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Purification of metallic ions from technological solutions before sorption recovery of rhenium under JSC Almalyk MMC

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ABSTRACT

In this article, rhenium's distinct physicochemical characteristics, which make it essential for petrochemistry, electrical technology, rocket and aviation engineering, and the manufacturing of catalysts and high-precision tools, account for the metal's rising demand. The main source of rhenium at JSC "Almalyk MMC" is the off-gases produced when molybdenum concentrates are roasted, where rhenium is mostly found as Re_2O_7. High selectivity and overall efficiency are ensured by optimizing the process parameters at each of the multiple subsequent technical phases involved in rhenium recovery. Perrhenate sorption is less efficient when organic molecules and Mn^{2+} and Cu^{2+} ions are present in the process fluids. Oxidative-precipitation techniques were used for the first purification: Mn ions were oxidized and precipitated using potassium permanganate, and Cu^{2}+ ions were selectively precipitated using an ammonium sulfide solution $(NH_4)_2S$. ICP-OES was used to assess the composition of the solutions, while SEM and EDS were used to examine the roasting gas-dust products. Using contemporary analytical methods, a thorough investigation of the relevant phases of selective purification was conducted for the first time at JSC "Almalyk MMC." It was shown that treating the solutions with KMnO4 and $(NH_4)_2S$ efficiently eliminates interfering elements without causing rhenium or molybdenum losses, thereby creating ideal conditions for the sorption of perrhenate ions later on. Manganese and copper concentrations dropped from 1.44 to 0.0039 and 2.68 to 0.0036 g/l, respectively, demonstrating the great purification process efficiency. Rhenium and molybdenum concentrations did not alter during these phases, suggesting that they were fully preserved.

Keywords: sorption, ICP-OES, SEM, EDS, oxidative-precipitation treatment, molybdenum, rhenium, and selective purification

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Introduction

The unique physicochemical properties and high thermal stability of rhenium determine its wide application in rocket engineering, aviation, electrical engineering, petrochemistry, and in the production of catalysts and precision tools [[1], [2], [3], [4], [5]]. The primary industrial source of rhenium is the off-gases generated during the roasting of sulfide copper–molybdenum

concentrates (MoS_2), in which rhenium is predominantly present in the form of Re_2O_7 [[6], [7], [8], [9], [10], [11]]. Industrial recovery of rhenium involves several sequential technological stages, each requiring careful optimization of process parameters to ensure high selectivity and economic efficiency [[12], [13], [14], [15], [16]].

At JSC "Almalyk MMC," copper–molybdenum ores are processed by flotation [[17], [18], [19], [20]], producing sulfide copper and molybdenum concentrates. The initial processing stage includes oxidative roasting of molybdenum sulfide concentrates in electric tubular rotary furnaces at temperatures of 600–650 °C to convert sulfides before the subsequent leaching stage [21]. During roasting, molybdenum disulfide is oxidized to molybdenum trioxide according to the reaction:

$MoS_2 + 3.5O_2 \rightarrow MoO_3 + 2SO_2$

Simultaneously, rhenium sulfides are oxidized to volatile rhenium oxides, predominantly Re_2O_7:

$Re_2S_7 + 10.5O_2 \rightarrow Re_2O_7 + 7SO_2$

The off-gases generated during roasting contain sulfur dioxide and other volatile components, as well as minor amounts of sublimated oxides of rhenium and molybdenum [[22], [23]].

These gases undergo multi-stage purification using dry dust-collection systems followed by wet scrubbing units before being directed to sulfuric acid production [24]. As a result, acidic technological solutions enriched with rhenium are formed [[25], [26], [27], [28]]. During wet gas cleaning, dust particles are transferred into the scrubber slurry (cake), which is subsequently collected and stored as technogenic waste. However, the high acidity, complex chemical composition, and unfavorable physicochemical characteristics of this material, including high moisture content and poor filterability, complicate its comprehensive processing and limit its economic utilization.

Acidic scrubber solutions typically contain a range of impurity ions that may interfere with downstream hydrometallurgical processes. In particular, the presence of transition metals such as manganese, copper, and iron can influence solution chemistry, promote competing reactions, and reduce the efficiency of subsequent recovery stages. Therefore, preliminary purification represents an important step in preparing technological solutions for further processing.

Oxidative–precipitation methods are widely used in hydrometallurgy for the removal of interfering components due to their operational simplicity, technological reliability, and compatibility with strongly acidic media. Their application to complex technogenic solutions requires careful evaluation of purification efficiency and the behavior of valuable components during treatment.

This study aims to evaluate the effectiveness of oxidative precipitation purification for the targeted removal of interfering ions from acidic scrubber solutions before downstream recovery processes under the conditions of JSC "Almalyk MMC."

This study presents one of the first systematic assessments of oxidative precipitation purification performed under highly acidic industrial conditions (~300 g/L H_2SO_4), where the behavior of impurity ions differs substantially from that observed in conventional laboratory systems.

Selection and Characterization of Research Objects

Before the experimental work, the compositions of process gases, dust, and technological solutions formed during the roasting of molybdenum concentrates were analyzed to identify the primary sources of rhenium and associated impurity elements. Particular attention was given to the molybdenum-containing cake generated at the wet gas-cleaning stage, since its interaction with the scrubbing medium leads to the formation of acidic solutions subsequently subjected to purification.

The chemical composition of the molybdenum-containing cake is presented in Table 1. The analytical results indicate that the cake contains not only molybdenum and rhenium but also significant amounts of silicon, iron, copper, and other elements that may transfer into the liquid phase during wet gas treatment.

Table 1 - Chemical composition of molybdenum cake, %

Product (%)	Mo	Re	SiO_2	Fe	Cu	Zn	Au (g/t)	Ag (g/t)
Cake 1	41.1	0.025	26.14	4.92	0.01	1.7	17.6	48.76
Cake 2	39.8	0.08	24.1	4.06	0.02	1.5	18.2	42.00

The composition of gases and entrained dust produced during the roasting of molybdenum concentrate was also examined to better understand the origin of both valuable components and impurity ions in the scrubber solutions. Figure 1 shows a scanning electron microscope image of wet molybdenum dust, confirming the presence of fine particles capable of entering the scrubbing system and influencing the chemical composition of the resulting technological solutions.

Materials and Methods

Scrubber solutions collected from the industrial wet gas-cleaning system during the roasting of molybdenum concentrates at JSC “Almalyk MMC” were used as the primary research material. Prior to the experiments, the solutions were filtered through paper filters to remove suspended solids.

The technological solutions were characterized by a high sulfuric acid concentration (up to approximately 300 g/L), ensuring strongly acidic conditions throughout the experiments. The volumes of reagents used during purification were insufficient to cause significant dilution or neutralization; therefore, the solution remained strongly acidic during all treatment stages.

Purification Procedure

Selective purification was carried out in two sequential stages.

At the first stage, the pre-filtered solution was heated to 92 °C and treated with a 0.5 M potassium permanganate ($KMnO_4$) solution. The mixture was stirred using an electric mechanical stirrer at a speed of 200 rpm for 40 minutes to promote oxidation and precipitation of interfering ions. After completion of the reaction, the solution was cooled to room temperature and filtered to remove the formed precipitate.

At the second stage, the filtrate was treated with a 0.1 M ammonium sulfide ($(NH_4)_2S$) solution and stirred at 200 rpm for 20 minutes to facilitate the precipitation of copper ions. The resulting light-colored suspension was separated by paper filtration, yielding a clarified solution suitable for subsequent sorption processes.

Owing to the high initial sulfuric acid concentration (~300 g/L), the reagent volumes did not cause significant dilution or neutralization, and the solution remained strongly acidic throughout the purification process.

The purification procedure was repeated five times under identical conditions, and the reported results represent averaged values.

Analytical Methods

The morphology and elemental composition of solid particles were examined using scanning electron microscopy (SEM) coupled with energy-dispersive spectroscopy (EDS).

The chemical composition of the technological solutions before and after purification was determined by inductively coupled plasma optical emission spectrometry (ICP-OES).

Results

The analysis of off-gases and dust generated during molybdenum concentrate roasting confirmed their role as the primary source of rhenium and associated impurity elements entering the scrubber solutions (Table 2, Fig. 1).

Table 2 - Composition and characteristics of off-gases and dust generated during molybdenum concentrate roasting

Parameters of off-gases	Value	Unit
Oxygen flow rate V_0	565,8	m^3/h
Gas temperature T_r	140	°C
Dust loading z	3500	g/m^3
Dust particle size d, µm:	<5	20%
	5-10	22%
	10-20	24%
	20-40	16%

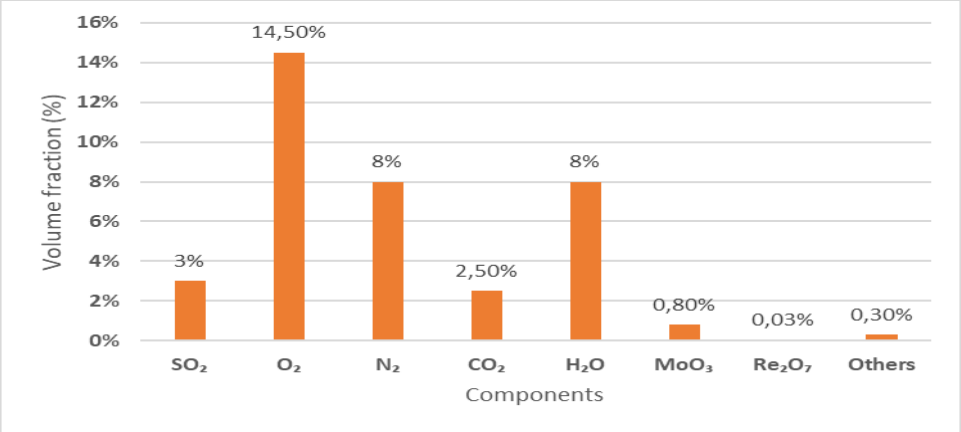

Fig. 1 - Composition of off-gases and dust contributing to the formation of scrubber solutions

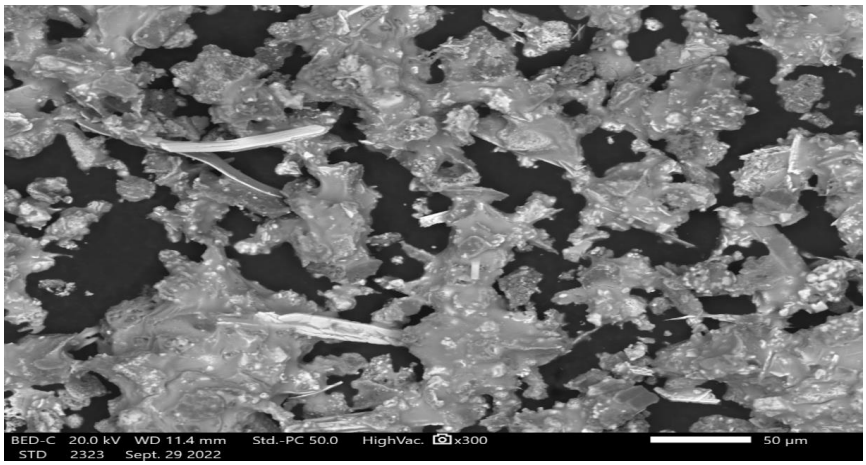

Fig. 2 - SEM image of wet molybdenum dust particles.

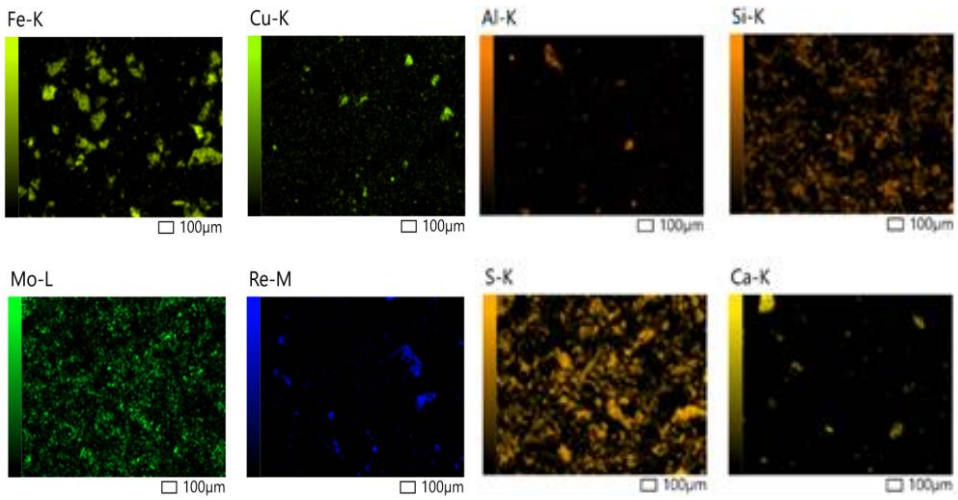

Fig. 3 - SEM images and EDS spectra of dust particles formed during molybdenum concentrate roasting.

SEM-EDS analysis revealed the presence of fine dust particles generated during the roasting of molybdenum concentrate. The particles exhibited heterogeneous morphology, indicating the entrainment of oxide phases from the high-temperature zone. Elemental spectra indicated the presence of Mo, S, O, Si, Fe, and trace amounts of rhenium, suggesting the transport of oxidized rhenium species into the dust phase. These observations support the role of roasting dust as a carrier of rhenium into the gas-cleaning system. These observations are consistent with the transfer of volatile rhenium species into the gas-cleaning system during concentrate roasting.

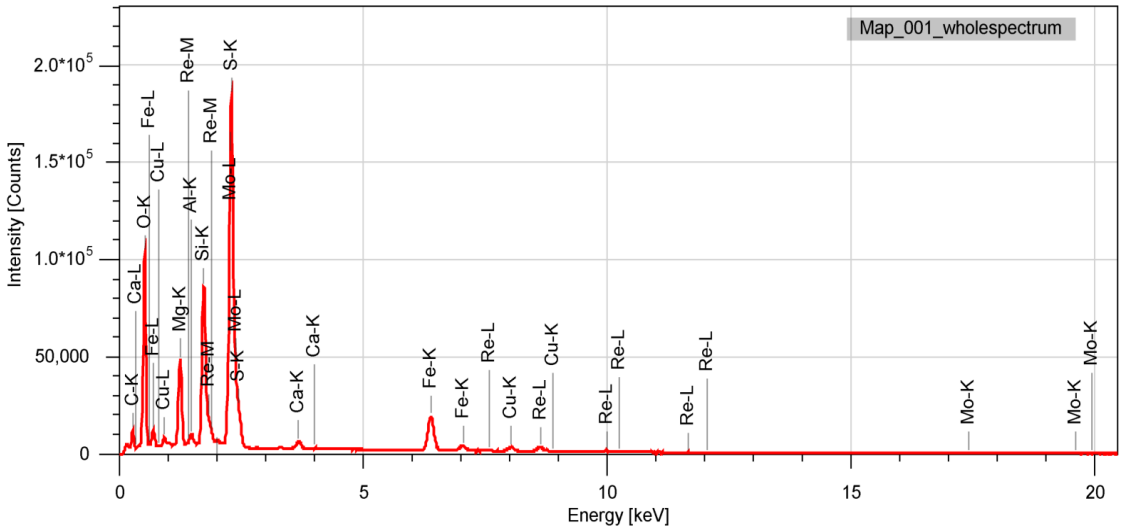

Fig. 4 - EDS spectra indicating the elemental composition of roasting dust particles.

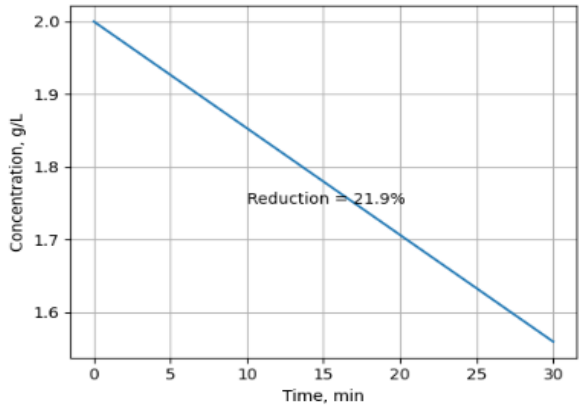

Fig. 5 - Changes in Fe^{3+} ion concentration during $KMnO_4$ treatment.

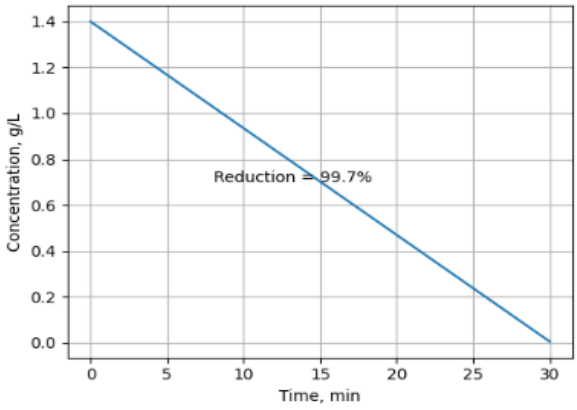

Fig. 6 - Changes in Mn^{2+} ion concentration during $KMnO_4$ treatment.

The presence of competing ions in strongly acidic technological solutions may negatively affect downstream recovery processes by occupying active sites of anion-exchange sorbents. Therefore, the removal or reduction of such components' during pretreatment is considered an important step in preparing solutions for further processing.

Scrubber solution samples were subjected to a two-stage oxidative–precipitation purification. Treatment with potassium permanganate resulted in the oxidation of Fe^{2+} to Fe^{3+}, while Mn^{2+} was converted into insoluble MnO_2, forming a colloidal precipitate that was subsequently removed by filtration. As a result, 21.9% of iron and 99.7% of manganese were removed (Figs. 5 and 6). The oxidation and precipitation processes can be described by the following reactions:

$$5Fe^{2+} + MnO_4^- + 8H^+ \rightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$$

$$3Mn^{2+} + 2MnO_4^- + 2H_2O \rightarrow 5MnO_2\downarrow + 4H^+$$

The limited removal of iron is attributed to the high stability of Fe^{3+} sulfate complexes in strongly acidic media, where iron predominantly remains in soluble form.

Equilibrium modeling was performed using Visual MINTEQ 3.1 to evaluate the speciation behavior of iron and manganese in strongly acidic sulfate media. The initial concentrations of the technological solution constituents (Table 4), together with the sulfuric acid concentration and temperature (25 °C), were used as input parameters. Modeling results (Fig. 7) indicate that under strongly acidic conditions, Fe^{3+} predominantly exists as soluble sulfate complexes ($FeSO_4^+$ and $Fe(SO_4)_2^-$), whereas Mn^{2+} is mainly stabilized as $MnSO_4$(aq). Under oxidizing conditions, manganese forms insoluble MnO_2, explaining the high degree of manganese removal observed experimentally.

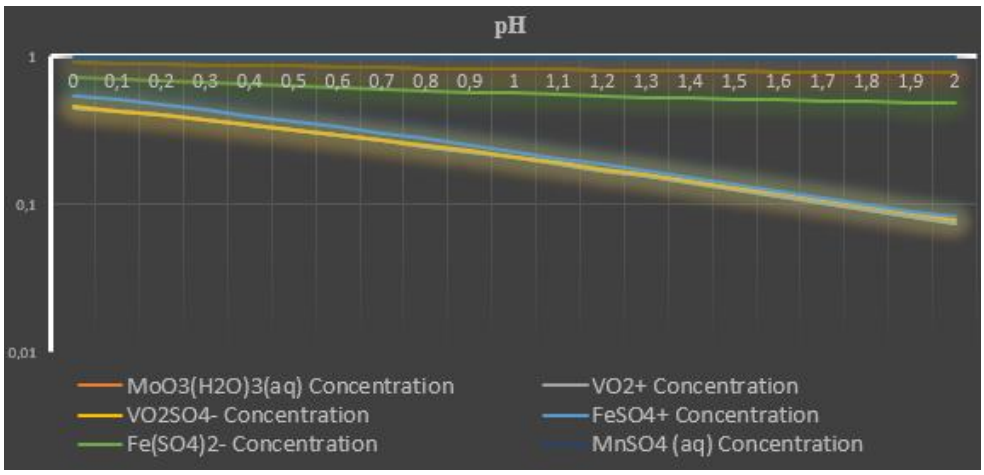

Fig. 7 - Distribution of iron and manganese species as a function of pH (0–2), calculated using Visual MINTEQ.

Table 4 - Elemental composition determined by ICP-OES (Thermo Scientific iCAP Pro)

Element	Initial solution (g/L)	Purified solution (g/L)
Nickel (Ni)	0.004	0.004
Iron (Fe)	2.01	1.57
Manganese (Mn)	1.44	0.0039
Rhenium (Re)	0.51	0.51
Molybdenum (Mo)	15.85	16.19
Vanadium (V)	0.019	0.019
Copper (Cu)	2.68	0.0036

At the second purification stage, treatment with ammonium sulfide precipitated Cu^{2+} ions as CuS, achieving a copper removal efficiency of approximately 95–100%, as confirmed by ICP-OES analysis and supported by equilibrium modeling.

The elemental composition of the initial and purified solutions was determined using ICP-OES (Thermo Scientific iCAP Pro, USA), and the results are presented in Table 4.

The results presented in Table 4 demonstrate a substantial reduction in the concentrations of major impurity ions following purification, particularly manganese and copper. Manganese concentration decreased from 1.44 g/L to 0.0039 g/L, while copper was reduced from 2.68 g/L to 0.0036 g/L, confirming the high efficiency of the oxidative–precipitation process.

The concentrations of rhenium and molybdenum remained essentially unchanged, indicating the absence of valuable metal losses during treatment. The slight increase in molybdenum concentration is within the analytical uncertainty of the ICP-OES method and does not indicate actual enrichment.

These findings confirm the effectiveness of oxidative precipitation treatment for removing interfering components from strongly acidic technological solutions while preserving valuable metals in the liquid phase.

Conclusion

Technological solutions obtained during the processing of molybdenum concentrates at JSC “Almalyk MMC” require preliminary purification to remove interfering components prior to downstream recovery stages. The results of this study demonstrate that oxidative precipitation treatment using potassium permanganate and ammonium sulfide effectively reduces the concentrations of major impurity ions, particularly copper and manganese. Copper concentration decreased from 2.68 g/L to 0.0036 g/L, while manganese was reduced from 1.44 g/L to 0.0039 g/L.

SEM-EDS analysis confirmed the presence of oxidized rhenium species and molybdenum-containing phases in the dust particles formed during concentrate roasting. The purification process did not result in detectable losses of rhenium and preserved valuable metals in the liquid phase.

These findings indicate that oxidative precipitation treatment represents an effective and technologically applicable approach for improving the chemical composition of strongly acidic process solutions by removing interfering ions while maintaining the stability of valuable components.

Conflict of interest. On behalf of all the authors, the corresponding author declares that there is no conflict of interest.

CRediT author statement: **Kh. Azizova:** conceptualization, designed the experimental methodology, writing – original draft; **O. Usmankulov:** writing -review and editing; **N. Kattaev:** writing -review and editing, literature review; **Z. Kadirova:** methodology and editing; **M. Yakubov:** writing – review and editing, supervision, methodology; **Kh. Akbarov:** writing - review and editing, data curation.

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Алмалық ТКМК АҚ жағдайында ренийді сорбциялық жолмен бөліп алу алдында технологиялық ерітінділерді металл иондарынан тазарту

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АННОТАЦИЯ

Бұл мақалада ренийдің ерекше физикалық және химиялық қасиеттерін қарастырып, мұнай-химиясы, электр техникасы, зымыран және авиация техникасында, сондай-ақ катализаторлар мен жоғары дәлдіктегі құралдар өндірісі үшін аса маңызды ететін ерекше физика-химиялық қасиеттеріне байланысты осы металға деген сұраныстың артып келе жатқаны қарастырылады.«Алмалық ТКМК» АҚ -да ренийдің негізгі көзі молибден концентраттарын күйдіру кезінде түзілетін түтін газдар болып табылады, мұнда рений негізінен Re_2O_7 түрінде болады. Ренийді бөліп алу бірнеше техникалық кезеңдерден тұратындықтан, процестің әрбір сатысындағы параметрлерді оңтайландыру арқылы жоғары селективтілік пен жалпы тиімділікке қол жеткізіледі. Технологиялық ерітінділерде органикалық молекулалар мен Mn^{2+} және Cu^{2+} иондары болған кезде перренат сорбциясының тиімділігі төмен болады. Алғашқы тазарту үшін тотығу-тұндыру әдістері қолданылды: Mn иондары калий перманганатының көмегімен тотықтырылып тұндырылды, ал Cu^{2+} иондары аммоний сульфиді $(NH_4)_2S$ ерітіндісін пайдаланып селективті түрде тұндыру жүргізілді. Ерітінділердің құрамын бағалау үшін ICP-OES әдісі, ал күйдіру кезіндегі газ-тозаң өнімдерін зерттеу үшін SEM және EDS әдістері пайдаланылды. Заманауи аналитикалық әдістерді қолдана отырып, «Алмалық ТКМК» АҚ жағдайында селективті тазартудың тиісті кезеңдеріне алғаш рет жан-жақты зерттеу жүргізілді. Ерітінділерді $KMnO_4$ және $(NH_4)_2S$ реагенттерімен өңдеу рений мен молибденнің шығынынсыз кедергі келтіретін қоспаларды тиімді түрде жоятындығы, соның нәтижесінде кейінгі перренат иондарының сорбциясы үшін оңтайлы жағдайлар жасалатыны көрсетілді. Марганец пен мыс концентрациясы сәйкесінше 1,44-тен 0,0039 г/л-ға дейін және 2,68-ден 0,0036 г/л-ге дейін төмендеді, бұл тазарту процесінің жоғары тиімділігін дәлелдейді. Бұл фазаларда рений мен молибден концентрациясы өзген жоқ, бұл олардың толық сақталғанын көрсетеді.

Түйін сөздер: *сорбция, ICP-OES, SEM, EDS, тотығу-тұндыру арқылы өңдеу, молибден, рений, селективті тазарту.*

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Очистка технологических растворов от ионов металлов перед сорбционным извлечением рения в АО Алмалыкский ГМК

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АННОТАЦИЯ

В этой статье рассматриваются особые физико-химические характеристики рения, которые делают его незаменимым для нефтехимии, электротехники, ракетной и авиационной техники, а также для производства катализаторов и высокоточных инструментов, что объясняет растущий спрос на этот металл. Основным источником рения на АО "Алмалыкский ГМК" являются отходящие газы, образующиеся при обжиге молибденовых концентратов, где рений в основном содержится в виде Re_2O_7. Высокая селективность и общая эффективность обеспечиваются за счет оптимизации параметров процесса на каждом из множества последующих технических этапов, связанных с извлечением рения. Сорбция перрената менее эффективна, когда в технологических жидкостях присутствуют органические молекулы и ионы Mn^{2+} и Cu^{2+}. Для первой очистки были использованы методы окислительного осаждения: ионы марганца были окислены и осаждены с использованием перманганата калия, а ионы Cu^{2+} были выборочно осаждены с использованием раствора сульфида аммония $(NH_4)_2S$. ICP-OES использовали для оценки состава растворов, в то время как SEM и EDS использовались для изучения состава растворов. обжиг газопылевых продуктов. Впервые в условиях АО "Алмалыкский ГМК" с использованием современных аналитических методов было проведено тщательное исследование применимых стадий селективной очистки. Было показано, что обработка растворов $KMnO_4$ и $(NH_4)_2S$ эффективно устраняет мешающие элементы, не вызывая потерь рения и молибдена, что обеспечивает идеальные условия для последующей сорбции перренат-ионов. Концентрации марганца и меди снизились с 1,44 до 0,0039 и с 2,68 до 0,0036 г/л соответственно, что свидетельствует о высокой эффективности процесса очистки. Концентрации рения и молибдена не изменялись в течение этих фаз, что позволяет предположить, что они полностью сохранились.

Ключевые слова: сорбция, ICP-OES, SEM, EDS, окислительно-восстановительная обработка, молибден, рений и селективная очистка

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