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Research article

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## Hydrometallurgical recovery of nickel from oxidized ores

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**Abstract:** A significant portion of the world's reserves of Ni-containing raw materials (40–66 %) is concentrated in oxidized nickel ores. One of the alternatives to the high-cost pyrometallurgical and ammonia-carbonate methods for processing such ores could be the chlorammonium recovery of nickel from relatively low-grade ores. The halide-ammonia decomposition and recovery technology of nickel from oxidized nickel ores, supplemented by a sorption process, is less stage-intensive and simpler in practical implementation. Nickel adsorption recovery is feasible using carbon sorbents that exhibit high chemical stability, withstand high-temperature exposure, and strong acidic treatment. Sorbents were obtained through steam-gas activation of extracted carbonizates from fossil coals. The sorption capacity for Ni(II) ions was studied, and the patterns and characteristic parameters of the process on carbon sorbents were identified using adsorption isotherms while varying experimental conditions. The experimental results were processed using the Freundlich and Langmuir equations. The sorbents have several distinctive features determined by their predominant microporous structure and multifunctional surface with active complex-forming atomic groups, characteristic of ampholytes with cation- and anion-exchange properties. The adsorption process is described by a pseudo-first-order equation with rate constants ranging from 0.204 to 0.287 s<sup>-1</sup>. For the adsorption recovery of Ni(II), a scheme with two adsorbers and a pseudo-fluidized sorbent bed is proposed. Nickel desorption and sorbent regeneration were carried out with a 2.3 % sulfuric acid solution, desorbing 95 to 98 % of nickel. Standard chemical machinery and equipment are recommended for these processes.

**Keywords:** oxidized ores, carbon sorbents, nickel recovery.**For citation:** Dudarev V.I., Dudareva G.N., Yakovleva A.A. Hydrometallurgical recovery of nickel from oxidized ores. *Izvestiya. Non-Ferrous Metallurgy*. 2024;30(3):25–33. <https://doi.org/10.17073/0021-3438-2024-3-25-33>

## Гидрометаллургическое извлечение никеля из окисленных руд

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**Аннотация:** Большая часть мировых запасов Ni-содержащего сырья (40–66 %) сосредоточена в окисленных никелевых рудах. Одной из альтернативных высокочрезвычайно затратным пирометаллургическому и аммиачно-карбонатному методам переработки таких руд может быть хлораммонийное извлечение никеля из относительно бедных по содержанию металла руд. Технология галогенидно-аммиачного разложения и извлечения никеля из окисленных никелевых руд, дополненная сорбционным процессом, является менее длительной по стадийности и проще в практическом исполнении. Адсорбционное извлечение никеля возможно углеродными сорбентами, обладающими высокой химической устойчивостью, выдерживающими высокотемпературное воздействие и сильноокислотную обработку. Сорбенты получены путем парогазовой активации выделенных карбонизатов иско-

паемых углей. Изучена сорбционная способность ионов Ni(II), выявлены закономерности и характеристические параметры процесса на углеродных сорбентах с помощью изотерм адсорбции при варьировании условий проведения экспериментов. Обработку экспериментальных результатов выполняли с использованием уравнений Фрейндлиха и Ленгмюра. Сорбенты имеют ряд особенностей, определяемых преобладающей микропористой структурой и полифункциональной поверхностью с активными комплексообразующими группировками атомов, характерными для амфолитов с катионо- и анионообменными свойствами. Процесс адсорбции описан уравнением псевдопервого порядка с константами скорости от 0,204 до 0,287 с<sup>-1</sup>. Для адсорбционного извлечения Ni(II) предложена схема с двумя адсорберами и псевдооживленным слоем сорбента. Десорбция никеля и регенерация сорбента проведены 2,3 %-ным раствором серной кислоты. При этом десорбируется от 95 до 98 % никеля. В процессах рекомендуются стандартные химические машины и аппараты.

**Ключевые слова:** окисленные руды, углеродные сорбенты, извлечение никеля.

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## Introduction

In the global reserves of Ni-containing raw materials, a significant portion (from 40 to 66 %) is composed of oxidized nickel ores (ONO) [1–3], in which the average Ni content is only 0.7–1.2 %. Therefore, their targeted processing by pyrometallurgical methods is high-cost and, as a rule, unprofitable [2–4]. The combined scheme for processing ONO developed by scientists at the Ural Federal University named after the first President of Russia B.N. Yeltsin (Yekaterinburg) [5] (thermochemical treatment — aqueous leaching — hydroxide precipitation) allows obtaining a nickel concentrate, which enhances the economic feasibility of applying pyrometallurgical technology. However, the scheme involves thermochemical treatment of the entire ore mass, including barren rock, as well as requiring preliminary treatment of ONO with significant volumes of hydrochloric acid and roasting to obtain insoluble forms of interfering components. All this significantly complicates the process of nickel recovery.

Researchers at Tomsk Polytechnic University [6; 7] propose using a single reagent, ammonium chloride, for the initial processing of ONO. The main ore mass, including SiO<sub>2</sub> oxides (51 %) and Al<sub>2</sub>O<sub>3</sub> (5 %), does not interact with the reagent, while oxides of other associated metals start interacting at a temperature of 473 K. Nickel oxide transitions to a water-soluble nickel chloride through an intermediate product NiCl<sub>2</sub>·nNH<sub>4</sub>Cl at  $T = 600$  K. Subsequent aqueous leaching of the thermally treated ore and ammonia precipitation of hydroxides allow sequential separation of ONO into individual target components [6]. Incorporating a sorption process into such innovative technology can significantly enhance the efficiency of nickel recovery from ONO [8; 9].

The adsorption recovery of nickel is possible with sorbents from various raw materials, including car-

bon sorbents, which have a developed porous structure with a specific surface area of more than 500 m<sup>2</sup>/g [10–21]. Carbon sorbents of the AD type, synthesized at INRTU, possess high chemical stability, withstand high-temperature exposure, and resist strong acid treatment [22; 23]. They have several features defined by their microporous structure and multifunctional surface with active complex-forming atomic groups, characteristic of ampholytes with cation- and anion-exchange properties [24].

Sorbents obtained through steam-gas activation of extracted carbonizates from fossil coals are dark granules of irregular shape with an average particle size of 1 to 2 mm, a specific surface area of 550 m<sup>2</sup>/g, mechanical abrasion resistance of 82 %, a total pore volume (by water) of 0.61 cm<sup>3</sup>/g, iodine adsorption activity of 84 %, and a bulk density of 560 g/dm<sup>3</sup> [23].

The aim of this work was to study the adsorption characteristics of the AD carbon sorbent and to develop recommendations for the hydrometallurgical recovery of nickel from oxidized ores.

## Research methodology

The analysis of the sorption capacity for nickel ions, the identification of regularities, and characteristic parameters of the process were carried out using adsorption isotherms while varying the experimental conditions in both static and dynamic modes. The structure, porosity, and surface compound properties of the sorbents were studied using various physicochemical methods with modern instrumentation [24–26].

The AD sorbent possesses amphoteric ion-exchange properties. The ion-exchange capacity was determined by reverse titration methods using 0.1 N solu-

tions of HCl and NaOH. The cation-exchange capacity for  $H^+$  was 0.92 meq/g, and the anion-exchange capacity for  $OH^-$  was 7.52 meq/g. These data indicate that the sorbent surface contains functional groups with exchangeable  $H^+$  and  $OH^-$  ions that can be replaced by metal ions. The optimization of nickel sorption recovery was carried out by varying the acidity and temperature of the solutions, as well as considering the nature of the adsorption on the porous material surface.

The effect of medium acidity on sorption capacity was evaluated under static conditions. A 0.5 g sample of the adsorbent was placed in 100 cm<sup>3</sup> of a solution prepared from reagent-grade nickel chloride. The reaction flask volume was 250 cm<sup>3</sup>, and the initial metal concentration in the solution was 0.51 mmol/dm<sup>3</sup>. The pH value in the range of 3 to 11 was adjusted using ammonia-acetate buffer solutions. A magnetic stirrer was used for mixing until adsorption equilibrium was established.

Quantitative analysis of nickel was performed by spectrophotometric methods using analytical reagents such as dimethylglyoxime or N-acyl-acetohydrazone [26]. Periodically, additional control of nickel ion concentration in solutions and the metal content in the sorbent after thermo-acid decomposition of the dried sorbent was carried out by atomic absorption analysis according to standard methodology [27]. The adsorption amount ( $A$ , mmol/g) was calculated using the formula

$$A = (C_0 - C_p)V/m, \quad (1)$$

where  $C_0$  and  $C_p$  are the initial and equilibrium concentrations of the adsorbate, respectively, mmol/dm<sup>3</sup>;  $m$  is the mass of the adsorbent, g;  $V$  is the volume of the working solution, dm<sup>3</sup>.

Studies at elevated temperatures were conducted in thermostated cells.

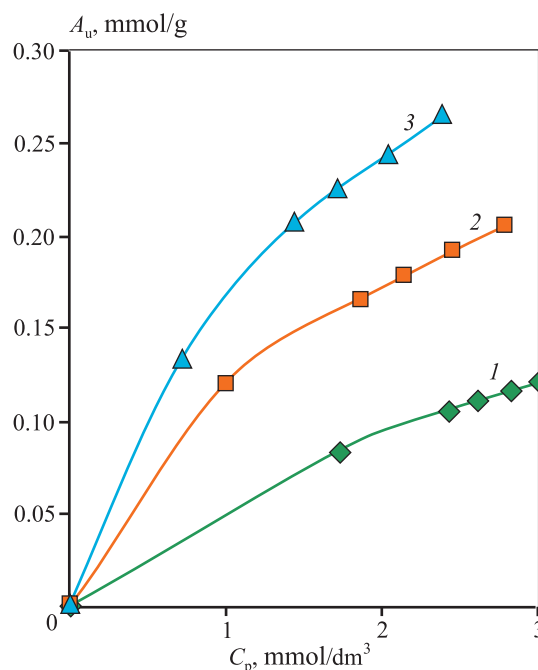
## Results and discussion

The nature of the sorption interaction, observed at temperatures of 298, 318, and 338 K, is shown by the adsorption isotherms of Ni(II) ions on the carbon sorbent (Fig. 1).

The processing of experimental results in the low nickel concentration range in solutions was performed using the classical Freundlich equation:

$$A = K_F C_p^{1/n}. \quad (2)$$

After performing arithmetic transformations and



**Fig. 1.** Adsorption isotherms of Ni (II) ions at temperatures 298 K (1), 318 K (2), and 338 K (3)

$A_u$  – concentration of metal on the sorbent

**Рис. 1.** Изотермы адсорбции ионов Ni(II) при температурах 298 К (1), 318 К (2) и 338 К (3)

$A_u$  – концентрация металла на сорбенте

plotting the linear dependence of the logarithmic form of the equation:

$$\lg A = \lg K_F + 1/n \lg C_p \quad (3)$$

both constant parameters  $K_F$  and  $n$  were determined graphically. The constant  $K_F$  represents the millimolar adsorption coefficient, as it corresponds to the adsorption value at an adsorbate concentration  $C = 1$  mmol/dm<sup>3</sup>. The exponent  $1/n$  is a proper fraction and generally characterizes the degree of the adsorption isotherm's approach to the abscissa axis. From the data presented in Table 1, it follows that with increasing temperature, both constants of the Freundlich equation (2)

**Table 1. Constants of the Freundlich equation ( $R^2 = 0.92$ )**

Таблица 1. Константы уравнения Фрейндлиха ( $R^2 = 0,92$ )

Constants	Temperature, K		
	298	318	338
$K_F$	0.19	0.25	0.29
$n$	1.73	1.84	1.94

increase. The value of the coefficient  $K_F$  suggests that the adsorption of Ni(II) ions in the initial period occurs with high efficiency.

To describe the process upon reaching the adsorption limit, the Langmuir equation was used. In its linear form, it is represented as a straight-line equation:

$$1/A = 1/A_{\infty} + 1/A_{\infty}K_p C, \quad (4)$$

where  $C$  is the concentration of the metal in the solution, mmol/dm<sup>3</sup>;  $A_{\infty}$  are the ultimate adsorption values, mmol/g;  $K_p$  is the adsorption equilibrium constant.

By plotting the dependence in the coordinates  $1/A = f(1/C)$ , the constants  $A_{\infty}$  and  $K_p$  in the Langmuir equation were determined graphically. The results are presented in Table 2.

It is evident that temperature affects both the ultimate adsorption value and the adsorption equilibrium constants, which also increase with temperature.

Analysis of the results shows that the process of nickel adsorption on the carbon sorbent is not purely physical adsorption. The standard thermodynamic parameter, Gibbs free energy ( $\Delta G$ ), was calculated using the classical chemical affinity equation:

$$\Delta G = -RT \ln K_p, \quad (5)$$

where  $R = 8.314 \text{ J/mol}\cdot\text{K}$  is the universal gas constant;  $T$  is the process temperature in K.

From the data presented in Table 2, it is evident that with increasing temperature, the likelihood of the spontaneous adsorption process increases.

For the graphical determination of the adsorption enthalpy of Ni(II), the isobar equation in its differential form was used:

$$\ln K_p = \frac{\Delta H}{RT}. \quad (6)$$

As a result, its value was  $-8.96 \text{ kJ/mol}$  (see Table 2).

The evaluation of kinetic regularities was conducted using the classical method of selecting the axes of kinetic equations. The linear dependencies  $\ln C = f(t)$  at all temperatures (Fig. 2) confirm that the adsorption process is described by a pseudo-first-order equation. The values of the rate constants ( $k_c$ ) obtained in this way are presented in Table 2.

Contrary to the Van't Hoff rule for homogeneous chemical reactions, the adsorption rate constant grows significantly slower, indicating the complexity of the process mechanism. This is also confirmed by the acti-

Table 2. **Thermodynamic sorption constants ( $R^2 = 0.96$ )**

Таблица 2. Термодинамические константы адсорбции ( $R^2 = 0,96$ )

Parameter	Temperature, K		
	298	318	338
$A_{\infty}, 10^{-4} \text{ mmol/g}$	1.01	1.53	1.66
$K_p$	394	416	524
$k_c, \text{ s}^{-1}$	0.204	0.229	0.287
$\Delta G, \text{ kJ/mol}$	-20.5	-22.1	-24.7
$\Delta H, \text{ kJ/mol}$		-8.96	
$E_a, \text{ kJ/mol}$		7.1	

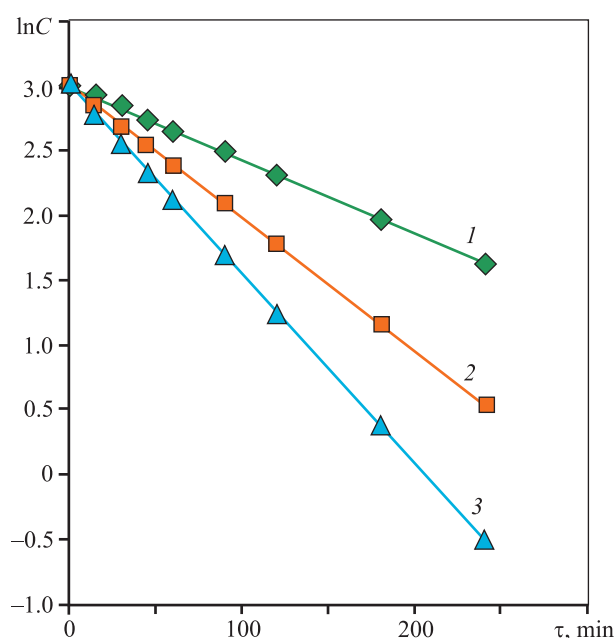


Fig. 2. Kinetic dependences of Ni(II) ion sorption at temperatures 298 K (1), 318 K (2), and 338 K (3)

Рис. 2. Кинетические зависимости сорбции ионов Ni(II) от температуры 298 К (1), 318 К (2) и 338 К (3)

vation energy ( $E_a$ ), graphically determined by linearizing the Arrhenius equation:

$$\ln k = \ln k_0 \frac{E_a}{RT}. \quad (7)$$

The experimentally determined activation energy for the adsorption process was  $7.1 \text{ kJ/mol}$ , indicating the process occurs in the diffusion region [28].

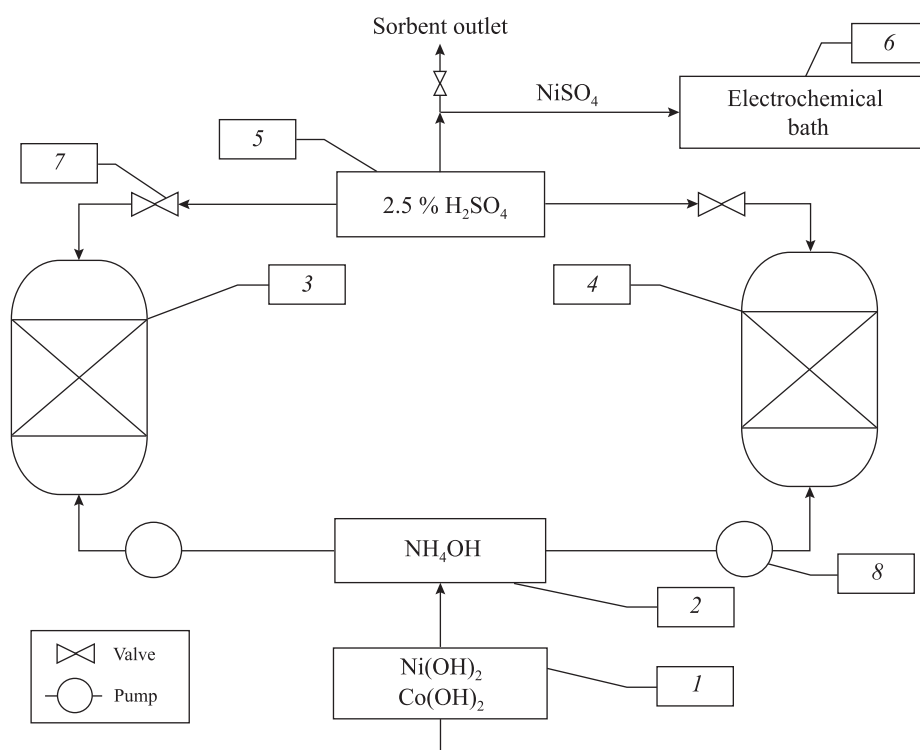
To address the question of the limiting stage of adsorption, experiments with process interruption in dynamic mode were conducted for a specified time. The results showed that in all cases, a 24-hour inter-

ruption of the adsorption flow resulted in a decreased concentration of the adsorbed metal in the solution exiting the column. The discontinuity in the adsorption exit curves suggests that the limiting stage of the adsorption kinetics of Ni(II) ions on the carbon adsorbent under the studied conditions is diffusion within the adsorbent granules, i.e., adsorption occurs under “gel” kinetics, and its rate is hindered by intradiffusion processes. The possible sorption mechanism can be considered as the diffusion process of complex Ni(II) ions, specifically the hydroxo-pentaammin cation  $[\text{Ni}(\text{NH}_3)_5(\text{OH})]^+$ , into the granules and their interaction with functionally active groups on the sorbent surface through ion exchange.

Studies using IR spectroscopy methods showed that the observed results of metal ion sorption involve the participation of double bonds of compound fragments located on the carbon surface. Changes were observed in the overtones of  $=\text{CH}_2$ ,  $=\text{C}-\text{NH}_2$ , and  $=\text{C}-\text{OH}$  bonds, as well as in the conjugated fragments of  $=\text{C}=\text{C}=\text{C}=\text{}$  and  $\text{R}-\text{C}=\text{O}$ ,  $\text{R}-\text{OH}$  bonds. This indicates the participation of  $\pi$ -electrons of these molecular fragments or their exchange parts in the reactions. They likely act as electron pair donors, with

the free  $d$ -orbitals of the adsorbed metal serving as acceptors in the corresponding complexes. This conclusion confirms the nature of metal ion interaction with the adsorbent surface. The appearance of bands in its spectrum after metal ion sorption at  $\nu = 574, 604$ , and  $836 \text{ cm}^{-1}$ , corresponding to the characteristic deformation vibration bands of  $\text{Me}-\text{O}-\text{C}$  and  $\text{Me}-\text{O}-\text{Me}$  bonds, indicates the presence of such bonds in the investigated compounds.

The analysis of results also showed that during adsorption, with increasing temperature, the sorbent's capacity for nickel increases. The adsorption of Ni(II) ions in this case does not correspond to the classical behavior of metal ions: for many of them, adsorption results in an exothermic effect [29]. It is likely that the high stability of associated nickel compounds: amine complexes ( $K_u = 5,3 \cdot 10^8$ ), aqua-amine complexes ( $K_u = 4,2 \cdot 10^6$ ), and hydroxo-amine complexes ( $K_u = 5,8 \cdot 10^6$ ) [26], predetermines the need for additional energy to break them down before sorption. This energy may be greater than the energy of the exothermic ion exchange reaction when Ni(II) is fixed on the sorbent surface, making the overall sorption process appear endothermic.



**Fig. 3.** Schematic diagram of the apparatus chain for nickel sorption extraction

1, 2, 5, 6 – solution tanks; 3, 4 – adsorbents; 7 – valves; 8 – pumps

**Рис. 3.** Схема цепи аппаратов для сорбционного извлечения никеля

1, 2, 5, 6 – емкости для растворов; 3, 4 – адсорберы; 7 – вентили; 8 – насосы

In the chlorammonium technology for nickel recovery from oxidized ores, the active reagent used for the technological transformation of metal oxides is regenerated and returned to the head of the process for the decomposition of a new batch of ONO [6]. The selectivity of ammonium chloride allows for the initial stage to free most of the ore. During ore opening, 75 % of nickel is extracted as hydroxide [7]. It seems reasonable to introduce a stage of adsorption recovery of nickel in the process of separating the technological solution, where precipitation and separation of nickel hydroxide will occur at  $\text{pH} = 8.0\div 8.5$  [8]. For the adsorption recovery of Ni(II), a scheme with two adsorbers with a pseudo-fluidized sorbent bed is proposed (Fig. 3).

During the adsorption studies, insights into the process mechanism were obtained, enabling the development of practical recommendations for implementing the proposed scheme. Before initiating the process, it is necessary to adjust the acidity of the technological solution to optimal values, which is achieved by adding an ammonium hydroxide solution. The solution is pumped from the bottom into the adsorber for the adsorption process. Compared to other designs, adsorbers with a pseudo-fluidized sorbent bed offer advantages such as an increased phase contact area with the same loading volume and a longer phase contact time.

For the adsorption recovery of metal, a cylindrical column design with a conical bottom and distribution grids inside the apparatus was chosen. To organize a continuous recovery process, two adsorbers are used: after the sorbent in the first unit becomes saturated, it switches to reloading while the second unit switches to adsorption. After reloading, the sorbent is directed to a desorber equipped with a stirrer, where a diluted (1 : 20, or 2.3 %) sulfuric acid solution is supplied. It was found that using  $\text{H}_2\text{SO}_4$  at this concentration as the eluent desorbs 95 to 98 % of the nickel. The saturated Ni(II) solution after desorption is proposed for use in the electrolytic recovery of the metal.

When operating the sorption columns, it should be noted that the degree of nickel recovery from solutions decreases by 3–5 % after each cycle. The adsorbers should be switched before complete saturation of the load, as at times close to full saturation of the adsorbent, the concentration of metals in the solution approaches equilibrium, and the adsorption rate realistically decreases. The estimated total operating time of one adsorber before reloading the carbon is 648 hours. It is advisable to use only standard chemical machinery and apparatus in the processes.

## Conclusion

The chlorammonium hydrometallurgical technology for nickel recovery from oxidized ores is based on the solid-phase chlorination of oxidized nickel ores with ammonium chloride at  $T = 473 \text{ K}$ , followed by aqueous leaching of soluble nickel chlorides and other valuable components. It is advisable to include a nickel adsorption recovery process in the above-mentioned technological scheme at the stage of obtaining ammonia solutions ( $\text{Ni}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ). It is most likely that after precipitation, the metals are present in the technological solution as complex amines.

The study showed that adsorption interaction in the “metal-containing solution — carbon sorbent” system proceeds quite intensively: the rate constants range from 0.204 до 0.287  $\text{s}^{-1}$ . The activation energy value ( $E_a = 7.1 \text{ kJ/mol}$ ) indicates that the adsorption process occurs in the diffusion region. The change in Gibbs free energy ( $\Delta H = -8.96 \text{ kJ/mol}$ ) suggests that the likelihood of the spontaneous adsorption process increases with temperature.

For the adsorption recovery of Ni(II), a scheme with two adsorbers with a pseudo-fluidized sorbent bed and bottom supply of a weakly alkaline technological solution is proposed. Nickel desorption is carried out with a 2.3 % sulfuric acid solution, desorbing 95 to 98 % of the nickel.

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**G.N. Dudareva** — performed calculations, tested samples, and prepared the text of the article.

**A.A. Yakovleva** — provided scientific guidance, revised the text, and refined the conclusions.

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