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Д.В.Сокольский атындағы «Жанармай,
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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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NEWS

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OF THE REPUBLIC OF KAZAKHSTAN
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NAS RK is pleased to announce that News of NAS RK. Series of chemistry and technologies scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of chemistry and technologies in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of chemical sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Химия және технология сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Web of Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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INVESTIGATION OF CONCENTRATION METHOD OF SCANDIUM-CONTAINING SOLUTIONS

Abstract. In current times, enterprises in Kazakhstan that extract uranium by in-situ leaching (ISL) method do not simultaneously recover valuable components. Cardinal problems in this area are the issues of reserves estimation and the availability of effective technology that allows for cost-effective operations. The peculiarities of processing solutions of ISL uranium should also include extremely low contents of valuable components and a complex chemical composition. To solve some of these problems, the authors are working on the development and optimization of technological solutions.

This article presents studies results of further concentration and purification of scandium strippants of the first operations of scandium sorption from uranium sorption filtrates of the ISL cycle. Concentration effect of sodium carbonate on the kinetics and desorption efficiency of scandium from the resin was studied using the example of SE-DEHPA resin under static conditions. Solution's pH influence on second stage operation of sorption on SE-DEHPA resin under static conditions is recognized, and the possibility of subsequent carbonate desorption of scandium was investigated.

As a result of the laboratory studies, a basic technological scheme of scandium associated extraction was developed within the framework of the ISL uranium cycle and the main technological parameters and modes were determined that formed the basis of the basic technical and economic assessment.

Key words: scandium, sorption, desorption, extraction, degree of extraction, exchange capacity, filtrate.

Introduction. Scandium is a rare element and a very expensive metal due to the complex metallurgical processes of its extraction, purification and reduction. General application of scandium oxide is solid oxide fuel cells production. Scandium consumption in light sources production, in nuclear energy, medicine, in the production of refractory materials, phosphors is promising. However, the active use of scandium unique properties is currently constrained by the high cost of its production [1-3].

Due to its low content, scandium is typically recovered as a by-product in the processing of aluminum and rare metal materials. Currently, hydrometallurgical processes, which are mainly associated with leaching, liquid extraction and precipitation, are usually used to extract scandium. One of the major scandium sources is uranium ores, during the processing of which using sulfuric or nitric acid scandium is extracted together with uranium and transferred to leaching solutions. The next time uranium is cleaned, the scandium is separated. When processing uranium-containing leaching solutions, extraction and sorption methods are most often used to extract scandium [4-13].

Separation and purification of scandium is carried out using acidic, alkaline, neutral and chelating extractants. A promising area of extraction chemistry is the use of binary extractants, characterized by high separation and distribution coefficients, a high extraction rate, as well as ease of re-extraction and reagent consumption reduction. Among extractants, the most selective with respect to scandium is di-2-ethylhexylphosphoric acid (DEHPA) [14-22].

The aim of this work is to develop a highly efficient, economically acceptable technology for scandium associated extraction from uranium sorption filtrates.

Experimental procedure

Consumables. In our previous investigations, we determined the effectiveness of a binary extractant prepared on the basis of di-(2-ethylhexyl) phosphoric acid (DEHPA) for scandium extraction from uranium sorption filtrates and established the main approaches to desorption. This article discusses the technological issues of further concentration and purification of scandium strippants of the first and second sorption operations.

Table 1 presents the characteristics of DEHPA sorbent used in the work, which underwent preliminary thorough conditioning.

Table 1 – Characteristics of the used sorbent DEHPA

Ionite	Functional group	Matrix	Granules size in the air-dry state, mm
SE- DEHPA	C ₁₆ H ₃₅ PO ₄	macroporous, cross-linked polystyrene	0.63÷2.50

Scandium sorption in static conditions. Taking into account the extremely low concentrations of the target components, it is advisable to use a large solution: sorbent phase ratio (L:S) for studies under static conditions. Static sorption was made in a container with a volume of 5 dm³ using a mixing device with a ratio of L:S 1000:1 (5 dm³:5 g). Experiment duration was 24 hours. Samples for analysis were taken at certain time intervals without interrupting the experiment. The change in system volume as a result of sampling did not exceed 5%.

The values of the static exchange capacity of the ion exchanger was calculated by the formula:

$$SEC = \frac{C_0 - C_p}{C_0} \cdot \frac{V}{m} \quad (1)$$

where C_0 and C_p – initial and equilibrium concentrations of the adsorbed component, mg/dm³ or mmol/dm³; V – volume of solution, dm³; m – weight of resin, g.

Scandium sorption I from a model solution. Research of the first stage of scandium sorption was carried out on a model solution simulating real solutions composition of the “Irkol” mine.

The composition of model solution with a volume of 60 dm³ and a scandium concentration of 0.15 mg/dm³:

- 4.602 mg of scandium oxide Sc₂O₃ with a purity of 99.9%;
- an aqueous solution of sulfuric acid (60%) of "chemical pure" grade 94.6.

Scandium oxide dissolution was done in a hot sulfuric acid solution at a temperature of 80°C for 0.5 hours.

The process of scandium sorption on DEHPA resin in a static mode was conducted research on a laboratory shaker LOIP LS110 at a rotation speed of 190 rpm at room temperature.

Scandium sorption II from strippant stage I. Kinetic studies of second stage process of sorption at different pH were carried out in a model solution with a concentration of scandium of 15.81 mg / dm³ simulating stage I strippant. Solution preparation included the dissolution of a selected sample of scandium oxide (99.9%) weighing 0.0153g in 1 cm³ of concentrated sulfuric acid, followed by dilution of the solution to 1 dm³. The resulting solution was divided into 5 equal portions of 200 cm³, which underwent a pH adjustment to the desired value. Solution pH was adjusted to the acidic side with sulfuric acid and alkaline - with sodium carbonate. At sodium carbonate concentration of 200 g/dm³, the pH was 10.73, while the resin formed large pieces with a diameter of 0.5-1.0 cm, which subsequently stuck together into one large agglomerate.

Studies were carried out at pH values of solutions 1,3,5,10,13.

The process of scandium secondary sorption from the strippant of stage I on resins in the static mode was taken on a laboratory shaker LOIP LS110 at a rotation speed of 190 rpm at room temperature.

Scandium desorption from saturated DEHPA resin under static conditions. The processes of primary and secondary desorption of scandium were carried out under static conditions using various concentrations of Na₂CO₃ sodium carbonate desorbing solution: 100, 150, 200 and 250 g/dm³.

In desorption study, the ratio L:S=50:1 (250 cm³:5 g) was used with a duration of 12 hours. The change in system volume as a result of sampling also did not exceed 5%.

The element content in the resin was calculated based on the material balance taking into account element concentration in the initial solution.

The scandium content in liquid and solid samples was analyzed using plasma atomic emission spectroscopy (Optima 8300 DV, PerkinElmer, LLC).

Results and discussion

As a result of scandium sorption I from model solutions on DEHPA resin, saturation was established (table 2).

Table 2 – Results of scandium sorption under static conditions from model solutions on DEHPA resin

Number of resin's aliquot	Initial Sc concentration, mg/dm ³	Final Sc concentration, mg/dm ³	Estimated Sc concentration on resin, kg/m ³
1	0.12	0.0015	0.118
2	0.12	0.0012	0.119
3	0.12	0.0015	0.118
4	0.12	0.00095	0.119

Scandium I was desorbed from saturated DEHPA resin using a sodium carbonate Na₂CO₃ stripping solution of various concentrations: 100, 150, 200 and 250 g/dm³ (table 3, figure 1).

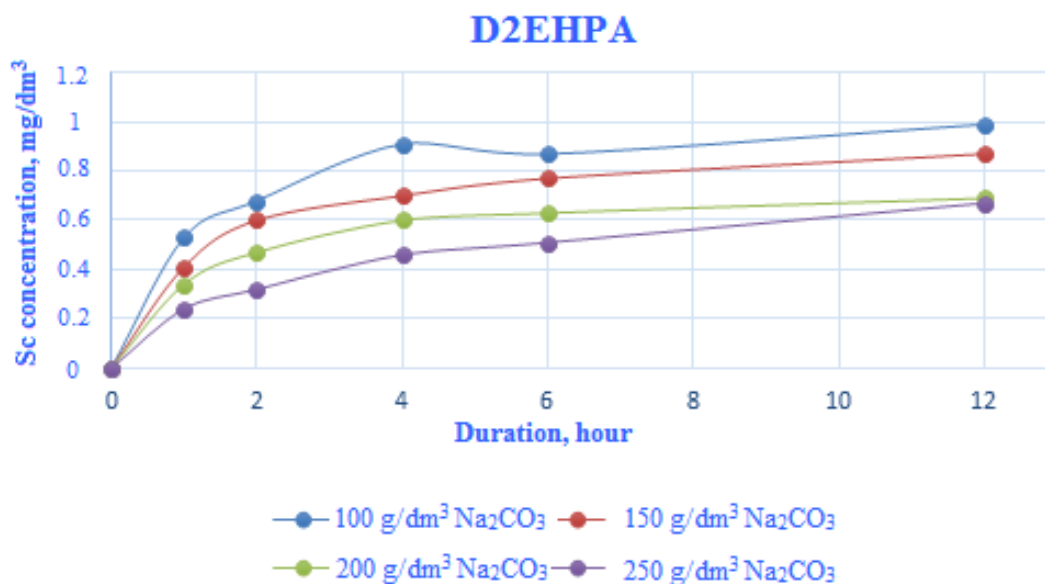


Figure 1 – Scandium desorption I kinetic curves at various concentrations of Na₂CO₃.

Table3 – Researc findings of the kinetics of scandium desorption I at various concentrations of a desorbing agent

Duration	Initial Sc content in DEHPA resin, kg/m ³			
	0.118	0.119	0.118	0.119
	Scandium concentration in solution, mg/dm ³			
	100 g/dm ³ Na ₂ CO ₃	150 g/dm ³ Na ₂ CO ₃	200 g/dm ³ Na ₂ CO ₃	250 g/dm ³ Na ₂ CO ₃
1 hour	0.53	0.41	0.34	0.24
2 hours	0.68	0.60	0.47	0.32
4 hours	0.91	0.70	0.60	0.46
6 hours	0.87	0.77	0.63	0.51
12 hours	0.99	0.87	0.69	0.67
Extraction, %	33.56%	29.24%	23.39%	22.52%

It is seen from table 3, the obtained degree of scandium extraction determined in the static mode, is low and is at the level of 30%, which is probably due to the significant affinity of scandium to the resin and laboratory scale of the tests. The most optimal concentration of a stripping solution providing this extraction degree is 100 g/dm³ of Na₂CO₃.

During the second stage of scandium sorption from the strippant of stage I, pH influence of the solution on the kinetic sorption properties of the DEHPA resin was determined. The composition and preparing method of model solutions are described above. The results of testing the operation of scandium sorption II are shown in table 4 and in figure 2.

Table 4 – The results of scandium sorption II kinetics studies with DEHPA resin at various pH solutions

Duration	Scandium concentration in solution, mg/dm ³				
	pH 1	pH 3	pH 5	pH 10	pH 13
1 hour	0.012	0.0012	0.66	7.94	9.42
2 hours	0.020	0.0022	0.95	7.79	8.69
4 hours	0.0072	0.0053	0.79	6.46	10.54
6 hours	0.0037	0.0014	0.56	7.01	10.15
12 hours	<0.00003	<0.00003	0.054	8.01	10.03
24 hours	0.00048	<0.00003	0.0011	7.76	10.05
Extraction, %	100.00%	100.00%	99.99%	50.92%	36.43%
Sc content in resin, kg/m ³	0.474	0.474	0.468	0.400	0.377

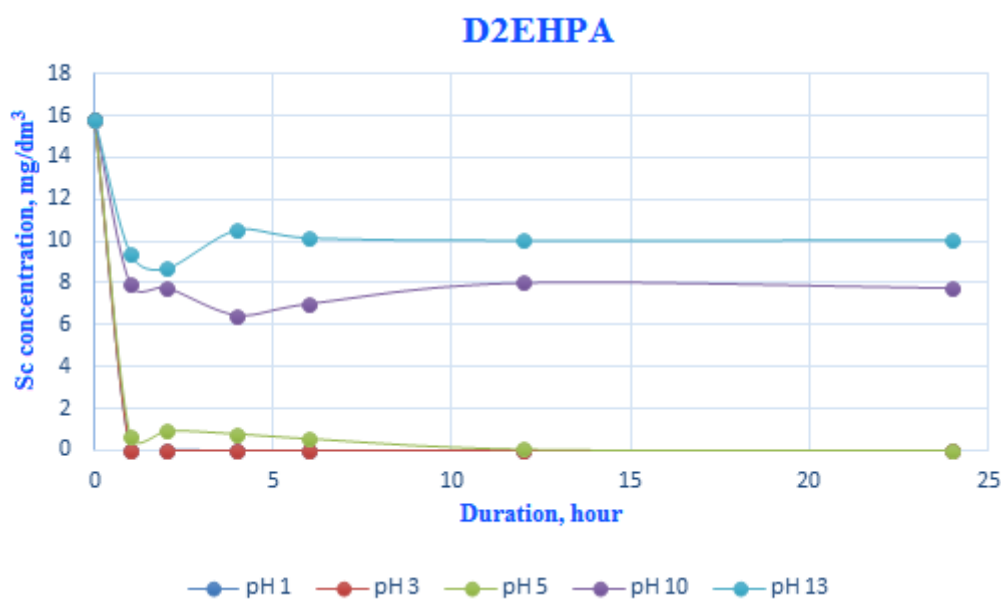


Figure 2 – Kinetic curves of scandium sorption II with DEHPA resin at various pH of the solution

As can be seen from table 4, when the pH of the solution is 5, there is a decrease in the sorption properties of the DEHPA resin and with a further increase in the pH of the solution, the capacity of the resin decreases significantly (up to 70% of the capacity in an acidic environment), which is due to the type of ion-exchange groups.

Studies results confirm the feasibility of carrying out the first operation of solutions desorption using 100 g/dm³ Na₂CO₃, since in the future, a minimum amount of deoxidizing agent will be required to bring the pH of stage I strippants to 2-3. This solution will allow for efficient sorption extraction in the second stage with the lowest operating cost.

DEHPA resin obtained as a result of scandium sorption II was desorbed in a static mode using 100 g/dm³ Na₂CO₃. The results of testing scandium desorption operation II are shown in table 5 and figure 3.

Table 5 - Results of kinetics studies of scandium desorption II at 100 g/dm³ Na₂CO₃

Duration	Scandium concentration in solution, mg/dm ³				
	Resin from pH 1	Resin from pH 3	Resin from pH 5	Resin from pH 10	Resin from pH 13
The initial content of Sc in the resin, kg/m ³	0.474	0.474	0.468	0.400	0.377
1 hour	0.65	1.20	2.03	0.18	0.11
2 hours	1.24	1.47	1.85	0.25	0.11
4 hours	1.90	2.33	2.73	0.30	0.098
6 hours	2.50	2.54	2.41	0.35	0.076
12 hours	3.42	3.09	2.58	0.39	0.1
24 hours	4.99	3.39	3.80	0.42	0.13
Extraction, %	35.66%	25.93%	29.30%	3.89%	1.30%

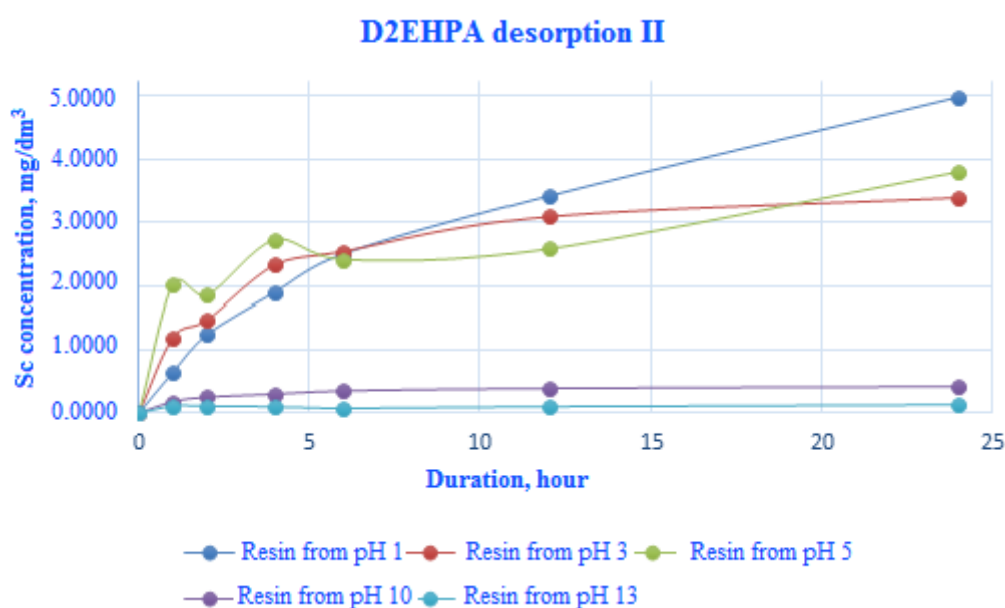


Figure 3 – Kinetic curves of scandium desorption II from DEHPA resin saturated at various pH

As in the case of desorption I, the obtained degree of scandium extraction, determined in static mode, is at the level of 30%, which can be gave a reason by laboratory test scope. It is noteworthy that scandium practically did not desorb from a resin saturated at an alkaline pH.

Derived strippants can be further processed by oxalic acid precipitation. The advantage of this method is scandium precipitation, while calcium (the main impurity element) during oxalic acid deposition remains in solution, which ultimately allows for a cleaner product.

As a result of laboratory studies, a conceptual technological schema was developed for the associated extraction of scandium from uranium sorption filtrates. The main technological parameters and modes, which formed the basis of the base technical and economic assessment, were determined. Indicators given in this assessment are preliminary and cannot be applied directly for project purposes. However, these results will allow giving a sense of feasibility of continuing work in this area.

Considering that the initial and remaining reserves of scandium in the contaminated uranium ore are unknown, at this stage we can assume that scandium equilibrium concentration in solution is 0.22 g/m³. It makes sense to make an assumption about the feasibility of processing 250 m³/h of mother liquids for uranium sorption, which is convenient both in hardware design and in terms of minimizing financial risks at an acceptable level of profitability.

The scandium extraction technology is based on sorption I on an impregnated sorbent of the brand SE-DEHPA, carbonate desorption I, neutralization and subsequent sorption II on SE (SE-solid extractant). Desorption II is carried out by sodium carbonate and subsequently scandium oxalate is produced by oxalic deposition in an acidic medium (figure 4).

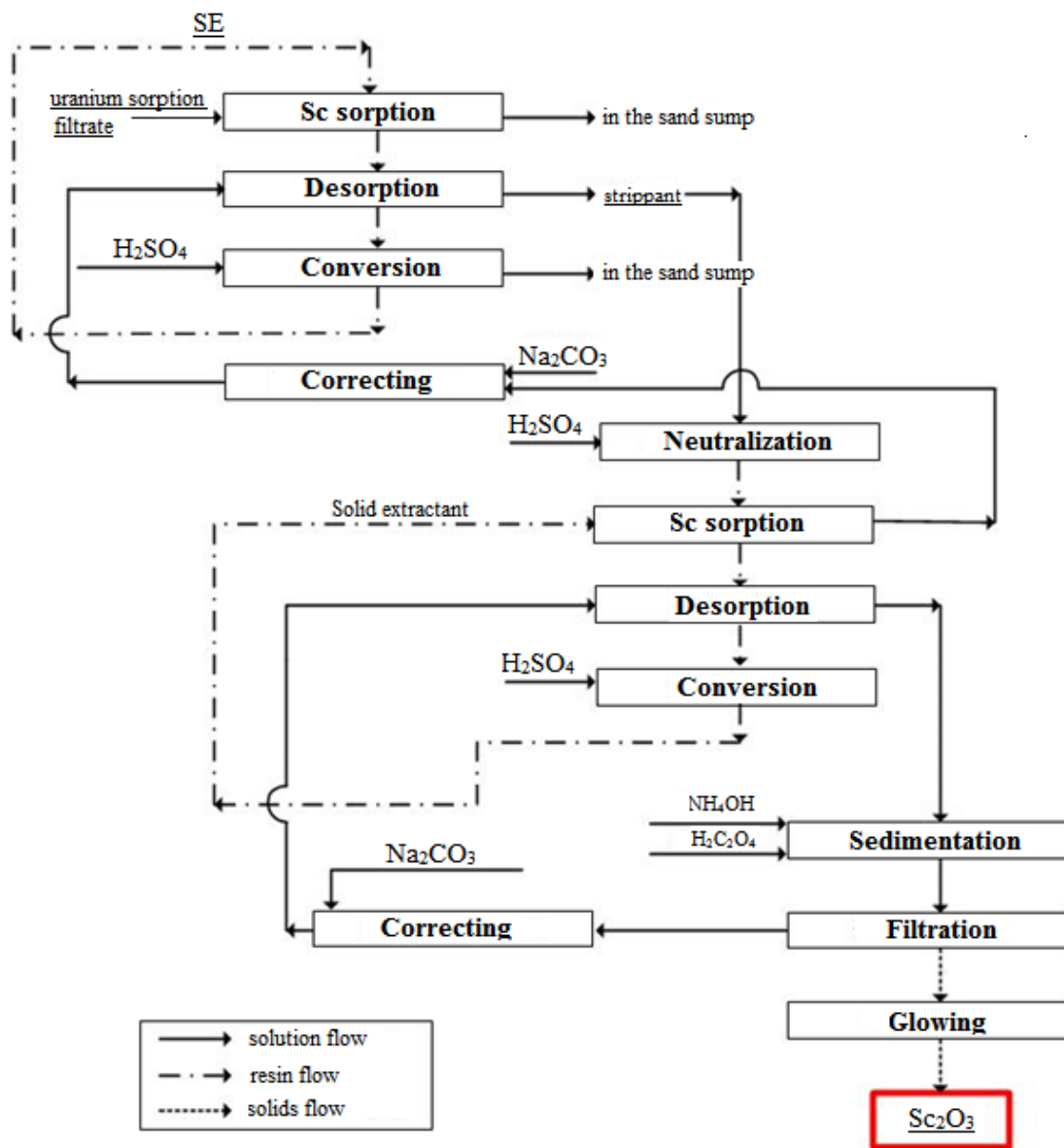


Figure4 – Process flow sheet of scandium extraction from uranium sorption filtrates

The scandium-containing solution (uranium sorption filtrate) is passed through PSC-3m type pressure sorption column filled with SE resin, before the scandium breaks through in the sorption filtrates with a flow of 250 m³/h.

2 m³ of scandium-saturated resin is discharged from the column every 24 hours. Sc content in the reloaded resin is 590 g/m³. Desorption is carried out using sodium carbonate solution with a concentration of 100 g/dm³ by passing 8 specific volumes of the solution through 2 m³ of resin in the corresponding column. An extraction degree of 40% sorbed scandium to obtain 16 m³ of strippant with an average content of 29.7 g/m³.

The resin is returned to the primary sorption operation. Strippant before subsequent sorption II is deoxidized with sulfuric acid concentrated solution. The conditioned solution is collected in a collection tank and then, with a flow of 0.25 m³/h, is sent to the sorption of stage II, also carried out on SE in the corresponding column. 640 specific volumes (16 m³) of the stage solution are passed through 0.025 m³ of the processed resin for 64 hours, after which the scandium content in the resin reaches 18.6 kg/m³.

At the end of sorption, desorption is carried out in the same column by passing a desorbing solution of 100 g/dm³ Na₂CO₃. Solution's 8 beats are flowed through 0.025 m³ of resin with the extraction of at least 50% of the adsorbed scandium. As a result, 0.2 m³ of solution is formed with an average concentration of 1.13 kg/m³. The resulting strippant is adjusted in pH to a value of 2÷3 with a solution of ammonia and oxalic acid dihydrate is added. Precipitated sediments are filtered on a nutsche-filter and calcined at a temperature of 900°C to obtain draft quality scandium oxide.

Thus, the productivity of the section will be about 0.214 kg of scandium in 24 hours, or 71.47 kg of scandium per year, which in terms of scandium oxide will be 109.35 kg.

The specified parameters are indicative and require confirmation during conducting semi-industrial tests on real solutions.

Conclusion

Based on the analysis of the obtained experimental data, sorption and desorption processes optimal operating parameters were determined with the simultaneous extraction of scandium from uranium sorption filtrates, which formed the basis of the developed conceptual technological scheme for scandium extraction. A preliminary technical and economic estimate of the process indicates the feasibility of further research on the associated extraction of scandium from real uranium sorption filtrates.

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СКАНДИЙ БАР ЕРІТІНДІЛЕРДІҢ ШОҒЫРЛАНДЫРУ ӘДІСІН ЗЕРТТЕУ

Аннотация. Қазіргі уақытта Қазақстандағы жерастылық ұңғымалық шаймалау әдісімен уран өндіретін кәсіпорындар (ЖҰШ) бір уақытта байланысты компоненттерді шығаруды жүзеге асырмайды. Қорларды бағалау және рентабельдік әрекеттерді жүргізуге мүмкіндік беретін тиімді технология бұл саладағы негізгі мәселелері болып табылады. Уранды ЖҰШ технологиялық ерітінділерінің ерекшеліктеріне пайдалы құрамдастың өте төмен мөлшері және күрделі химиялық құрамы кіруі керек. Осы мәселелердің кейбірін шешу үшін авторлар технологиялық шешімдерді әзірлеу және оңтайландыру бойынша жұмыс жасады.

Бұл мақалада ЖҰШ циклінің уран сорбциялық фильтраттарынан скандий сорбциясының алғашқы операцияларының скандий десорбаттарын әрі қарай шоғырландыру және тазарту зерттеулерінің нәтижелері келтірілген. Статикалық жағдайларда натрий карбонаты концентрациясының кинетикаға әсері және

шайырдан скандийдің десорбциясының тиімділігі ҚЭ-Д2ЭГФК шайырының мысалын қолдана отырып зерттелді. Сорбцияның екінші сатысының жұмысына статикалық жағдайда ҚЭ-Д2ЭГФК шайырында ерітіндінің рН әсер етуі қарастырылды және де скандийдің кейінгі карбонатты десорбциясы мүмкіндігі зерттелді.

Зертханалық зерттеулердің нәтижесінде ЖҮШ уран циклінің аясында скандийдің ілеспе өндірісінің негізгі технологиялық сызбасы жасалды және негізгі техникалық-экономикалық бағалаудың негізін құрайтын басты технологиялық параметрлер мен режимдер анықталды.

Түйін сөздер: скандий, сорбция, десорбция, экстракция, алудәрежесі, алмасу сыйымдылығы, сүзінді.

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ИССЛЕДОВАНИЕ СПОСОБА КОНЦЕНТРИРОВАНИЯ СКАНДИЙСОДЕРЖАЩИХ РАСТВОРОВ

Аннотация. В настоящее время предприятия в Казахстане, добывающие уран методом подземного скважинного выщелачивания (ПСВ), не осуществляют попутное извлечение ценных компонентов. Основными проблемами данного направления являются вопросы подсчета запасов и наличие эффективной технологии, позволяющей вести рентабельную деятельность. К особенностям переработки растворов ПСВ урана следует также отнести крайне низкие содержания ценных компонентов и сложный химический состав. Для решения некоторых из этих проблем авторами ведутся работы по разработке и оптимизации технологических решений.

В данной статье представлены результаты исследований дальнейшего концентрирования и очистки скандиевых десорбатов первой операций сорбции скандия из фильтратов сорбции урана цикла ПСВ. В статических условиях на примере смолы ТВЭКС-Д2ЭГФК изучено влияние концентрации карбоната натрия на кинетику и эффективность десорбции скандия из смолы. Рассмотрено влияние рН раствора на операцию второго этапа сорбции на смоле ТВЭКС-Д2ЭГФК в статических условиях и исследована возможность последующей карбонатной десорбции скандия.

В результате проведенных лабораторных исследований была разработана принципиальная технологическая схема попутного извлечения скандия в рамках цикла ПСВ урана и определены основные технологические параметры и режимы, которые легли в основу базовой технико-экономической оценки.

Ключевые слова: скандий, сорбция, десорбция, экстракция, степень извлечения, обменная емкость, фильтрат.

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