

Review of modern scientific developments in the field of molybdenum recovery from spent catalysts

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The widespread use of rare metals in industry requires an increase in the amount of their production and expansion of methods for obtaining them. In this regard, the use of secondary raw materials in modern production is growing rapidly and steadily.

Molybdenum is a refractory metal, which is used as an alloying element in various steels, heating elements of electric furnaces, special purpose alloys, electrovacuum equipment and other fields. Data on the volume of consumption of catalysts in the oil refining and petrochemical industries, as well as on the import of molybdenum-containing catalysts are given.

The technologies for processing spent hydro-treating catalysts with production of molybdenum oxide are considered, including oxidative roasting of the feedstock, as well as such operations as: leaching by sulfuric, hydrochloric, and nitric acids, sodium carbonate [7]. Hydrometallurgical methods of decomposition of raw materials are usually combined with the subsequent extraction of molybdenum by extraction with citric acid [16], MTAA carbonate [15], or ion-exchange sorption.

High purity molybdenum is characterized by a number of unique properties that are important for its use in modern technology. Most of the hard-to-remove impurities get into molybdenum oxide from the feedstock, so one of the important tasks is to optimize the leaching process at the stage of uncovering of technogenic raw materials. Recently, more and more attention has been paid to obtaining high-purity molybdenum oxide, as well as to the separation of molybdenum and tungsten. One of the promising processes seems to be ion exchange, which makes it possible to purify solutions from metal impurities from solutions and pulps formed during the decomposition of molybdenite concentrates or during leaching of raw materials.

Key words: spent catalysts, molybdenum, roasting, leaching, pulp, reagent, extraction, solvent, ion exchange.

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Introduction

Molybdenum is a refractory metal that is often used in metallurgy [1]. It is at number 42 in the D. I. Mendeleev periodic table of chemical elements. According to the source [1], the mass content of metal in the earth's crust is $3 \times 10^{-4}\%$, however, molybdenum is not found in free form.

High-purity molybdenum oxide is in demand in the production of molybdenum-containing catalysts, which are widely used in oil refining (cracking, hydrotreatment, reforming), conversion of methanol to formaldehyde, vapor-phase oxidation of propylene to acrolein, ammonolysis of toluene, epoxidation of various alkenes, and others [2].

Currently, there is an increase in the demand for catalysts in the oil refining and petrochemical industries (Fig. 1). Thus, in 2020, the consumption of catalysts in Russia amounted to more than 17.5 thousand tons. According to the forecast, by 2030 this figure will reach 26 thousand tons [3].

The schedule of imports of high-purity molybdenum oxide was built on the basis of data from the customs service of the Russian Federation [4] (Fig. 2).

According to Fig. 2 imports of high-purity molybdenum oxide are increasing every year.

The volume of catalysts imports in the Russian Federation is shown in Fig. 3.

The prices for the cobalt–molybdenum catalyst without customs duties and VAT ranged from \$12.6 to \$22.8/kg, for the nickel–molybdenum catalyst from \$12.1 to \$27.6/kg, and for the iron–molybdenum catalyst from \$6.3 to \$14.7/kg [4].

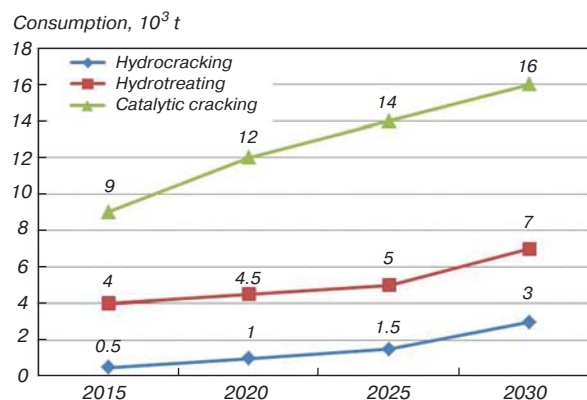


Fig. 1. Demand for catalysts by types, thousand tons per year

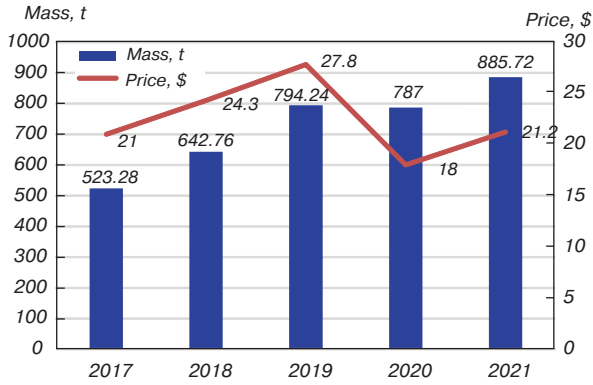


Fig. 2. Import of high-purity molybdenum oxide to the Russian Federation

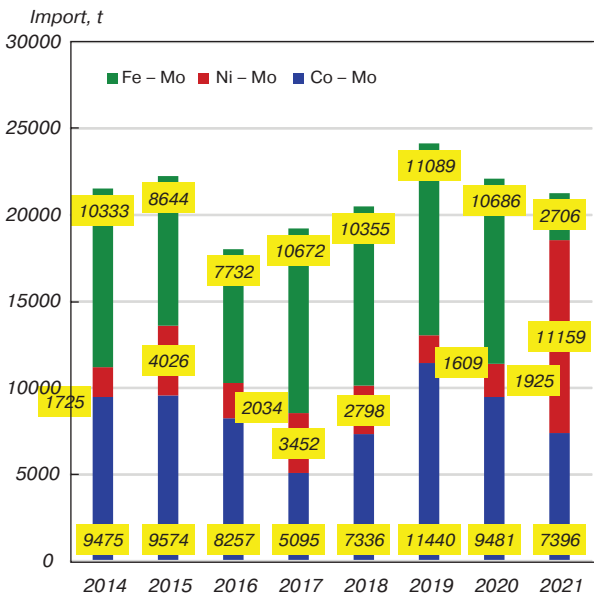


Fig. 3. Volumes of imported molybdenum-containing catalysts in the Russian Federation based on the customs service data

As for our own production, the molybdenum oxide produced according to the existing technology at JSC Company Wolfram by the hydrometallurgical method has the following composition, %: 60.1 Mo, 1.29 W, 0.044 Cu, 0.064 P, 0.23 S, 0.0010 Pb. This technology does not provide the production of high-purity molybdenum oxide with an impurity content of less than 0.1% [5].

Processing of technogenic raw materials, in the form of spent catalysts, will reduce the ecological burden on the environment. And the production of high-purity molybdenum oxide from them will, firstly, reduce the import of high-purity molybdenum oxide and ready-made molybdenum-containing catalysts in the Russian Federation, and secondly, it will allow to organize the production of domestic catalysts for the hydrotreatment of petroleum products (molybdenum-nickel, molybdenum-cobalt and iron-molybdenum), also completely imported into the Russian Federation.

Thus, spent catalysts can become a promising source of molybdenum production. Pyrometallurgical and hy-

drometallurgical methods for processing such raw materials have been developed.

Methods

In [6], the authors conducted several series of experiments to identify an effective molybdenum lixiviant from spent catalysts. Before leaching, the initial catalyst was crushed, treated with H_2O_2 , and calcined at a temperature of 700 °C.

In the first series of experiments, leaching was carried out with a 34% ammonia solution for 1 hour, after which the solution was filtered off and the precipitate was re-treated with ammonia. After that, both solutions were mixed and treated with nitric acid $pH = 3$, then filtered again. After precipitation of molybdic acid, part of the solution was evaporated for chemical analysis of the content of molybdenum. The results of chemical analysis showed that 1% of molybdenum remained in the solution. Thus, ammonia leaching does not provide complete recovery of molybdenum.

In the second series of experiments, leaching was carried out with alkali (NaOH) in an autoclave. The results of chemical analysis showed that 1.7% of molybdenum remained in the washed filter residue.

In the third series of experiments, the authors used two-stage leaching: soda and alkali, the scheme of which is shown in Fig. 4.

When boiling for 3 hours from the sodium molybdate agglomerate formed during the calcination of granules at 700 °C, sodium molybdate goes into solution. It is removed from the solution by filtration. The results of

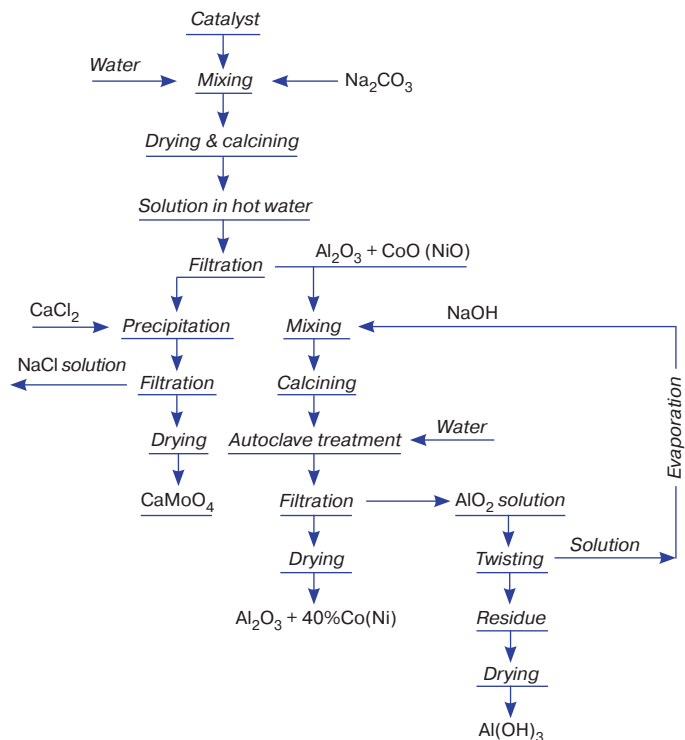


Fig. 4. Technological scheme of two-stage leaching of molybdenum from spent catalysts

chemical analysis showed that 0.8% of molybdenum remained in the washed filter residue.

In [7], the process of oxidative roasting of a spent catalyst for the petroleum products hydrotreatment with addition of lime was studied. Spent catalysts containing wt. %: Mo, 9.7; Co – 3.1; V – 0.1 were used as raw materials. Oxidative firing was carried out in a muffle furnace at temperatures of 550–600 °C with an air supply rate of 3 l/min to increase mass transfer. Also in the work, the firing parameters were optimized for the process duration and lime consumption.

As a result of the study, it was found that at a firing temperature of 575 °C for 40 min with a mass ratio of CaO : spent catalyst = 0.4, the best indicators for the purification of the product from sulfur and carbon are achieved, and the formation of calcium molybdate, suitable for further leaching process, was established. In addition, the paper emphasizes the need to classify sintering products by fractions: less than 5 microns and more than 5 microns. The fraction of less than 5 µm contains CaMoO₄ and oxides of aluminum and cobalt, and the fraction of more than 5 µm, consists mainly of calcium carbonates and sulfates and unreacted lime, in order to reduce contamination of the products of further processing.

The authors of [8] developed a complex technology for the extraction of high purity vanadium and molybdenum from spent oil catalysts. The feedstock was washed with acetone to remove organic matter, dried, calcined for 4 hours, and crushed. The catalysts were then leached with a solution of an extractant in kerosene such as D2EHPA(di-(2-ethylhexyl) phosphoric acid, cationic extractant), Alamine336 (N,N-dioctyl-1-octylamine, anionic extractant), Aliquat336 (N-methyl-N,N,N-trioctylammonium chloride, ionic liquid), as well as a solution of ALi-D2 (N-methyl-N,N,N-trioctylammonium bis(2-ethylhexyl)phosphate, bi-functional ionic liquid) and H₂O₂. The optimal modes for extracting V and Mo into the solution were: C(ALi-D2) = 0.4 mol/l, volume ratio H₂O₂ : ALi-D2 = 2.5, temperature 60 °C, duration 15 minutes, stirring speed 300 rpm.

After that, molybdenum was precipitated from the filtrate with a BaCl₂ solution. Insoluble residues from leaching were also leached to further extract valuable components from solution. This technology makes it possible to obtain vanadium and molybdenum with a purity of 99.5% and 84.1%, respectively, with a reduction in the cost of processes and environmental problems.

In [9], a study was conducted on the extraction of molybdenum from spent refinery Ni/Al₂O₃-based catalysts crushed to 100 µm containing in mass. %: Al – 27.1, Mo – 11.6, Ni – 2.5, S – 9.7, P – 1.9, C – 4.8 and traces of Fe, Co, Si. Sulfuric acid was used as a leaching agent. To study the effect of technological parameters on the valuable component extraction, the temperature and duration of the process, the pH of the solution, and the ratio of *S* : *L* were varied.

As a result of the study, it was found that during acid leaching, the maximum extraction of Mo into solution is 65% at a temperature of 95 °C, a duration of 2 hours and using an 20% concentration acid. On the contrary, preliminary calcination of the spent Mo – Ni/Al₂O₃ catalyst with H₂SO₄ followed by concentrate leaching (in excess of 1.2 of the stoichiometry) at 300 °C for 1 h contributed to an increase in the recovery of Mo at similar leaching parameters up to 96%. It was also found that with an increase in the sintering time of a powdered catalyst with sulfuric acid, a decrease in the degree of leaching is observed, which the authors attribute to the volatilization of the reagents during sintering.

Thus, the authors of the work propose the following technological parameters for the highest degree of extraction: the use of pre-sintering of crushed spent oil refining catalysts with an excess of sulfuric acid (1.2 of stoichiometric) at a temperature of 300 °C for 1 h, followed by weak acid leaching at a temperature of 80 °C, H₂SO₄ concentration of 2 vol. % and process duration of 30 min.

In [10], a study of the possibility of extracting molybdenum using acid leaching was also made. Co – Mo/Al₂O₃ catalysts were used as feedstock. For acid leaching, sulfuric acid (1.5 M) and a mixture of sulfuric and nitric acids were used as agents. Also, to select the leaching mode that provides the maximum degree of Mo extraction into the solution, the following parameters were optimized: particle size, process temperature and duration, acid concentration, and *S* : *L* ratio. In the course of the study, it was found that with direct sulfuric acid leaching at a temperature of 50 °C for 5 h, the degree of Mo extraction into the solution did not exceed 20%, while for a mixture of acids H₂SO₄ (0.5 M) and HNO₃ (1.0 M) at the same parameters, the degree of extraction was 78.2%, and the authors of the work noted the effect of the nitric acid concentration on the extracting molybdenum process: with a further increase in the HNO₃ concentration to 4 M, the degree of leaching increases to 97.9%, while with a change in the sulfuric acid concentration, the degree of Mo leaching did not change. Also, when studying the dependence of the extraction degree on the size of the initial particles of the catalyst, it was found that with an increase in the size of the initial particles, the degree of leaching decreases from 97.9% to 93% for particles with a size of 50 and 250 µm, respectively, which is explained by an increase in the specific surface area of the particles and the area of contacts between solid and liquid phases.

On the basis of the studies carried out, the authors of the work proposed the following technological parameters of the leaching process: temperature of 50 °C, pulp density of 10%, the use of a mixture of H₂SO₄ (0.5 M) and HNO₃ (4.0 M) acids, particle size of 50-71 microns with a process duration of 5 hours.

In [11], the vanadium and molybdenum recovery from spent catalysts by leaching with a NaOH solution under microwave irradiation was studied. Preliminary preparation of raw materials was grinding to a particle

size of 150 microns, sifting to determine the particle size distribution.

Based on the studies, it was found that the vanadium and molybdenum extraction into the solution reached 94 and 96%, respectively, at a microwave power of 600 W, a leaching time of 10 minutes, a process temperature of 90 °C, a solid-to-liquid ratio of 0.2 g/ml, constant stirring of 400 rpm. Moreover, such a high recovery is achieved by leaching using microwaves. The leaching process by the traditional method takes longer.

The authors of [12] studied the molybdenum extraction from spent aluminum-molybdenum-cobalt (AMC) oil refining catalysts. To extract valuable components from AMC, leaching with aqueous solutions of sodium and ammonium carbonates, as well as solutions of sodium and ammonium hydroxide was used. According to the results of the study, it was found that the surface film of organic compounds (coke) formed during the catalysts operation reduces the degree of molybdenum transition into solution: the highest degree of molybdenum extraction at a temperature of 70 °C and a ratio of $S : L = 1:50$ was 20%.

Preliminary roasting before the leaching process for 1 h and at a temperature of 400–500 °C contributed to the removal of organic compounds and sulfur from the catalysts, which made it possible to increase the degree of molybdenum extraction with an Na_2CO_3 aqueous solution to 99.9% under the same process conditions. It is also noted that during leaching with sodium carbonate, a joint extraction of aluminum occurs (up to 15%), in sodium systems the cobalt extraction into solution was not found.

The molybdenum extraction from carbonate solutions was carried out using toluene solutions of carbonates and methyltrioctylammonium (MTOA) molybdate in the presence of gaseous CO_2 . It has been established that the presence of gaseous CO_2 promotes the polymerization of molybdate anions in the organic phase with the formation of polymolybdate anions, which increases the Mo distribution and purification coefficient from aluminum impurities, due to the displacement of Al by Mo atoms from the extract. In the case of using MTOA molybdate, the Mo purification coefficient from Al was 100. The back-extraction process can be carried out to obtain ammonia solutions of ammonium molybdate with a concentration of 100–300 g/l or to obtain a crystalline hydrate $\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$.

To extract cobalt from an insoluble precipitate after the process of molybdenum leaching with an Na_2CO_3 aqueous solution, the authors of [12] used an ammonium carbonate solution with the addition of a 0.2 M aqueous solution of H_2O_2 to transfer Co(II) to Co(III). It has been established that an increase in temperature and the ratio of $S : L$ leads to an increase in the degree of cobalt extraction into the solution and, under optimal conditions, is 80%. To isolate cobalt from the solution, the resulting solutions are boiled to form $\text{Co}[(\text{NH}_3)_6]_2(\text{CO}_3)_3\cdot 4\text{H}_2\text{O}$ precipitates and their further thermal decomposition. It was noted that

Table

Elemental composition of the catalyst

Element	Na	Fe	Ni	Mo	Al	Si
Content, %	1.04	0.58	2.47	8.13	46.58	2.88

the addition of NaOH during boiling promotes the Co_3O_4 formation.

As a feedstock, the author of [14] used samples of the most common hydrotreating catalysts of the GKD-202 and GKD-205 grades, which are cylindrical gray-brown, blue, and black granules. The **Table** shows the elemental composition of the GKD-205 catalyst [13].

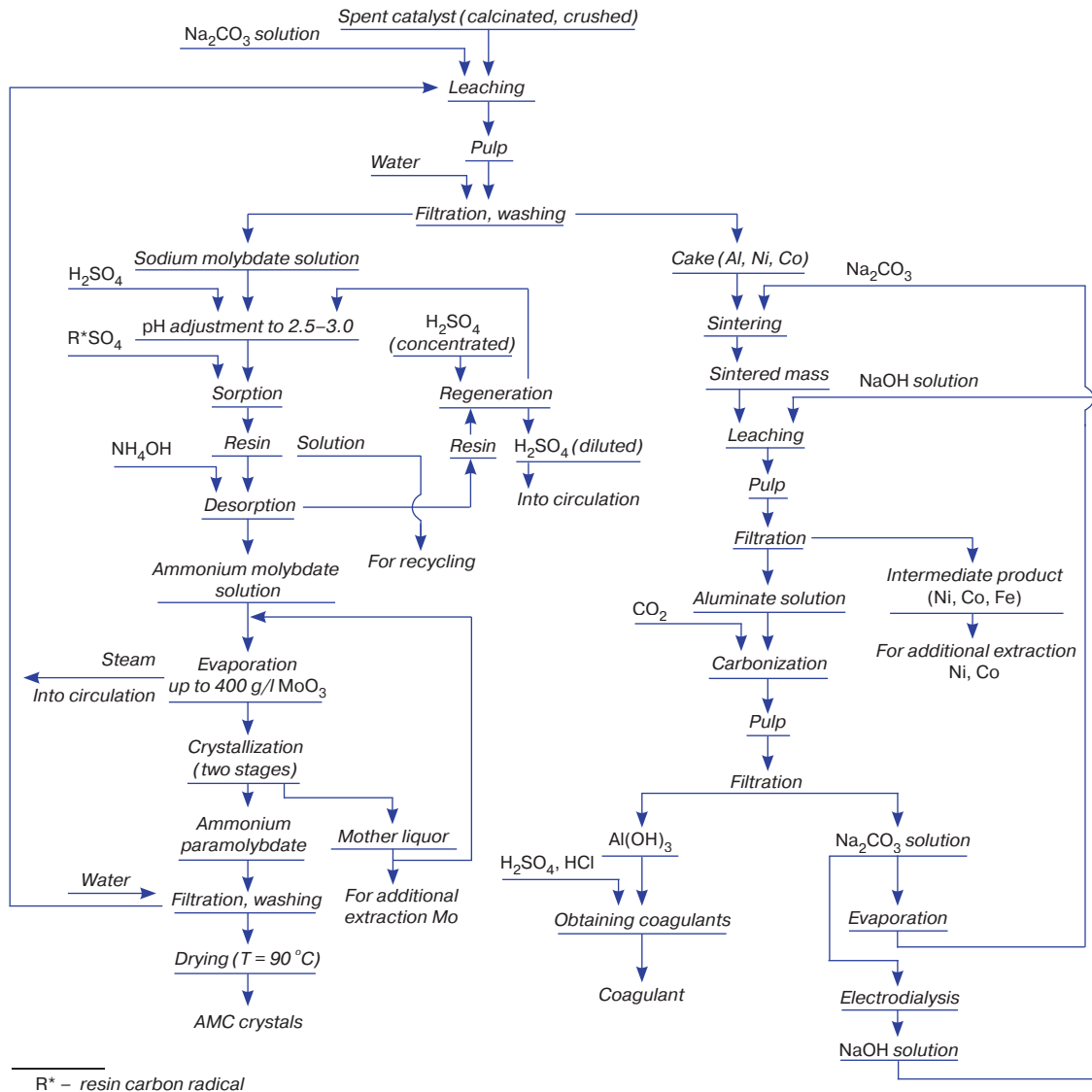
As can be seen from the table, the catalyst is based on aluminum (46.58%), molybdenum (8.13%), and nickel (2.47%).

Using X-ray diffraction analysis, it was found that the basis of spent catalysts is represented by two modifications of aluminum oxide $\alpha\text{-Al}_2\text{O}_3$ and $\gamma\text{-Al}_2\text{O}_3$, as well as in the composition of NiAl_2O_4 , $\text{NiAl}_{26}\text{O}_{40}$ и $\text{Na}_2\text{Al}_{2x}\text{O}_{3x}$. Molybdenum is represented by the NiMoO_4 and Na_2MoO_4 modifications, and also in the form of MoS_2 . In accordance with the basic technological scheme (**Fig. 5**), the calcined, crushed catalyst of the GKD-205 grade is supplied for leaching with an aqueous solution of sodium carbonate with a concentration of 5–10 wt.% at $S : L = 1:10$, the process duration of 60–90 minutes and temperature of 85–100 °C [13]. The use of soda leaching makes it possible to reduce the amount of nickel and aluminum compounds in solution and improve the quality of the products obtained, and also reduces the resin consumption during subsequent sorption. Next, the resulting pulp is filtered, washed, and the sodium molybdate solution is sent to the sorption extraction of molybdenum. Then, the resulting solution is sent for purification from phosphorus, arsenic, iron, nickel.

The technological scheme also provides for the processing of the alumina residue to obtain aluminum oxide and various coagulants for the purification of drinking and waste water, including sintering of the alumina residue, leaching of the resulting sintered mass, filtration, carbonization of the aluminate solution and subsequent filtration.

To ensure the highest degree of extraction of aluminum oxide into the aluminate solution, the alumina residue is sintered at a molar ratio in the charge of $\text{Na}_2\text{O}:\text{Al}_2\text{O}_3 = 1.1$ at a temperature of 1250 °C and a sintering time of 2 hours. Next, the sintered mass is crushed and leached with a sodium hydroxide solution at $S : L = 1 : 3$ and a process temperature of 90 ± 5 °C.

Further, the resulting pulp is filtered and the aluminate solution (35–40 g/l) is fed to carbonization at temperatures of 25–30 °C for 1.5–2 hours. The resulting pulp is filtered and aluminum hydroxide is sent to the synthesis of coagulants — sulfate, hydroxosulfate and aluminum hydroxochloride. The considered scheme for the processing of catalysts by soda leaching followed by sintering the insoluble residue with soda and leaching the sintered mass



R* – resin carbon radical

Fig. 5. Principal process flow diagram for processing spent hydrotreating catalysts

with a solution of sodium hydroxide is characterized by a low extraction of molybdenum 75–85% and the extraction of aluminum into the by-product (aluminum hydroxide) also does not exceed 85%. The applied mode of sintering the insoluble residue of soda leaching for 2 hours at 1250 °C and the Na₂O : Al₂O₃ ratio of 1.1 leads to high energy consumption.

The authors of [14] studied the extraction removal of molybdenum from carbonate solutions of the processing of spent catalysts with methyltrioctylammonium salts. Extraction of molybdenum from aluminumcobaltmolybdenum catalysts was carried out using a solution of carbonate and molybdate MTAA (MTOA) in toluene. Methyl sulfate MTAA (MTOA) acted as the initial extractant. Carbonate and alkaline solutions were obtained, depending on the spent catalyst leaching conditions.

Extraction from carbonate solutions was carried out in the pH range = 7.5–12, as pH above the upper limit of

the interval, the concentration of OH ions increases, as a result D_{Mo} decreases.

Extraction from an alkaline solution was carried out with MTAA carbonate at pH = 12.3, which contributed to the Mo extraction of 38% in one step. Upon carbonization of the alkaline solution with gaseous CO₂, the pH dropped from 12.3 to 6.9, but upon extraction from the bicarbonate solution with MTAA carbon, the Mo extraction was 55%. Thus, by lowering the pH and bubbling gaseous CO₂, D_{Mo} rises. The end result of the extraction process is the obtaining of 99.9% Mo from the raffinate.

In [15], the object of study is a spent catalyst from the Indonesian PT company. Leaching was carried out using citric acid at pH (1.5, 2, 2.5) for 120 minutes at temperatures of 60, 80 and 90 °C. The result of the experiment showed that the maximum Mo extraction 71.2% is carried out at a temperature of 90 °C and pH 1.5 for 2 hours. Thus citric acid has potential as a leaching agent.

Discussion

The review of the scientific and technical literature shows that most researchers for the extraction of molybdenum from spent catalysts choose a hydrometallurgical processing scheme, in particular leaching methods, followed by the extraction of molybdenum from solution by extraction or sorption methods, which provide more pure molybdenum compounds compared to traditional precipitation methods by sulfides and fractional crystallization of ammonium paramolybdate.

Extraction is characterized by high selectivity with respect to the extracted target metal [16]. Thus, the degree of extraction of molybdenum from carbonate solutions of spent catalysts leaching with an extractant in the form of a solution of methyltrioctylammonium salts [14] was 99.9%. But the extraction technology is more complex than the sorption one and is used to a lesser extent. Also, a significant disadvantage is that the solvents used for extraction are toxic and flammable, as a result it is a rather expensive process to organize industrial production. That is why the most attractive are environmentally friendly and easy-to-maintain sorption methods.

At the same time, sorption methods also have disadvantages - high cost and short service life of ion-exchange resins and insufficient selectivity with respect to molybdenum ions.

It was also proposed to use citric acid as an organic extractant to extract molybdenum [15], but the degree of molybdenum extraction using this technology does not exceed 72%, which is lower than that of sorption methods.

Conclusion. Sorption methods for extracting molybdenum are the most promising for large-scale production.

The results of R&D were achieved during the implementation of the project using measures of state support for the development of cooperation between Russian educational institutions of higher education, state scientific institutions and organizations of the real sector of the economy implementing complex projects to create high-tech production, provided for by Decree of the Government of the Russian Federation dated April 9, 2010 No. 218. An agreement on the provision of subsidies from the federal budget for the development of cooperation between a Russian educational organization of higher education and an organization in the real sector of the economy in order to implement a comprehensive project to create a high-tech production "Creation of an import-substituting production of high purity vanadium oxide for deep processing of hydrocarbon raw materials" was concluded between the Ministry of Science and Higher Education of the Russian Federation and JSC "Company Wolfram" dated June 25, 2021 no. 075-11-2021-053.

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