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Sorption properties of a copolymer based on divinyl sulphide and 4-vinylpyridine

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Abstract: Organic polymers with N, S-functional groups are widely used as effective materials for the adsorption of transition metal ions. The adsorption of noble metals by copolymer of divinyl sulphide with 4-vinylpyridine has been studied in solutions of mineral acids. The copolymer under study has displayed high values of static sorption capacities, amounting to 1200, 340, 1040, 520 mg/g in Au (III), Ag (I), Pt (IV) and Pd (II), respectively. A comparative study of the Langmuir, Freundlich and Dubinin-Radushkevich adsorption models has been carried out to describe the experimental adsorption isotherms of noble metal ions on the surface of the copolymer under investigation. Constants and parameters of the considered adsorption models have been identified. Analysis of the values of the coefficients of determination R^2 indicates that the adsorption of noble metal ions from sulphuric acid solutions and Ag from nitric acid solutions is most adequately described within the Langmuir model. The obtained results demonstrate that the adsorption of noble metal ions occurs on an energetically heterogeneous surface and represents a monomolecular process.

Keywords: ion exchange resins, copolymer of divinylsulphide and 4-vinylpyridine, noble metals, Langmuir, Freundlich, Dubinin-Radushkevich adsorption models

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Сорбционные свойства сополимера на основе дивинилсульфида и 4-винилпиридина

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Резюме: Органические полимеры с N,S-функциональными группами находят широкое применение в качестве эффективных адсорбционных материалов для ионов переходных металлов. Исследована адсорбция благородных металлов сополимером дивинилсульфида с 4-винилпиридином в растворах минеральных кислот. Показано, что исследуемый сополимер характеризуется высокими значениями статических сорбционных емкостей, которые для Au(III), Ag(I), Pt(IV) и Pd(II) достигают 1200, 340, 1040, 520 мг/г, соответственно. Проведено сравнительное исследование адсорбционных моделей Ленгмюра, Фрейндлиха и Дубинина – Радускевича для описания экспериментальных изотерм адсорбции ионов благородных металлов на поверхности исследуемого сополимера. Определены константы и параметры рассмотренных моделей адсорбции. Анализ значений коэффициентов де-

терминации R^2 свидетельствует, что адсорбцию ионов благородных металлов из сернокислых растворов и Ag из азотнокислых растворов наиболее адекватно описывает модель Ленгмюра. Полученные результаты указывают на то, что адсорбция ионов благородных металлов происходит на энергетически неоднородной поверхности и является мономолекулярным процессом.

Ключевые слова: ионообменные смолы; сополимер дивинилсульфида и 4-винилпиридина; благородные металлы; модели адсорбции Ленгмюра, Фрейндлиха, Дубинина – Радушкевича

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INTRODUCTION

Divinylsulphide (DVS) is one of the classes of monomers having the potential to create ion exchange resins due to the presence of sulphide sulphur atoms ("soft" easily-polarised centres of basicity and complexation) within the framework of the polymer matrix, which endows the ion exchanger with a highly-selective complexing affinity with respect to transition metals. The combined reaction of DVS with 4-vinylpyridine (4-VP) enables the synthesis of an ion exchanger containing a "pyridine" nitrogen atom and sulphide sulphur in the polymer chain, which ensures high sorption activity of the copolymerisation products with respect to noble metal ions.

The propensity of noble metal ions to form stable complexes with N- and S-donor ligands is commonly used to create sorption materials [1, 2]. Thus, functional resins containing guanylthiourea and 2-mercapto-1-methylimidazole exhibited high selectivity towards gold(I) and silver(I) from ammonium solutions. However, they did not absorb ammonia complexes of copper(II). The kinetics of gold(I) sorption on these resins was improved by immobilising the ligands of N, N-dimethylethanolamine, which were responsible for increasing the hydrophilicity of the polymeric material. The degree of gold sorption was 99.5% [3].

A series of eighteen copolymers of 4-vinylpyridine and 4-methyl-4'-vinylpyridine used for the sorption of Ag(I), Au(III), Pd(II) and Pt(IV) ions and having various crosslinking agents in aqueous solutions were investigated. Chelating polymers have a significant sorption selectivity for Au(III) over Ag(I) and for Pd(II) over Pt(IV) [4].

A new chelating resin was synthesised from a macroreticular polystyrene-divinylbenzene copolymer incorporating the β -hydroxydithiocoric acid functional group. The study of adsorption capacity showed that the synthesised resin was highly selective for Ag(I), Hg(II), Au(III) and Pt(IV) in a strongly acidic aqueous solution [5].

A series of highly effective sorbents of noble metals were obtained by sol-gel synthesis based on organosilicone N,S-functional monomers. A distinc-

tive feature of such polymers, along with outstanding complexing activity, is high chemical and thermal stability [6–8]. In general, N- and S-containing ion-exchange and complexing sorbents dominate in the vast group of polymeric materials proposed for the extraction of noble metal ions.

The aim of this work was to study the adsorption of noble metals by ion exchangers on the basis of the copolymer of divinylsulphide and 4-vinylpyridine.

EXPERIMENTAL PART

The copolymers of divinylsulphide and 4-vinylpyridine were obtained by free-radical copolymerisation in glass ampoule reactors with azobisisobutyronitrile at 60 °C [9, 10].

Insoluble copolymers with quantitative yields for all ratios of the monomer mixture were obtained by free-radical copolymerisation of divinylsulphide with vinylpyridine. The compositions of the copolymers, calculated from nitrogen and sulphur contents, are satisfactorily in agreement with each other. The number of DVS units in the copolymer decreases with a decrease of its concentration in the initial mixture. With the increase of the content of divinylsulphide in the initial mixture, a decrease in the yield of the target product is observed.

Since previous experiments had shown that a divinylsulphide-4-vinylpyridine copolymer with a composition ratio of 38,77:61,23 mol.%, containing 8,78% of nitrogen and 12,67% of sulphur, has a higher sorption activity, we studied the sorption properties of this copolymer sample.

The initial solutions of noble metal ions with a concentration of 10 mg/ml were prepared from chemically pure solutions of $H_2PtCl_6 \cdot 6H_2O$, $PdCl_2 \cdot 2H_2O$, $HAuCl_4 \cdot 4H_2O$, $Ag(NO_3)$.

The sorption characteristics of divinylsulphide-4-vinylpyridine copolymers were studied in a static mode with respect to the acid complexes of platinum(IV), palladium(II), gold(III) in solutions of hydrochloric and sulphuric acid. The adsorption of silver(I) ions was investigated in nitric acid solutions at the temperature of 298 K.

To construct isotherms, the constant mass

(0,02g) bottle-point method was used. The solutions under study were prepared with a volume of 20 ml. The mass ratio of liquid and solid phases in the adsorption process was 1:1000. Static sorption capacity (A , mg/g,) was calculated by the following formula:

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

where C_0 and C_e are the initial and equilibrium concentrations of metal ion in solution, respectively, mg/dm³; V is the volume of solution, dm³; m is the mass of adsorbent, g.

The content of gold(III) and silver(I) in the solution was monitored using the atomic absorption method on the C-115M1 device (Research and Production Enterprise "Ukrospribor"), that of platinum and palladium was monitored using the photometric method by the reaction of formation of their tin-chloride complexes [11]. To measure the optical density, a «Promecolab PE-5400V» spectrophotometer was used.

RESULTS AND DISCUSSION

The study of the adsorption of silver ions on the divinylsulphide-4-vinylpyridine copolymer in solutions of nitric acid was carried out, where the metal

has the form of the Ag^+ cation. The extraction of Pt, Pd and Au ions was studied in hydrochloric and sulphuric acid solutions, in which these metals are present in the form of acid complexes of different composition [1, 2].

For Pt, Pd, Au chloro-complexes, with an increase in the concentration of acids, a slight decrease in the degree of extraction is observed with the exception of the gold tetrachloride complex in sulphuric acid (Fig. 1).

A decrease in the degree of extraction of chloro-complexes of platinum and gold is more manifested in hydrochloric acid solution (see Fig.1). The given type of the dependence is evidence for the anion exchange at the pyridine nitrogen atom and the effect of competing acid anions, considering their size and mobility. Coordination interaction of metal ions by nitrogen and sulphur atoms, which is more characteristic of the cationic form of silver, cannot be ruled out.

The time needed to establish sorption equilibrium in metal extraction from 1 M acid solutions is 60 minutes at the half-sorption time, $\tau_{1/2}$: for the silver cation, it is 15 min; for Pt and Pd anionic complexes, it is 30 min; for Au chloro-complexes, it is 5–10 min.

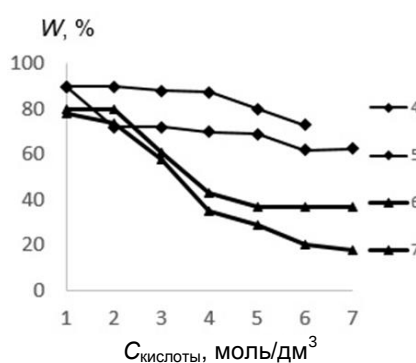
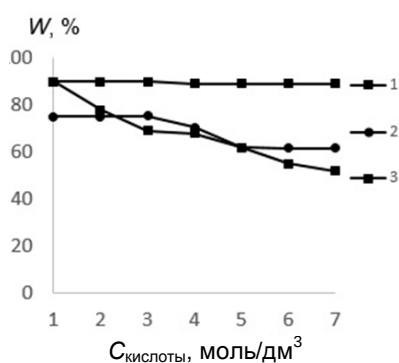


Fig. 1. Influence of the nature and concentration of acids on the extraction degree of ions: Au(III) in H_2SO_4 (1) and HCl (3); Ag(I) in HNO_3 (2); Pd(II) in H_2SO_4 (4) and HCl (5); Pt(IV) in H_2SO_4 (6) and HCl (7)

Рис. 1. Влияние природы и концентрации кислот на степень извлечения ионов: Au(III) в H_2SO_4 (1) и HCl (3); Ag(I) в HNO_3 (2); Pd(II) в H_2SO_4 (4) и HCl (5); Pt(IV) в H_2SO_4 (6) и HCl (7)

Since the distribution of metal ions in liquid and solid phases in the equilibrium state is characterised by adsorption isotherms in 1 M acid solutions (Fig. 2), we were able to calculate the values of the sorption capacity and metal phase distribution ratios (Table 1).

Experimental isotherms of noble metal ion adsorption by divinylsulphide-4-vinylpyridine copolymer are interpreted by several adsorption models: the Langmuir model for limited adsorption on the monolayer, the Freundlich model of unrestricted adsorption and the Dubinin-Radushkevich model for adsorption on porous microreticular sorbents [12].

The Langmuir monomolecular adsorption equation is most often applied to the solid/liquid inter-

face:

$$A = A_{\infty} \cdot (K \cdot C_e / (1 + K C_e)), \quad (2)$$

where A is the current value of adsorption, mmol/g; A_{∞} is the maximum value of adsorption, mmol/g; K is the adsorption equilibrium constant; C_e is the equilibrium concentration of metal ion, mmol/dm³.

The experimental data were processed using the Langmuir equation, reduced to linear form:

$$1/A = (1/A_{\infty}) + ((1/A_{\infty} \cdot K) \cdot (1/C_e)). \quad (3)$$

The isotherms of noble metal ion adsorption by the considered ion exchanger belong to the monomolecular adsorption isotherms (Fig. 3).

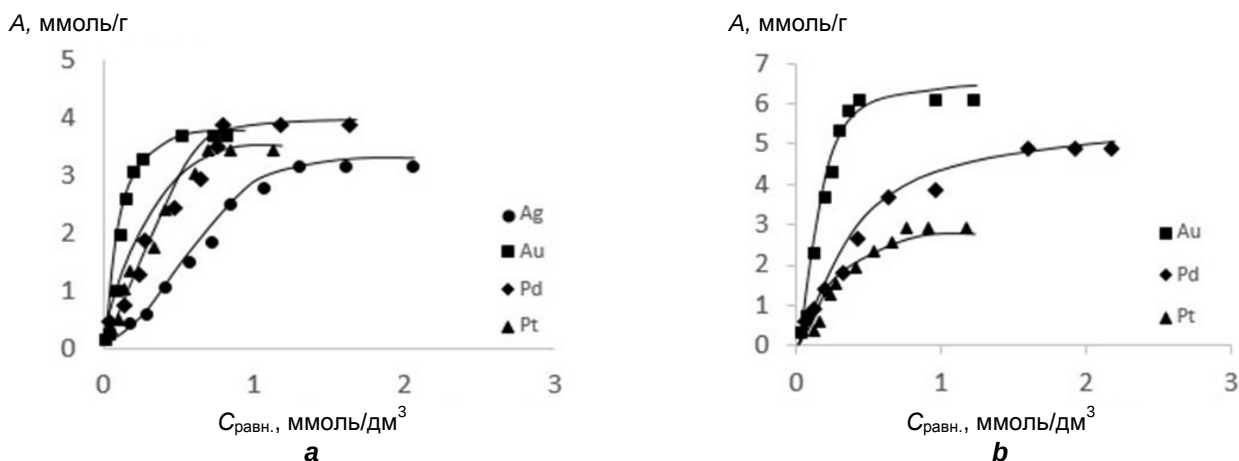


Fig. 2. Adsorption isotherms: of Ag(I) ions in 1 M HNO₃, Au(III), Pt(IV), Pd(II) in 1 M HCl (a) and in 1 M H₂SO₄ (b) ($m = 10$ mg; $V = 20$ ml; $\tau = 2$ h)

Рис. 2. Изотермы адсорбции: ионов Ag(I) в 1 M HNO₃, Au(III), Pt(IV), Pd(II) в 1 M HCl (a) и в 1 M H₂SO₄ (b) ($m = 10$ мг; $V = 20$ мл; $\tau = 2$ ч)

Table 1

Values of sorption capacity (A) and metal distribution ratios (D) in 1 M acid solutions

Таблица 1

Значения сорбционной емкости (A) и коэффициентов распределения металлов (D) в 1 M растворах кислот

Me	A, mg/g	D, cm ³ /g	Me	A, mg/g	D, cm ³ /g
Ag (HNO ₃)	330	$3,8 \cdot 10^5$	Au (H ₂ SO ₄)	1200	$1,6 \cdot 10^4$
Au (HCl)	730	$1,5 \cdot 10^5$	Pd (H ₂ SO ₄)	520	$12,1 \cdot 10^5$
Pd (HCl)	410	$1,3 \cdot 10^5$	Pt (H ₂ SO ₄)	570	$4,0 \cdot 10^4$
Pt (HCl)	670	$1,1 \cdot 10^5$	-	-	-

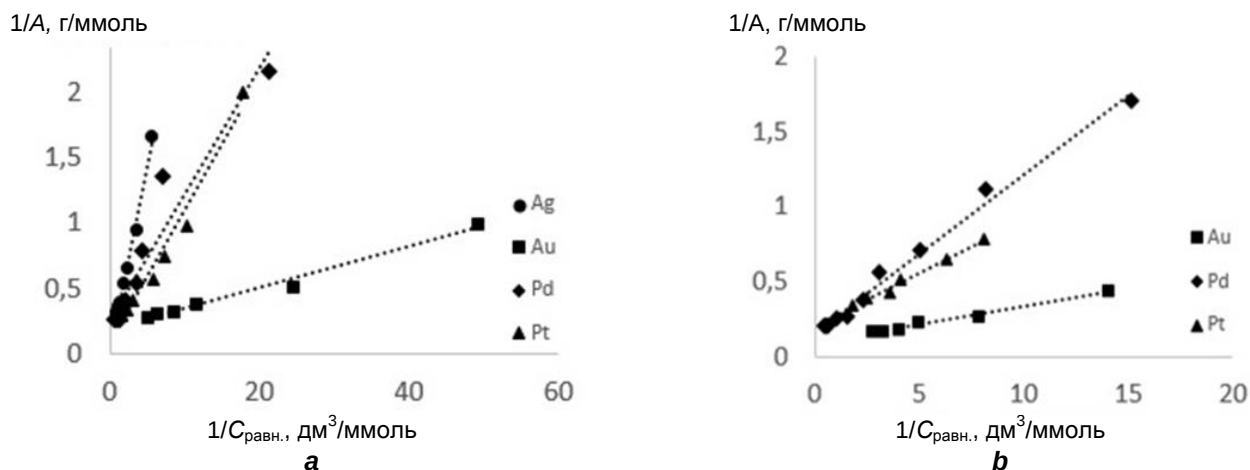


Fig. 3. Adsorption isotherms in the coordinates of the linear Langmuir equation's form: Adsorption isotherms: Ag(I) ions in 1 M HNO₃, Au(III), Pt(IV), Pd(II) in 1 M HCl (a) and in 1 M H₂SO₄ (b) at 298 K

Рис. 3. Изотермы адсорбции в координатах линейной формы уравнения Ленгмюра: Ag(I) в 1 M HNO₃, Au(III), Pt(IV), Pd(II) в 1 M HCl (a) и в 1 M H₂SO₄ (b) при 298 K

From the graphical linear dependencies shown in Fig. 3, the limiting value of adsorption A_{∞} was determined from the value of the straight line

segment intersecting the ordinate axis, and the adsorption equilibrium constant K was determined from the slope of straight line, the values of which

are presented in tab. 2.

The determination coefficient values given in Table 2 indicate that the adsorption of noble metals from sulphate solutions ($R^2=0,991$) and silver from nitric acid solutions ($R^2=0,995$) is most adequately described by the Langmuir adsorption model.

To the medium loading area of the heterogeneous surface of adsorbents comprising the considered copolymers with localised functional groups, the Freundlich empirical equation is commonly applied. Freundlich's equation in the logarithmic form is:

$$\lg A = \lg K_f + 1/n \cdot \lg C_e, \quad (4)$$

where A is the adsorption value, mmol/g; C_e is

equilibrium concentration of metal ions, mmol/dm³; K_f and n are constants.

Freundlich's theory suggests that adsorption on each type of adsorption centres complies with the Langmuir equation. According to the Freundlich model, the adsorption centres have different energy values, with the active adsorption centres having maximum energy being loaded first. Due to the constants K_f and n , the adsorption capacity towards different adsorbates can be compared. When the concentration of metal ions in the solution is 1 mmol/dm³, the adsorption value of these ions is equal to a constant K_f (molar adsorption ratio); the parameter n indicates the intensity of the adsorbent-adsorbate interaction (Table 3).

Table 2

Parameters of the Langmuir adsorption model

Таблица 2

Параметры модели адсорбции Ленгмюра

Me	A_∞ , mmol/g	K	R^2	Me	A_∞ , mmol/g	K	R^2
Ag (HNO ₃)	4,762	0,742	0,955	Au (H ₂ SO ₄)	1200	4,000	0,991
Au (HCl)	5,405	11,563	0,983	Pd (H ₂ SO ₄)	520	1,481	0,991
Pd (HCl)	4,184	2,464	0,939	Pt (H ₂ SO ₄)	570	2,887	0,991
Pt (HCl)	5,208	1,901	0,971	-	-	-	-

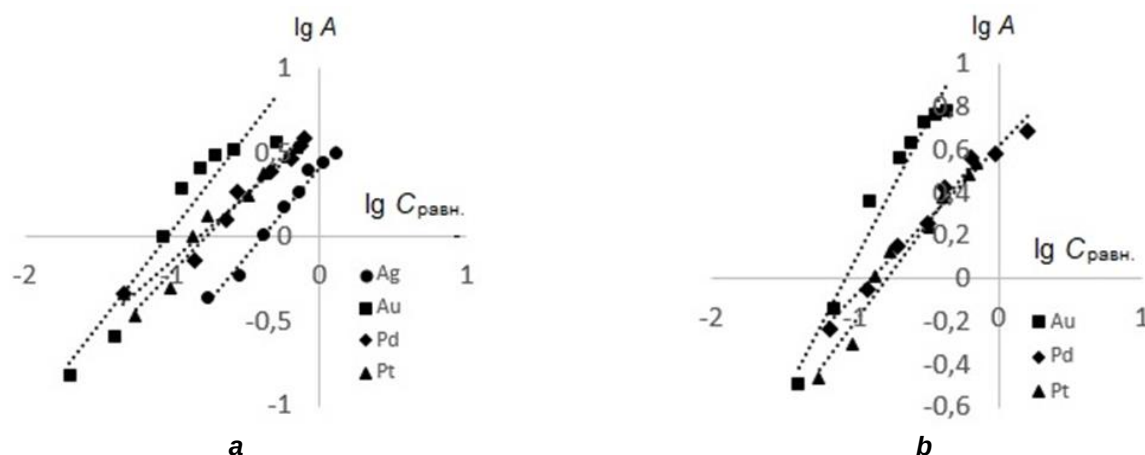


Fig. 4. Adsorption isotherms in the coordinates of the linear Freundlich equation form: Adsorption isotherms: Ag(I) ions in 1 M HNO₃, Au(III), Pt(IV), Pd(II) in 1 M HCl (a) and in 1 M H₂SO₄ (b) at 298 K

Рис. 4. Изотермы адсорбции в координатах линейной формы уравнения Фрейндлиха: Ag(I) в 1 М ННО₃, Au(III), Pt(IV), Pd(II) в 1 М НСl (а) и в 1 М Н₂SO₄ (б) при 298 К

Table 3

Parameters of the Freundlich adsorption model

Таблица 3

Параметры модели адсорбции Фрейндлиха

Me	K_f	n	R^2	Me	K_f	n	R^2
Ag (HNO ₃)	2,612	0,942	0,986	Au (H ₂ SO ₄)	23,442	0,781	0,967
Au (HCl)	14,723	0,881	0,908	Pd (H ₂ SO ₄)	4,150	1,435	0,975
Pd (HCl)	4,295	1,294	0,969	Pt (H ₂ SO ₄)	4,507	0,949	0,931
Pt (HCl)	2,553	1,104	0,966	-	-	-	-

The data in Table 3 demonstrate that the maximum value of the molar ratio is achieved through the adsorption of Au ions from sulphate and hydrochloric acid solutions, which agrees with the data in Table 1. According to the intensity of interaction with the ion exchanger under consideration, metal ions form the following series: Pd(II) > Pt(IV) > Ag(I) > Au(III). The values of the determination coefficients indicate that the adsorption of silver ions from nitric acid solutions can be described by the Freundlich adsorption model.

The equilibrium data of the adsorption process were processed using the Dubinin-Radushkevich isotherm model:

$$A = A_m \exp(-k\varepsilon^2), \quad (5)$$

where k is a constant (mol^2/kJ^2) associated with the energy of adsorption; ε is the Polanyi potential (kJ/mol), reflecting the isothermal transfer of one mole of metal ions from the equilibrium solution to the adsorbent surface and determined from the

expression $\varepsilon = RT \cdot \ln(1 + 1/C)$ where R is the universal gas constant, kJ/mol ; T is absolute temperature, K.

Based on the Dubinin-Radushkevich equation in logarithmic form

$$\ln A = \ln A_m - k\varepsilon^2 \quad (6)$$

the dependences $\ln A = f(\varepsilon^2)$ (Fig. 5) are plotted.

The constants k and A_m (Table 4) were determined from the slope of straight lines and the line segment on the axis of ordinates.

The Dubinin-Radushkevich model, developed to describe the adsorption of gas on microporous solids based on the theory of adsorption potential, is used when considering the adsorption of both complex organic molecules [13] and inorganic ions [14] on solid support. The Dubinin-Radushkevich model is considered to be a more general model whose assumption is not based on a homogeneous surface or a constant potential of adsorption [15], which coincides with the idea of energetic heterogeneity of synthetic sorbents with chemically active groups localised on the surface.

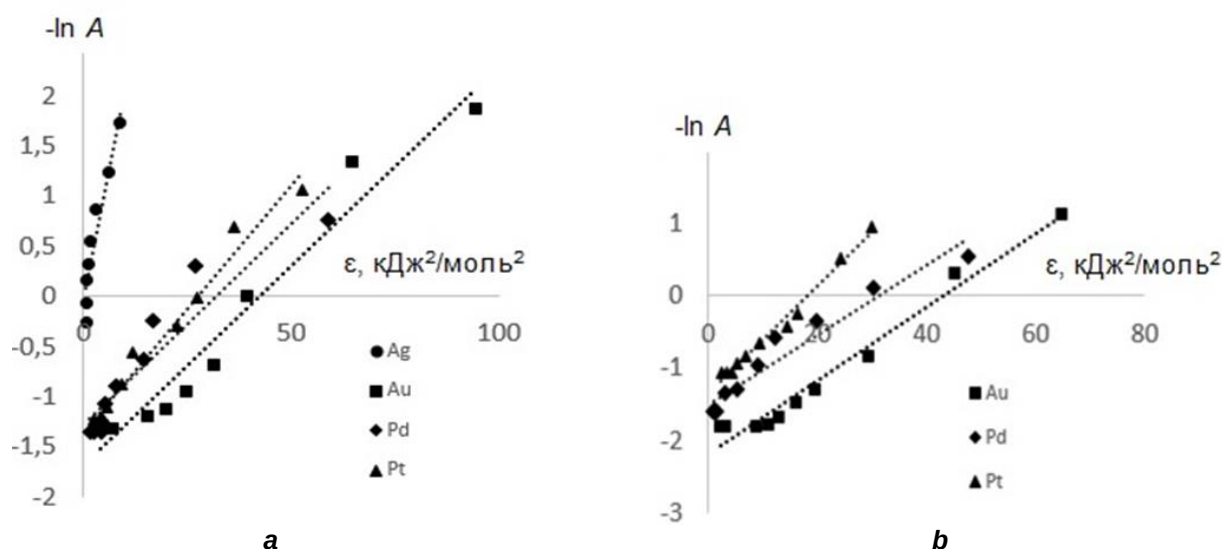


Fig. 5. Adsorption isotherms in the coordinates of the linear Dubinin-Radushkevich equation form: Adsorption isotherms: Ag(I) ions in 1 M HNO₃, Au(III), Pt(IV), Pd(II) in 1 M HCl (a) and in 1 M H₂SO₄ (b) at 298 K

Рис. 5. Изотермы адсорбции в координатах линейной формы уравнения Дубинина – Радушкевича: Ag(I) в 1 М ННО₃, Au(III), Pt(IV), Pd(II) в 1 М НСl (a) и в 1 М Н₂SO₄ (b) при 298 К

Table 4

Parameters of the Dubinin-Radushkevich adsorption model

Таблица 4

Параметры модели адсорбции Дубинина – Радушкевича

Me	A_m , mmol/g	k , mole ² /kJ ²	R^2	Me	A_m , mmol/g	k , mole ² /kJ ²	R^2
Ag (HNO ₃)	1,012	0,214	0,901	Au (H ₂ SO ₄)	10,431	0,055	0,990
Au (HCl)	6,018	0,042	0,961	Pd (H ₂ SO ₄)	4,477	0,048	0,942
Pd (HCl)	3,888	0,031	0,928	Pt (H ₂ SO ₄)	3,884	0,075	0,987
Pt (HCl)	3,849	0,049	0,992	-	-	-	-

Analysing the values of the determination coefficients in hydrochloric acid solutions of all the three considered adsorption models, we can conclude that the Dubinin-Radushkevich model is adequate under the adsorption of Pt ions from hydrochloric acid solutions ($R^2=0,992$).

In general, the divinylsulphide-4-vinylpyridine copolymers display high values of static sorption capacities amounting to 1200, 340, 1040, 520 mg/g for Au(III), Ag(I), Pt(IV) and Pd(II), respectively. It is essential that under the sorption of noble metal ions from rich or poor solutions, the value of the exchange capacity of the copolymers remains constant. The high distribution coefficient values (see Table 1) demonstrate the efficiency of sorption and the possibility of group extraction of noble metals.

The adequacy of the adsorption of noble metal ions from sulphuric acid solutions and silver from nitric acid solutions by the divinylsulphide-4-vinylpyridine copolymer of the Langmuir model enables us to characterise the process under study as a monomolecular adsorption on the energetically heterogeneous surface of a porous micro-

reticular sorbent; this result is in full conformity with the structural characteristics of this type of copolymers.

CONCLUSIONS

1. The divinylsulphide-4-vinylpyridine copolymer exhibits high static sorption capacities amounting to 1200, 340, 1040, 520 mg/g for Au(III), Ag(I), Pt(IV) and Pd(II), respectively.

2. The adsorption of noble metal ions from sulphuric acid solutions and silver from nitric acid solutions by the divinylsulphide-4-vinylpyridine copolymer of the Langmuir model is most adequately described within the Langmuir adsorption model, making it possible to characterise the process under study as a monomolecular adsorption on the energetically heterogeneous surface of the porous microreticular sorbent, which is in full conformity with the structural characteristics of this type of copolymers.

3. The adsorption of Pt ions from hydrochloric acid solutions is most adequately described within the Dubinin-Radushkevich adsorption model.

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Contribution

Elena G. Filatova, Nina S. Shaglaeva, Yuriy N. Pozhidaev, Denis B. Baginov, Liubov M. Stanevich carried out the experimental work, on the basis of the results summarized the material and wrote the manuscript. Elena G. Filatova, Nina S. Shaglaeva, Yuriy N. Pozhidaev, Denis B. Baginov, Liubov M. Stanevich have equal author's rights and bear equal responsibility for plagiarism.

Conflict of interests

The authors declare no conflict of interests regarding the publication of this article.

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