



BENEFICIATION AND PROCESSING OF NATURAL AND TECHNOGENIC RAW MATERIALS

Research paper

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The effect of clay minerals on in-situ leaching of uranium

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Abstract

In recent years, with the development of techniques and methods for in-situ leaching (ISL), additional uranium extraction from previously worked-out blocks is becoming not only relevant, but also quite achievable. In this case, the extraction of residual uranium reserves from previously worked-out blocks does not require additional costs for the necessary infrastructure. One of the most important factors in the formation of residual uranium reserves in worked-out blocks is the presence of clay minerals in the ore horizon. In this regard, we conducted a number of studies on the adverse and positive effects of clay minerals on ISL process. Water permeability and relatively good filtration (not less than 0.5–1 m/day) of ores and rocks of a productive horizon (aquifer) is the most important hydrogeological factors affecting the performance of uranium ISL. The second most important hydrogeological factor is the lack of fluid communication between the productive aquifer and non-productive aquifers, i.e., the obligatory presence of aquicludes. The role of clays in these hydrogeological factors is twofold. On the one hand, the presence of clays negatively affects both the solutions filtration rate and uranium extraction. On the other hand, the presence of clay minerals (forming an aquiclude) enhances the effect of ISL. The study findings allowed the role of clay minerals in uranium ISL to be assessed. The diffusion coefficients of nitrate ions in the clays were determined, and the protective effect of aquicludes was calculated. The effect of the clay content in the ore sand horizon on the solutions filtration coefficients was also established. The static uranium exchange capacity of clays was determined by studying the process of uranium sorption by clay samples from sulfate and bicarbonate solutions. The studies established the diffusion coefficients of nitrate ions in montmorillonite and kaolinite clays, which amounted to $3.34 \cdot 10^{-6}$ and $2.14 \cdot 10^{-6}$ cm²/s. Taking into account the calculated values of diffusion coefficients, the protective time of the clayey aquiclude for nitrate ions was 43 years. At 20 % clay minerals content, the solution filtration coefficient decreases to values where ISL conditions become unfavorable. It was found experimentally that the sorption of uranium by clay minerals depends on both the nature of the clays and the composition of the solution. Uranium sorption from sulfate solutions proceeds noticeably better than that from bicarbonate solutions. The highest values of the static uranium exchange capacity were obtained for bentonite (104 mg/g).

Keywords

uranium, ISL, clay, filtration, diffusion coefficient, water permeability, sorption

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ОБОГАЩЕНИЕ, ПЕРЕРАБОТКА МИНЕРАЛЬНОГО И ТЕХНОГЕННОГО СЫРЬЯ

Научная статья

Влияние глинистых минералов
на процесс подземного выщелачивания уранаО. Ф. Петухов¹, И. У. Халимов¹  , В. П. Истомин², Н. М. Каримов¹ ¹ Навоийский государственный горно-технологический университет, г. Навои, Узбекистан² Навоийский горно-металлургический комбинат, г. Навои, Узбекистан khalimov_i@bk.ru

Аннотация

В последние годы с развитием техники и технологии подземного выщелачивания (ПВ) доизвлечение урана из ранее отработанных блоков становится не только актуальной, но и вполне достижимой задачей, так как извлечение остаточного запаса урана из ранее отработанных блоков не требует дополнительных расходов на необходимую инфраструктуру. Одним из важнейших факторов фор-



мирования остаточного запаса урана в отработанных блоках является присутствие глинистых минералов в рудном горизонте. В связи с этим нами был проведен ряд исследований по изучению отрицательного и положительного влияния глинистых минералов на процесс ПВ. Водопроницаемость и относительно хорошая фильтрация (не менее 0,5–1 м/сут) руд и пород продуктивного горизонта занимают первое место в ряду гидрогеологических факторов, влияющих на эффективность ПВ урана. Вторым по значимости гидрогеологическим фактором является отсутствие гидрологической связи продуктивного водоносного горизонта с непродуктивными горизонтами, то есть обязательное наличие водоупоров. При этом роль глин в указанных гидрогеологических факторах – двоякая. В первом случае наличие глин негативно сказывается как на скорости фильтрации растворов, так и на извлечении урана. Во втором – наличие глинистых минералов (в качестве водоупоров) усиливает эффект ПВ. В результате исследования дана оценка роли глинистых минералов в процессе подземного выщелачивания урана; определены коэффициенты диффузии нитрат-ионов в глинах и рассчитано защитное действие водоупоров; установлено влияние содержания глин в рудном песчаном горизонте на коэффициенты фильтрации растворов; определены статические обменные емкости глин по урану путем исследования процесса сорбции урана на глинистых образцах из сернокислых и бикарбонатных растворов. Проведенными исследованиями установлены коэффициенты диффузии нитрат-ионов в монтмориллонитовых и каолининовых глинах, которые составили $3,34 \cdot 10^{-6}$ и $2,14 \cdot 10^{-6}$ см²/с. С учетом полученных расчетами значений коэффициентов диффузии защитное время глинистого водоупора для нитрат-ионов составило 43 года. При 20%-ном содержании глинистых минералов коэффициент фильтрации растворов снижается до значений, когда условия ПВ становятся неблагоприятными. Экспериментально установлено, что сорбция урана глинистыми минералами зависит как от природы глин, так и от состава раствора. Из сернокислых растворов сорбция урана протекает заметно лучше, чем из бикарбонатных растворов. При этом наибольшие значения статической обменной емкости по урану получены для бентонита (104 мг/г).

Ключевые слова

уран, подземное выщелачивание, глина, фильтрация, коэффициент диффузии, водопроницаемость, сорбция

Для цитирования

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Introduction

In-situ leaching (ISL) mining methods find large-scale implementation and ensure the effective operation of mining industry in many countries [1–4]. The properties of the rocks and minerals involved in ISL processes determine its performance. Therefore, research aimed at studying the key physical and chemical processes in ISL process is of great importance.

In our opinion, insufficient attention has been paid in historical studies to the influence of clay properties on the ISL process [5–8]. For example, in [5, 6], the emphasis was placed on the influence of clays on the filtration of solutions through an ore horizon, while quantitative characteristics of the filtration coefficients were not presented. In [7], for the first time, the role of clayey aquicludes was included in the list of the classification factors for deposit developed by ISL method. However, the numerical values of the diffusion coefficients of elements were not presented, and no examples of calculating the thickness of the protective aquiclude were given. In [8], the influence of clays on the water permeability of an ore layer was described, but similar to [5–7], nothing was said about the influence of clay minerals on the sorption of uranium from pregnant solutions, leading to losses of leached metal.

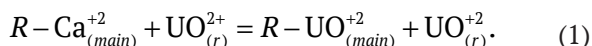
The purpose of our study was: a) to estimate the coefficients of diffusion in clay minerals and calculate the thickness of a protective aquiclude; b) to study the effect of the content of clay minerals on the solutions filtration coefficient; and c) to study the sorption of uranium by clay minerals.

Positive effect of the properties of clay minerals on ISL processes

In order to establish the thickness (height) of a clayey aquiclude as a screen to protect against the harmful effects of leaching solutions on natural waters, we conducted studies to determine the diffusion coefficients of some chemical components in clays. Since clay aquicludes are mainly represented by montmorillonite (bentonite), less often kaolinite, these clay minerals were considered in the study. First, it was taken into account that the rate of penetration of chemical components through clays is limited by diffusion [9]. Second, a relatively toxic NO_3^- – anion (MPC = 45 mg/L) present in the pregnant solutions of uranium ISL was selected as the chemical component to be studied.

It is incorrect to use metal cations (e.g., UO_2^{2+}) as chemical components in diffusion studies, since the diffusion process is accompanied by cation-exchange

sorption of metals by clays (in exchange for Ca^{2+} , Mg^{2+} , Na^+) by reaction:



As for anions, according to data of [9] and our studies [10], NO_3^- anions are not sorbed by bentonite and kaolinite.

Samples of intact bentonite and kaolinite clays were used in the experimental studies. The identification of constituent minerals of the clays was carried out using a “Dron-3.0” X-ray diffractometer with a tube 03BSV – 25-Cu. The content of exchangeable forms of cations and the total cation-exchange capacity (CEC) of the clays were determined based on their interaction with a 0.1N solution of ammonium acetate at pH = 7.0 [11]. The composition of the clay samples is presented in Table 1. It shows that alkaline-earth bentonite contains 93 % of montmorillonite, while the content of the main mineral in the kaolinite clay sample is 88.2 %.

The studies to determine the diffusion coefficients in the clays were carried out according to the technique described in [12, 13] in a laboratory setup, the schematic of which is shown in Fig. 1.

An intact clay sample with known area S and thickness h was placed in a glass tube. Gauze swabs were placed on both sides of the sample for stability and destruction prevention. The tube containing the

clay sample was connected to the vessels through the rubber stoppers. Vessel A was filled with a solution of volume V_A with known concentration NO_3^- . Vessel B was filled with distilled water of volume V_B . The same level of the liquids in the vessels was maintained on both sides. Magnetic stirrers were used to stir the solutions, in order to create a uniform concentration of NO_3^- throughout the whole volume. The conditions of the experiments were constant: $t = 200^\circ\text{C}$, stirring solutions at a rate of $n = 50$ rpm; $V_A = V_B = 1$ L.

The diffusion coefficients were calculated based on the experimental data obtained using Fick's first law equation:

$$D = \Delta m(h / \tau)S(C_A - C_B), \quad (2)$$

where D is diffusion coefficient, cm^2/s ; Δm is weight of substance transferred during diffusion, mg, $\Delta m = C_B V_B$; h is thickness of clay sample, cm; τ is diffusion time, s; S is area of clay sample, cm^2 ; C_A is equilibrium concentration in vessel A, mg/cm^3 ; C_B is equilibrium concentration in vessel B, mg/cm^3 .

The conditions and findings of the experiments and the calculated diffusion coefficients of NO_3^- – ions in the clays are presented in Table 2.

As can be seen from Table 2, the values of the diffusion coefficients obtained agree well with the findings of [14, 15]. In particular, according to [15], the diffusion coefficient of Cl^- in bentonite was $3,0 \cdot 10^{-6} \text{ sm}^2/\text{s}$.

Table 1

Clay composition

Clay sample	Mineral composition, %				Content of counterions, g/kg				CEC, g-eq/kg
	Montmorillonite	Kaolinite	Sericite	Quartz	Ca^{2+}	Mg^{2+}	Na^+	K^+	
Alkali-earth bentonite	93.0	2.0	5.0	–	6.32	0.60	0.05	0.15	0.372
Kaolinite	–	88.2	5.8	6.0	1.85	0.19	0.07	0.12	0.113

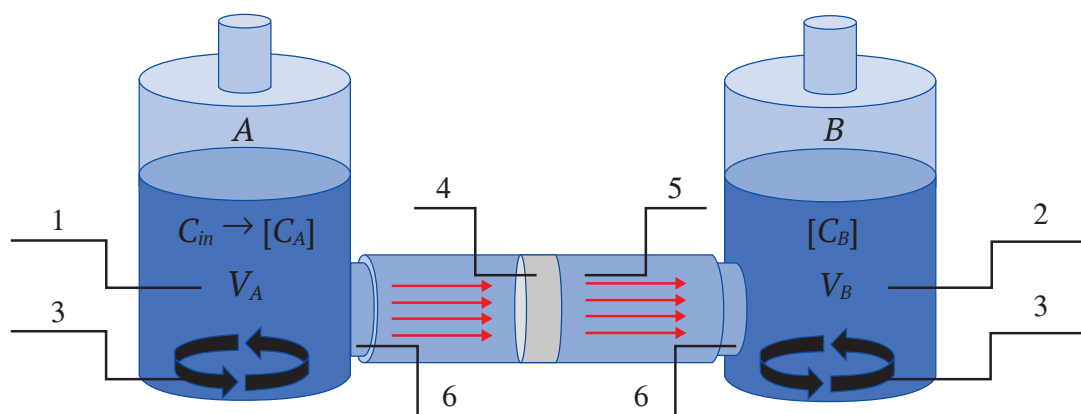


Fig. 1. Schematic of the laboratory setup for determining the diffusion coefficients in clays: 1 – vessel A with initial concentration C_{in} , equilibrium concentration C_A and volume V_A ; 2 – vessel B with equilibrium concentration C_B and volume V_B ; 3 – stirring devices; 4 – intact clay sample; 5 – glass tube connecting the vessels; 6 – rubber stoppers

Secondly, the diffusion coefficient in bentonite is about 1.5 times higher than that in kaolinite. This is explained by the fact that kaolinite has a strong, fixed lattice, the interpacket distance in which is 0.71–0.72 nm. In montmorillonite, the crystal lattice packet is movable, three-layered; the interpacket distance is 1.5 nm and can expand up to 14 nm. The difference in the lattices of these clays can be clearly seen in Fig. 2.

The established values of diffusion coefficients are of great practical importance, as they allow calculating the clay aquiclude protective action time. For example, the concentration of NO_3^- -ions in the pregnant solutions of the ore horizon $C_{in} = 2,0 \text{ g/l}$ (2 mg/cm^3) and does not change in the process due to flow renewal. The concentration of NO_3^- -ions in natural waters should not exceed $C_B = 0.045 \text{ g/l}$ (0.045 mg/cm^3). The area of the bentonite aquiclude $S = 1000 \text{ m}^2$ ($1 \cdot 10^7 \text{ cm}^2$), and the thickness $h = 10 \text{ m}$ ($1 \cdot 10^3 \text{ cm}$). The volume of natural water is taken to be

2000 m^3 ($2 \cdot 10^9 \text{ cm}^3$). The weight of NO_3^- , transferred by diffusion to natural waters, $\Delta m = 9 \cdot 10^7 \text{ mg}$. According to formula (2), the time of the protective action of the aquiclude is determined as follows:

$$\begin{aligned} \tau &= m(h/D)S(C_A - C_B) = \\ &= 9 \cdot 10^7 \cdot 1 \cdot 10^3 / 3,34 \cdot 10^6 \cdot 1 \cdot 10^7 \cdot (2,0 - 0,045) = \\ &= 1,37 \cdot 10^9 \text{ s or 10 years.} \end{aligned}$$

However, right there the positive effect of clays on the ISL process ends.

Negative effect of the properties of clay minerals on ISL processes

The other three properties of clay minerals: extremely low filtration coefficient ($K_f = 10^{-4} - 10^{-6} \text{ m/day}$ [15, p. 3–6]), noticeable sorption properties in relation to metal cations [10], and the tendency to form colloidal solutions have a negative impact on the process of uranium ISL.

Table 2

Diffusion coefficients determination results

Clay sample	Experimental conditions				Findings				
	$h, \text{ cm}$	$S, \text{ cm}^2$	$C_{\text{NO}_3} (\text{in}), \text{ mg/cm}^3$	$\tau, \text{ s}$	$[C_A], \text{ mg/cm}^3$	$[C_B], \text{ mg/cm}^3$	$C_A - C_B, \text{ g/cm}^3$	$\Delta m, \text{ mg}$	$D, \text{ cm}^2/\text{s}$
Bentonite	0.5	7.0	1.0	$2.6 \cdot 10^5$	0.65	0.35	0.30	350	$3.34 \cdot 10^{-6}$
Kaolinite	0.5	7.0	1.0	$2.6 \cdot 10^5$	0.70	0.30	0.40	300	$2.14 \cdot 10^{-6}$

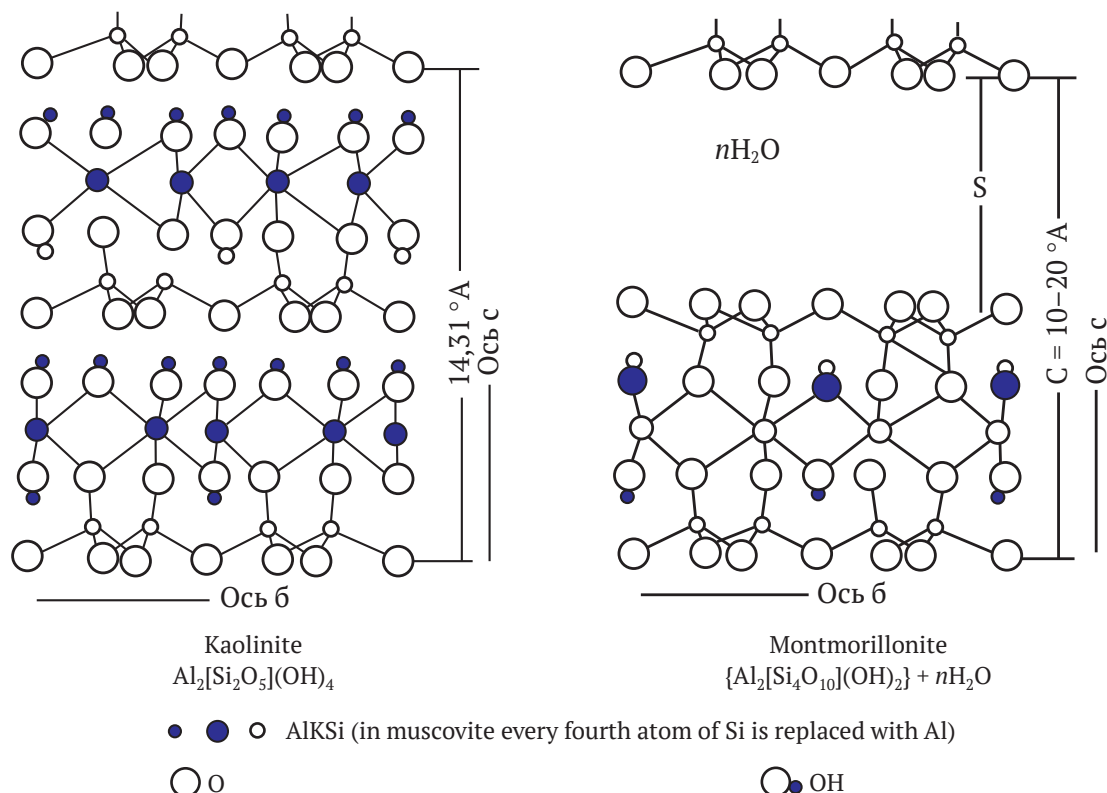


Fig. 2. Crystal lattices of clay minerals



Table 3

Effect of clay content in sand on water and solution filtration coefficients

Clay content in sand, %	K_f in the presence of bentonite, cm/s · 10 ⁻²			K_f in the presence of kaolinite, cm/s · 10 ⁻²		
	H ₂ O	10 g/l H ₂ SO ₄	1 g/l NaHCO ₃	H ₂ O	10 g/l H ₂ SO ₄	1 g/l NaHCO ₃
0.0	2.2	2.2	2.2	2.2	2.2	2.2
3.0	1.2	1.1	1.0	1.3	1.3	1.1
6.0	0.9	0.8	0.6	1.1	1.0	0.9
15.0	0.4	0.3	0.2	0.6	0.5	0.4
20.0	0.2	0.1	0.05	0.3	0.2	0.1

First, let us elaborate on low filtration coefficients of clays. Permeable ore horizons mainly consist of sand. In addition to sand, an ore horizon often contains clay minerals that cement the sand (hence the name “sandstone-type deposits” has arisen [16]). The content of clay minerals limits the rate of solution filtration through the ore horizon.

We performed experimental studies, in order to determine the coefficients of filtration of water and solutions through sand and a mixture of sand with different content of clay minerals (bentonite and kaolinite). The studies were conducted in steady-state regime (the filtering material layer was unchangeable in all experiments) at a constant head gradient according to GOST 25584–2016¹. In the experiments, a thoroughly washed sand of 0.25–0.50 mm fraction and powdered clays were used (see Table 1). Samples of the sand with the given clay content were thoroughly mixed before being loaded into the cylinder of the filtration unit. Water and solutions of 10 g/l H₂SO₄ and 1 g/l NaHCO₃ were used as filtering liquids. Filtration coefficient K_f was calculated by Darcy equation based on the experimental data obtained (using the determined filtration rate).

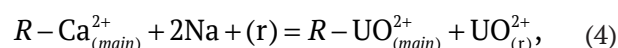
$$v = K_f (\Delta P / h), \quad (3)$$

where K_f is filtration coefficient, cm/s; v is filtration rate, cm/s, $v = V/S\tau$; V is volume of filtered liquid, cm³; S is filtration area, cm²; τ is filtration time, s; $\Delta P/h$ is pressure gradient in the layer (slope ratio of the filtration rate versus filtration time plot); ΔP is water pressure fall, cm; h is thickness of filtering layer, cm.

The findings of the studies to determine the filtration coefficients are shown in Table 3.

The data presented in Table 3 shows that K_f of the liquids decreases markedly with increasing the clay content. Notice that K_f in the presence of bentonite

is lower than in the presence of kaolinite. This is due to the different swelling properties of the clays, as mentioned above. The use of sulfate and sodium bicarbonate solutions also affects the K_f , and the use of NaHCO₃ solution (even at a lower concentration than that of the sulfate solution) has a stronger effect. This is explained by the fact that, due to the cationic exchange of Na⁺ ions for Ca²⁺ counterions in the clays,



clay swelling takes place. At the content of clay of 20 % K_f decreases to the values, at which the conditions become unfavorable for ISL [6, p. 405].

c) Next, let us focus on the sorption properties of clays, which produce adverse impact on uranium ISL process. Table 4 presents the data on CEC values of some clay minerals [17].

As shown in Table 4, vermiculite and montmorillonite (the main mineral of bentonite clays, being the most characteristic of uranium ISL conditions), have the highest CEC values.

Table 4

Cation exchange capacities of clay minerals

Clay type	Mineral	CEC, g-eq/kg
Kaolinite group	Kaolinite	0.02–0.10
Illite group	Muscovite	0.105
	Illite	0.13–0.42
Fibrous clay	Attapulgitite	0.18–0.22
Montmorillonite group	Nontronite	0.57–0.64
	Saponite	0.69–0.81
	Montmorillonite	0.8–1.5
Resinous derivatives	Biotite	0.03
	Vermiculite	1.0–1.5

¹ GOST–2016 25584. Soils. Methods for laboratory determination of filtration coefficient. Moscow: Standardinform; 2016.



We have conducted studies specifically on the sorption of uranium from different solutions by bentonite and kaolinite clays. The studies were performed under static conditions with the use of a thermostatically controlled stirred reactor at a temperature of 20 °C, Solid / Liquid ratio of 1000, and the agitation time of 1 h (saturation regime) [17]. After agitation, the suspension was filtered, the sediment was washed with water, dried, and analyzed for uranium by X-ray spectral analysis at a ARF-7 spectrometer. The experiments used actual production solutions taken from sulfate and bicarbonate uranium ISL sites. The composition of the solutions is presented in Table 5. Table 6 presents the experimental data on the uranium static exchange capacity (SEEC) of the clays.

The data presented in Table 6 shows that the sorption of uranium by clays depends significantly on the nature of both the clay and the test solution. Uranium SEEC of bentonite is markedly higher than that of kaolinite. This can be explained by the higher values of bentonite CEC (see Table 1). The uranium sorption from sulfate solutions is also markedly higher than that from bicarbonate solutions. This is because the stability constants of sulfate anionic uranyl complexes ($K = 76\text{--}2500$) is much lower than those of the carbonate complex ($K = 2,0 \cdot 10^{18}$). It is very likely that the sorption of uranium from bicarbonate solutions proceeds not by the ion-exchange mechanism, but as adsorption.

Clayey rocks of sedimentary strata often contain carbonaceous organic matter, which is also a good sorbent for uranium.

It should be noted that laboratory studies do not fully simulate natural conditions. The differences between the laboratory experimental conditions and the natural processes consist in the lack of rock pressure and the associated changes in the values of porosity, moisture, and anisotropic component of the artificially prepared clay layer.

The simulation of rock pressure during the research requires the use of special installations. As for the isotropy of samples, it is clear that the quantitative relationships between individual parameters can be established only when the composition is homogeneous and its characteristics are known.

In any case, in natural ISL conditions, along with the process of uranium leaching, a parallel process of partial sorption of uranium by clay minerals ensues. This is both by the boundary surface of clay aquicludes, and by the clays contained in a sandy ore horizon.

In conclusion, let us consider three other properties of clay minerals which produce an adverse impact on the ISL process. First of all, this is the ability of clays to form colloidal species. When clays interact with a large volume of solutions that takes place at ISL, peptization, the process of forming clay colloidal species (gels) proceeds. The gels entrained by the flow of pregnant solutions, move towards extraction wells. Some of the gels are sorbed on the surface of the host rock, some are deposited on filters, and some are pumped out with the pregnant solution for sorption uranium extraction. In order to prevent the gels and other solids from entering the sorption columns, at the ISL mines, settling sumps are provided. However, in those cases where the concentration of colloidal species is high (turbid opalescent solutions are formed), the area of settling sumps is not enough, a clogging of the sorption resin with these species happens. This leads to reducing the performance of sorption columns, and sometimes to their shutdown. In this case, the uranium sorbed by colloidal species of clay is lost with the sediments at the bottom of the sumps.

When drilling and constructing ISL process boreholes, clay layers and interlayers are often intersected in the stratigraphic sequence. During the drilling process, the clay drowning takes place, causing them to swell inside the borehole, resulting in reducing the specified borehole diameter. This, in turn, prevents the casing from descending. In practice, we have to use technical means to eliminate this problem that leads to additional labor costs when casing boreholes.

Drilling, including the penetration of a productive horizon, is carried out with the use of clay mud as a washing fluid, creating a clay crust on the borehole

Table 5

Composition of ISL production solutions

Solution	Concentration, mg/l						pH
	U	SO ₄ ²⁻	HCO ₃ ⁻	Fe ³⁺	Fe ²⁺	Al ³⁺	
Sulfate	40.4	12750	–	730	240	544	1.2
Bicarbonate	36.2	67	665	30	–	–	9.3

Table 6

The findings of determining static uranium exchange capacity of clays

Clay sample	Uranium SEEC of clays, mg/g	
	from sulfate solution	from bicarbonate solution
Bentonite	104	15
Kaolinite	32	5



walls. In the filter installation interval, claying of the near-filter zone also takes place. Preflushing the borehole with clean water and pumping after casing are carried out for the filter cake removal. These measures do not allow the near-filter zone to be completely cleared from clays which subsequently adversely affect the borehole productivity. It is especially difficult to clean a borehole from clays in the conditions of low groundwater head on the productive horizon roof.

When constructing the process sumps, a special attention is paid to waterproofing of their bottoms. The bottom is waterproofed using bentonite clays, which, as mentioned above, also sorb uranium that leads to its losses.

Conclusions

1. Clay minerals have a great deal of both positive and adverse effects on uranium ISL process.
2. Clay minerals are very effective as aquicludes, preventing the flow-out of pregnant and extracting solutions. The studies conducted enabled the diffusion coefficients of nitrate ions in montmorillonite

and kaolinite clays to be determined, amounting to $3,34 \cdot 10^{-6}$ and $2,14 \cdot 10^{-6} \text{ cm}^2/\text{s}$.

3. Taking into account the calculated values of the diffusion coefficients, the protective time of the clayey aquiclude for nitrate ions was 43 years.

4. Clay minerals present in a sand ore horizon adversely affect the filtration coefficient of the solutions. The decrease in the filtration coefficients of the solutions with increasing the content of montmorillonite and kaolinite clays in a sand ore horizon has been experimentally established. At 20 % content of the clay minerals, the solution filtration coefficient decreases to values at which the conditions become unfavorable for the ISL.

5. Exhibiting cation-exchange sorption abilities, clay minerals also adversely affect uranium extraction. It was found experimentally that the sorption of uranium by clay minerals depends on both the nature of the clays and the composition of the solution. The uranium sorption from sulfate solutions proceeds noticeably better than from bicarbonate solutions. The highest values of the static uranium exchange capacity were obtained for bentonite (104 mg/g).

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