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Sorption of cobalt by anion exchangers from solutions for complex processing of rhenium-containing superalloys waste

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Abstract: The possibility of cobalt sorption of by anion exchangers from hydrochloric acid solutions in the presence of nickel was evaluated in the paper. The highest value of sorption capacity was observed in the gel anion exchange resin with pyridinium functional groups (Axionit VPA G.2.4) and the macroporous anion exchange resin with functional groups of secondary and tertiary amine (Indion 850). Processing of cobalt sorption isotherms with Indion 850 and Lewatit MP 800 anion exchangers was carried out using the Langmuir equation with constant values of $(7 \cdot 10^{-2} \pm 0.03)$ and (0.2 ± 0.02) L·mg^{-1}, the values of the maximum sorption capacity were (109 ± 5) and (74 ± 3) mg·g^{-1}, respectively. The value of the Freundlich constant during the treatment of the sorption isotherm of cobalt with anionite Axionit VPA G.2.4 was (9.6 ± 0.3) mg·g^{-1}. The kinetics of cobalt sorption by Axionit VPA G.2.4 and Indion 850 anionites was studied; the kinetic data of cobalt sorption with the highest value of the coefficient of determination R^2 are described using the pseudo-second order equation. Using mathematical processing of the kinetic data of cobalt sorption according to the Weber-Morris equation, the intradiffusion nature of sorption was determined. The analysis of the results of the influence of elements accompanying the complex processing of waste rhenium-containing superalloys – iron, aluminum and chromium – suggests that iron and chromium have a competitive effect on the sorption of cobalt due to the possibility of the formation of their anionic forms in hydrochloric acid solutions for processing secondary rhenium-containing raw materials.

Keywords: ion exchange; cobalt; nickel; rhenium-containing superalloys; complex processing; anion exchangers; kinetics; isotherm.

Сорбционное извлечение кобальта анионитами из растворов комплексной переработки отходов ренийсодержащих никелевых суперсплавов

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Аннотация: В работе оценена возможность сорбционного извлечения кобальта анионитами из солянокислых растворов в присутствии никеля. Наибольшее значение сорбционной емкости наблюдается у гелевого анионита с пиридиниевыми функциональными группами (Axionit VPA G.2.4) и макропористого анионита с функциональными группами вторичного и третичного амина (Indion 850). Обработка изотерм сорбции кобальта анионитами Indion 850 и Lewatit MP 800 осуществлена с использованием уравнения Ленгмюра со значениями констант $(7 \cdot 10^{-2} \pm 0,03)$ и $(0,2 \pm 0,02)$ л/мг, значения максимальной сорбционной емкости составили (109 ± 3) и (74 ± 2) мг/г, соответственно. Значение константы Фрейндлиха при обработке изотермы сорбции кобальта анионитом Axionit VPA G.2.4 составило $(9,6 \pm 0,3)$ мг/г. Изучена кинетика сорбции кобальта анионитами Axionit VPA G.2.4

и Indion 850, кинетические данные сорбции кобальта с наибольшим значением коэффициента детерминации R^2 описываются с применением уравнения псевдо-второго порядка. С помощью математической обработки кинетических данных сорбции кобальта по уравнению Вебера–Морриса определен внутри диффузионный характер сорбции. Анализ результатов влияния элементов, сопутствующих при комплексной переработке отходов ренийсодержащих суперсплавов – железа, алюминия и хрома, позволяет утверждать, что железо и хром оказывают конкурирующее влияние на сорбцию кобальта из-за возможности образования их анионных форм в солянокислых растворах переработки вторичного ренийсодержащего сырья.

Ключевые слова: кобальт; ионный обмен; никель; ренийсодержащие суперсплавы; комплексная переработка; аниониты; кинетика; изотерма.

1. Introduction

The ongoing global trend towards the transition from the fossil-based systems of energy production to renewable energy sources causes the need to store the energy produced, since renewable sources are characterized by their intermittent generation. Currently, lithium-ion batteries (LIBs) are most commonly used for energy storage. Cobalt is one of the key components for the production of rechargeable LIBs, and therefore the demand for it for the production of batteries has grown 26 times in the first two decades of the 21st century [1–4].

The world production of cobalt in 2022 amounted to ~200 thousand tons. About 73 % of the cobalt production falls on the Democratic Republic of the Congo, where it is a by-product of copper mining from stratiform deposits [5]. According to the USGS, for 2021, ~7.5 thousand tons of this metal was produced in the Russian Federation.

In 2020, about 57 % of global cobalt consumption was for the manufacture of lithium-ion batteries, while about 13 % of it was for the production of nickel-based alloys, including rhenium-containing ones. Other important applications of cobalt were catalysts [6], pigments [7], magnets [8], and special steels [9].

Increasing the limits of operating temperatures and heat resistance for structural materials of aircraft gas turbine parts made from heat-resistant rhenium-containing nickel alloys makes it possible to increase the fuel efficiency of engines and has a positive effect on the environmental friendliness of their use due to more complete combustion of fuel.

Waste from the treatment and operation of such alloys is a promising type of secondary rare-metal raw materials, since they include tungsten, tantalum, and rhenium – one of the most expensive elements, the content of the latter can reach 10 wt. %. Treatment of waste rhenium-containing superalloys consists in the recovery of rhenium using pyro- and hydrometallurgical methods. Complex treatment of lumpy waste of rhenium-containing superalloys often includes anodic dissolution with the transfer of rhenium and most of the non-ferrous metals into solution, extraction of rhenium and precipitation of nickel-cobalt concentrate in the form of hydroxides [10, 11], or cathodic reduction of nickel to obtain nickel concentrate after anodic dissolution of lumpy waste [12]. The closeness of the physicochemical properties of nickel and cobalt makes it difficult to recover these elements. Complex processing of rhenium-containing raw materials makes it possible to recover not only individual rhenium compounds, but also cobalt, which, in turn, can be returned to the production of alloys or used as a component of lithium-ion batteries.

Various hydrometallurgical methods are used to recover and separate cobalt and nickel. In [13], the possibility of extraction of nickel and cobalt from sulfuric acid solutions using (2,4,4-trimethylpentyl) phosphinic acid (Cyanex-272) and dialkylphosphinic acid INET-3 [14] was presented. The extraction of nickel and cobalt is also carried out from chloride media [15]. Sorption of cobalt from acidic media in the presence of nickel can be carried out using Lewatit TP-272 impregnate [16]. The tendency of cobalt to form anionic complexes in chloride media is used to extract it and separate it from nickel using membrane technologies [17].

This study aims to develop a method for absorbing cobalt with anion exchangers with enhanced capacitance characteristics from model solutions for the complex processing of rhenium-containing superalloys waste.

2. Materials and Methods

2.1. Materials and substances

The following anion exchangers were tested in the study: Axionit VPA G.2.4 (Axion – Rare and Precious Metals JSC, Russian Federation); Indion

850, 830, GS 400 (Indion, India); LewatitMonoPlus MP 800, K 6367, M 800 (Lanxess AG, Germany); Purolite A500U, A600 (Purolite Inc., Germany); Ambersep 920U (DuPont, USA); Tokem 845, 400 (TOKEM LLC); AMP; AM-2B.

Some characteristics of the used anion exchangers are presented in Table. 1.

To prepare a model solution, we used: $NiCl_2 \cdot 6H_2O$ (pure), $CoCl_2 \cdot 6H_2O$ (analytically pure, reagent grade), $FeCl_3 \cdot 6H_2O$ (pure), $CrCl_3 \cdot 6H_2O$ (pure), $AlCl_3 \cdot 6H_2O$ (CASno.7784-13-6), HCl (chemically pure). For analytical research, we used: $CH_3COONa \cdot 3H_2O$ (analytically pure, reagent grade), HNO_3 (chemically pure), nitroso-R-salt (pure).

2.2. Methodology for conducting experiments

Cobalt sorption experiments were carried out under static conditions. Sorbent weighed portions were placed in sealed flat-bottom flasks with a model solution with a known cobalt content at a phase ratio (sorbent weight : solution volume) in the range of 1 g : 5–50 mL, placed on a shaker heated to 70 °C, and stirred for 2 h, after whereupon the solution was separated from the anion exchanger and analyzed for cobalt content. The concentration of cobalt in the initial and equilibrium solutions was determined by the photometric method using the nitroso-R-salt. The concentration of elements in the eluate prepared during the sorption of cobalt from a real nickel-cobalt concentrate processing solution was determined by inductively coupled plasma mass spectrometry using an ICPMS-2030 mass spectrometer (Shimadzu, Japan).

Cobalt sorption isotherms of from a model hydrochloric acid solution (C_{HCl} − 4M) with a cobalt concentration of 5 g·L^{-1}, were prepared at a temperature of −70 °C and the phase ratio of the sorbent (g) : solution (mL): 1 : 5, 10, 15, 25, 30, 35, 50. Time contact was 2 hours.

Table 1. Properties of anion exchangers

Anion exchanger	Matrix type	Anion exchanger type	Dispersion of the effective size fraction, mm	Chloride-ion total exchange capacity, mg-eq·g^{-1}, not less than	Functional groups
Lewatit MP 800	Macroporous		0.6−0.7	1.0	Quaternary ammonium
Purolite A500U			0.3−1.2	1.2	Quaternary ammonium
Indion 830			0.315−1.25	0.8	Quaternary ammonium
Indion GS 400			0.3−1.2	−	Benzyldimethylamine
Ambersep 920U			0.6−0.8	1.25	Tertiaryamine
Lewatit M 800	Gel	Strong-base	0.65−0.75	1.2	Quaternary ammonium
Purolite A600			0.3−1.2	1.4	Quaternary ammonium
Axionit VPA G.2.4			0.8−2.0	4.0	Pyridinium
AMP			0.8−2.0	3.1	Benzylpyridinium
Lewatit K 6367			0.8−1.2	1.4	Quaternary ammonium
Tokem 845			0.55−0.65	1.2	Quaternary ammonium
AM-2B	Macroporous	Medium-base	0.8−2.5	3.3	Benzyldimethylamine and dibenzyldimethylammonium
Tokem 400	Gel		0.315−1.250	1.6	Tertiaryamine
Indion 850	Macroporous Gel	Weak-base	0.32−1.25	1.5	Tertiary amine and quaternary ammonium

Experiments to study the kinetic characteristics were carried out under static conditions using the method of a limited volume of solutions at a temperature of 70 °C and a phase ratio of sorbent (g) : solution (mL) – 1 : 250. This phase ratio was chosen so that the change in the solution volume during sampling did not exceed 5 %.
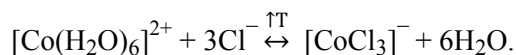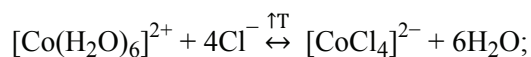
3. Results and Discussion

To determine the sorption capacity of anion exchangers for cobalt, we used a model hydrochloric acid solution $C_{Co} - 5$ g·L^{-1}, $C_{HCl} - 4$ M. The phase ratio was 1 g : 15 mL of the solution, the temperature was 70 °C.

Based on the data presented in Fig. 1, the sorption capacity of anion exchangers for cobalt increased in the series: Tokem 400 < Purolite A600 < < Lewatit Mono Plus M 800 < Ambersep 920U < < Tokem 845 < Indion GS 400 < Indion 830 < < Lewatit K 6367 < AMP < AM-2B < Purolite A500U < Lewatit Mono Plus MP 800 < Indion 850 < < Axionit VPA G.2.4.

The highest sorption capacity for cobalt was observed when using gel anion exchangers Axionit VPA G.2.4, Lewatit Mono Plus MP 800, and macroporous anion exchanger Indion 850. These anion exchangers were used for further study of sorption characteristics.

Cobalt was able to form anionic coordination compounds with the chloride ion by the reactions:

$$[Co(H_2O)_6]^{2+} + 4Cl^- \overset{\uparrow T}{\leftrightarrow} [CoCl_4]^{2-} + 6H_2O;$$

$$[Co(H_2O)_6]^{2+} + 3Cl^- \overset{\uparrow T}{\leftrightarrow} [CoCl_3]^- + 6H_2O.$$

The factor that directly affects the formation of complexes of this type is the concentration of the chloride ion in the solution.

The sorption capacity of the selected anion exchangers for cobalt was studied at a concentration of hydrochloric acid in the initial model solution in the range of 2–5 M.

The study of the influence of the concentration of hydrochloric acid (Fig. 2) showed that the highest sorption capacity of anion exchangers was observed at a concentration of hydrochloric acid equal to 4M.

A further increase in the concentration of hydrochloric acid did not lead to an increase in the sorption capacity, which, apparently, can be associated with the predominance of the formation of the $[CoCl_4]^{2-}$, which interacts with two functional groups of the sorbent.

Cobalt sorption isotherms with Axionit VPA G.2.4, Lewatit Mono Plus MP 800, and Indion 850 anion exchangers are shown in Fig. 3.

The mathematical description of cobalt sorption isotherms with anion exchangers was carried out using the Freundlich (Fig. 4a) and Langmuir (Fig. 4b) models.
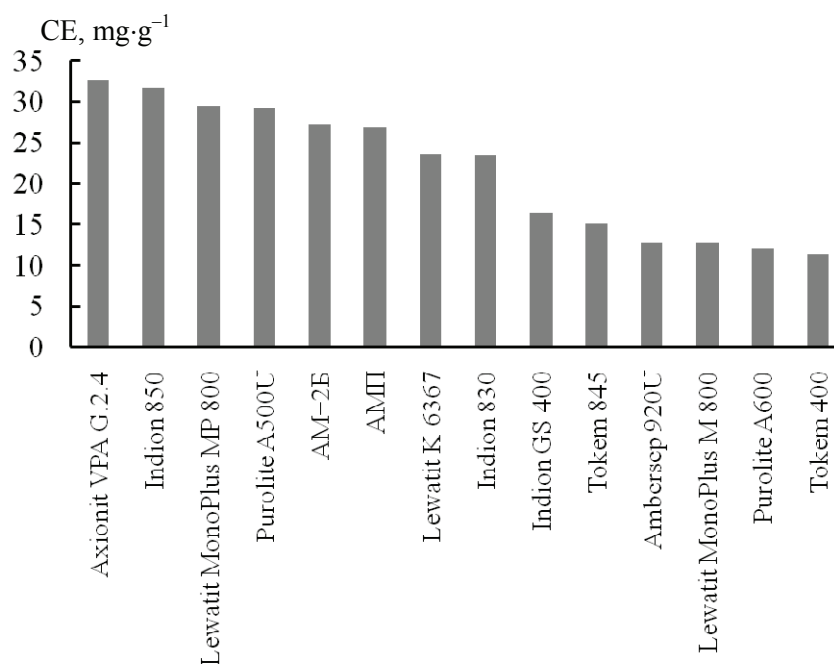

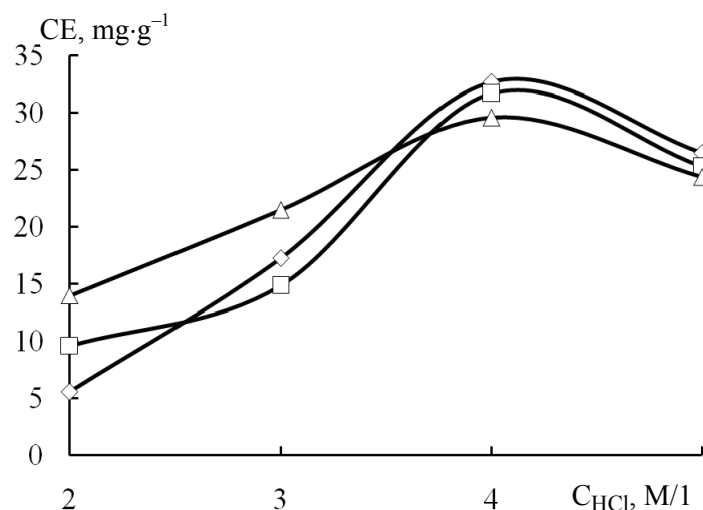**Fig. 1.** Sorption capacity of anion exchangers for cobalt from a model hydrochloric acid solution (4 M HCl)

Fig. 2. Influence of hydrochloric acid concentration on the sorption capacity of anion exchangers for cobalt:
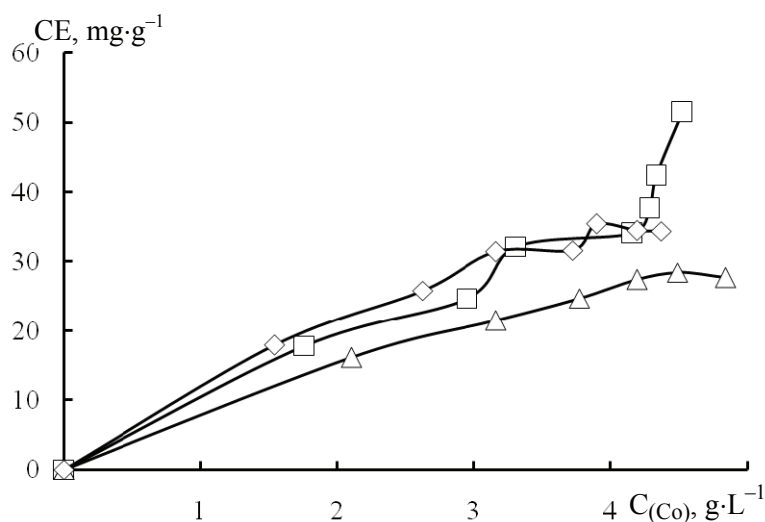◊ – Indion 850; △ – Lewatit MP 800; □ – Axionit VPA G2.4

Fig. 3. Cobalt sorption isotherms on anion exchangers from hydrochloric acid solutions (4 M):
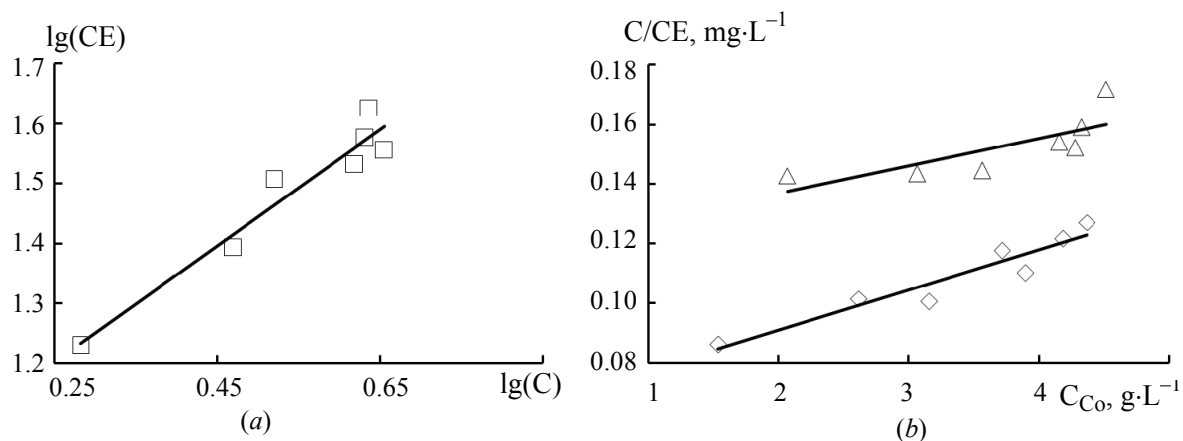◊ – Indion 850; △ – Lewatit MP 800; □ – Axionit VPA G2.4

Fig. 4. Anamorphoses of cobalt sorption isotherms with anion exchangers: *a* – Freindlich model; *b* – Langmuir model;
◊ – Indion 850; △ – Lewatit MP 800; □ – Axionit VPA G2.4

Table 2. Equilibrium characteristics of cobalt sorption according
to the Freindlich and Langmuir models

Anion exchanger	Freundlich constant, mg·g^{-1}	n	R^2	Langmuir constant, L·mg^{-1}	CE$_\infty$, mg·g^{-1}	R^2
Axionit VPA G.2.4	9.6 ± 0.3	0.51	0.9511		–	
Lewatit MP 800				0.077 ± 0.03	109 ± 5	0.6264
Indion 850		–		0.206 ± 0.02	74 ± 3	0.9065

The processing of cobalt sorption isotherms with Axionit VPA G.2.4, Lewatit MP 800, and Indion 850 anion exchangers according to the Langmuir and Freundlich equations made it possible to determine the equilibrium constants with an estimate of the determination coefficients and the maximum sorption capacity of anion exchangers using the Langmuir model (Table 2).

The cobalt sorption isotherms prepared with Axionit VPA G.2.4 and Indion 850 anion exchangers with a high determination coefficient were described by the Freundlich and Langmuir models, respectively (Table 2). In this case, only the cobalt sorption isotherm on the Axionit VPA G.2.4 anion exchanger had a concave character and was suitable for linearization in Freundlich coordinates, the value of the parameter n was 0.51, the value of the Freundlich constant was (9.6 ± 0.3) mg·g^{-1}.

As can be seen from Table 2, the Langmuir constant was ~ 2.7 times higher during the sorption of cobalt by the anion exchanger Indion 850 than the Langmuir constant during the sorption of cobalt by the anion exchanger Lewatit MP 800.

The cobalt sorption kinetics was studied using anion exchangers that showed the best capacitive properties – Axionit VPA G.2.4 and Indion 850. The sorption kinetic data allow make it possible to measure the process rate and how quickly the equilibrium state is reached, but they can be useful for evaluating the mechanism sorption [18].
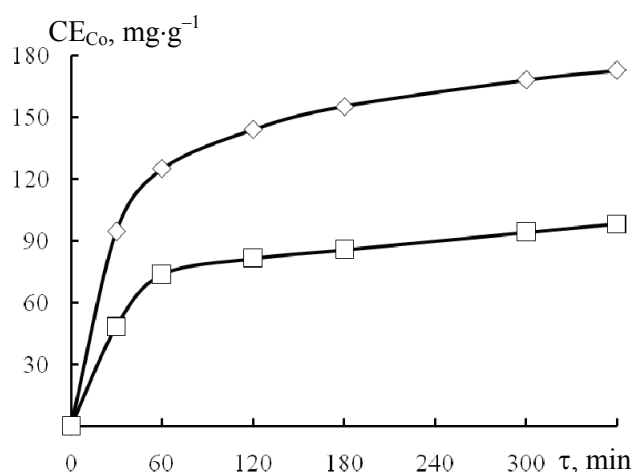
The integral kinetic curves of cobalt sorption are shown in Fig. 5

The kinetic data of cobalt sorption were processed using mathematical models:

Pseudo-first order chemical kinetics model [19]

$$\log(Q_e - Q_\tau) = \log Q_e - \frac{k_1\,\tau}{2.303}, \qquad (1)$$

where Q_e, Q_τ is equilibrium sorption capacity and sorption capacity at time τ, mg·g^{-1}; k_1, is the pseudo first order rate constant, min^{-1}.

Fig. 5. Integral kinetic curves of cobalt sorption by anion exchangers from hydrochloric acid solutions (4 M): ◊ – Indion 850; □ – Axionit VPA G2.4

Pseudo-second order chemical kinetics model [19]

$$\frac{\tau}{Q_\tau} = \frac{1}{k_2 Q_e^2} + \frac{1}{Q_e}\tau, \qquad (2)$$

where k_2 is the pseudo second order rate constant, g·(mg·min)$^{-1}$.

Elovich chemical kinetics model [20]

$$Q_\tau = \frac{1}{\beta}\ln(1 + a \times \beta \times \tau), \qquad (3)$$

where β is the Elovich constant; a is the initial rate of the sorption process.

Internal diffusion model (*Weber–Morris*) [21]

$$Q_\tau = k_p \times \tau^{0.5} + B, \qquad (4)$$

where k_p is the internal diffusion rate constant, mg·g^{-1}×min$^{1/2}$; B is constant related to the boundary layer thickness, mg·g^{-1}.

The results of processing kinetic data using mathematical models are presented in Table 3.

Table 3. Rate constants and coefficients of determination
of linearized direct cobalt sorption by mathematical models

Pseudo-first order model		Pseudo-second order model		Internal diffusion model		Elovich model	
$k_1 \cdot 10^{-3}$, min^{-1}	R^2	$k_2 \cdot 10^3$, mg·g^{-1}·min^{-1}	R^2	k_{id}, mg·g^{-1}·min$^{-0.5}$	R^2	β, g·mg^{-1}	R^2
Indion 850							
2.8	0.9093	119.8	0.9963	42.0	0.8479	0.041	0.9294
Axionit VPA G.2.4							
3.3	0.9637	185.3	0.9985	78.6	0.9125	0.057	0.9879

The highest values of the determination coefficients in the linearization of experimental data were observed when using the pseudo-second order model (Table 3). This fact may indicate that a second-order chemical reaction makes a certain contribution to the mechanism of cobalt sorption.

The intradiffusion model equation, in contrast to the pseudo-second order model or pseudo-first order model, makes it possible to identify the sorption mechanism. If the straight line linearized according to the Weber-Morris model does not pass through the origin of coordinates, which is observed when studying the kinetics of cobalt sorption with Indion 850 and Axionit VPA G.2.4 anion exchangers, then the sorption process is limited by internal diffusion.

Chromium, aluminum, iron, etc. are present in the waste of rhenium-containing superalloys. Some of these elements in chloride solutions for the processing of waste alloys may be present in the form of anions. To study the effect of these elements on the sorption of cobalt, we used hydrochloric acid solutions (4 M) of the composition, g·L^{-1}: Ni : Co : Fe = 40 : 9 : 4, Ni : Co : Al = 40 : 9 : 4, Ni : Co : Cr = 40 : 9 : 7, Ni : Co : Fe : Al : Cr = 40 : 9 : 4 : 4 : 7.

The results of cobalt sorption by Indion 850 and Axionit VPA G.2.4 anion exchangers from solutions of complex composition at a temperature of 70 °C and a phase ratio of 1 : 25 (g : mL) are shown in Fig. 6.

The analysis of the data from Fig. 6 suggests that the greatest influence on the sorption capacity of Axionit VPA G.2.4 anion exchanger is exerted by iron and chromium impurities, while chromium impurities affect the sorption capacity of Indion 850 anion exchangers. This may be due to the formation of iron ($FeCl_4^{2-}$) and chromium (CrO_4^{2-} (chromate-) and/or $Cr_2O_7^{2-}$ dichromate) anions and their competing effect on cobalt sorption.

To determine the possibility of iron sorption, the iron sorption capacity of the selected anion exchangers was also determined (Fig. 7).
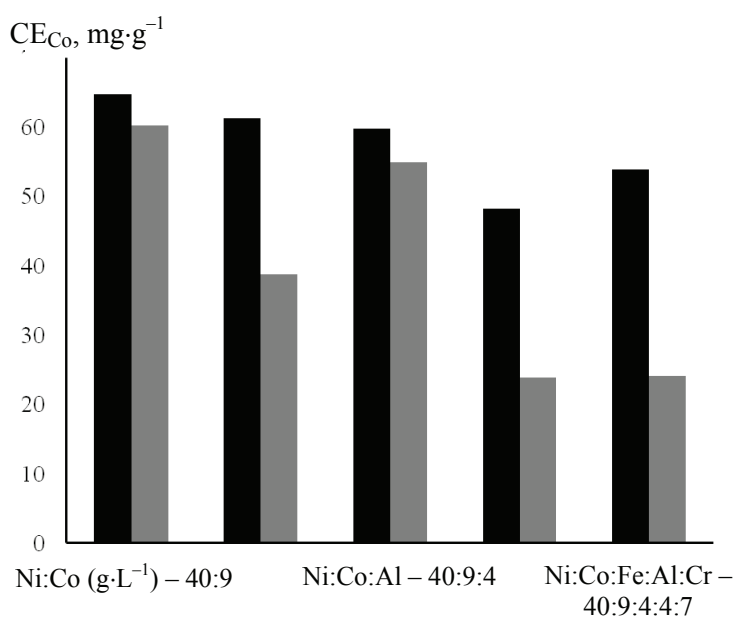
Fig. 6. Dependence of the sorption capacity of anion exchangers Axionit VPA G.2.4 (■)
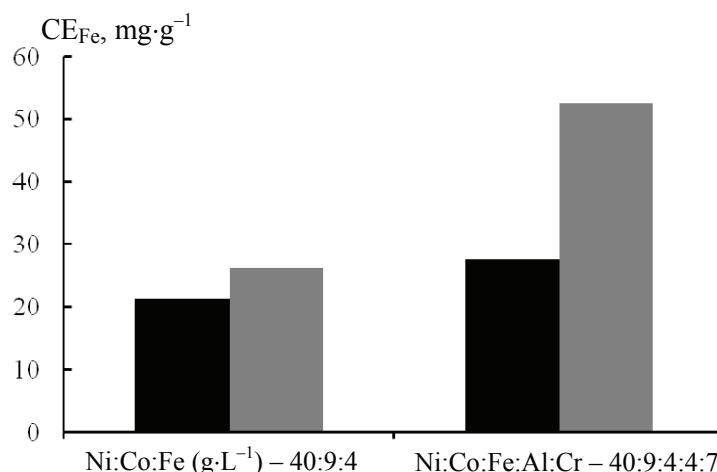and Indion 850 (▪) for cobalt on the concentration of impurities

Fig. 7. Sorption capacity for iron anion exchangers: Axionit VPA G.2.4 (■) and Indion 850 (■)

The data in Fig. 7 shows that the Indion 850 anion exchanger has a lower sorption capacity for iron than the Axionit VPA G.2.4 anion exchanger, the value of the sorption capacity of which for iron is 2 times higher than for cobalt when sorption from a solution containing, in addition to nickel and cobalt, Fe, Al and Cr. This may be due to the greater affinity of the functional groups of the tertiary amine and quaternary ammonium of the Indion 850 anion exchanger for cobalt ions than for iron ions.

The method of cobalt sorption by anion exchangers was tested from a real chloride solution (C_{HCl} − 4M) of nickel-cobalt concentrate leaching, which was formed during the complex processing of wastes of rhenium-containing superalloys, using the Indion 850 weakly basic anion exchanger. In the concentrate leaching solution, the Ni:Co ratio ($g \cdot L^{-1}$) is 40 : 5.5; in addition to the main elements, Al, Fe, and Cr were also present in the solution. The cobalt sorption from the indicated solution was done at a phase ratio at the stage of sorption sorbent : solution = 1 (g) : 20 (mL), subsequent washing of the sorbent and desorption of cobalt with water made it possible to prepare 10 mL of eluate with a concentration ratio of Ni : Co = 1.1 ($g \cdot L^{-1}$) : 5.6 ($g \cdot L^{-1}$). The Fe and Cr content in the eluate was 2.0 and 0.25 $g \cdot L^{-1}$, respectively, the Al concentration was below the detection limit.

The separation coefficient Co/Ni, calculated as the ratio of the distribution coefficients of cobalt and nickel, was 35 for one sorption cycle. Since the Fe content, an impurity that limits the quality of cobalt commercial products, was quite high in the eluate, its additional purification was necessary, for example, sorption using a cation exchanger after adjusting its acidity with an HCl solution.

4. Conclusion

Under static conditions, the cobalt sorption by anion exchangers of various types from hydrochloric acid solutions at elevated temperatures was studied. It has been established that the highest value of the capacity of anion exchangers for cobalt is observed from hydrochloric acid solutions with a concentration of 4M HCl and a temperature of 70 °C, increasing in the series: Tokem 400 < Purolite A600 < < LewatitMonoPlus M 800 < Ambersep 920U < < Tokem 850 < Indion GS 400 < Indion 830 < < Lewatit K 6367 < AMP < AM-2B < Purolite A500U < Lewatit Mono Plus MP 800 < Indion 850 < < Axionit VPA G.2.4.

The value of the Langmuir constant during the cobalt sorption by the Indion 850 anion exchanger was (0.2 ± 0.02) $L \cdot mg^{-1}$, the value of the maximum sorption capacity was (74 ± 3) $mg \cdot g^{-1}$. The processing of the cobalt sorption isotherm according to the Freundlich model with the Axionit VPA G.2.4 anion exchanger made it possible to determine the value of the Freundlich constant, which was − (9.6 ± 0.3) $mg \cdot g^{-1}$, as well as the value of the parameter n − 0.51.

The analysis of the kinetic data processing of cobalt sorption by anion exchangers has shown that the highest value of the determination coefficient is observed when the kinetic data of cobalt sorption are linearized using the internal diffusion equation and a pseudo-second order, which may indicate that the process of cobalt sorption by Indion 850 and Axionit VPA G.2.4 anion exchangers is limited in intradiffusion region with some contribution from the chemical reaction.

Tested on real leaching solutions of nickel-cobalt concentrate formed during the complex

processing of rhenium-containing superalloy wastes, the method of cobalt sorption using the Indion 850 anion exchanger has a high potential for use in the separation of nickel and cobalt from hydrochloric acid solutions after the recovery of rhenium.

5. Funding

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6. Conflict of interests

The authors declare no conflict of interest.

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