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B. T. Altaibayev[1], M. K. Kylyshkanov[1], *K. S. Togizov[2]
[1]Kazakh-British Technical University (Almaty, Kazakhstan),
[2]Satbayev University (Almaty, Kazakhstan)

INVESTIGATION OF THE ACID DESILICATION PROCESS OF INDUSTRIALLY SPENT ANION EXCHANGE RESIN

Abstract. Silicon fouling of strong-base anion-exchange resins reduces the efficiency of uranium recovery from pregnant solutions generated during in situ leaching. This study examined the acid desilication of industrially spent resin using ***HF*** and ***HF–HNO$_3$*** solutions. The effects of reagent concentration, solid-to-liquid ratio, and treatment time were evaluated. Increasing the ***HF*** concentration and the solid-to-liquid ratio enhanced silicon removal. The highest desilication efficiency, 65.5%, was achieved with ***2.0 M HNO$_3$ + 0.5 M HF*** at an ***S:L*** ratio of 1:5, reducing the residual ***Si*** content to 3.81 wt.%. Most silicon was removed within the first 30–90 min, after which the rate decreased due to intraparticle diffusion limitations. Particle-size analysis showed that the resin beads largely retained their integrity after treatment. The ***HF–HNO$_3$*** system therefore offers a practical approach for regenerating silicon-fouled anion-exchange resins used in uranium hydrometallurgy.
Key words: *uranium, underground leaching, sorbent, silicon, desilication, acid treatment, sorbent regeneration.*

Өндірісте пайдаланылған анионалмастырғыш шайырдың қышқылды десиликация процесін зерттеу

Аңдатпа. Кремний қосылыстарымен ластанған күшті негізді анионалмастырғыш шайырлар жерасты шаймалау өнімді ерітінділерінен уранды сорбциялық бөліп алу тиімділігін төмендетеді. Жұмыста өнеркәсіптік пайдаланылған шайырды ***HF*** және ***HF–HNO$_3$*** ерітінділерімен қышқылдық десиликациялау зерттелді. Реагенттер концентрациясының, Қ:С қатынасының және өңдеу ұзақтығының әсері бағаланды. ***HF*** концентрациясы мен Қ:С қатынасының артуы кремнийдің бөліну дәрежесін жоғарылатты. Ең жоғары десиликация дәрежесі ***65,5% 2,0 M HNO$_3$ + 0,5 M HF*** ерітіндісінде және Қ:С = 1:5 кезінде алынды, ал ***Si*** қалдық мөлшері 3,81 мас.% дейін төмендеді. Кремнийдің негізгі бөлігі алғашқы 30–90 мин ішінде жойылды, кейін түйіршік ішіндегі диффузиялық шектеулерге байланысты процесс жылдамдығы төмендеді. Гранулометриялық талдау шайыр түйіршіктерінің тұтастығы сақталғанын көрсетті. ***HF–HNO$_3$*** жүйесін уран гидрометаллургиясында кремниймен ластанған анионалмастырғыш шайырларды регенерациялау үшін қолдануға болады.
Түйінді сөздер: *уран, жерасты шаймалау, сорбент, кремний, десиликация, қышқылды өңдеу, сорбенттерді регенерациялау.*

Исследование процесса кислотной десиликации промышленно отработанной анионообменной смолы

Аннотация. Загрязнение сильноосновных анионообменных смол соединениями кремния снижает эффективность сорбционного извлечения урана из продуктивных растворов подземного выщелачивания. Исследована кислотная десиликация промышленно отработанной смолы растворами ***HF*** и ***HF–HNO$_3$***. Оценено влияние концентрации реагентов, соотношения Т:Ж и продолжительности обработки. Увеличение концентрации ***HF*** и соотношения Т:Ж повышало степень удаления кремния. Максимальная десиликация 65,5% достигнута при ***2,0 M HNO$_3$ + 0,5 M HF*** и Т:Ж = 1:5; остаточное содержание ***Si*** составило 3,81 мас. %. Основное удаление кремния происходило в первые 30–90 мин, после чего скорость снижалась из-за внутригранульных диффузионных ограничений. Гранулометрический анализ подтвердил сохранение целостности гранул. Система ***HF–HNO$_3$*** может использоваться для регенерации кремнезагрязненных анионообменных смол в урановой гидрометаллургии.
Ключевые слова: *уран, подземное выщелачивание, сорбент, кремний, десиликация, кислотная обработка, регенерация сорбентов.*

Introduction

Global demand for natural uranium is expected to rise as nuclear power capacity expands. Under the WNA scenarios, uranium requirements could reach about 130 ktU/year by 2040 in the base-rate scenario and around 183 ktU/year in the high-rate scenario. The same analysis indicates that, with rapid growth of nuclear power, uranium production could peak around the middle of this century, increasing the risk of a widening gap between reactor requirements and primary uranium supply [1].

Amid growing uranium demand, ISL technology has become dominant in the global mining structure, accounting for more than half of global uranium production [2]. ISL's widespread use is due to its relatively low capital intensity, reduced environmental impact, and high economic efficiency compared to traditional open-pit and underground mining methods [3]. In sulfuric acid ISL processes, widely used at sandstone uranium ore deposits in Kazakhstan and other countries, uranium is transferred to productive solutions primarily in the form of anionic uranyl sulfate complexes [4].

In industrial practice, uranium extraction from productive ISL solutions is typically accomplished by ion-exchange sorption using strongly basic anion-exchange resins that exhibit high selectivity for uranyl sulfate complexes, stability in acidic environments, and suitability for continuous column operation. The efficiency of the sorption-desorption process is largely determined by the preservation of the sorption and mechanical properties of the ion-exchange material under long-term cyclic operation. One of the main factors limiting the operational stability of anion-exchange resins in uranium ISR systems is their contamination with silicon compounds entering the productive solutions as a result of the sulfuric acid decomposition of silicate and aluminosilicate host rocks. Silicon compounds present in the solution, in the form of silicic acid and reactive silicate particles, are capable of being sorbed onto the functional groups of the resin, followed by intrapore polycondensation and the formation of polymerized siloxane structures within the polymer matrix. This process leads to the blocking of active ion-exchange centers, deterioration of mass transfer, a decrease in the uranium sorption capacity, and a gradual degradation of the mechanical strength of the sorbent granules [5–8]. According to industrial and laboratory studies, the silicon content in spent uranium anion exchange resins can reach 12–17 wt. % or more, which is accompanied by a significant decrease in the technological efficiency of the sorption process [8]. In this regard, the regeneration of silicon-contaminated sorbents is one of the key tasks in ensuring the stability and cost-effectiveness of ion-exchange uranium extraction.

The most common industrial method for desiliconizing anion-exchange resins is alkaline treatment with sodium hydroxide solutions, based on the hydrolytic destruction of siloxane bonds and the conversion of silicon into soluble silicate forms [6]. However, numerous studies show that the effectiveness of alkaline desilication is limited in the presence of highly polymerized silicon structures, while an increase in the alkali concentration and more stringent processing conditions lead to the degradation of the polymer matrix and functional groups of strongly basic anion exchange resins [8–10]. Despite the significant practical significance of the problem, the development

of alternative methods for the deep regeneration of silica-contaminated uranium sorbents remains understudied. In particular, the number of studies devoted to the use of acidic methods for processing silica-contaminated anion-exchange resins is limited, and the impact of acid treatment on the degree of silica removal, the state of the polymer structure, and the restoration of sorbent performance requires further systematic study.

Therefore, the objective of this study is to investigate the acidic reprocessing of anion-exchange resins contaminated with silica compounds during the processing of uranium ISL (in-situ leaching) productive solutions and to evaluate the effectiveness of this approach for desiliconizing sorbents and restoring their performance properties.

Materials and Methods

Materials

The object of the study was an industrially spent strongly basic anion exchange resin applied in the extraction of uranium from productive solutions of in-situ leaching. The silicon content in the studied industrially spent sorbent was 11.04 wt. %. According to the results of granulometric analysis (Table 1), the studied sorbent was characterized by the predominance of granules of the medium fraction. Solutions of hydrofluoric acid ***HF*** and ***HF–HNO$_3$*** mixtures of chemically pure grade were used as reagents. The choice of ***HF*** solutions and ***HF–HNO$_3$*** mixtures as desilicate reagents is due to the ability of hydrofluoric acid to interact with silica to form soluble fluorosilicate compounds, whereas nitric acid promotes the oxidation of silicon compounds and increases the efficiency of their dissolution, which is consistent with the literature data on the chemical dissolution of silicon in ***HF/HNO$_3$*** systems [11, 12].

Equipment

The sorbent was processed in a chemically resistant polypropylene flask. Stirring was performed using a magnetic stirrer. After processing, the sorbent was separated by filtration on a laboratory filter.

Desilication Procedure

Desilication was carried out in a static mode with stirring at room temperature. Treatment was carried out at a solid:liquid ratio of 1:3–1:5, a contact time of 15–180 min, and a sorbent sample weight of 25 g. After treatment, the sorbent was separated by filtration, washed with warm distilled water to a neutral pH, and dried. The filtrate was sent for analysis. All experiments for each point were carried out in at least three independent replicates. The sorbent desilication process flow chart is shown in Figure 1. Additionally, after treatment, a comparative analysis of the sorbent's particle size distribution was performed to assess possible changes in granule distribution under the influence of desilication solutions.

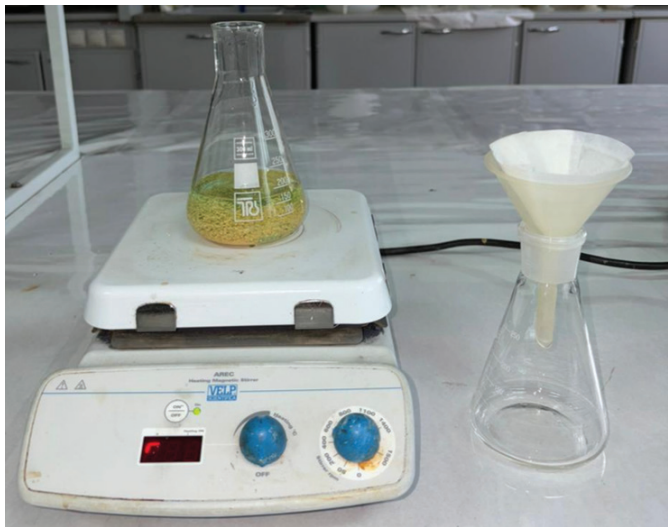


Figure 1. Scheme of the process of desilication of the sorbent and subsequent filtration.
Сурет 1. Сорбентті десиликациялау процесінің және кейінгі сүзудің сызбанұсқасы.
Рис. 1. Схема проведения процесса десиликации сорбента и последующей фильтрации.

Analytical Methods

The silicon content released into solution after desilication was determined gravimetrically by analyzing the filtrate. Based on the data obtained, the degree of silicon removal from the sorbent was calculated using the formula:

$$R = (C_0 - C) / C_0 \cdot 100\ \%,$$

where C_0 is the ***Si*** content in the original resin, wt. %;

C is the residual ***Si*** content in the resin after treatment, wt. %.

To monitor the effect of reagent treatment, the particle size distribution of the sorbent after desilication was additionally assessed.

Results and Discussion

The results showed that treatment of the anion exchange resin with ***HF*** and ***HF–HNO$_3$*** solutions at a solid-to-liquid ratio of ***S:L = 1:3*** contributed to a decrease in silicon content within the sorbent structure. As shown in Figure 2, increasing the ***HF*** concentration was accompanied by an increase in desilication efficiency. When using ***1.0 M HF***, the degree of

Table 1

Granulometric composition of the industrially spent sorbent

Кесте 1

Өндірісте пайдаланылған сорбенттің гранулометриялық құрамы

Таблица 1

Гранулометрический состав промышленно отработанного сорбента

Sieve cell size, mm	*2.0*	*1.8*	*1.6*	*1.4*	*1.25*	*1.0*	*0.8*	*0.63*	*0.5*	*0.4*	*0.315*
Fraction content, %	0	1	4	10	35	31	14	3	1	1	0

silicon removal reached 33.2% with a residual ***Si*** content of 7.38%, whereas increasing the ***HF*** concentration to ***2.0 M*** enhanced the silicon removal degree to 46.9% and reduced the residual ***Si*** content to 5.86%. The obtained results are associated with silica dissolution and the formation of soluble fluorosilicate compounds.

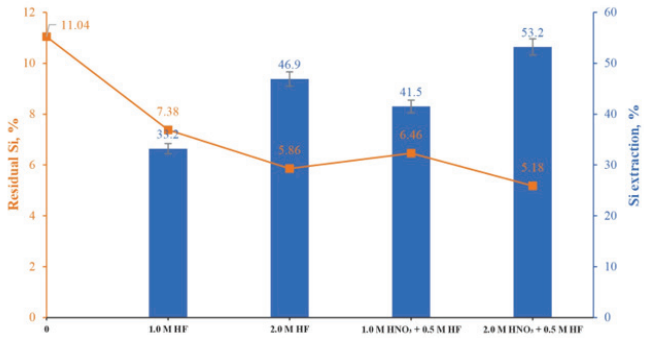


Figure 2. Effect of HF concentration and HF–HNO_3 systems on the degree of silicon removal and residual Si content in the sorbent.
Сурет 2. HF концентрациясы мен HF–HNO_3 жүйелерінің кремнийді жою дәрежесіне және сорбенттегі қалдық Si мөлшеріне әсері.
Рис. 2. Влияние концентрации HF и систем HF–HNO_3 на степень удаления кремния и остаточное содержание Si в сорбенте.

Higher desilication rates were observed using combined HF–HNO_3 solutions. For the ***1.0 M HNO_3 + 0.5 M HF*** system, the silicon extraction rate was 41.5% with a residual Si content of 6.46%. Increasing the ***HNO_3*** concentration to 2.0 M while maintaining a constant ***HF*** concentration (0.5 M) increased the silicon removal rate to 53.2% and reduced the residual ***Si*** content to 5.18%. This increased efficiency is likely due to the combined action of the acidic environment and fluoride ions, which ensure more intensive destruction of silicate deposits in the sorbent structure. The use of more concentrated acidic systems was also not considered to minimize potential degradation of the ion-exchange resin structure during processing.

As shown in Figure 3, increasing the solid:liquid ratio has a positive effect on desilication efficiency. For a ***1.0 M HNO_3 + 0.5 M HF*** solution, an increase in the S:L ratio from 1:3 to 1:5 was accompanied by a decrease in the residual ***Si*** content from 6.46 to 5.29% and an increase in the degree of silicon extraction from 41.5 to 52.1%. A similar pattern was observed for a more concentrated system of ***2.0 M HNO_3 + 0.5 M HF***, where an increase in the ***S:L*** ratio from 1:3 to 1:5 led to a decrease in the residual ***Si*** content from 5.18 to 3.81% and an increase in the degree of silicon extraction from 53.2 to 65.5%.

The obtained results indicate that increasing the solution volume relative to the sorbent mass facilitates more complete interaction of the reagent with silicate deposits in the resin structure. This is likely due to improved mass transfer conditions, increased availability of the active reagent, and reduced local saturation of the solution with silica dissolution products.

As can be seen in Figure 4, the desilication process at a solid:liquid ratio of 1:5 is characterized by uneven silicon removal rates at different stages of processing. At the initial stage of the process, intensive dissolution of silicon compounds is observed: within the first 30 minutes, the silicon extraction rate reaches 44.9%, with the residual Si content decreasing to 6.09%. It is likely during this period that the most accessible and weakly bound silicate deposits localized on the surface and in the subsurface layers of the sorbent are removed.

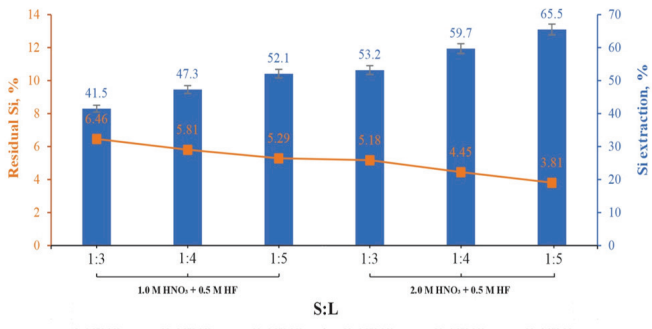


Figure 3. The influence of solid:liquid ratio on the efficiency of sorbent desilication.
Сурет 3. Қатты:сұйық қатынасының сорбентті десиликациялау тиімділігіне әсері.
Рис. 3. Влияние соотношения твердое:жидкое на эффективность десиликации сорбента.

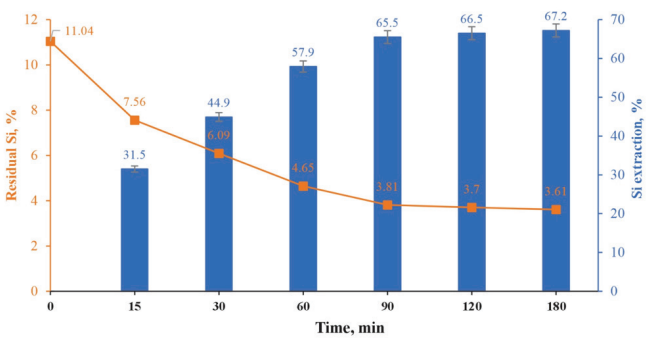


Figure 4. Effect of treatment time on sorbent desilication.
Сурет 4. Өңдеу уақытының сорбентті десиликациялау процесіне әсері.
Рис. 4. Влияние продолжительности обработки на десиликацию сорбента.

In the 30–90 min interval, the process rate decreases, but desilication continues to proceed quite intensively. By 90 min, silicon extraction reaches 65.5%, and the residual Si content decreases to 3.81%, indicating further dissolution of more stable forms of silica within the resin structure.

A further increase in processing time to 120–180 min does not significantly improve process efficiency: extraction increases only from 65.5 to 67.2%, while the residual Si content changes only slightly – from 3.81 to 3.61%. The obtained results indicate the gradual achievement of a quasi-equilibrium state and the process being limited by internal diffusion of the reagent deep into the sorbent granules.

Thus, the optimal desilication duration is approximately 90–120 min, since this interval achieves maximum silicon removal with minimal increase in processing time. After desilication of the sorbent, a comparative analysis of the particle

Table 2

Comparative granulometric composition of the sorbent before and after desilication

Кесте 2

Десиликацияға дейінгі және кейінгі сорбенттің салыстырмалы гранулометриялық құрамы

Таблица 2

Сравнительный гранулометрический состав сорбента до и после десиликации

Sieve cell size, mm	*2.0*	*1.8*	*1.6*	*1.4*	*1.25*	*1.0*	*0.8*	*0.63*	*0.5*	*0.4*	*0.315*
Before desilication, %	0	1	4	10	35	31	14	3	1	1	0
After desilication, %	0	1	4	11	36	28	12.5	5.5	1	1	0

size distribution was performed to assess the effect of reagent treatment on the granule size distribution (Table 2). The appearance of the sorbent before and after desilication is shown in Figure 5.

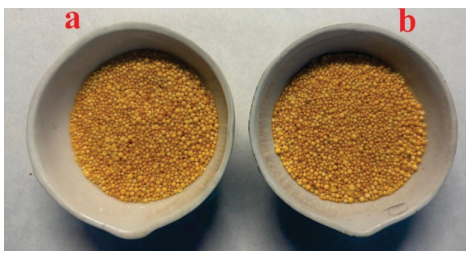


Figure 5. Appearance of the sorbent before (a) and after (b) treatment.
Сурет 5. Өңдеуге дейінгі (a) және өңдеуден кейінгі (b) сорбенттің сыртқы көрінісі.
Рис. 5. Внешний вид сорбента до (a) и после (b) обработки.

It was established that after processing, no significant change in the granulometric composition was observed, while no noticeable destruction, deformation or change in the color of the sorbent granules was visually detected, which indicates the preservation of the structural integrity of the polymer matrix under the studied desilication conditions. The majority of the sorbent, both before and after desilication, was composed of medium-sized granules, indicating that the material's structural integrity was maintained under the selected processing conditions. Minor changes in the content of individual fractions may be due to mechanical action during mixing and partial surface modification of individual granules during processing.

Conclusion

This study investigated the feasibility of acid desilication of industrially used strongly basic anion exchange resin used for uranium extraction from productive in-situ leaching solutions. Treatment with ***HF*** and ***HF–HNO$_3$*** solutions was found to partially remove silicon compounds from the sorbent structure and reduce residual silicon content.

Increasing the ***HF*** concentration and increasing the solid:liquid ratio were shown to intensify the desilication process. The highest silicon removal rate was 65.5% using a ***2.0 M HNO$_3$ + 0.5 M HF*** system and a solid:liquid ratio of 1:5, while the residual Si content decreased to 3.81 wt. %.

Kinetic studies showed that the most intense silicon removal occurs during the first 30–90 minutes of treatment, while further increases in process duration are accompanied by a slight increase in the degree of desilication. A reasonable treatment duration is 90–120 minutes. The results of particle size analysis and visual inspection showed no significant granule degradation after acid treatment. Unlike alkaline regeneration methods, which can lead to degradation of the sorbent's polymer matrix, the ***HF*** and ***HF–HNO$_3$*** systems ensure silicon removal while maintaining the resin's structural integrity.

These results confirm the potential of ***HF*** and ***HF–HNO$_3$*** systems as an alternative method for the partial regeneration of silica-contaminated anion exchange resins used in uranium production.

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Information about the authors:
Altaibayev B. T., Ph.D, Senior Researcher, Kazakh-British Technical University (Almaty, Kazakhstan), *bagdataltai9@gmail.com;* https://orcid.org/0000-0002-7405-6854
Kylyshkanov M. K., Doctor of Physico-Mathematical Sciences, Professor, Kazakh-British Technical University (Almaty, Kazakhstan), *kylyshkanov@mail.ru;* https://orcid.org/0000-0002-8304-7124
Togizov K. S., Ph.D, associate professor, Satbayev University (Almaty, Kazakhstan), *k.togizov@satbayev.university;* https://orcid.org/0000-0002-4830-405X

Авторлар туралы мәліметтер:
Алтайбаев Б. Т., Ph.D, аға ғылыми қызметкер, Қазақ-Британ техникалық университеті (Алматы қ., Қазақстан)
Кылышканов М. К., физика-математика ғылымдарының докторы, профессор, Қазақ-Британ Техникалық Университеті (Алматы қ., Қазақстан)
Тогизов К. С., Сәтбаев университетінің Ph.D докторы, қауымдастық профессоры (Алматы қ., Қазақстан)

Сведения об авторах:
Алтайбаев Б. Т., Ph.D, ст. научный сотрудник, Казахстанско-Британский технический университет (г. Алматы, Казахстан)
Кылышканов М. К., доктор физико-математических наук, профессор, Казахстанско-Британский технический университет (г. Алматы, Казахстан)
Тогизов К. С., Ph.D, ассоциированный профессор, Satbayev University (г. Алматы, Казахстан)