characterization of clay and carbonate assemblages is essential for assessing their response to leaching solutions and the potential development of colmatation [42].

Permeability deterioration is not caused exclusively by chemical precipitation. Mechanical colmatation may occur as a result of migration and accumulation of fine-grained clay and silt particles under the hydrodynamic influence of the injected lixiviant [43], [44]. This mechanism becomes particularly important in low-permeability and clay-rich sandstone formations. Various approaches have therefore been proposed to improve filtration characteristics and enhance mass transfer during ISL, including blasting-enhanced permeability techniques [45], [46], electrokinetic treatment, ultrasonic stimulation, and solution pulsing [47]-[50]. At the same time, mineralogical characterization of ores and host rocks remains an essential component of ISL design because the initial distribution of carbonate, clay, and other reactive minerals largely determines the subsequent geochemical response of the productive horizon to leaching [51], [52].

Reactive transport studies further indicate that precipitation behavior can be controlled by a relatively limited number of mineralogical and geochemical parameters. For example, three-dimensional modeling of the Tortkuduk uranium deposit demonstrated the importance of initial beidellite, calcite, and iron hydroxide contents for reproducing the evolution of the leaching system [53]. Streamline-based modeling approaches have also been proposed to substantially reduce the computational requirements of large-scale ISL simulations [54], [55]. Thermodynamic and kinetic models have provided additional insight into the sequence of mineral dissolution and precipitation and the saturation behavior of gypsum, calcite, and iron-bearing secondary phases under changing lixiviant compositions [39], [56]. Such results demonstrate the potential of numerical modeling for predicting colmatation-prone zones; however, the reliability of these predictions ultimately depends on representative mineralogical data obtained from actual operating systems.

The restoration of well productivity requires decolmation methods capable of disrupting or dissolving the mineral framework of accumulated deposits. Previous studies have shown that carbonate phases may constitute a major fraction of colmatants formed during uranium ISL and that their composition should be considered when selecting chemical treatment solutions [8], [57]. Physical intensification techniques, including alternating electric current, magnetic treatment, and ultrasonic vibrations, have also been investigated to improve mass transfer and metal recovery [47], [58]. More recently, fluoride-based chemical treatments have received particular attention because of their ability to interact with siliceous and other poorly soluble mineral components. Ammonium fluoride in combination with a sodium carbonate buffer has been proposed as an alternative decalcification reagent [59]. At the same time, hydroxyethylidene diphosphonic acid (HEDP) has been investigated for preventing gypsum precipitation during ISL operation [60]. These studies indicate that both preventive and restorative approaches can help maintain well performance; however, their effectiveness depends strongly on the mineralogical composition and structural characteristics of the colmatant. The long-term performance of uranium ISL well fields also depends on the construction, maintenance, repair, and regeneration of technological wells, which remain important areas for improving the operational efficiency of uranium recovery [61]–[64].

Despite significant progress in reactive transport modeling and the development of different well stimulation and decolmation techniques, a direct relationship between the mineralogical composition of colmatants formed under actual ISL operating conditions and their structural response to specific chemical treatment solutions remains insufficiently characterized. In particular, comparative experimental data that combine quantitative phase identification with microscopic evaluation of colmatant destruction are limited for uranium deposits in the Syrdarya province. Such information is necessary not only for identifying the dominant mechanisms of colmatation but also for selecting treatment formulations according to the specific mineralogical composition of deposits formed at individual uranium mining sites.

Therefore, this study aims to determine the mineralogical composition of colmatants collected from production wells at the North Kharasan, North Karamurun, and Irkol uranium deposits in Syrdarya province and to evaluate their response to two chemical decolmatation solutions using X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results obtained are further used to substantiate deposit-specific approaches to chemical well treatment, based on the predominant mineral phases and the observed structural changes in the colmatants.

2. Materials and methods

2.1. Geological and mineralogical characteristics of the productive horizons

The mineralogical composition of the ores and host rocks is an important factor controlling the interaction between the lixiviant and the productive horizon during uranium ISL. Integrated geological and satellite remote sensing data have also been used to forecast roll-front uranium provinces and delineate prospective uranium-bearing areas [65]. The host rocks of the Syrdarya depression are characterized by a heterogeneous mineral composition dominated by quartz, feld-

spars, rock fragments, clay minerals, carbonates, organic matter, sulfides, and uranium-bearing phases (Table 1). Numeric values represent quantitative ranges (wt.%).

Quartz constitutes the dominant mineral component of the host rocks, with contents ranging from 30 to 80%. Feldspars and clay minerals may also occur in substantial proportions, reaching up to 40 and 45%, respectively. Their abundance is important for the filtration behavior of the productive horizon because fine-grained clay material may migrate with the solution flow and contribute to mechanical colmatation, while swelling and ion-exchange processes may additionally reduce pore-space accessibility.

Table 1. Mineral composition of ores and host rocks of the Syrdarya depression deposits

Mineral / mineral aggregate	Content, %
Quartz	30-80
Feldspars	5-40
Rock fragments	5-30
Accessory minerals	minor
Clay minerals (illite, kaolinite, montmorillonite, glauconite, chlorite)	5-45
Carbonates (calcite, siderite, dolomite, ankerite)	0-5
Organic matter (plant detritus, bitumens, etc.)	5-15
Sulfides (pyrite, marcasite, minor Mo, Cu, Zn, Pb sulfides)	abundant
Uranium mineralization	minor

Carbonate minerals, including calcite, siderite, dolomite, and ankerite, generally account for up to 5% of the host-rock composition. Despite their relatively low bulk abundance, these phases are highly reactive under sulfuric acid leaching conditions and therefore play an important role in the formation of secondary mineral precipitates. According to [8] and [24], carbonate contents above approximately 1.5% can promote chemical scale formation upon interaction with sulfuric acid solutions.

The coexistence of quartz, aluminosilicate minerals, clay phases, and carbonates creates favorable conditions for the formation of compositionally heterogeneous colmatants. Accordingly, the mineralogical characteristics of the productive horizons provide the necessary background for interpreting the phase composition of deposits collected from the technological equipment of the studied wells.

2.2. Sample collection and preparation

Colmatant samples were collected from the inner surfaces of submersible pump assemblies installed in production wells at three uranium deposits of the Syrdarya province: North Kharasan, North Karamurun, and Irkol. All three deposits are operated using *in situ* leach (ISL) technology with sulfuric acid as the principal leaching reagent.

Three representative samples were selected for further mineralogical and microstructural analyses. Sample 1, collected from the North Kharasan deposit, and Sample 3, obtained from the Irkol deposit, consisted of reddish, pulverulent precipitates containing relatively coarse homogeneous fragments and individual sand particles. Sample 2, collected from the North Karamurun deposit, was characterized by a white powder with beige inclusions.

Before X-ray diffraction analysis and decolmation experiments, the collected precipitates were dried and subsequently ground in an agate mortar to obtain a homogeneous fine

powder suitable for further laboratory investigation. This preparation procedure was applied to reduce sample heterogeneity and ensure representative conditions for subsequent mineralogical and microstructural analyses.

2.3. X-ray diffraction analysis

The phase composition of the colmatant samples was determined by X-ray diffraction (XRD) using a PANalytical X'Pert MPD PRO diffractometer. Measurements were performed with CuK α radiation using a Ni filter. The accelerating voltage was 30 kV, and the tube current was 10 mA. Diffraction patterns were recorded over a 2θ range of 10–90° at a scanning rate of 1°/min.

The interplanar spacings were calculated according to Bragg's law:

$$n\lambda = 2d \sin \theta, \quad (1)$$

where:

- n – order of reflection;
- λ – X-ray wavelength;
- d – interplanar spacing;
- θ – diffraction angle.

Phase identification was performed using the ICDD PDF-2 diffraction database. Diffraction peak intensities were evaluated on a 100-point scale.

2.4. Decolmation treatment and SEM analysis

The prepared colmatant samples were subjected to laboratory treatment with two decolmation solutions. Solution 1 consisted of 10% HF and 90% process water, whereas Solution 2 contained 5% HF, 10% H₂SO₄, 1% surfactant, and 84% process water. For each experiment, 0.5 g of powdered colmatant was placed on a glass slide and treated with 2 mL of the corresponding solution at room temperature (22 ± 2°C). The contact time was 30 min. After treatment, the samples were rinsed with deionized water and dried at room temperature.

The use of hydrofluoric acid was associated with its high reactivity toward gypsum, aluminosilicate, and siliceous components present in colmatant deposits [66]. In Solution 2, sulfuric acid was additionally introduced to promote the dissolution of aluminum- and iron-bearing phases, while the surfactant was used to improve the reagent's penetration into the pore space of the deposits.

The surface morphology of the colmatant samples before and after chemical treatment was examined using a TESCAN MIRA 3 FEG-SEM scanning electron microscope equipped with a Schottky field-emission cathode. The electron beam energy range was 200 eV–30 keV, and the beam current was continuously adjustable from 2 pA to 400 nA. The microscope resolution in high-vacuum mode was 1.2 nm at 30 keV using the SE detector and 3.5 nm at 1 keV using the In-Beam SE detector. The maximum field of view exceeded 8 mm at a working distance of 10 mm.

3. Results and discussion

3.1. Mineralogical composition of colmatants

3.1.1. North Kharasan deposit

The X-ray diffractogram of the colmatant sample collected from the North Kharasan deposit is characterized by distinct and stable diffraction reflections, indicating a relatively high degree of crystallinity of the precipitated mineral phases (Fig. 1).

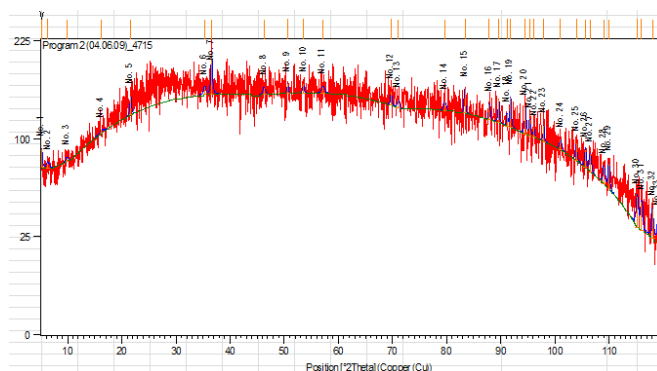


Figure 1. Diffractogram of the colmatant from the North Kharasan deposit

Phase identification confirmed the presence of carbonate, oxide, phosphate, sulfide, and aluminosilicate-related components within the deposit. The quantitative phase composition of the sample is presented in Table 2.

Table 2. Mineralogical composition of the colmatant from the North Kharasan deposit

Component	Chemical formula	Content, %
Quartz	SiO ₂	2
Aluminum phosphate	AlPO ₄	11
Iron oxide (magnetite)	Fe ₃ O ₄	14
Magnesium sulfide	MgS	11
Calcium aluminum hydroxide	CaAl ₂ (OH) ₈ ·2H ₂ O	1
Ankerite	Ca(Mg,Fe,Mn)(CO ₃) ₂	51
Dolomite	CaMg(CO ₃) ₂	6
Amorphous phase	—	4

The mineralogical composition indicates that ankerite is the dominant phase, accounting for 51% of the total sample mass. Together with dolomite, the total carbonate fraction reaches 57%, demonstrating the predominant role of carbonate minerals in the formation of the North Kharasan colmatant. The high carbonate content is consistent with chemical interactions between the sulfuric acid lixiviant and the carbonate-bearing components of the productive horizon, which promote the dissolution of primary carbonate minerals, followed by the formation and accumulation of secondary mineral phases. The remaining crystalline phases include magnetite (14%), aluminum phosphate (11%), and magnesium sulfide (11%), while calcium aluminum hydroxide occurs only in a minor amount (1%). The amorphous fraction constitutes 4% of the sample. The amorphous fraction may correspond to poorly crystallized iron oxyhydroxide phases.

Overall, the XRD results indicate that the North Kharasan colmatant is predominantly chemical in origin, with Fe-bearing carbonate phases constituting the principal component of the deposit. This composition supports the interpretation that reactions between sulfuric acid solutions and carbonate minerals of the host rocks are among the key mechanisms responsible for colmatant formation under ISL conditions. The obtained results are consistent with previously reported observations of secondary mineral precipitation during acid in situ leaching [3], [13], [26].

3.1.2. North Karamurun deposit

The X-ray diffractogram of the colmatant sample collected from the North Karamurun deposit is characterized by relatively weak diffraction reflections, indicating a more

heterogeneous mineral structure than that of the North Kharasan sample (Fig. 2). The diffraction pattern suggests a mixed composition involving carbonate, phosphate, siliceous, and sulfide phases. The quantitative phase composition of the sample is presented in Table 3.

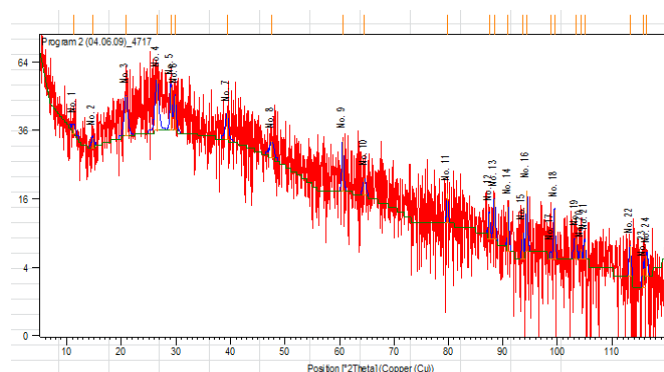


Figure 2. Diffractogram of the colmatant from the North Karamurun deposit

Table 3. Mineralogical composition of the colmatant from the North Karamurun deposit

Component	Chemical formula	Content, %
Magnesite	MgCO ₃	16
Dolomite	CaMg(CO ₃) ₂	13
Quartz	SiO ₂	14
Sphalerite (zinc sulfide)	ZnS	6
Iron pyrophosphate	Fe ₂ P ₂ O ₇	15
Berlinite	AlPO ₄	16
Monetite	CaHPO ₄	20

The mineralogical composition differs markedly from that of the North Kharasan sample. The total carbonate fraction is approximately 29% and consists of magnesite (16%) and dolomite (13%). At the same time, phosphate-bearing phases represent the dominant group in the sample: monetite accounts for 20%, berlinite for 16%, and iron pyrophosphate for 15%, giving a combined phosphate fraction of 51%. Quartz constitutes an additional 14%, while sphalerite is present at 6%.

The coexistence of carbonate, phosphate, and siliceous phases indicates that the North Karamurun colmatant has a more complex mineralogical origin. Carbonate and phosphate phases are primarily formed by chemical precipitation during the interaction of the leaching solution with reactive mineral components. In contrast, the presence of quartz indicates an additional contribution from mechanically transported fine mineral particles. Accordingly, the colmatant can be interpreted as having a mixed chemical-mechanical origin.

The relatively high proportion of phosphate minerals is a distinctive feature of the North Karamurun sample. Previous studies have shown that interactions between acidic lixiviants and phosphate-bearing minerals in sandstone aquifers may promote the formation of secondary phosphate precipitates, which can contribute to pore-space clogging alongside the more commonly reported carbonate-sulfate phases [67].

Overall, the XRD results demonstrate that colmatation at the North Karamurun deposit is controlled by a multicomponent assemblage rather than by a single dominant carbonate phase. The substantial contribution of phosphate minerals, together with carbonates and quartz, should therefore be taken into account when selecting chemical decolmatation reagents, since the treatment must be capable of acting on mineral phases with different chemical reactivities.

3.1.3. Irkol deposit

The X-ray diffractogram of the colmatant sample collected from the Irkol deposit is characterized by sharp and well-defined diffraction reflections, indicating the predominance of well-crystallized mineral phases (Fig. 3).

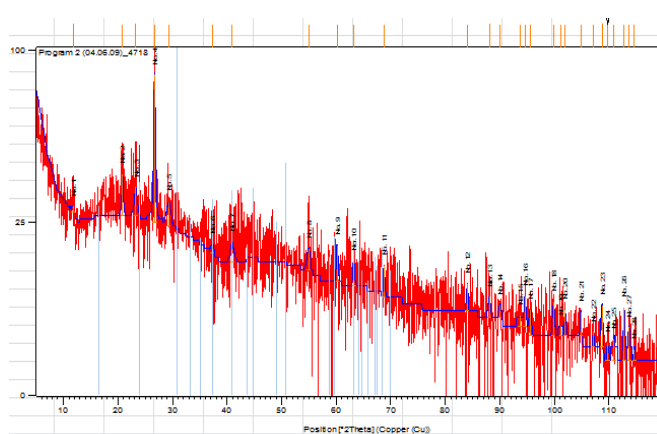


Figure 3. Diffractogram of the colmatant from the Irkol deposit

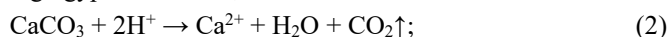
The identified mineral assemblage includes carbonate, sulfate, phosphate, and siliceous components. The quantitative phase composition of the sample is presented in Table 4.

Table 4. Mineralogical composition of colmatant from the Irkol deposit

Component	Chemical formula	Content, %
Quartz	SiO ₂	23
Gypsum	CaSO ₄ ·2H ₂ O	18
Berlinite	AlPO ₄	14
Ankerite	Ca(Mg,Fe,Mn)(CO ₃) ₂	7
Fe-bearing dolomite	Ca(Mg,Fe)(CO ₃) ₂	14
Dolomite	CaMg(CO ₃) ₂	24

The Irkol colmatant is characterized by a substantial carbonate fraction of approximately 45%, comprising dolomite (24%), Fe-bearing dolomite (14%), and ankerite (7%). In contrast to the North Kharasan sample, where ankerite is the dominant carbonate phase, the Irkol sample is dominated by dolomite and Fe-bearing dolomite.

A distinctive feature of the Irkol sample is the relatively high gypsum content, which reaches 18%.



Gypsum precipitation is particularly favored where dissolution of calcium-bearing minerals provides Ca²⁺ for interaction with sulfate ions present in the lixiviant [38], [39]. Its accumulation within the pore space can contribute to a progressive reduction in permeability. The presence of gypsum indicates intensive interaction between calcium-bearing mineral phases and sulfate-rich leaching solutions under sulfuric-acid ISL conditions. This observation is consistent with published reactive transport studies showing that gypsum precipitation can be an important mechanism for secondary mineral accumulation and permeability reduction during leaching.

Quartz accounts for 23% of the sample, while berlinite constitutes 14%. The relatively high quartz content indicates that mechanically transported siliceous particles contribute to the formation of the colmatant, in addition to chemically precipitated phases. Thus, the Irkol deposits can be consi-

dered to have a mixed origin, with chemical precipitation remaining dominant but mechanical particle transport also contributing to the final mineralogical composition.

Overall, the XRD results show that the Irkol colmatant is distinguished by the simultaneous presence of a large carbonate fraction and a significant gypsum component. This combination is particularly important from the standpoint of subsequent decolmatation treatment, because carbonate and sulfate phases may respond differently to acidic and fluoride-containing reagents. The identified mineral assemblage, therefore, provides the mineralogical basis for interpreting the SEM results obtained after chemical treatment and for developing deposit-specific decolmatation strategies.

3.1.4. Comparative analysis of colmatant composition

Comparative analysis of the XRD results reveals substantial differences in the mineralogical composition of colmatants formed at the three studied deposits. Despite these differences, carbonate minerals constitute a major component in all samples, accounting for approximately 57% at North Kharasan, 29% at North Karamurun, and 45% at Irkol. This general tendency indicates that interaction between sulfuric-acid lixiviants and carbonate-bearing components of the productive horizons plays an important role in colmatant formation throughout the studied part of the Syrdarya uranium province.

At the same time, each deposit is characterized by a distinct mineral assemblage. The North Kharasan colmatant is strongly dominated by ankerite, which constitutes 51% of the sample and determines its predominantly carbonate character. In contrast, the North Karamurun sample contains a considerably lower carbonate fraction. It is distinguished by a high proportion of phosphate phases, including monetite, berlinite, and iron pyrophosphate, which together account for 51% of the phases. The Irkol colmatant occupies an intermediate position in terms of carbonate content but differs from the other samples by the simultaneous presence of a substantial gypsum fraction of 18%.

The proportion of quartz also varies considerably between the deposits, increasing from only 2% at North Kharasan to 14% at North Karamurun and 23% at Irkol. Together with the differences in carbonate and phosphate phases, this variation indicates that colmatant formation is controlled not by a single precipitation mechanism but by a combination of

chemical reactions and the accumulation of mineral particles transported within the filtration flow. Nevertheless, the predominance of reactive carbonate and secondary mineral phases suggests that chemical processes remain the principal mechanism of colmatation in the studied wells.

According to [68], the CO₂ content of the host rocks increases from approximately 0.45% in the upper part of the Maastrichtian ore-bearing zone to 1.11% in the lower Campanian zone. In comparison, individual intervals may contain 3-4%. The proportion of silty-clay particles smaller than 0.05 mm varies from 8 to 25%. Such heterogeneity provides different conditions for mineral dissolution, secondary precipitation, and fine-particle migration during ISL operation.

Thus, the XRD results demonstrate that the composition of colmatants is deposit-specific: North Kharasan is characterized by an ankerite-dominated carbonate assemblage, North Karamurun by a carbonate-phosphate assemblage, and Irkol by a carbonate assemblage with a pronounced gypsum component. These differences are important for subsequent decolmatization treatment because the effectiveness of chemical reagents depends on the mineral phases that form the colmatant framework. Accordingly, the mineralogical results provide the basis for interpreting the structural changes observed during SEM analysis after treatment with the two decolmatation solutions.

3.2. SEM evaluation of decolmatation efficiency

3.2.1. North Kharasan deposit

The untreated colmatant sample from the North Kharasan deposit is characterized by a dense framework composed predominantly of tabular crystalline formations with clearly expressed striated surfaces (Fig. 4a). The measured crystal dimensions range from approximately 15 to 37 µm, including L1 = 20.03 µm, L3 = 37.34 µm, and L5 = 15.09 µm. The crystals are randomly oriented and tightly packed, with visible cracks and open voids practically absent, indicating a compact structure in the untreated colmatant.

Treatment with Solution 1 resulted in pronounced destruction of the original framework (Fig. 4b). Deep cavities and large cracks formed between the crystalline particles, while the initial tabular morphology became strongly deformed.

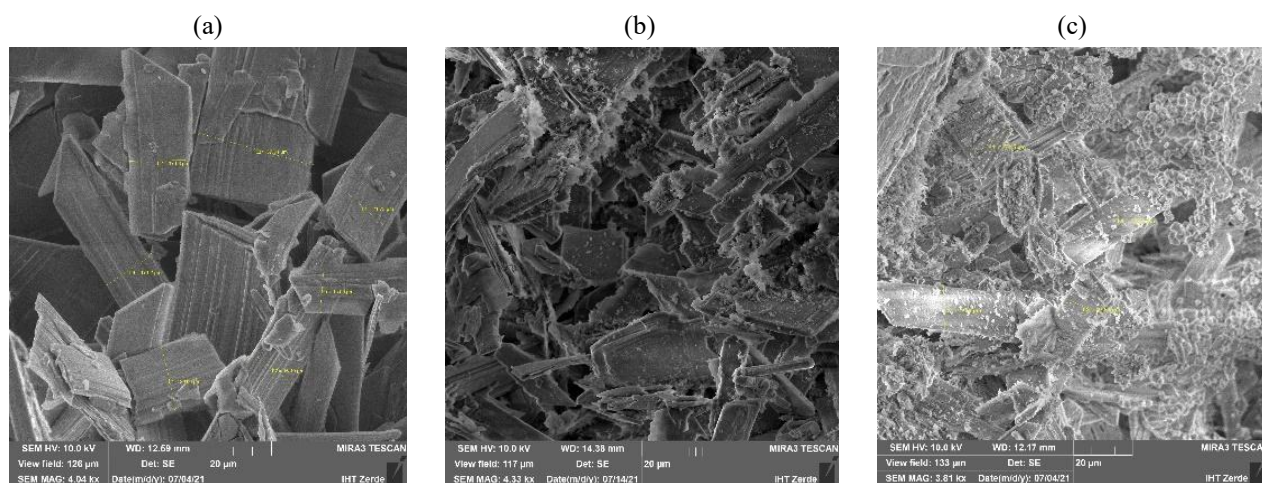


Figure 4. SEM images of the North Kharasan colmatant: (a) untreated sample; (b) after treatment with Solution 1; (c) after treatment with Solution 2

Numerous fine-grained products were also observed on the crystal surfaces, indicating intensive interaction between the reagent and the mineral components of the colmatant. Treatment with Solution 2 also caused substantial structural changes (Fig. 4c). The dimensions of the preserved crystalline fragments decreased to approximately 13-17 μm , with measured values of $L1 = 13.25 \mu\text{m}$, $L2 = 14.73 \mu\text{m}$, $L3 = 13.87 \mu\text{m}$, and $L4 = 17.45 \mu\text{m}$. Numerous globular aggregates of secondary reaction products appeared on the surface, while channels and voids developed within the inter-crystalline space.

Thus, both solutions effectively disrupted the dense structure of the North Kharasan colmatant. Solution 1 produced more pronounced framework destruction, whereas Solution 2 also generated a developed system of voids and channels while reducing the size of the original crystalline formations.

3.2.2. North Karamurun deposit

The untreated North Karamurun sample is characterized by densely packed elongated platy crystals with relatively smooth surfaces and uniform relief (Fig. 5a). The measured crystal dimensions range from approximately 5 to 19 μm , including $L1 = 19.23 \mu\text{m}$, $L2 = 17.90 \mu\text{m}$, $L3 = 4.74 \mu\text{m}$, $L5 = 9.34 \mu\text{m}$.

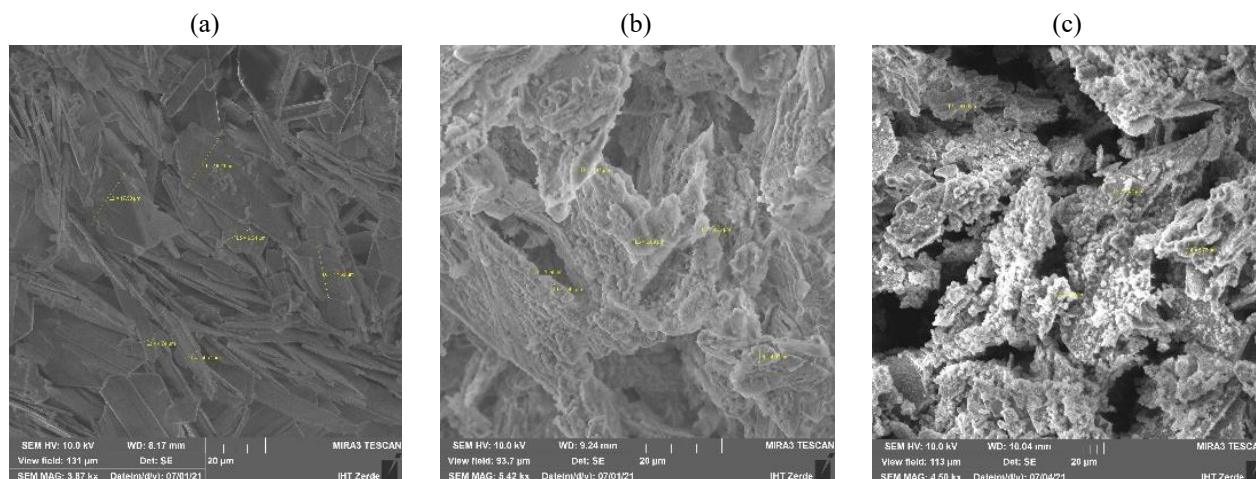


Figure 5. SEM images of the North Karamurun colmatant: (a) untreated sample; (b) after treatment with Solution 1; (c) after treatment with Solution 2

Among the samples studied, treatment with Solution 1 caused particularly severe structural damage at North Karamurun (Fig. 5b). The original platy morphology was largely lost, and the dimensions of the remaining fragments decreased to approximately 1.5-6.7 μm . The measured values were $L1 = 2.42 \mu\text{m}$, $L2 = 4.44 \mu\text{m}$, $L3 = 3.33 \mu\text{m}$, $L4 = 1.59 \mu\text{m}$, and $L6 = 6.69 \mu\text{m}$. The treated surface developed a porous, spongy morphology with deep cavities and rounded etch pits, indicating intensive chemical attack on the colmatant framework.

Treatment with Solution 2 also caused considerable destruction (Fig. 5c). The size of the remaining fragments decreased to approximately 3.5-8 μm , with $L1 = 8.06 \mu\text{m}$, $L2 = 7.04 \mu\text{m}$, $L3 = 5.52 \mu\text{m}$, and $L4 = 5.76 \mu\text{m}$. Granular debris was formed over the treated surface, although some platy fragments retained recognizable morphological features. The degree of cavern development was therefore somewhat lower than that observed after treatment with Solution 1.

The SEM observations demonstrate that the North Karamurun colmatant is highly susceptible to both decolimation formulations. Nevertheless, Solution 1 resulted in greater morphological degradation and a larger reduction in fragment size than Solution 2.

3.2.3. Irkol deposit

The untreated Irkol colmatant consists mainly of densely packed, elongated prismatic and acicular crystalline forms (Fig. 6a). Spherical formations observed on individual crystal surfaces indicate the presence of additional finely dispersed or poorly crystallized phases within the colmatant structure. After treatment with Solution 1, the original structure was substantially destroyed (Fig. 6b). Deep cavities and through-going voids developed within the colmatant framework, while pre-

served fragments decreased to approximately 3.0-7.6 μm . The measured dimensions were $L1 = 7.10 \mu\text{m}$, $L2 = 7.61 \mu\text{m}$, $L3 = 5.53 \mu\text{m}$, $L4 = 3.07 \mu\text{m}$, and $L5 = 3.02 \mu\text{m}$.

Treatment with Solution 2 also caused pronounced morphological changes (Fig. 6c). The treated surface contained numerous globular and spongy aggregates of secondary reaction products, along with large cavities approximately 20 μm in diameter. At the same time, some crystalline fragments measuring approximately 7-10 μm remained within the altered structure. Thus, both solutions produced substantial degradation of the Irkol colmatant structure. Solution 1 predominantly caused fragmentation and development of through-going voids, whereas Solution 2 resulted in extensive cavern formation accompanied by the accumulation of secondary reaction products.

3.2.4. Comparative efficiency of the decolimation solutions

Comparative analysis of the SEM images from all three deposits demonstrates that both investigated solutions are capable of substantially disrupting the initial colmatant framework. In all cases, chemical treatment resulted in fragmentation of crystalline formations, the development of cracks, cavities, and voids, and the formation of secondary reaction products on the treated surfaces.

Solution 1, containing 10% HF, generally caused somewhat more severe structural damage. This effect was particularly evident for the North Karamurun sample, where the dimensions of crystalline fragments decreased to approximately 1.5-6.7 μm , compared with approximately 3.5-8 μm after treatment with Solution 2.

However, the difference between the two treatments was not fundamental.

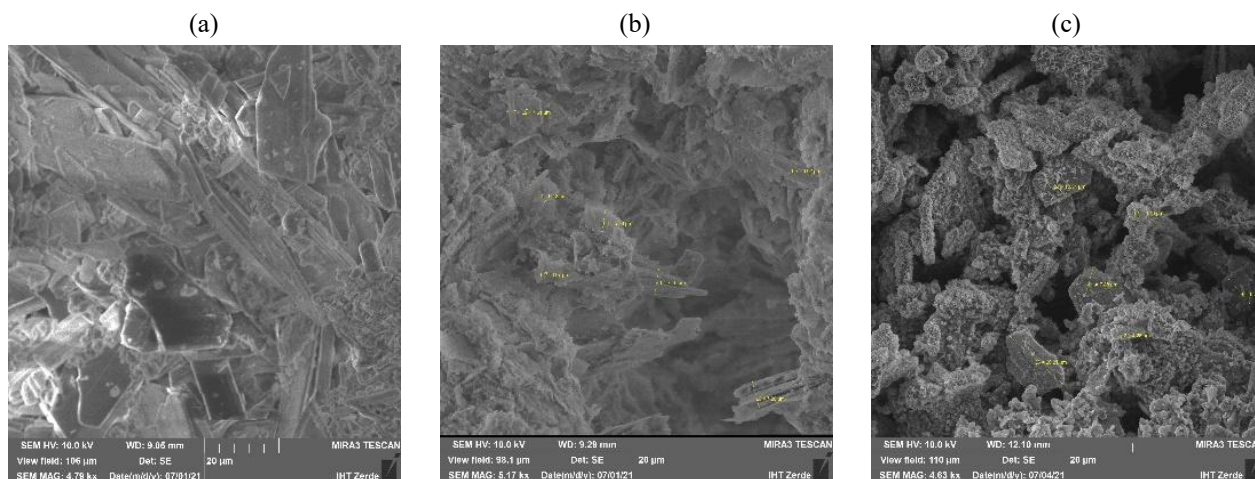


Figure 6. SEM images of the Irkol colmatant: (a) untreated sample; (b) after treatment with Solution 1; (c) after treatment with Solution 2

Solution 2 also produced extensive destruction of the colmatant structure at all three deposits, including the formation of channels, cavities, voids, and fragmented mineral aggregates. Its principal technological advantage is reducing HF concentration from 10% to 5%, corresponding to a two-fold decrease in hydrofluoric acid consumption.

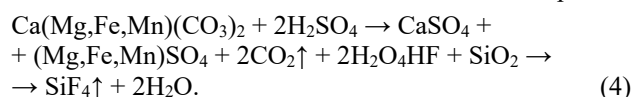
According to the experimental formulation, the lower HF concentration in Solution 2 is supplemented with 10% H₂SO₄ and 1% surfactant. The sulfuric acid component contributes to interactions with carbonate and iron-bearing phases, while the surfactant improves the solution's penetration into the pore space of the colmatant. This combined action helps compensate for the lower HF content and explains the comparable degree of structural disruption observed in the SEM images.

Overall, the SEM results indicate that Solution 1 causes somewhat greater local destruction of the colmatant framework, whereas Solution 2 achieves a comparable overall decolmatation effect with substantially lower HF consumption. From the perspective of reagent consumption and subsequent field application, this difference provides the basis for considering the composite formulation in the deposit-specific treatment strategy discussed in Section 3.3.

3.3. Deposit-specific decolmatation strategy

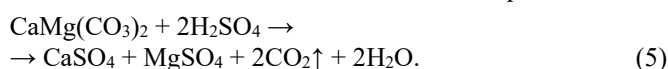
The combined XRD and SEM results demonstrate that the selection of a decolmatation treatment should account for the mineralogical composition of the colmatant formed at a particular deposit. Although both investigated solutions produced substantial structural disruption, the composite formulation containing 5% HF, 10% H₂SO₄, and 1% surfactant (Solution 2) achieved an overall effect comparable to that of 10% HF while reducing the HF concentration by half. Therefore, Solution 2 can be considered the principal experimentally evaluated formulation for further field-oriented assessment at the studied deposits.

At the North Kharasan deposit, the colmatant is dominated by ankerite, which accounts for 51% of the total mineral composition. The predominance of Fe-bearing carbonates indicates that dissolution of carbonate phases is a key requirement for effective treatment. The sulfuric acid component of Solution 2 can promote the decomposition of carbonate minerals, whereas HF contributes to the dissolution of siliceous components:



This combination is consistent with the mineralogical composition of the North Kharasan colmatant and with the structural destruction observed after treatment with Solution 2.

At the North Karamurun deposit, the colmatant has a more complex multicomponent composition, including approximately 29% carbonates, 51% phosphate phases, and 14% quartz. This mineralogical heterogeneity requires a reagent system capable of interacting with several mineral groups. The combined HF-H₂SO₄ formulation is therefore particularly relevant: HF acts on siliceous and phosphate-bearing phases, while sulfuric acid contributes to the dissolution of the carbonate fraction. The interaction with dolomite can be represented as:



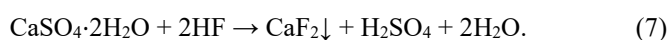
For this type of multicomponent colmatant, ammonium bifluoride (NH₄HF₂) has also been proposed in the literature as a possible alternative to HF because it can generate hydrofluoric acid in situ:



However, NH₄HF₂ was not experimentally investigated in the present study, and its potential application should therefore be regarded as a literature-based option rather than as a result established by the current experiments.

The Irkol deposit requires particular consideration because its colmatant contains approximately 45% carbonate minerals, 18% gypsum, and 23% quartz. For the Irkol deposit, a combined approach is proposed that involves preventing gypsum accumulation during routine ISL operations and periodically removing accumulated carbonate-siliceous deposits. HEDP-based inhibition has previously been proposed to limit gypsum precipitation, whereas the experimentally investigated HF-H₂SO₄-surfactant formulation can be considered for the removal of existing deposits.

A specific limitation of HF-containing treatment in gypsum-rich systems is the possible formation of insoluble calcium fluoride:



Formation of CaF₂ may lead to secondary colmatation of the near-wellbore zone. Therefore, treatment of gypsum-rich colmatants with HF-containing reagents should be accompanied by sufficient pumping and subsequent flushing to remove newly formed solid particles before resumption of normal well operation. This consideration is particu-

larly important for the Irkol deposit due to its relatively high gypsum content.

The relationship between predominant colmatant composition and the corresponding treatment approach is summarized in Table 5. The proposed treatment scheme combines the experimental results obtained in the present study with previously published approaches for mineral

phases that were not independently tested here. In particular, the use of NH_4F + Na_2CO_3 for siliceous colmatants and of NH_4HF_2 -based formulations for mixed deposits is derived from the literature [59], [66]. By contrast, Solution 2 was experimentally evaluated for the carbonate-, phosphate-, and gypsum-bearing colmatants considered in this study.

Table 5. Recommended chemical treatment methods according to predominant colmatant type

Predominant type	Key mineral(s)	Recommended reagent	Basis
Carbonate, Fe-bearing	Ankerite	5% HF + 10% H_2SO_4 + 1% surfactant	This study
Carbonate, Mg-bearing	Dolomite, magnesite	5% HF + 10% H_2SO_4 + 1% surfactant	This study
Phosphate-rich mixed	AlPO_4 , CaHPO_4 , $\text{Fe}_2\text{P}_2\text{O}_7$	5% HF + 10% H_2SO_4 + 1% surfactant	This study
Siliceous	SiO_2	NH_4F + Na_2CO_3 (buffer)	[59]
Gypsum, removal	$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	5% HF + 10% H_2SO_4 + pumping	This study
Gypsum, preventive	CaSO_4 precipitate	HEDP inhibitor	[60]
Mixed multicomponent	All types	NH_4HF_2 + H_2SO_4 + surfactant + pumping	[66], this study

Overall, the results indicate that a deposit-specific decolorization strategy should be based on the dominant mineral phases rather than on a universal reagent formulation. The XRD data identify the mineralogical controls on colmatant composition, whereas the SEM observations provide direct evidence of the structural response to chemical treatment. Their combined application therefore provides a basis for selecting and further validating decolmatation treatments under field conditions.

4. Conclusions

The combined XRD and SEM investigation demonstrated that colmatants formed during uranium in situ leaching at the North Kharasan, North Karamurun, and Irkol deposits have markedly different mineralogical compositions. However, carbonate phases constitute a major fraction in all three cases. The total carbonate content reached approximately 57% at North Kharasan, 29% at North Karamurun, and 45% at Irkol. North Kharasan was characterized by ankerite-dominated colmatants, North Karamurun by a mixed carbonate-phosphate assemblage, and Irkol by a carbonate-rich composition with a significant gypsum component.

The SEM observations confirmed that both investigated decolmatation solutions substantially disrupted the initial colmatant structure. Treatment with 10% HF generally produced somewhat more intensive local destruction, particularly for the North Karamurun sample, where the dimensions of residual fragments decreased to approximately 1.5-6.7 μm . At the same time, the composite solution containing 5% HF, 10% H_2SO_4 , and 1% surfactant produced a comparable overall structural effect while reducing the HF concentration by half.

The results indicate that the efficiency of decolmatation treatment should be evaluated in relation to the predominant mineral phases that form the deposits. Fe-bearing carbonate colmatants such as those at North Kharasan require effective dissolution of carbonate phases. In contrast, the multicomponent carbonate-phosphate-siliceous composition at North Karamurun requires a reagent system capable of interacting with several mineral groups. At Irkol, the simultaneous presence of carbonates and 18% gypsum requires particular attention because HF-containing treatment may generate insoluble CaF_2 , creating a potential risk of secondary colmatation. Adequate pumping and flushing should therefore accompany such treatment under field conditions.

The proposed treatment matrix provides a mineralogy-based framework for selecting decolmatation approaches. The experimentally investigated composite HF- H_2SO_4 -surfactant formulation can be considered for further field validation at the studied deposits, while NH_4F -, NH_4HF_2 -, and HEDP-based approaches included in the treatment strategy are derived from previously published studies and require independent verification under the specific geochemical conditions of the deposits.

Further research should focus on pilot-scale testing of the composite decolmatation solution under operational well conditions, comparative evaluation of HF- and NH_4HF_2 -based formulations, and integration of experimentally determined colmatant compositions into reactive transport models. Such studies would make it possible to relate mineral precipitation dynamics to well productivity decline and to refine deposit-specific treatment protocols for uranium ISL operations.

Author contributions

Conceptualization: ZS, ZK; Data curation: ZK; Formal analysis: ZS, ZK; Funding acquisition: BT; Investigation: YP, AS; Methodology: ZK, YP; Project administration: AM, BT; Resources: AS; Software: BT; Supervision: ZS; Validation: ZK; Visualization: ZS, YP; Writing – original draft: ZS, ZK; Writing – review & editing: ZS, YP, AS, AM, BT. All authors have read and agreed to the published version of the manuscript.

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Conflicts of interest

The authors declare no conflict of interest.

Data availability statement

The original contributions presented in the study are included in the article, further inquiries can be directed to the corresponding author.

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Мінералогічний склад кольматантів при підземному свердловинному вилуговуванні урану: XRD-характеристика та SEM-оцінювання ефективності декольматації

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Мета. Визначити мінералогічний склад кольматантів, сформованих у процесі підземного свердловинного вилуговування урану на родовищах Північний Харасан, Північний Карамурун та Ірколь Сирдар'їнської урановорудної провінції, а також оцінити їх структурну реакцію на хімічну декольматацийну обробку для обґрунтування диференційованих підходів до обробки свердловин з урахуванням особливостей конкретного родовища.

Методика. Зразки кольматантів, відібрані з внутрішніх поверхонь вузлів занурювальних насосів видобувних свердловин, досліджували методами рентгенівської дифракції (XRD) та сканувальної електронної мікроскопії (SEM). Кількісний фазовий склад визначали за допомогою дифрактометра PANalytical X'Pert MPD PRO. Зразки обробляли двома декольматацийними розчинами:

Розчином 1, що містив 10% HF, та Розчином 2, що містив 5% HF, 10% H₂SO₄ і 1% поверхнево-активної речовини. За результатами SEM-досліджень порівнювали зміни морфології поверхні, фрагментацію кристалічних утворень, а також формування каверн і порожнин до та після хімічної обробки.

Результати. Встановлено, що карбонатні мінерали становлять значну частку досліджених кольматантів: близько 57% на родовищі Північний Харасан, 29% – Північний Карамурун і 45% – Ірколь. Для Північного Харасану характерна перевага анкериту, для Північного Карамуруну – змішаний карбонатно-фосфатний склад із часткою фосфатних фаз 51%, а для Ірколю – карбонатний склад зі значним вмістом гіпсу (18%). Обидва декольматційні розчини забезпечили істотне руйнування структури кольматантів. Розчин із 10% HF загалом спричиняв більш інтенсивне локальне руйнування, тоді як композиційний розчин забезпечував порівнянний загальний структурний ефект за зменшення концентрації HF удвічі. Для кольматантів із високим вмістом гіпсу обробка HF може сприяти утворенню осаду CaF₂, що потребує забезпечення достатнього обсягу відкачування та подальшого промивання свердловини.

Наукова новизна. Комплексно поєднано кількісну XRD-характеристику кольматантів, сформованих у реальних умовах експлуатації свердловин, із SEM-оцінюванням їх структурної реакції на два хімічні декольматційні розчини. Встановлено взаємозв'язок між специфічним для кожного родовища мінералогічним складом кольматантів і вибором підходу до їх декольматції.

Практична значимість. Отримані результати формують мінералогічно обґрунтовану основу для вибору способів декольматційної обробки свердловин на родовищах урану, що розробляються методом підземного свердловинного вилугування. Композиційний розчин 5% HF + 10% H₂SO₄ + 1% поверхнево-активної речовини може бути рекомендований для подальшої перевірки в промислових умовах, тоді як для систем із підвищеним вмістом гіпсу необхідно передбачати додаткові заходи щодо запобігання вторинній кольматції, пов'язаній з утворенням CaF₂.

Ключові слова: підземне свердловинне вилугування; видобування урану; кольматация; кольматанти; мінералогічний склад; рентгенівська дифракція; декольматация; осаждение гипсу; обробка свердловин

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