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Technology for increasing the oxidation potential of recycled solutions for uranium in-sit leaching

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Abstract. This article presents theoretical studies of the physicochemical mechanisms of leaching processes related to the oxidation-reduction conditions of the environment, the ability of elements to form compounds with other elements, form ions of varying valence, yield soluble or gaseous compounds. The Semizbay deposit's ores are visually indistinguishable from the host rocks and are distinguished only by sampling. Uranium is found in these ores in both mineral and sorbed forms. The ores are primarily loose and poorly cemented; carbonate ores make up 20% of the deposit's ores and are the primary acid absorbers during sulfuric acid leaching. The ionic composition of the uranium leaching solution during the acidification stage of the formations was analyzed, and the interaction of sulfuric acid solutions with the ore-hosting rocks was assessed.

Keywords: research, regularity, in-situ leaching, physicochemical processes, oxidation potential, elements, compounds, uranium
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Технология повышения окислительного потенциала регенерированных растворов для подземного выщелачивания урана

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Резюме. В данной статье представлены теоретические исследования закономерностей физико-химических процессов выщелачивания, которые связаны с окислительно-восстановительной обстановкой среды, способностью элементов вступать в соединения с другими элементами, образовывать ионы различной валентности, давать растворимые или газообразные соединения и т.д. При этом вещественно-минералогический состав рудообразующих элементов и состав растворителей, участвующих в процессе выщелачивания, определяют характер химических реакций, расход реагентов и в целом геохимические условия выщелачивания урана. Руды месторождения Семизбай визуально не отличаются от вмещающих пород и выделяются только по результатам опробования. Уран в них находится в минеральной и сорбированной формах. Руды представлены в основном рыхлым и слабо сцементированным материалом; руды на карбонатном цементе - карбонатные руды составляют 20% руд месторождения и являются основными поглотителями кислоты при сернокислотном выщелачивании. Проведен анализ формирования ионного состава раствора выщелачивания урана на стадии закисления пластов и дана оценка взаимодействия сернокислотных растворов с рудовмещающими породами. Анализ выпол-

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нен по данным полученным с полигонов ПВ Казахстана. При начале работ на месторождении Семизбай способом ПВ, ионный состав растворов будет уточняться.

Ключевые слова: исследования, закономерность, подземное скважинное выщелачивание, физико-химические процессы, окислительный потенциал, элементы, соединение, уран

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Introduction

The efficiency of mining the Semizbay uranium deposit using sulfuric acid leaching depends on the correct selection of in-situ leaching (CSL) technology parameters. This, in turn, depends on a proper understanding of the physical and physicochemical processes that determine the geochemical conditions for the dissolution, transport, and precipitation of substances during uranium leaching.

Sorbed uranium in ores is associated with carbonized plant fragments, clay minerals of cement, and iron hydroxides. Uranium minerals include coffinite, pitchblende, uranium black, and rare secondary minerals. In addition, uranium-bearing ilmenite, titanomagnetite, and leucoxene are present in small quantities [1-3].

The ore-bearing rocks of the deposit contain both poorly soluble and readily soluble minerals-carbonates, sulfides, etc. The presence of CO_2 in the environment leads to the dissolution of calcium and magnesium carbonates and the formation of readily soluble bicarbonates [3, 4]:

$$CaCO^3 + H^2O + CO^2 \rightarrow Ca(HCO^3)^2 \text{or}$$
$$2CaCO^3 + H^2SO^4 \rightarrow CaSO^4 + Ca(HCO^3)^2 \quad (1)$$

The primary acid absorbers during leaching are the minerals that cement the rock. The main cement minerals are: hydromicas, kaolinite, montmorillonite, ferrous chlorites or mixed-layer minerals of chlorite-montmorillonite composition, carbonates, iron hydroxides, allophane group minerals, and other difficult-to-detect minerals.

The most common clay mineral, found in virtually all types of ore-bearing rocks, is hydromica. Its content varies depending on the lithological type of the rock, from 3-5% to 15-25%. Hydromica forms cryptocrystalline aggregates and develops primarily after detrital micas and feldspars.

Kaolinite is found almost everywhere, but in relatively smaller quantities than hydromica. Its highest contents, up to 15-20%, are observed in gray-colored and bleached gray-colored fine-grained rocks. In the permeable part of the section, its content does not exceed 1-10%. Montmorillonite, like hydromica and kaolinite, is one of the main components of the clay cement of ore-bearing rocks. Its content ranges from 0 to 10-15%, averaging 6-7% in individual sections (including clay-silt deposits) [5-8].

According to the activity of interaction with sulfuric acid, the minerals that make up the rock cement can be arranged in the following order: calcite, ferrous dolomite, iron oxides, mixed-layer chlorite-montmorillonite, siderite, chlorite, iron oxides, montmorillonite, hydromica, kaolinite, carbonized organic matter.

Research methods

Clay minerals dissolve significantly more slowly, throughout virtually the entire leaching process. The proportion of reagent consumed by this group of minerals is complexly dependent on their quantity, active surface area (size of crystalline aggregates), and contact time with the solvent. With prolonged leaching, reagent consumption for this group of minerals can reach significant levels. The proportion of acid consumed by other minerals, due to their small quantities, is insignificant. Table 1 shows the solubility product of some chemical compounds in the ore-bearing rocks of the Semizbay deposit.

Table 1 / Таблица 1

Solubility of some chemical compounds in the ore-bearing rocks of the Semizbay deposit / Растворимость некоторых химических соединений в рудовмещающих породах месторождения Семизбай

Sulfides, g/l			
FeS	$4{,}0 \times 10^{-19}$	MnS	$1{,}4 \times 10^{-15}$
PbS	$1{,}0 \times 10^{-29}$	CuS	$8{,}0 \times 10^{-37}$
Sulfates, g/l			
$PbSO_4$	$2{,}0 \times 10^{-8}$	$CaSO_4$	$6{,}1 \times 10^{-5}$
Carbonates, g/l			
$PbSO_3$	$1{,}5 \times 10^{-13}$	$FeSO_3$	$2{,}5 \times 10^{-11}$
$MnCO_3$	$1{,}0 \times 10^{-10}$	$MgCO_3$	$1{,}1 \times 10^{-5}$
$CaCO_3$	$4{,}8 \times 10^{-9}$	$CaSO_3$	$1{,}4 \times 10^{-10}$

An analysis of the ionic composition of uranium leaching solutions during the acidification stage of the formations was conducted, and the interaction of sulfuric acid solutions with ore-bearing rocks was assessed. The analysis was based on data obtained from leaching sites in Kazakhstan. The ionic composition of the solutions will be refined when leaching operations at the Semizbay deposit commence.

Below is a brief summary of the work performed.

In solutions, uranium can exist in four valence states: U^{3-}, U^{4+}, U^{5+}, and U^{6+}. Uranium's multivalence is due to the different energy bonds of the electrons in its shells. This variable valence leads to the formation of various uranium complexes. When leached in an acidic medium, uranium forms divalent uranyl cations (UO^{2+}), and in an alkaline medium, uranite anions. It has a weak affinity for sulfur and a strong affinity for oxygen.

Aqueous solutions of trivalent uranium salts are highly unstable. Tetravalent uranium salts form more stable solutions. Atmospheric oxygen oxidizes tetravalent uranium in aqueous solutions to hexavalent uranium. Pentavalent uranium forms the uranyl radical (UO) in aqueous solutions in an extremely unstable form, converting into tetravalent and hexavalent uranium ions. Compounds of hexavalent uranium are of practical interest for uranium extraction using sulfuric acid leaching. All such compounds are characterized by the formation of the uranyl ion, which determines the properties of aqueous solutions of uranyl salts during leaching.

Analysis of the graph shows that increasing the solution pH from 0.5 to 1.5-2.0 leads to a decrease in the uranyl cation content

and a sharp increase in the content of uranyl sulfate complexes in the solution. A further increase in the solution pH from 2.0 to 2.5 leads to the virtual disappearance of the uranyl cation and the replacement of neutral uranyl sulfate molecules with uranyl disulfate and uranyl trisulfate anions. In the pH range of 2.0-2.5, the uranyl trisulfate content increases to 90-100%.

A further increase in pH to 3.0-3.5 leads to the polymerization of ions with the formation, as a result of hydrolysis, of complex ions of uranyl dimer tridisulfate $[UO_2O_5(SO_4)_3]^{4-}$, $[UO_2O_5(SO_4)_2]^{2-}$. Experience in processing leach solutions allows us to conclude that conducting sorption in the pH = 3.0 – 3.5 mode leads to an increase in the sorption capacity of the resin for uranium due to the fact that the resin sorbs predominantly divalent metal complexes, which are absorbed by strongly basic anion exchangers more intensively than tetravalent ones [3, 6].

Based on the above, the leaching process must be carried out with constant monitoring of the pH, Eh of the solution, and the content and percentage ratio of uranyl sulfate ion complexes, maximizing the formation of uranyl dimer disulfate complex ions $[UO_2O_5(SO_4)_2]^{2-}$ in productive solutions.

In addition to useful metals, macroimpurities of SO_4^{2-}, $HSO_4^-, CO_3^{2-}, HCO_3^-$, Ca_2^+, Mg_2^+, Fe_2^+, NO_3^-, Al_3^+, Cu^+, Mn_4^+, Ti, Ni, Zr, Bi, Li, Zn and other ions are formed in sulfuric acid leach solutions during the acidification stage of the formations.

It is important to note the depressant effect of certain anions in the leaching solution on sorption. This effect is exerted by the following ions: SO_4^{2-}, $NO_3^-, Cl^-, P^-$and PO_4^{3-}.

Low-polymer (mono- and dimeric) silicic acids (α and β-forms) formed in the solution have a strong toxic effect on resins. The most stable regions of existence of low-polymer silicic acids are formed at pH +1 to +3. Cases of resin poisoning by the accumulation of VO_3^-, VO_4^{3-}, $MoO_4^{2-}$and MoO_4^2 - anions have also been observed. Removal of these anions is only possible by leaching the resin with special solutions ($NaOH$, NH_4NO_3, $NaNO_3$, NaN, etc.).

Results

Since the primary goal of this study is to significantly reduce sulfuric acid consumption during uranium leaching, it is necessary to briefly examine the leaching process.

The leaching process is significantly influenced by the interaction of the sulfuric acid solution injected into the formation with non-metallic minerals in sedimentary rocks. Moreover, the redox environment in the leaching zone changes significantly at different stages of the process and directly depends on changes in the ionic composition of the solution and its changing ionic strength.

During the first stage of leaching (acidification), the presence of carbonates in the rocks accounts for the majority of acid consumption, creating a different CSL environment than during the subsequent leaching stage. Only after the carbonates have disappeared do the much slower reactions with aluminosilicates become decisive. The stage-by-stage change in the ionic strength of the resulting solutions, the moments of equilibrium and carbonate disappearance, and the changes in Eh and pH can be calculated using thermodynamic analysis using specialized computer programs (in particular, "Selector").

The relatively high rate of interaction of sulfuric acid solutions with carbonates, on the one hand, and the significant duration (from several weeks to several months) of the stay of solutions in the subsoil make it possible to interpret the chemistry of the CSL process at the acidification stage using calculation methods, which is much more difficult to do at the subsequent dynamic stages of CSL.

Since the process flow diagram used at CSL sites and in Kazakhstan involves the use of recycled solutions containing a number of petrogenic elements (Ha, K, Ca, Mg, Al, Fe), as well as C^- and NO_3^- ions, their influence on the change in ORP during interaction with carbonates in mineralized sulfuric acid solutions cannot be ignored. The interaction of sulfuric acid solutions with carbonates is accompanied by a significant increase in pH, and in addition to simple metal ions and their sulfate complexes, well-known hydrocomplexes, as well as bicarbonate and carbonate complexes, appear in the solutions.

Calculating equilibria in such complex systems, necessary for developing management decisions for the CSL process, is impossible without the use of a computer. The equilibrium principle allows for the application of the free energy minimization method (I.K. Karpov's method, applied to multicomponent heterogeneous systems containing an aqueous electrolyte solution and a gas phase) for the quantitative analysis of irreversible chemical processes. The "Selektr" program allows for the quantitative characterization of not only the overall equilibrium in the system but also the concentration of components in the CSL solution, as well as the molar amounts of substances in the solid phase, depending on the degree of reaction. This will allow us to describe the intermediate state of the system as it progresses toward full equilibrium and to objectively assess the dynamics of CSL changes.

Based on available experimental data, production data, and publications [9-12], the interaction of sulfuric acid solutions with ore-bearing rocks can be characterized as follows:

The deposition of material and the formation of zoning near the injection well continue until calcite completely disappears from all zones containing newly formed formations, after which the newly formed formations begin to dissolve and redeposit in calcite-bearing rocks closer to the pumping wells. When fresh portions of solution enter the injection well, the process continues with the dissolution of the redeposited formations and the movement of zoning toward the pumping well until the gypsum of the first zoning completely reaches the pumping well. The composition of the solutions entering the pumping well and subsequently reaching the surface depends on the composition of the zone closest to the pumping well. As long as carbonates are present in the rocks, the flow of solutions into the pumping well is accompanied by the release of carbon dioxide, which should lead to a significant increase in the solution pH and the precipitation of dolomite, calcite, and nesquegonite ($CaCO_3$; $CaMg(CO_3)_2, MgCO_3, 3H_2O$).

Discussion of Results

Since the solution remains in the pumping well for a short time, it can be assumed that carbonate precipitation due to CO_2 removal occurs primarily at the surface. The precipitated carbonates should soon dissolve—after acidic solutions begin to flow from the pumping wells. After the carbonates disappear from the rocks, solutions enriched in aluminum (pH 4.5-5.0) and ferric iron (pH 2-2.5) will successively flow to the surface. The calcium contained in the carbonates is converted into gypsum, and only a small portion is carried out into the pumping well.

The formation of solid phases results in chemical clogging of

the pore space of ore-bearing rocks, which significantly impacts the subsequent uranium leaching process. Precipitates containing aluminum and trivalent iron form in relatively small quantities and persist for a short time. Gypsum clogging is the main cause of complications during SSL.

From the above, it's clear how complex the acidification process is. It's drawn out over time, dissolution alternates with precipitation and then dissolution again, and so on. At each stage, the equilibrium in the system of chemical reactions is disrupted, and the system's pH and Eh change. It's virtually impossible to clearly and quantitatively track these changes in space-time coordinates (from injection to production wells). Therefore, all possible theoretical solutions are, and will remain, purely approximate.

During uranium leaching, the reaction of the solvent with rock-forming minerals forms mobile geochemical barriers (acid-base and redox) with alternating events of uranium dissolution and precipitation due to changes in the pH and Eh of the environment. In this process, acid is consumed not only and not so much for uranium leaching, but also for dissolving the rock-forming minerals, reaching tens and hundreds of kilograms per kilogram of uranium in real conditions. The main acid-intensive minerals are carbonates (calcium, dolomite, magnesite) and some types of clay minerals.

Due to the consumption of H_2SO_4 in reaction with rock-forming minerals, its concentration front moves significantly more slowly than if only uranium leaching were occurring. The concentration front of dissolved uranium tends to overtake the acid front, but this does not occur because, as the solution's acidity decreases, uranium hydrolysis occurs on the acid front, leading to redeposition according to the following scheme [3, 7]:

$$\begin{gathered} UO_2^{2+} + OH^- = UO_2OH^+ \\ UO_2OH^+ + OH^- = UO_2(OH)_2 \downarrow \end{gathered} \quad (2)$$

In this case, the acid concentration front acts as a mobile alkaline barrier. Then, as new portions of acid approach, the redeposited substance dissolves again, i.e., the reaction proceeds in reverse. Repeated steps lead to an increase in the H_2SO_4 concentration at the mobile barrier to the saturation concentration.

It is known that the oxidation-reduction potential is directly related to the concentration of the potential-determining components of the system:

$$E = E_0 + \frac{0.059}{n} lg\ lg\ \frac{[O_k]}{[B_0]} \quad (3)$$

From the perspective of potential-determining systems, the association of uranium and iron is of greatest interest at uranium deposits in formation oxidation zones. The reaction between uranium and iron in an acidic environment proceeds toward the oxidation of uranium and the reduction of iron.

Oxidation potentials for iron:

$$\begin{gathered} Fe - 0.44 \longrightarrow Fe^{2+} + 0.771 \longrightarrow \\ Fe^{3+} + 1.9 \longrightarrow FeO_4^{2-} \end{gathered} \quad (4)$$

In turn, at Eh +0.35, tetravalent uranium transforms into hexavalent hydrated oxide $UO_2(OH)_2$ and further at Eh +0.407 into the hexavalent divalent uranyl cation. The oxidation of uranium with trivalent iron ions proceeds according to the reaction where uranium is oxidized to the hexavalent form and iron is reduced to the divalent form [3, 8, 13-15].

During the SSL process, complexation significantly influences changes in the oxidation environment. During acid leaching, in addition to uranyl sulfate complexes UO_2SO_4, $[UO_2(SO_4)_2]^{2-}, [UO_2(SO_4)_3]^{4-}$, about half of the calcium, magnesium, and manganese are bound into sulfate complexes. Up to 80% of aluminum and over 90% of iron are bound into sulfate complexes. The proportion of simple ions decreases with increasing total concentration in the sulfate sulfur solution, pH and Eh change, and the content of free SO_4 ions decreases. Complexation causes a shift in the acidity of the SSL solution at geochemical barriers toward increasing pH values, and to maintain the oxidation and dissolution of uranium, it is necessary to increase the consumption of H_2SO_4.

However, given that the repeated circulation of sulfuric acid solutions between pumping and injection wells accumulates up to 0.05 mol/L or more of ferrous sulfate ($FeSO_4$) in the circulating solutions, maintaining the required CSL level can be accomplished without additional consumption of sulfuric acid or other oxidizing reagents. To achieve this, consideration should be given to the chemical effects of ultrasound on liquids during cavitation. Cavitation occurs in liquids due to the creation of negative pressures during the propagation of ultrasonic waves. Acoustic cavitation is characterized by small bubble sizes (10-3 – 10-2 cm). The pressure inside a cavitation bubble is initially very low compared to the pressure in the liquid. The liquid then rushes toward its center, and the bubble collapses (annihilates). As it collapses, a pressure peak forms in the central region of the bubble, and a spherical shock wave travels outward into the liquid. The pulsation and collapse of cavitation bubbles creates an exceptionally high energy density, stimulating a variety of chemical reactions.

Conclusion

Unlike traditional uranium mining methods, borehole leaching plants can be built in sequential stages using a modular approach. Capital investment in establishing a borehole leaching plant is 3-4 times lower, operating costs are 2-3 times lower, and the production cost is 5-6 times lower. The operating cost structure of a borehole leaching plant is also unique and consists of drilling and surface pipeline costs (17-23%), reagent costs (30-45%), and energy costs (8-15%). The specific costs of drilling and equipping wells are determined by the depth of the ore-bearing strata and the design features of the constructed wells.

The final assessment of the industrial and operational value of a deposit is based on the results of pilot work and observations of the leaching process parameters at the test site. Dissolution of clay minerals occurs significantly more slowly, occurring virtually throughout the entire leaching process, and the proportion of reagent consumed for this group of minerals is complexly dependent on their quantity, active surface area (size of crystalline aggregates), and contact time with the solvent. With prolonged leaching, the reagent consumption for this group of minerals can reach significant levels. The share of acid consumption for other minerals, due to their small quantities, is insignificant.

Determination of the chemical composition of productive solutions for each pumping well and for the collecting manifold, as well as uranium – daily; pH - once per day; Eh - once every 5-10 days; total mineralization, $SO_4^{2-}, Fe_2^+, Fe_3^+$ - once every 10-30 days; $Al_3^+, HCO_3^-, CO_3^{2-}$ – once every 5-10 days (during the formation preparation stage for leaching); free H_2SO_4, an

oxidizer - once every 5-10 days (during the leaching stage). Samples of collecting manifold solutions are taken once every 10-30 days for a full chemical analysis, including determination of $Ca_2^+, Mg_2^+, NO_3^-, Cl^-, P, Si$ and other components.

Determination of sulfuric acid, pH, and oxidizer content in working solutions is performed daily. Total mineralization of working solutions is measured every 10-30 days.

Suspended solids content is determined monthly in working solutions, and once every 10 days in pumping well solutions and the collection manifold.

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