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SORPTION EQUILIBRIA OF HEAVY METALS ON POLYSILOXANE WITH GRAFTED 2-AMINOETHYLPYRIDINE FUNCTIONAL GROUPS

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A new silica-based organic-inorganic hybrid material with grafted 2-aminoethylpyridine functional groups (SiAEP) has been synthesized by sol-gel method. Methods of the elemental analysis, ¹³C and ¹H NMR spectroscopy have been used in order to find substance composition; surface-structural characteristics of the SiAEP have been defined by method of nitrogen adsorption-desorption isotherms. The effect of pH on the sorption of copper (II), nickel (II), cobalt (II), zinc (II), cadmium (II), and lead (II) ions has been studied. A selectivity order of SiAEP at pH=6.5 is as follows: Cu>Co>Zn>Ni>Pb>Cd. Sorption isotherms of heavy metal ions by SiAEP have been obtained, their mathematical processing by models of Langmuir, Freundlich, and Redlich-Peterson has been carried out. The values of the maximum uptake SiAEP capacity for Cu²⁺, Co²⁺, Zn²⁺, Ni²⁺, and Cd²⁺ ions have been defined to be of 2.04, 1.76, 1.46, 1.06, and 0.76 mmol/g, respectively.

INTRODUCTION

Silica-based sorbents with functional groups containing donor nitrogen atoms are applied to separation and preconcentration of heavy metal ions with a success [1–7]. There are two ways of covalent grafting of functional groups on an inorganic matrix: 1) chemical modification of a silica surface with organosilanes (in particular, halogen- and alkoxy-silanes); 2) incorporation of functional groups *via* sol-gel methodology followed (if necessary) by postmodification [8, 9]. An advantage of the first method is the arrangement of all functional groups on the matrix surface, however, the content of an organic component in such sorbents is insignificant what leads to a low metal uptake capacity. The second method allows us to synthesize sorbents with a high content of the functional groups in regular intervals distributed through all the bulk of a matrix. So, the preference has been given to the sol-gel method of synthesis of a sorbent with 2-aminoethylpyridine functional groups grafted on a silica-like matrix in this work.

The main purpose of the present work is to research sorption properties of a silica-based organic-inorganic hybrid material with grafted 2-aminoethylpyridine functional groups (SiAEP).

EXPERIMENTAL

Preparation of polysiloxane with grafted 2-aminoethylpyridine functional groups (SiAEP). The mix containing 10.8 cm³ (0.10 mol) of 2-vinylpyridine, 92.7 cm³ (1.24 mol) of 2-propanol, 2.35 g (0.044 mol) of ammonium chloride, and 11.6 cm³ (0.05 mol) of 3-aminopropyltriethoxysilane was refluxed for 8 h. Then 21.5 cm³ (0.10 mol) of tetraethylorthosilicate and 5.0 cm³ (0.28 mol) of water were added. The obtained gel was left at a room temperature for 24 h, then crushed and washed out with 2-propanol in a Soxhlet apparatus. 1.01 g (0.044 mol) of sodium preliminary dissolved in 50.0 cm³ of 2-propanol was added to the formed compound. The salt obtained was washed with sufficient portion of water up to negative reaction of washing waters on chloride-ions. Finally, the product was dried at 100°C to constant weight.

The elemental analysis of the polysiloxane was carried out using a Perkin Elmer RE-2400 automatic analyzer. For obtaining ¹H and ¹³C NMR spectra, SiAEP was destructed by NaOH, dissolved in D₂O and registered with a BRUKER DRX 400 spectrometer in relation to TMS at the frequency of 400 and 100 MHz,

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respectively. Determining the parameters of porous structure and specific surface area of SiAEP was carried out by obtaining the nitrogen adsorption-desorption isotherms found at liquid nitrogen boiling point using a Micromeritics Tristar II 3020 analyzer.

Initial metal (II) solutions (0.1 mol/dm^3) were prepared by dissolving $\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and $\text{Pb}(\text{NO}_3)_2$ (puriss. p.a.) in distilled water. Exact concentration of metal ions was found by titration with EDTA [10]. Solutions with lesser concentration of metal ions were prepared by dilution of the initial solutions. Necessary pH value was created by ammonia-acetic buffer solution and controlled using an I-130 2M pH-meter with glass (ESL-43-07) and Ag/AgCl reference (EVL-1M3.1) electrodes.

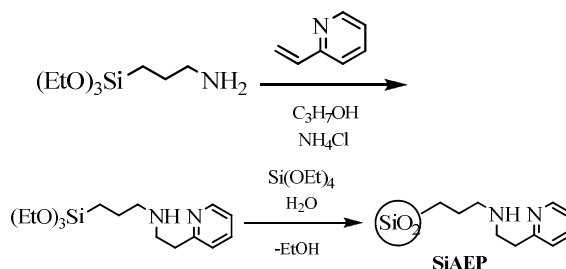
To study the effect of pH on the uptake of heavy metal ions, 0.02 g of polysiloxane was added in 50 cm^3 of solution with $C(\text{Me (II)}) = 1 \cdot 10^{-4} \text{ mol/dm}^3$ ($\text{Me} = \text{Ni, Co, Cu, Zn, Cd, Pb}$) and a certain pH value in the range from 4.0 to 8.0 and left for 7 days under periodic shaking. Then solutions were filtered off and measurements of the metal ion concentration in supernatant were carried out by atomic absorption spectrometry with flame atomization (FAAS) using a Thermo Solaar M6 spectrometer. The amount of sorbed metal ions was found using the difference of ion concentrations in the solution over a sorbent before and after sorption.

Selectivity of sorption was studied by the same method except there were all considered metal ions at in the initial solution.

The sorption isotherms of heavy metal ions ($\text{Ni (II), Co (II), Cu (II), Zn (II), Cd (II)}$) were obtained using a limited volume method under static conditions with periodic shaking. For this purpose 0.02 g of sorbent was put in 50 cm^3 of the solution of metal salt of certain concentration and acidity, left for 7 days at the temperature of $20 \pm 2^\circ\text{C}$. Further, the solutions were filtered off, concentrations of metal ions in supernatant were determined using FAAS method. The equilibrium metal uptake a_e was calculated by using the expression: $a_e = (C_i - C_s) \cdot V/m$ where C_i and C_s were the initial and final (after sorption) concentration of metal ions in the solution respectively, mol/dm^3 ; V – solution volume, dm^3 ; m – mass of the sorbent, g .

RESULTS AND DISCUSSION

Synthesis of the polysiloxane was carried out according to the following scheme:



The structure of SiAEP has been defined by means of ^1H and ^{13}C NMR spectroscopy. For transfer in a liquid phase, polysiloxane was preliminary destructed by NaOH, thus spectra have been received for $\text{Si}(\text{ONa})_3(\text{CH}_2)_3\text{NH}(\text{CH}_2)_2\text{C}_5\text{H}_4\text{N}$ compound. Resonance signals of protons and carbon atoms of various groups of the compound were referenced to on the basis of literary data [2, 6, 11–15] and also using the ACD/C+HNMR Predictor (v.12.01) software package [16] (Table 1).

Table 1. Assignment of bands in ^1H and ^{13}C NMR spectra of the decomposition product of SiAEP in NaOH

Core	δ , ppm	Assignment of signals
^1H	0.40–0.52	$\text{Si}-\underline{\text{CH}_2}-$
	1.45–1.60	$\text{SiCH}_2-\underline{\text{CH}_2}-$
	2.50–2.62	$\text{Si}(\text{CH}_2)_2-\underline{\text{CH}_2}-\text{NH}-$
	1.91	$-\text{CH}_2-\underline{\text{NH}}-\text{CH}_2-$
	2.86–2.97	$-\text{NH}-\underline{\text{CH}_2}-\text{CH}_2-\text{Py}$ $-\text{NH}-\text{CH}_2-\underline{\text{CH}_2}-\text{Py}$
	7.26–7.32	$-\text{CH}_2-\underline{\text{C}_5\text{H}_4\text{N}}$
	7.32–7.38	
	7.75–7.83	
	8.40–8.46	
^{13}C	14.01	$\text{Si}-\underline{\text{CH}_2}-$
	25.68	$\text{SiCH}_2-\underline{\text{CH}_2}-$
	54.35	$\text{Si}(\text{CH}_2)_2-\underline{\text{CH}_2}-\text{NH}-$
	50.96	$-\text{NH}-\underline{\text{CH}_2}-\text{CH}_2-\text{Py}$
	39.43	$-\text{NH}-\text{CH}_2-\underline{\text{CH}_2}-\text{Py}$
	124.80	$-\text{CH}_2-\underline{\text{C}_5\text{H}_4\text{N}}$
	126.77	
	140.79	
	151.00	
161.97		

Also, SiAEP was investigated using the elemental analysis method. Found (%): C, 23.83; N, 5.30; H, 3.26; Si, 28.02; calculated (%): C, 35.80; N, 8.35; H, 4.51; Si, 25.11. The quantity of 2-aminoethylpyridine functional groups in SiAEP

was found due to data of NMR spectroscopy and elemental analysis to be of 2.43 mmol/g.

Specific surface area of SiAEP was determined from the BET (Brunauer, Emmett and Teller) multi-point method [17] and also total volume of pores and pore size distribution were obtained using the BJH (Barret, Joyner and Halenda) method [18]. The value of specific surface area of SiAEP is of 121.71 m²/g, total amount of pores – 0.32 cm³. Dominating pores in the sorbent are those with sizes of 10–30 nm (mesopores).

The study results of on the effect of solution pH on the uptake of copper (II), nickel (II), cobalt (II), zinc (II), cadmium (II), and lead (II) ions by SiAEP are shown in Fig. 1.

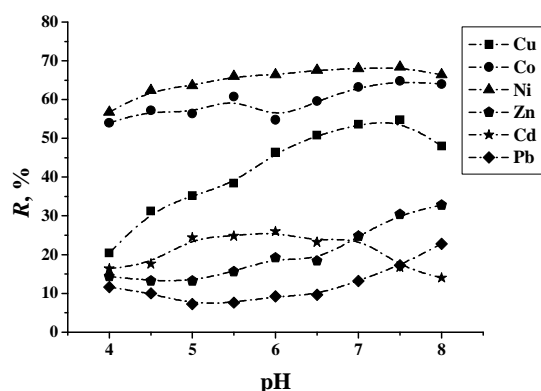


Fig. 1. Effect of pH on the retention of heavy metal on SiAEP. $C_i(\text{Me (II)})=1 \cdot 10^{-4}$ mol/dm³. Ammonia-acetic buffer solution. $T=293 \pm 2$ K

Extraction of cobalt (II) and nickel (II) ions by investigated sorbent depends a little on the acidity of solution and only slightly increases with increase in pH without exceeding 64.8 and 68.4%, respectively (Fig. 1). The plot of extraction of copper (II) ions has an extreme character with a maximum at pH=7.5 (54.8%). The optimum pH interval for sorption of cadmium (II) ions is of 5.0 to 7.0. Extraction of zinc (II) and lead (II) ions increases with increase in pH of solution and reaches the maximum value (32.8 and 22.8%, respectively) at pH=8.0. Since extraction curves are similar, it is theoretically possible to assume that in case of simultaneous presence of all considered ions in solution, SiAEP would be a group sorbent for extraction of nickel (II), cobalt (II), and copper (II) ions.

The research of selectivity of modified polysiloxane was carried out under simultaneous presence of copper (II), nickel (II), cobalt (II), zinc (II), cadmium (II), and lead (II) ions in the solution. The results are presented in Fig. 2.

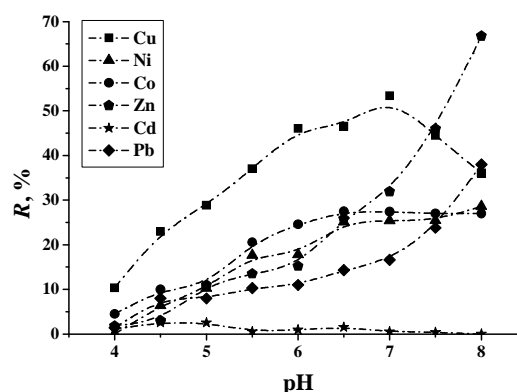


Fig. 2. Effect of pH on the retention of heavy metal on SiAEP (simultaneous presence of all considered metals). $C_i(\text{Me (II)})=1 \cdot 10^{-4}$ mol/dm³. Ammonia-acetic buffer solution. $T=293 \pm 2$ K

SiAEP shows a selective extraction of copper (II) ions in the presence of a number of preventing of heavy metal ions in a wide range of pH. The greatest preventing action is influenced by of zinc (II) ions in neutral and alkaline solutions. Under experimental conditions, extraction of cobalt (II) and nickel (II) ions is rather lower than their extraction from individual solutions (Fig. 1). Sorption of these ions is depressed by the presence of copper (II) and zinc (II) which probably form more stable complexes with functional groups of the sorbent.

For the further research of the sorption properties of SiAEP a value pH=6.5 have been chosen. Under these conditions, the content of metal ions in the sorbent phase in relation to their total quantity looks as follows: Cu(33.03%)> Co(19.52%)> Zn(18.34%)> Ni(17.86%)> Pb(10.14%)> Cd(1.11%).

The sorption isotherms of metal ions are shown in Fig. 3.

It is seen from the data presented that with increase in initial concentration of sorbate degree of its extraction increases too. There are three sites on the isotherms. The first of them, linear rising upwards site of the curve shows that for a small concentration, sorption is described by Henry's isotherm model ($a_e=K_H \cdot C_e$, where K_H – Henry constant, dm³/g; a_e – equilibrium metal uptake, mmol/g; C_e – equilibrium concentration, mmol/dm³). There is a monolayer filled by sorbate on the sorbent surface. Almost horizontal site corresponding to the high concentration of metal ion conforms that the sorbent surface is completely filled by sorbate. Thus, the quantity of sorbed substances practically ceases to depend on

concentration. The middle site of the curve corresponds to intermediate degrees of filling surface.

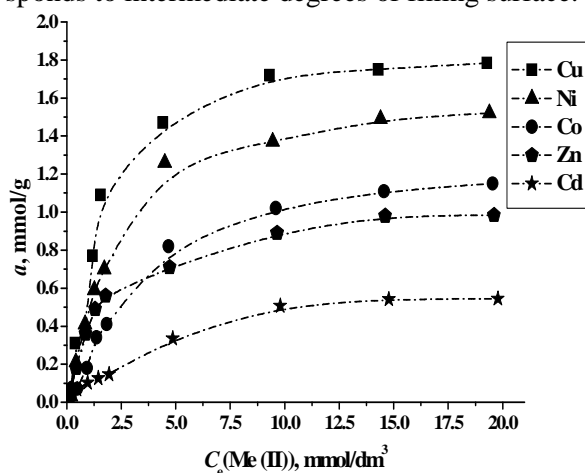


Fig. 3. Metal sorption isotherms of SiAEP. Ammonia-acetic buffer solution (pH=6.5). $T=293\pm 2$ K

The isotherms obtained were described using Langmuir

$$a_e = \frac{a_m \cdot K_L \cdot C_e}{1 + K_L \cdot C_e}, \quad (1)$$

Freundlich

$$a_e = K_F \cdot C_e^{1/n}, \quad (2)$$

and Redlich-Peterson models [19–23]

$$a_e = \frac{K_R \cdot C_e}{1 + a_R \cdot C_e^\beta}, \quad (3)$$

where K_L , K_F , K_R , a_R – Langmuir, Freundlich and Redlich-Peterson constants, dm^3/g , $\text{dm}^3/(\text{mmol}^{1-1/n} \cdot \text{g})$, dm^3/g , $\text{dm}^3/\text{mmol}^{1-1/\beta}$, respectively; $1/n$ and β – heterogeneity coefficients; a_e и a_m – equilibrium and maximum metal uptake, mmol/g ; C_e – equilibrium concentration, mmol/dm^3 .

The Langmuir sorption isotherm is based on the following assumptions: 1) sorption does not proceed beyond monolayer coverage; 2) all the sites available on the sorbent surface are equivalent and the surface is perfectly uniform; 3) there is no interaction between adjacent adsorbed molecules [20, 21].

The Freundlich model is an empirical equation employed to describe heterogeneous systems. This isotherm is another form of the Langmuir one, an approach for adsorption on an "amorphous" surface. The amount of sorbed material is the sum of adsorption on all sites. It describes

reversible adsorption and is not restricted to the formation of a monolayer [22].

The Redlich-Peterson isotherm model combines elements from both the Langmuir and Freundlich models, and the mechanism of adsorption is a hybrid unique and does not follow ideal monolayer adsorption. The Redlich-Peterson equation is widely used as a compromise between Langmuir and Freundlich systems [23].

The procedure of minimization was carried out using mathematical package Origin 8.0 [24] for definition of the values of the parameters entering into the equations of the Langmuir, Freundlich, and Redlich-Peterson isotherms (Table 2). Correlation factors r^2 were compared for finding an optimum model describing sorption of metal ions by SiAEP for each ion.

As a whole, sorption of metal ions by SiAEP is described in the best way by model of Redlich-Peterson. So, it is possible to conclude that the sorbent surface is chemically non-uniform, i.e. functional groups are of various chemical natures. Thus, sorption process proceeds due to Langmuir mechanism, i.e. there is formed a monomolecular layer on the surface.

The Langmuir isotherm model allows us to determine the maximum exchange capacities of SiAEP for copper (II), nickel (II), cobalt (II), zinc (II), and cadmium (II) ions (Table 2) which decrease as follows: $\text{Cu} > \text{Ni} > \text{Co} > \text{Zn} > \text{Cd}$.

Table 2. Parameters of Langmuir, Freundlich and Redlich-Peterson isotherm models and values of correlation factors for SiAEP

Ion	Parameter				
	$K_{L, F, R}$	$1/n$ or β	a_R	a_m	r^2
(1) Langmuir					
Cu (II)	0.500	-	-	2.037	0.970
Ni (II)	0.402	-	-	1.757	0.990
Co (II)	0.220	-	-	1.465	0.989
Zn (II)	0.570	-	-	1.064	0.992
Cd (II)	0.162	-	-	0.757	0.984
(2) Freundlich					
Cu (II)	0.676	0.368	-	-	0.888
Ni (II)	0.521	0.397	-	-	0.922
Co (II)	0.296	0.492	-	-	0.939
Zn (II)	0.385	0.344	-	-	0.947
Cd (II)	0.130	0.517	-	-	0.956
(3) Redlich-Peterson					
Cu (II)	0.890	1.100	0.330	-	0.973
Ni (II)	0.600	1.082	0.273	-	0.996
Co (II)	0.250	1.253	0.079	-	0.996
Zn (II)	0.678	0.948	0.738	-	0.992
Cd (II)	0.117	1.050	0.132	-	0.984

CONCLUSIONS

The present paper describes a silica-based organic-inorganic hybrid material with grafted 2-aminoethylpyridine functional groups (SiAEP) prepared by sol-gel method. This material was characterized by ^1H and ^{13}C NMR spectroscopy and elemental analysis techniques. The structure of SiAEP was investigated using method of nitrogen adsorption-desorption isotherms found at liquid nitrogen boiling point. The effect of pH on the uptake of heavy metal ions was studied. It is found that the sorbent possesses a selectivity in relation to copper (II) ions in the presence of a number of heavy metal ions. The values of maximum uptake capacity of copper (II), nickel (II), cobalt (II), zinc (II), and cadmium (II) ions have been defined; it has been shown that sorption of metal ions by SiAEP is described in the best way by the Redlich-Peterson model.

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Сорбційна рівновага важких металів на полісілоксані з прищепленими 2-аміноетилпіридиновими групами

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Золь-гель методом синтезовано новий орґано-неорґанічний матеріал з прищепленими 2-аміноетилпіридиновими функціональними групами (SiAEP). Методи елементного аналізу, ^{13}C та ^1H ЯМР-спектроскопії використано для визначення складу речовини; структурно-адсорбційні характеристики SiAEP визначено за ізотермами адсорбції та десорбції азоту. Вивчено вплив рН на сорбцію іонів міді (II), нікелю (II), кобальту (II), цинку (II), кадмію (II) та свинцю (II). Ряд селективності SiAEP при рН=6,5 наступний: $\text{Cu} > \text{Co} > \text{Zn} > \text{Ni} > \text{Pb} > \text{Cd}$. Одержано ізотерми сорбції іонів важких металів за допомогою SiAEP, виконано їхню математичну обробку за моделями Ленгмюра, Фрейндліха та Редліха-Петерсона. Величини граничної адсорбції для іонів Cu^{2+} , Co^{2+} , Zn^{2+} , Ni^{2+} та Cd^{2+} виявились рівними відповідно 2,04, 1,76, 1,46, 1,06 та 0,76 ммоль/г.

Равновесие сорбции тяжелых металлов на полисилоксане с привитыми 2-аминоэтилпиридиновыми группами

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Золь-гель методом синтезовано новий орґано-неорґанічний матеріал з привитими 2-аміноетилпіридиновими функціональними групами (SiAEP). Методами елементного аналізу, ^{13}C та ^1H ЯМР-спектроскопії визначено склад речовини; структурно-адсорбційні характеристики SiAEP визначено за ізотермами адсорбції та десорбції азоту. Вивчено вплив рН на сорбцію іонів міді (II), нікелю (II), кобальту (II), цинку (II), кадмію (II) та свинцю (II). Ряд селективності SiAEP при рН=6,5 наступний: $\text{Cu} > \text{Co} > \text{Zn} > \text{Ni} > \text{Pb} > \text{Cd}$. Отримано ізотерми сорбції іонів важких металів на SiAEP, проведена їхня математична обробка за моделями Ленгмюра, Фрейндліха та Редліха-Петерсона. Величини максимальної сорбційної ємності SiAEP по іонам Cu^{2+} , Co^{2+} , Zn^{2+} , Ni^{2+} та Cd^{2+} склали відповідно 2,04, 1,76, 1,46, 1,06 та 0,76 ммоль/г.