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## Microspherical Carriers and Adsorbents for the Processes in Corrosive Media

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### Abstract

As the result of the aerodynamic separation of non-magnetic cenosphere fraction, we obtained and characterized narrow homogeneous fractions differing in chemical composition, bulk density (0.195–0.396 g/cm<sup>3</sup>), average diameter (133–153 μm), thickness (3.1–7.6 μm) and the porosity of globule shell. Regularities in the relationship between the composition and morphology of the globules were established. After processing the product of aerodynamic separation by a reagent based on hydrofluoric acid we obtained a microspherical carrier with available internal volume and pore size ranging within 1–5 μm. Basing on this, we obtained a microspherical adsorbent with an active component triisobutyl phosphine sulphide for selective palladium extraction within a wide range of concentrations (3.6–360 mg/L) from corrosive media.

**Key words:** cenospheres, microspherical carrier, adsorbent

### INTRODUCTION

Traditionally, researchers use various inorganic and organic materials such as silica gel [1–4], aluminium oxide of various modifications [2, 5–9], kieselguhr [2], copolymers of styrene and divinylbenzene [10], fibrous and granulated cellulose [11], polyacrylonitrile [12] and others as carriers of catalysts and sorbents. For using in corrosive environments (pulp, acidic technological solutions, liquid radioactive waste), the carriers are charged with additional requirements, such as a high mechanical strength, acid resistance, radiation resistance, which restricts applying the traditional media.

As promising carriers of sorption active components for the processes in corrosive media, one could use hollow aluminosilicate microspheres from power station produced ash such as cenospheres [13–15], those are characterized by the presence of internal cavities, ther-

mal stability, resistance with respect to acids, high strength and the porosity of regular glassy crystalline envelope, and thereby they favourably differ from traditionally offered carriers.

The aim of this work consisted in obtaining microspherical carriers with perforated shell, adjustable thickness and porosity, as well as a selective sorbent based on them for the processes in corrosive media.

### EXPERIMENTAL

#### *Obtaining microspherical carriers*

In developing advanced functional materials with predictable properties based on cenospheres, including carriers and adsorbents for processes in corrosive media, one need a detailed study of the physicochemical properties of raw materials used, intermediate and final

products at every stage of their preparation. In this connection, each fraction of cenospheres was described by a standard set of parameters, including average diameter of globules ( $D_{av}$ ), size distribution, the content of globules with certain morphological type, apparent shell thickness ( $\delta$ ), the material density ( $\rho_{mat}$ ), bulk density ( $\rho_{bulk}$ ), chemical composition.

The average diameter of globules, the size distribution and content of the different morphological types of cenospheres in each fraction were determined using the method of optical microscopy with the help of Msphere program. Digital images of more than 5000 particles were used as initial data for the calculation.

The apparent thickness of cenosphere shell ( $\delta$ ) was calculated from the formula

$$\delta = 0.5D_{av} \left(1 - \sqrt[3]{1 - \rho_{bulk}/(0.6\rho_{mat})}\right) \quad (1)$$

The density of the shell material ( $\rho_{mat} = 2.45 \text{ g/cm}^3$ ) was determined basing on thermodynamic calculations for macrocomponent composition.

Determining the bulk density ( $\rho_{bulk}$ ) was carried out as a simple average of three to five measurements using standard methods described in [16, 17].

The chemical composition of the initial fraction and the isolated narrow fractions of cenospheres, those include the content of silicon, aluminium, iron, calcium, magnesium, potassium, sodium, titanium, manganese, sulphur oxides, as well as the calcinations loss value were determined using standard methods [18].

The surface structure and shell morphology cenospheres were studied using the method of scanning electron microscopy employing JSM-6460 LV electron microscope (Jeol) with the accelerating voltage of 25 kV and the resolution of 40 nm.

In order to obtain carriers we used as raw material a non-magnetic fraction of cenospheres M-NM-1A with the size of  $(-0.16 + 0.125) \text{ mm}$  and the bulk density of  $0.33 \text{ g/cm}^3$ , isolated according to scheme [15] from the concentrate of cenospheres resulted from the combustion of the Kuznetsk coal at the Moscow Heat and Electric Power Plant (HEPP-22).

Figure 1 demonstrates a standard feature of this fraction. With the help of optical microscopy, we identified three morphological types of globules in the initial fraction of cenospheres

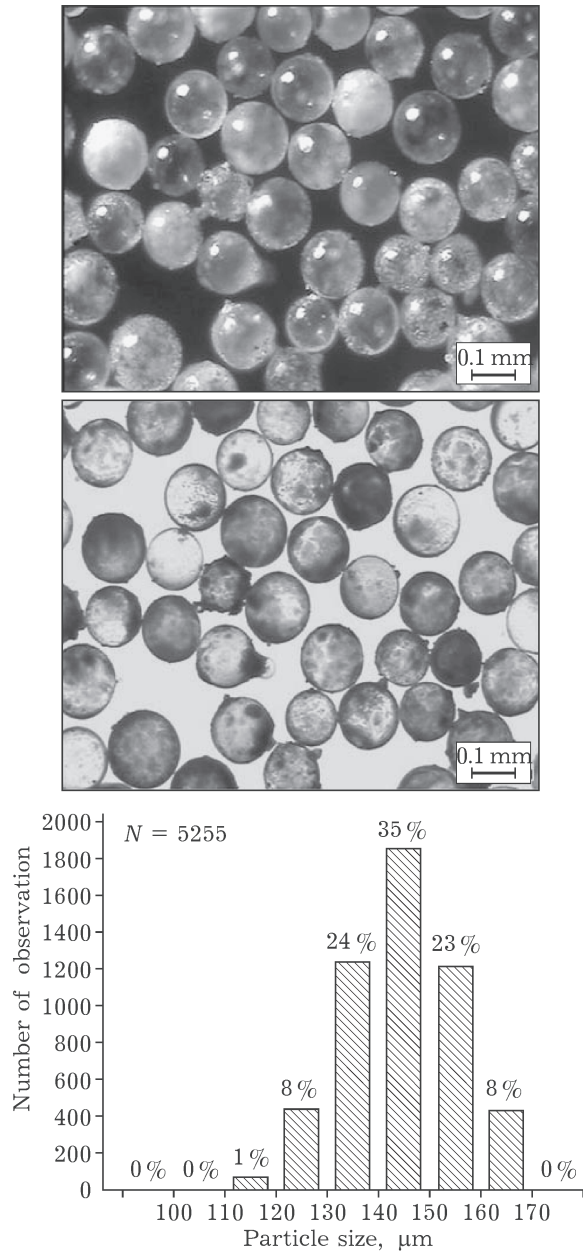


Fig. 1. Characteristics of the initial fraction NM-M-1A  $(-0.16 + 0.125) \text{ mm}$  ( $\rho_{bulk} = 0.33 \text{ g/cm}^3$ ) according to optical microscopy ( $N$  is the number of particles).

(Fig. 2). They are “transparent” – the cenospheres with a thin solid shell and a smooth surface, “gray” – smooth spheres with porous shell of varying thickness, “white” – the particles with highly porous shell and relief surface.

For obtaining microspherical carriers from the three established morphological types of cenospheres, of interest “gray” type are particles with a porous shell. Additional processing these globules with a reagent based on hydrof-

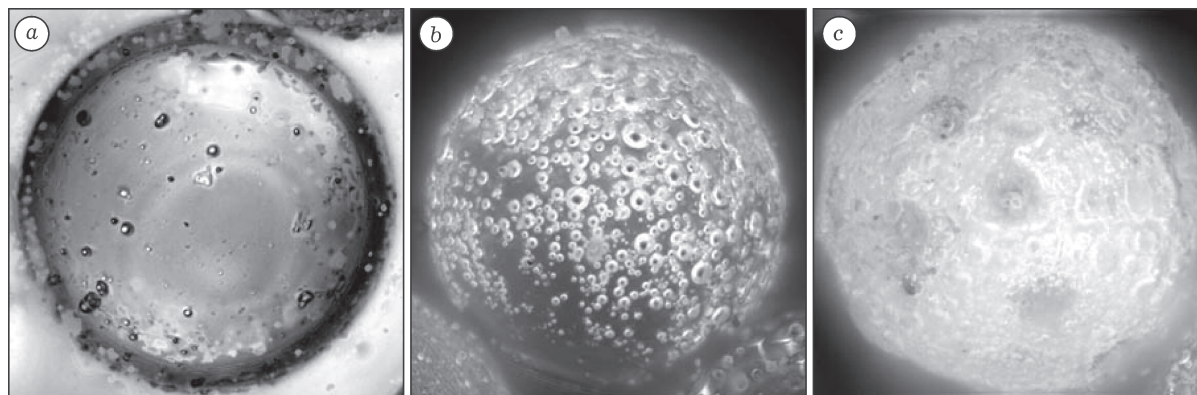


Fig. 2. Morphological types of cenospheres according to optical microscopy (reflected light): *a* – transparent, *b* – gray, *c* – white.

luoric acid resulted in removing glass-crystalline nanosize (30–50 nm) films from the surface of cenospheres [14, 19, 20]. As a result, the porous structure formed by gas inclusions becomes open, providing thus an access to the internal volume of the globules, wherein a highly specific active component of the sorbent is localized.

In order to isolate a morphologically homogeneous fraction of cenospheres with the maximum content of globules with porous shells we carried out an aerodynamic separation of the initial raw material ( $\rho_{\text{bulk}} = 0.33 \text{ g/cm}^3$ ) as described in [21]. *Via* aerodynamic separation based on carrying-over the particles with certain characteristics by a moving air flow, the initial cenospheres were separated into narrow fractions, characterized by bulk density, shell thickness, mean diameter and the content of “gray” globules.

A narrow fraction with the bulk the density of  $0.396 \text{ g/cm}^3$ , containing 91 vol. % of porous globules elected after aerodynamic separation of cenospheres was treated with a reagent based on hydrofluoric acid using the method described in [19], as it follows. A weighed portion of cenospheres was placed in a polypropylene beaker and treated with etching solution (100 mL contained 3.7 g  $\text{NH}_4\text{F}$  and 10 mL of HCl solution with the concentration of 12 mol/L) within 15 min, with occasional stirring. The volumetric S/L ratio was equal to 1 : 5. Upon the completion of processing, the cenospheres were divided into two layers: the lower one ( $\text{HF}_{\text{low}}$ ), wherein the internal volume of cenospheres was filled with the solution and the upper one ( $\text{HF}_{\text{up}}$ ). Both products were washed with distilled water to obtain a negative reaction with respect

to chloride ion with a solution of 0.1 M  $\text{AgNO}_3$  solution and dried in a drying oven at  $(100 \pm 5)^\circ\text{C}$  up to constant mass. The etching loss value amounted to 12.7 mass %. In order to obtain the sorbent we used a perforated HFlow product as a microspherical carrier.

#### *Obtaining the sorbent for palladium extraction from corrosive media*

The use of cenospheres as a carrier of the active component is demonstrated by the example of obtaining microspherical adsorbent for the extraction of palladium from acidic process solutions, slurries, and liquid radioactive waste.

As an active component, we used an organic extracting agent triisobutyl phosphine sulphide (TIBPS, Cyanex-471X) selective with respect to  $\text{Pd}^{2+}$ ,  $\text{Ag}^+$  and  $\text{Au}^{3+}$  ions in the presence of other platinum group metals, as well as a number of non-ferrous metals including  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Fe}^{3+}$ . Along with this, TIBPS is capable of reversible palladium sorption and therefore it can be reused [10]. The organic extractant TIBPS (chemical formula  $\text{C}_{12}\text{H}_{27}\text{PS}$ ) represents a white crystalline powder and exhibits the following properties: mass density (at  $22^\circ\text{C}$ )  $0.91 \text{ g/cm}^3$ , melting point  $58\text{--}59^\circ\text{C}$ , solubility (at  $24^\circ\text{C}$ )  $43 \text{ mg/L}$ . The introduction of the active component was performed by precipitation the extracting agent from a supersaturated alcohol extractant solution within the inner cavity of perforated cenospheres ( $\text{HF}_{\text{low}}$ ). A weighed portion of the perforated cenospheres was placed in a separatory funnel pumped with a water jet pump up to a residual pressure of 8.0 kPa

to hold at this rarefaction during 30 min. After this, to the bottom of the funnel via suction was supplied a hot (50 °C) alcohol solution of TIBPS whose 100 mL contained 40 g TIBPS. The products were held in the solution for 24 h, then filtered, washed with distilled water and dried in air at (20±5) °C for 1 day [19].

The amount of the extracting agent entered into the cenospheres was evaluated by infrared spectroscopy using a Bruker Vector 22 FT-IR spectrometer. As the analytical band we chose the absorption band (a. b.) corresponding to P=S bond stretching vibrations at 579 cm<sup>-1</sup>, whose value is directly dependent on the concentration of TIBPS.

The sorption characteristics of the resulting microspherical adsorbent were determined under static conditions for the model solutions of palladium nitrate at 23 °C. A weighed portion of the sorbent (approximately 0.1 g) was placed into a test tube with 10 mL of palladium nitrate solution, stopper and stirred during the entire sorption process. The concentration of palladium in the solution was determined spectrophotometrically with nitroso-R-salt (sodium 1-nitroso-2,3,6-naphtholdisulphonate) using a Spekol 20 spectrophotometer (Carl Zeiss, Jena, Germany) at the wavelength of 510 nm and the optical cell length of 1 cm [22].

## RESULTS AND DISCUSSION

### Characterization of microspherical carriers

The features of morphology and phase composition of the mineral cenospheres deter-

mine the prospects of obtaining microspherical carriers and adsorbents for processes in corrosive media basing on the cenospheres. Due to the internal cavity within and a high-strength of the glass-crystalline shell, the thermal stability and acid resistance, the cenospheres could be considered to be mechanically strong and stable microcontainers for the localization of active ingredient in the interior of the carrier.

Obtaining the microspherical carriers was performed in two stages: 1) aerodynamic separation into narrow fractions of cenospheres with certain shell thickness and porosity, and 2) acidic etching, providing an access to the internal volume of the globules.

Depending on the composition of cenospheres one can obtain a hollow microspherical carrier with certain shell thickness and with different porosity level. Promising carriers of active sorbent ingredients for corrosive media are presented by porous cenospheres belonging to the morphological type of "gray" cenospheres (see Fig. 2). In order to isolate morphologically homogeneous products with a high content of cenospheres with the porous shell the fraction of the initial cenospheres ( $\rho_{\text{bulk}} = 0.33 \text{ g/cm}^3$ ) were subjected to an additional aerodynamic separation. As a result, six products were obtained with the specific bulk density ranging from 0.195 to 0.396 g/cm<sup>3</sup>. The analysis of physico-chemical characteristics for the fractions obtained demonstrated that with increasing the bulk density of separation products, the average diameter of globules increases from 133 to 153 µm, the thickness of the cenosphere shell increases—from 3.1 to 7.6 µm, the volume frac-

TABLE 1

Physicochemical properties of raw materials and the products of aerodynamic separation

Fraction No.	Air flow rate, m/s	Yield, mass %	Physical parameters			Volume fraction of morphological types, %, according to optical microscopy		
			$\rho_{\text{bulk}}$ , g/cm <sup>3</sup>	$D_{\text{av}}$ , µm	$\delta$ , µm	Transparent	Grey	White
Initial	—	—	0.330	146	5.9	19	74	7
1	0.13	1.5	0.195	133	3.1	45	51	4
2	0.17	4.5	0.229	140	3.8	50	42	8
3	0.21	7.1	0.268	141	4.6	34	65	1
4	0.26	16.7	0.306	144	5.4	36	63	1
5	0.30	30.4	0.356	149	6.5	9	87	4
6	0.34	27.5	0.396	153	7.6	5	91	4

TABLE 2

Chemical composition of the initial fraction and the products of aerodynamic separation with different density

Bulk density, g/cm <sup>3</sup>	Mass fraction, %												
	Calcination loss	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	MnO	P <sub>2</sub> O <sub>5</sub>	Total
	<i>Initial fraction</i>												
0.330	0.56	66.28	25.49	2.80	1.57	0.92	0.27	0.47	1.52	0.12	n/d	0.22	100.22
	<i>Aerodynamic separation of narrow fractions</i>												
0.268	0.64	64.08	26.42	2.94	1.06	1.25	0.10	0.49	2.55	0.05	0.07	n/d	99.65
0.306	0.72	65.16	25.34	3.43	0.91	1.35	0.12	0.41	2.38	0.10	0.05	n/d	99.97
0.356	n/d	65.72	24.34	3.73	1.12	1.20	0.15	0.46	2.59	0.08	0.05	n/d	99.44
0.396	1.24	65.80	22.30	3.83	0.98	2.45	0.14	0.41	2.26	0.08	0.09	n/d	99.58

Note. n/d – not detected.

tion of particles with porous shells determined by optical microscopy in a number of “gray” globules increases from 51 to 91 % (Table 1).

Basing on physicochemical analysis for the aerodynamic separation of narrow fractions (Table 2) we confirmed the interrelation between the chemical composition and morphology of the globules [15]. The thickness of the shell for the products separation depending on Al<sub>2</sub>O<sub>3</sub> content in the cenospheres is described by a linear regression equation:  $\delta = 24.2 - 0.7[\text{Al}_2\text{O}_3]$ , the correlation coefficient being of  $-0.99$ . Thus, with decreasing Al<sub>2</sub>O<sub>3</sub> content from 26.42 to 22.30 mass % the thickness of globular shells increases from 4.6 to 7.6  $\mu\text{m}$ . In this case, according to optical microscopy, the content of cenospheres with porous shells (morphological type of gray cenospheres) increases from 65 to 91 vol. % (see Tables 1, 2).

Using the method of scanning electron microscopy (SEM) we confirmed the presence of cenospheres with solid and porous shells for aerodynamic separation products. For example, Fig. 3 demonstrates SEM images of destroyed globules from the two fractions with different bulk density, chemical composition, thickness and shell porosity. According to SEM, the thickness of the cenosphere shell from the fraction with the bulk density of 0.306 g/cm<sup>3</sup> on the average is equal to 5  $\mu\text{m}$  and the thickness of cenospheres with the density of 0.396 g/cm<sup>3</sup> increases up to 10  $\mu\text{m}$ , which agrees well with the values of the apparent shell thickness (see Table 1).

In order to obtain microspherical carriers we chose a homogeneous narrow fraction of cenospheres with the bulk density of 0.396 g/cm<sup>3</sup>,

characterized by a high yield (27.5 mass %) and containing 91 vol. % of the porous particles of gray type with the average globule diameter amounting to 153  $\mu\text{m}$  and the apparent shell thickness equal to 7.6  $\mu\text{m}$ . It should also be noted

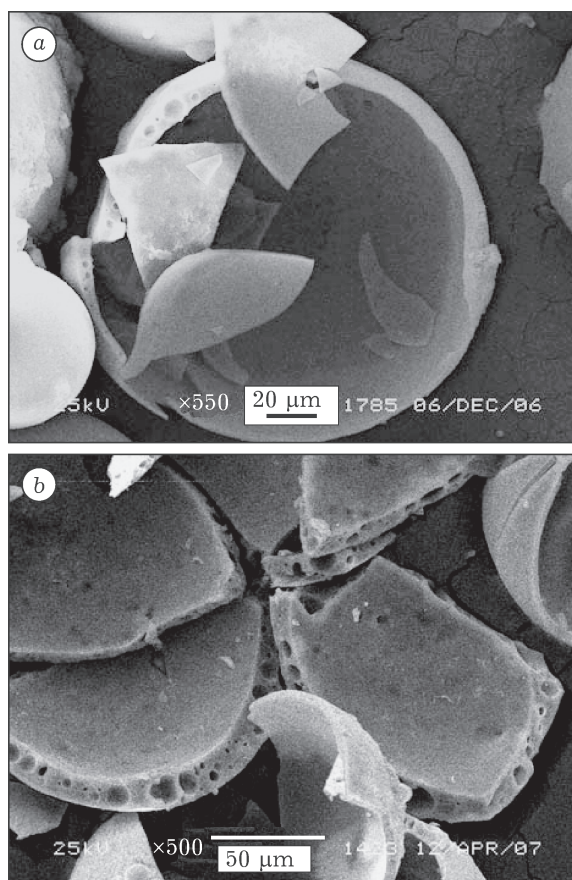


Fig. 3. SEM images for the destroyed globules of aerodynamically separated products.  $\rho_{\text{bulk}}$ , g/cm<sup>3</sup>: 0.306 (a), 0.396 (b).

TABLE 3

Characteristics of etching products, the reagent based on HF

Etching products	Yield, mass %	Physical parameters				Volume fraction of morphological types, %, according to optical microscopy		
		$\rho_{\text{bulk}}$ , g/cm <sup>3</sup>	$D_{\text{av}}$ , $\mu\text{m}$	$\delta$ , $\mu\text{m}$	$S_{\text{sp}}$ , m <sup>2</sup> /g	Transparent	Grey	White
Initial narrow fraction		0.396	153	7.6		5	91	4
HF <sub>low</sub>	64.7	0.367	152	6.9	0.4	6	89	5
HF <sub>up</sub>	22.6	0.355	153	6.7	0.3	3	93	4

that in order to increase the yield of the target fraction up to 58 mass % one could combine the products with the bulk density of 0.356 and 0.396 g/cm<sup>3</sup>, whose control parameters differ from each other only to a non-considerable extent.

In order to remove the surface film and, thus, to achieve the availability of the internal volume and open porosity of a crystalline-glass shell, the chosen narrow fraction of cenospheres was treated with a reagent based on hydrofluoric acid, using the method described in [19]. The characteristics of etching products are listed in Table 3. It is seen that this treatment results in decreasing the bulk density and the thickness of globule shells for the etching products, which could be connected with removing a nanoscale film and surface heterogeneity. Low values of specific surface area (0.3–0.4 m<sup>2</sup>/g) after etching of cenospheres indicate the absence of surface micro- and mesopores. The study of the shell morphology of perforated cenospheres by SEM revealed the

presence of through transport macropores of 1–5  $\mu\text{m}$  in diameter (Fig. 4).

Thus, as the result of the aerodynamic separation of narrow cenosphere fractions and the subsequent acid etching we obtained a morphologically homogeneous porous carrier with an accessible internal volume and with transport pores of 1–5  $\mu\text{m}$  in size, and characterized it by the methods of physicochemical analysis, which carrier could be used to obtain sorbents stable in corrosive media.

#### Parameters of microspherical adsorbent

Depending on the problem to be solved, basing on perforated cenospheres one could obtain microspherical sorbents with different active components, including both inorganic ion exchangers and organic extracting agents [23, 24]. In recent years, perforated cenospheres were used as carriers for the creation of sorbents for purifying liquid radioactive wastes [25–27]. The sorbents obtained basing on cenospheres and ammonium molybdophosphate (AMP) represent efficient trapping agents for <sup>137</sup>Cs, both under static and dynamic conditions, from acidic liquid wastes resulting from spent nuclear fuel [25–27]. Inorganic sorbents, such as transition metal ferrocyanides and zirconium phosphates localized within the inner cavity of cenospheres, can capture <sup>137</sup>Cs from radioactive waste to solve the problem of <sup>137</sup>Cs fixation within the structure of stable skeleton-framework aluminosilicates and phosphates with the formation of mineral-like compounds those are suitable for long-term disposal in granitoids [23].

The problem of selective extracting the palladium from slurries, acidic process solutions and liquid radioactive waste is extremely com-

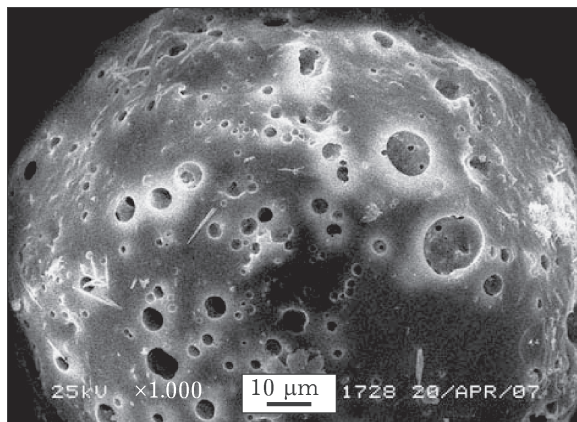


Fig. 4. SEM image of perforated (bottom) cenospheres.

plicated due to the instability of the traditional carriers of active components in corrosive environments. Thus, solid extraction agents (SEA) for extracting palladium, silver and mercury from solutions in the mode of extraction chromatography are usually obtained by applying triisobutyl phosphine sulphide onto an organic carrier based on a styrene and divinylbenzene copolymer [10] or silica [3]. Such SEA can efficiently extract silver, palladium, mercury from aqueous solutions, but they cannot be used for the extraction of these components in corrosive environments.

As a sorption-active component of the microspherical sorbent for the extraction of palladium from acidic solutions, slurries and liquid radioactive wastes we used in organic extractant triisobutyl phosphine sulphide (TIBPS, Cyanex-471X) which was placed into the internal volume of the perforated cenospheres. The content of the active component in the resulting sorbent, according to the FT-IR spectroscopy, was equal to 33 mass %.

The investigation of the adsorption characteristics for the microspherical sorbents obtained was carried out using model solutions under static conditions. Thus, the time dependence for palladium sorption ( $C_{Pd} = 120$  mg/L;  $C_{HNO_3} = 0.5$  mol/L;  $\tau = 0.5, 1, 1.5, 3, 24$  h) indicates reaching the maximum capacity of the sorbent after 3 h of the sorption procedure duration. The acid-base dependence of the palladium sorption ( $C_{Pd} = 120$  mg/L;  $\tau = 3$  h;  $C_{HNO_3} = 0.1, 0.5, 1, 2, 4$  mol/L) indicates the stability of the sorbent obtained within the entire range of nitric acid concentrations of with the maximum value of the sorption level from 0.5 M solution.

The sorption isotherm for palladium ( $C_{Pd} = 3.6$ – $360$  mg/L) (Fig. 5) corresponds to the process of sorption from dilute solutions and can be described either by the Langmuir equation:

$$A = \frac{A_m K C}{1 + K C}$$

or by the Freundlich equation:

$$A = K C^{1/n}$$

For testing the applicability of each model with respect to the experimental data, the empirical Langmuir and Freundlich equations were transformed into the linear form:

$C/A = 1/A_m K + C/A_m$  and  $\log A = \log K + 1/n \log C$ , respectively. Obtaining a straight line in

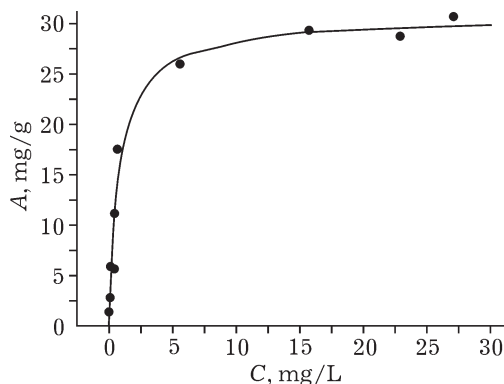


Fig. 5. Sorption isotherm of palladium ( $C_{HNO_3} = 0.5$  mol/L,  $\tau = 3$  h).

the coordinates  $C/A$ – $C$  with the correlation coefficient equal to 0.99 indicates that the experimental sorption isotherm corresponds to the Langmuir model (Fig. 6).

The adsorption equilibrium constant ( $K = 1.17$  L/mg) and limiting sorption ( $A_m = 30.8$  mg/g sorbent) and a high distribution coefficient ( $K_D = 8.1 \cdot 10^6$  mL/g) calculated from the Langmuir model allow one to predict to a satisfactory extent equilibrium and dynamic parameters for the sorption process at low palladium concentrations (1–10 mg/L). The dynamic sorption of palladium from 0.5 M  $HNO_3$  solution ( $C_{Pd} = 9.6$  mg/L) was equal to 29 mg/g of the sorbent and demonstrated a 10% breakthrough level after passing a 1100-fold column volume of palladium solution. In addition, a 1000-fold excess of  $Cu^{2+}$ ,  $Co^{2+}$ ,  $Ni^{2+}$  and  $Fe^{3+}$  cations does

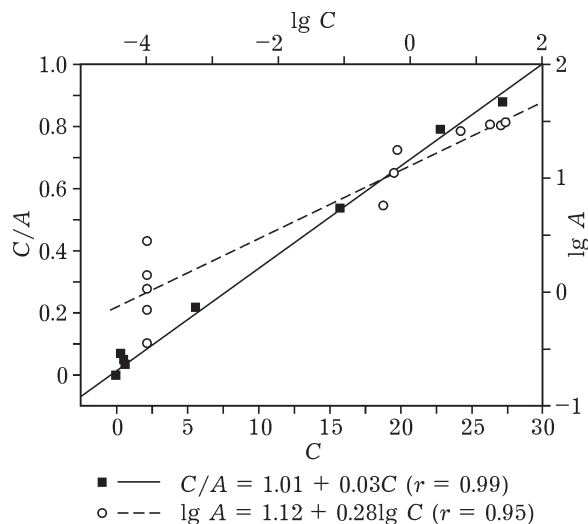


Fig. 6. Sorption isotherm for palladium, plotted in the linearized coordinates according to Langmuir and Freundlich equations.

not prevent the sorption of palladium and does not result in decreasing the sorption capacity of the sorbent.

For the desorption of palladium, we used nitrate solutions of thiourea (1 mol/L), those provided a 96 % level of palladium extraction with a 13-fold absolute palladium concentrating level.

Thus, a theoretical possibility was demonstrated for using a morphologically homogeneous narrow fraction of perforated cenospheres with a shell with variable thickness and porosity, as a carrier of an active component aimed at obtaining a highly efficient sorbent for the extraction of palladium from corrosive media.

## CONCLUSION

As the result of the aerodynamic separation of non-magnetic cenosphere fraction NM-M-1A with the size of  $(-0.16 + 0.125)$  mm ( $\rho_{\text{bulk}} = 0.33 \text{ g/cm}^3$ ) we obtained narrow cenosphere fractions with different bulk density, chemical composition, average diameter, thickness and porosity of the globule shells and characterized them using the methods of physico-chemical analysis. It was established that the increase in the bulk density of cenospheres from 0.195 to 0.396 g/cm<sup>3</sup> is accompanied by increasing the average diameter (from 133 to 153  $\mu\text{m}$ ) and the apparent thickness of globule shells (from 3.1 to 7.6  $\mu\text{m}$ ). In addition, with decreasing Al<sub>2</sub>O<sub>3</sub> content, a considerable increase in the number of globules with porous shells (up to 91 vol. %) was observed.

Basing on the morphologically homogeneous fractions of cenospheres containing 91 vol. % of particles with porous shells obtained by acidic etching, we obtained a macroporous carrier with available internal volume and the pore size of 1–5  $\mu\text{m}$  further used to obtain high-efficient (the sorption capacity of 8.30 mg/g) microspheric sorbent filled with an active component (triisobutyl phosphine sulphide) for the extraction of palladium within a wide range of concentrations (3.6–360 mg/L) from corrosive media.

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