
INORGANIC SYNTHESIS
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Sorption of Uranium Compounds by Zirconium-Silica Nanosorbents

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Abstract—Bitemplate (solubilization) synthesis was used to obtain new mesoporous zirconium-silica nanosorbents that can be successfully used to recover uranium compounds from sulfate and carbonate solutions.

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Studies concerned with sorption methods for recovery of uranium compounds from aqueous solutions, seawater, and wastewater pay primary attention to synthesis of new, or modification of already known, sorbents in order to raise their sorption capacity for uranium, improve their selectivity, and elucidate the sorption mechanism in relation to the pH of the medium and forms in which the sorbate is present in solution.

The following sorbents have been used: natural minerals (quartz, muscovite, labradonite [1], pozzolan [2]); zeolites [3], including organozeolites [4] produced by treatment of clinoptilolite-containing tuffs with polyhexamethylene guanidine; sorbents based on wood and lignin [5] with high content of phosphorus; polymers: polymeric chelate sorbents [6]; biopolymer chitosan [7]; Al, Si, Ti oxides, sulfides, and phosphates, including hydrogels based on the SiO₂–TiO₂ formulation (in hydrogen form) [8]; crystalline TiO₂ modified with amorphous aqueous SiO₂ [9], titanium phosphates and phosphosilicates [10]; ion-exchange resins: phosphoric acid resins and sulfocation-exchange resins [11]; anion-exchange resins: macroporous [12], low-basic [13], high-basic [14], and resins of gel and porous structure [15]; activated carbons: oxidized [16] and modified with arsenazo reagents [17].

The wide assortment of sorbents enables their choice for recovery of uranium compounds from aqueous media of various compositions. At the same time, there

hardly is any evidence about purposeful synthesis of sorbents with prescribed pore sizes that would match the size of molecules or ions of uranium compounds being sorbed. However, the number of studies in this area has noticeably increased recently (2007–2010) [18–22].

The goal of our study was to find whether recovery of uranium(VI) compounds from aqueous solutions by sorption with zirconium–silica nanosorbents is possible and can be efficient.

EXPERIMENTAL

Zirconium–silica nanosorbents were synthesized by bitemplate (solubilization) method from inexpensive reagent: sodium silicate, zirconyl chloride, and industrial fraction of a surfactant [23, 24]. Freshly synthesized starting samples were subjected to hydrothermal (980°C) and thermal (600°C) treatments. The specific surface area of the samples (Table 1) was determined from the argon sorption by the BET method [25] (sample nos. 1–10) and from isotherms of low-temperature sorption-desorption of nitrogen (Fig. 1, sample nos. 1 and 9), measured with Quantachrome NovaWin2 instrument. The pore size distribution was found from the desorption isotherms by using the BJH model.

As objects of study served sulfate and carbonate solutions of uranium(VI), which contained 2×10^{-4} mol (50 mg) of the metal per liter. The concentration of

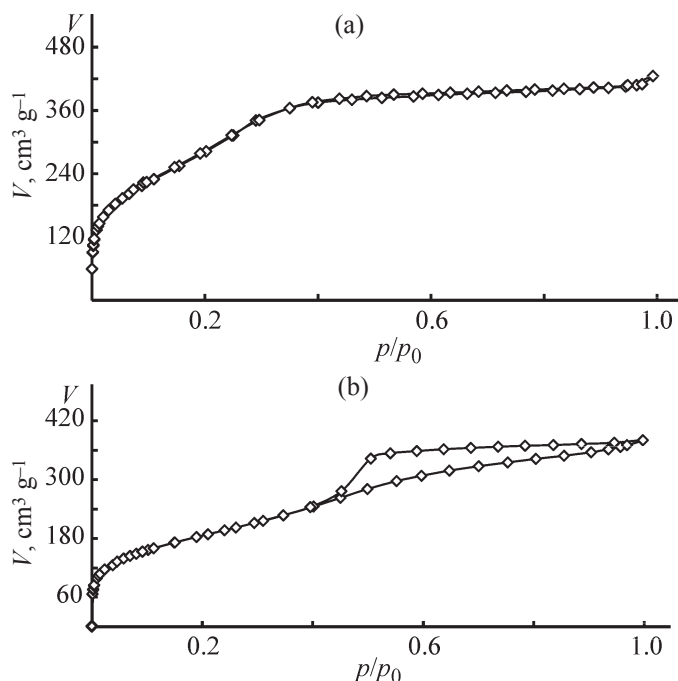


Fig. 1. Isotherms of nitrogen sorption-desorption by sorbent nos. (a) 1 and (b) 9. (V) Volume of sorbed nitrogen and (p/p_0) relative pressure.

sulfate and carbonate ions in the solutions was 0.02 M (except in experiments aimed to examine the effect of the concentration of carbonate ions on the efficiency of uranium sorption). The solutions were prepared from chemically pure salts [$\text{UO}_2(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, NaHCO_3] and chemically pure concentrated sulfuric acid. The natural pH value was 2 for the uranium-containing

Table 1. Effect of the specific surface area S_{sp} of zirconium-silica sorbents, their pore diameter d , $\text{ZrO}_2 : \text{SiO}_2$ molar ratio, and mass fraction W of ZrO_2 in samples on the degree S of sorption of uranium compounds from sulfate (A) and carbonate (B) solutions at natural pH values

Sample no.	S_{sp} , $\text{m}^2 \text{g}^{-1}$	d , nm	$\text{ZrO}_2 : \text{SiO}_2$	W , %	S , %	
					A	B
1	1300	3.18	1 : 6.5	24	30	20
2	1100	—	1 : 5	29	23	27
3	1070	—	1 : 5	29	25	63
4	1020	—	1 : 5	29	31	58
5	1020	—	1 : 5	29	35	64
6	990	—	1 : 5	29	28	56
7	940	—	1 : 5	29	38	25
8	900	—	1 : 3.5	37	44	65
9	800	7.59	1 : 3.5	37	45	30
10	700	—	1 : 3.5	37	48	15

sulfite solutions, and 8 for the carbonate solutions. Under the given experimental conditions, uranium was present (Figs. 2, 3) as a mixture of sulfate complexes of composition $[\text{UO}_2(\text{SO}_4)_2]^{2-}$ (33.3%), $[\text{UO}_2(\text{SO}_4)]$ (33.3%), and uranyl ions (33%) in the sulfate solutions, and as a mixture of complex compounds $[\text{UO}_2(\text{CO}_3)_2]^{2-}$ (87%) and $\text{pUO}_2(\text{CO}_3)_3]^{4-}$ (13%) in the carbonate solutions.

In separate experiments, as objects of study served model solutions that simulated (i) carbonate mining water containing (mg L^{-1}): U 7, Cl^- 140, NaHCO_3 462, Ca^{2+} 10, Mg^{2+} 65 [26], and (ii) technological solutions formed in sulfuric acid decomposition of lean uranium ores. These solutions contained (g L^{-1}): U 0.1, Ca^{2+} 0.5, Mg^{2+} 0.5, Fe^{3+} 0.5, Al^{3+} 2, and SO_4^{2-} [15 [27]].

The sorption of uranium by the sorbents under study was performed under static conditions at a sorbent expenditure of 0.5 g L^{-1} at room temperature (20°C) in the course of 5–7 h. We found that this mode is the optimal in preliminary experiments. The phase

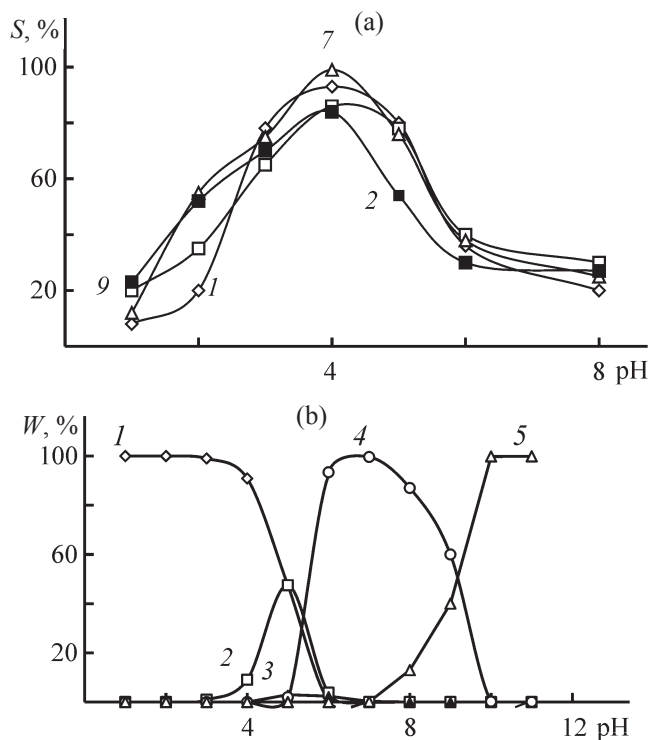


Fig. 2. Effect of pH on (a) degree of sorption, S , of uranium from its initially carbonate solutions by sample nos. 1, 2, 7, and 9 and (b) form of uranium existence in carbonate solutions: (1) UO_2^{2+} , (2) UO_2OH^+ , (3) $\text{UO}_2(\text{OH})_2$, (4) $[\text{UO}_2(\text{CO}_3)_2]^{2-}$, and (5) $[\text{UO}_2(\text{CO}_3)_3]^{4-}$. (W) Mass fraction of uranium compounds; the same for Fig. 3.

separation after sorption by settling or centrifugation proved to be inefficient. Therefore, the phases were separated by ultrafiltration with a parchment ultrafilter preliminarily soaked in twice-distilled water for 40 min, a time sufficient for effective operation of the ultrafilter. Then the filtrate was photocolometrically analyzed for the content of uranium by the standard procedure [28], with arsenazo III serving as indicator. The efficiency of the sorption recovery of uranium was judged from the degree of sorption, S (%).

The isoelectric point of the sorbents under study was determined by means of microelectrophoresis [29]. The pH values of the solutions was determined with 1 M KOH and HCl solution and measured with an EV-74 general-purpose ion-meter with a glass electrode.

Our study demonstrated (Fig. 1) that the total volume of nitrogen sorbed by sample no. 1 exceeds that for sample no. 9. The sorption-desorption isotherms obtained belong to type-IV physical sorption isotherms according to the BDDT classification (isotherms with a clearly pronounced capillary condensation), which is characteristic of mesoporous sorbents [30].

The initial portion of the nitrogen sorption isotherms corresponds to sorption over the entire accessible surface (at relative pressures $p/p_0 = 0-0.07$ for sample no. 1 and $p/p_0 = 0-0.1$ for sample no. 9). The ascending portion of the isotherms (linear portion at $p/p_0 = 0.07-0.35$ for sample no. 1 and $p/p_0 = 0.1-0.4$ for sample no. 9) corresponds to filling of mesopores in the bulk of mesophase particles of the sorbents. The plateau following the ascending portion ($p/p_0 = 0.35-0.98$ for sample no. 1) characterizes the sorption on the outer

surface remaining after the mesopores are filled [31]. There is no plateau in the nitrogen sorption isotherm of sample no. 9 (Fig. 1b). At $p/p_0 > 0.98$, the second steep rise is observed in the nitrogen sorption isotherm of sample no. 1, which is indicative [30] of a phase inhomogeneity of the sorbent.

Both isotherms have a sorption hysteresis loop whose shape strongly varies between the samples under study. According to the IUPAC classification [30], the capillary-condensation hysteresis loops in the isotherms of sample nos. 1 and 9 have shapes H4 and H2, respectively. The shape of the hysteresis loop H2 with a steep desorption branch is characteristic of “bottle-shaped” pores with sizes more than twice exceeding the bottleneck size [30]. The shape of the hysteresis loop H4 with nearly horizontal sorption and desorption branches and small loop area is characteristic of sorbents having slit-like pores [30].

The difference observed between the shapes of nitrogen sorption-desorption isotherms for sample nos. 1 and 9 can be attributed to different contents of ZrO_2 in the sorbents because an increase in the amount of ZrO_2 formed as a result of their multiple-stage synthesis favors development of the main mesoporous structure [30]. This is confirmed by the fact that the average diameter d and total pore volume V_{tot} , found from the sorption-desorption isotherms, grow as the $ZrO_2 : SiO_2$ molar ratio decreases, i.e., as the amount of ZrO_2 in the sorbent is raised:

Sample	d , nm	V_{tot} , $cm^3 g^{-1}$	$ZrO_2 : SiO_2$
1	3.18	0.63	1.0 : 6.5
9	7.59	0.90	1.0 : 3.5

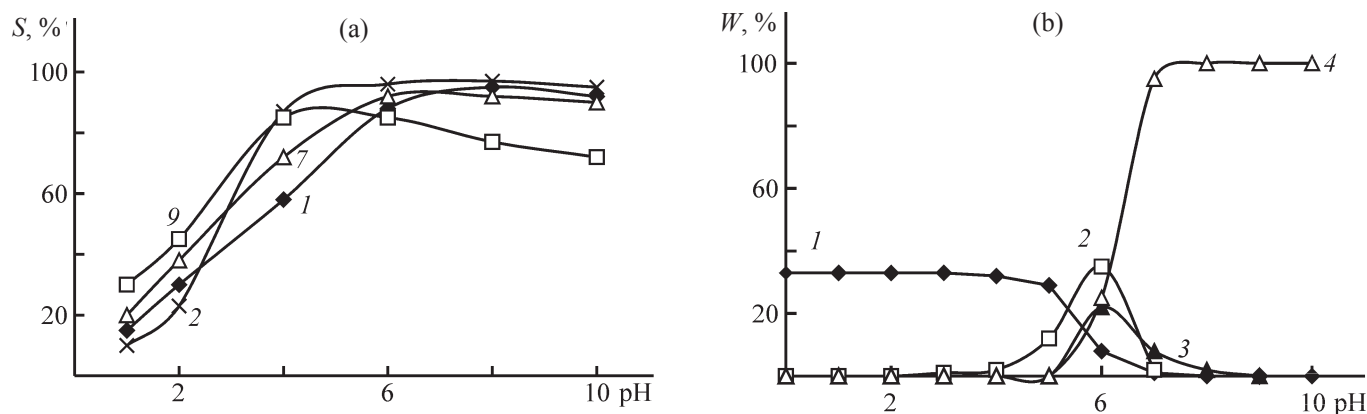


Fig. 3. Effect of pH on (a) degree of sorption, S , of uranium from its sulfate solutions by sample nos. 1, 2, 7, and 9 and (b) form of uranium existence in sulfate solutions: (1) UO_2^{2+} , $[UO_2(SO_4)]^0$, $[UO_2(SO_4)]^{2-}$; (2) UO_2OH^+ ; (3) $UO_2(OH)_2$; and (4) $[UO_2(OH)_3]^-$.

Further studies demonstrated (Table 1) that all the sorbents under study can recover 15–65% of uranium compounds from sulfate and carbonate solutions at their natural pH value, depending on the specific surface area of a sorbent and its zirconium oxide content.

It follows from the data in Table 1 that the sorption recovery of uranium compounds from their carbonate solutions is, on the whole, more efficient than that from sulfate solutions. With decreasing specific surface area of the sorbents, the degree of sorption of uranium compounds from sulfate solutions nearly steadily increases, from 23 to 48%. In recovery of uranium compounds from carbonate solutions with sorbents having intermediate values of the specific surface area (900–1070 m² g⁻¹), the degree of sorption is at a maximum, 56–65%. When sorbents with the minimum (700 m² g⁻¹) and maximum (1100 m² g⁻¹) specific surface areas are used, the degree of sorption sharply decreases to 15–27% (Table 1).

The observed phenomena can be understood if

Sorbate	UO ₂ ²⁺	[UO ₂ (SO ₄)]	[UO ₂ (SO ₄) ₂] ²⁻	[UO ₂ (CO ₃) ₂] ²⁻	[UO ₂ (CO ₃) ₃] ⁴⁻
<i>r</i> _{min} , nm	0.186	0.318	0.347	0.347	0.417
<i>r</i> _{max} , nm	0.325	0.406	0.516	0.531	0.532

These parameters were found using MarvinView program from the MarvinBeans software package (calculation was performed with the particle solvation disregarded). Comparison of the values of *r*_{min} and *r*_{max} with the pore sizes characteristic of mesoporous sorbents [32] (Table 1) and with data on existence forms of uranium in sulfate and carbonate solutions suggests that the composition of uranium-containing solutions and ZrO₂ content of sorbates are important in sorption of uranium compounds by the samples under study.

In order to search ways to improve the efficiency of the sorption recovery of uranium compounds from aqueous solutions, we studied the effect of the pH of the medium on the sorption of uranium compounds by the compounds under study. This effect may be due to changes of the existence forms of uranium compounds in solution (Figs. 2, 3), because of the complexation and hydrolysis, and to the influence exerted by the acidity of the medium on the properties of a sorbent. It was found experimentally that the isoelectric point of the sorbents under study falls within the pH range 3.0–3.5 and is

we take into account the relative contents of various forms of uranium in sulfate and carbonate solutions at their natural pH values and also changes in formation conditions of the mesoporous structure of the sorbents under the influence of the increasing amounts of ZrO₂ they contain [30]. It follows from the data in Table 1 that an increase in the content of ZrO₂ in the sorbents is accompanied by a decrease in their specific surface area. This is due to the appearance of coarser pores in the samples, which facilitates sorption of [UO₂(SO₄)] molecules and coarse complex ions [UO₂(SO₄)₂]²⁻, [UO₂(CO₃)₂]²⁻, and [UO₂(CO₃)₃]⁴⁻.

It is necessary to take into account that sorbate particles have a nearly linear structure. This favors their sorption in slit- and bottle-like pores of the sorbents under study. The particle size can be characterized by two parameters, radii of the minimum (*r*_{min}) and maximum (*r*_{max}) van der Waals projections of a particle onto the plane:

independent of their content of zirconium oxide. Thus, the sorbent surface is charged positively at pH < 3.0 and negatively at pH > 3.5.

Further studies demonstrated (Fig. 2) that the sorption of uranium from carbonate solutions (pH 6–8) is poorly efficient and does not exceed 40%. The reason is that, at pH 6–8, uranium is present in solution as negatively charged uranyl bi- [UO₂(CO₃)₂]²⁻ and tricarbonate [UO₂(CO₃)₃]⁴⁻ complexes and the sorbent surface is charged negatively in this case, and, therefore, the sorption occurs via dispersion interaction of the sorbate and sorbent.

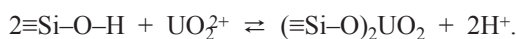
As the pH of the medium decreases, the degree of sorption noticeably increases reaches values of 90–100% (pH 4). The reason is that, as carbonate solutions are acidified to pH 4, negatively charged uranyl carbonate complexes of composition [UO₂(CO₃)₂]²⁻ and [UO₂(CO₃)₃]⁴⁻ disintegrate to give UO₂²⁺ (90.9%) and UO₂OH⁺ (9.1%) cations. Because the sorbent surface is negatively charged at pH 4, the sorption efficiency increases due to an electrostatic interaction of the

Table 2. Mass fraction W of bi- and tricarbonat complexes of uranyl in its 2×10^{-4} M solution at various concentrations of carbonate ions

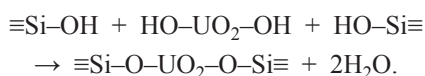
$c_{\text{CO}_3^{2-}}$, M	$W, \%$	
	$[\text{UO}_2(\text{CO}_3)_2]^{2-}$	$[\text{UO}_2(\text{CO}_3)_3]^{4-}$
0	0	0
0.01	93	7
0.02	87	13
0.04	62	38
0.06	43	57
0.20	5	95

sorbate and sorbent.

Taking into account that the zirconium-silica sorbents under study exhibit weakly acid cation-exchange properties, we can assume that uranium compounds are sorbed in the pH range 1–3 by the cation-exchange mechanism



The sorption of uranium compounds from sulfate solutions (pH 1–2) is insignificant (Fig. 3). This is probably due to the steric hindrance that appears in sorption of coarse uranyl sulfate complexes $[\text{UO}_2(\text{SO}_4)]^0$ and $[\text{UO}_2(\text{SO}_4)_2]^{2-}$ in sorbent pores. Upon alkalization of sulfate solutions to pH 10, the sorption of uranium compounds grows (Fig. 3) and reaches the maximum value of 85–96% in the pH ranges 6–10 (sorbent no. 1), 4–8 (sorbent no. 2), 6–8 (sorbent no. 7), and 4–6 (sorbent no. 9). A comparison of the data on how the pH value affects the degree of uranium sorption and existence forms of uranium in sulfate solutions (Fig. 3b) demonstrated that uranium is sorbed at the optimal pH values both as positive and negative hydroxo complexes of composition $\text{UO}_2(\text{OH})^+$, $\text{UO}_2(\text{OH})_3^-$ and as uranyl hydroxide $\text{UO}_2(\text{OH})_2$. In the latter case, the sorption mechanism is presumably associated with the copolymerization process [33] occurring by the scheme:



Thus, the following should be taken into account when determining the sorption mechanism of uranium compounds from their sulfate solutions by zirconium-

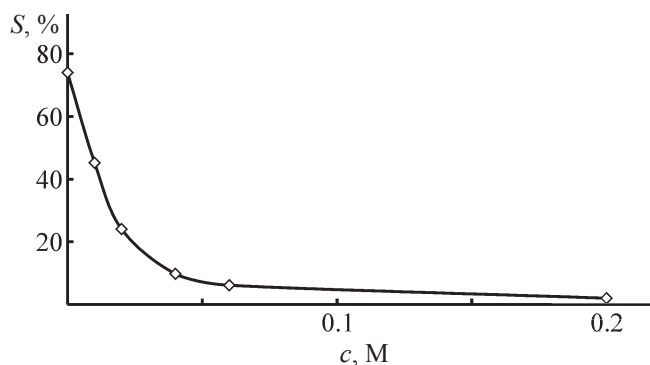
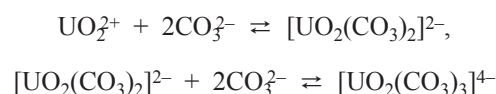


Fig. 4. Effect of the CO_3^{2-} concentration c on the degree S of uranium sorption from carbonate solutions by sample no. 7. Solution pH 8.

silica nanosorbents. At pH < 3.5, the sorbent surface is positively charged, and, therefore, sorption occurs due both to the electrostatic interaction of the sorbent with negatively charged sulfate complexes of uranyl and to the universal dispersion interaction of the sorbent with neutral uranyl sulfate complexes and uranyl cations. At pH > 3.5, the sorbent surface is negatively charged, with the result that there occur both the electrostatic (pH 4–6) and dispersion interaction between the sorbate and sorbent (pH 4–8).

It is known that carbonate ions are present in technological solutions and natural water. The content of these ions may widely vary and they can serve as ligands in complexation of uranium(VI). Therefore, it was of interest to determine the effect of their concentration on the degree of sorption of uranium compounds by zirconium-silica sorbents. It was found (Fig. 4) that, as the concentration of carbonate ions ($c_{\text{CO}_3^{2-}}$) increases, the degree of uranium sorption decreases from 74% ($c_{\text{CO}_3^{2-}} = 0$ M) to 2% ($c_{\text{CO}_3^{2-}} = 0.2$ M).

The results obtained can be understood as follows. The increase in the ligand concentration in solution shifts the equilibria



toward formation of the uranyl tricarbonat complex, whose size is large, which hinders its sorption by the sorbents under study.

To confirm this conclusion, we calculated the mass fractions of uranyl bi- and tricarbonat complexes in solutions with pH 8 and various initial concentrations of

carbonate ions (Table 2). We used in these calculations the stepwise hydrolysis constants K_h of the uranyl ion [34], instability constants of uranyl carbonate complexes [35], and stepwise dissociation constants of carbonic acid [35].

Results of our calculations (Table 2) demonstrated that, as the concentration of carbonate ions in solutions with pH 8 increases at a constant uranium concentration in solution, the amount of uranyl bicarbonate complexes decreases, and that of tricarbonic complexes grows. This leads to a sharp fall of the degree of uranium sorption (Fig. 4).

To study the sorption activity of uranium compounds from model solutions simulating carbonate mine water and technological solutions formed in sulfuric acid decomposition of lean uranium ores, we used sorbent no. 2. It was found that degree of sorption of uranium compounds from acid sulfate solutions (pH 2.6) is 62%, and that from alkaline carbonate solutions (pH 8.4), 55%. The sorption process can be optimized by alkalization of sulfate model solutions to pH 6–8 and acidification of carbonate model solutions to pH 4 at sorption duration of 5 h. Under the conditions studied, the degree of sorption of uranium compounds from model solutions reaches a value of 97%.

CONCLUSIONS

(1) It was shown that zirconium-silica sorbents produced by bitemplate (solubilization) synthesis are mesoporous sorbents (pore diameter 3.18–7.59 nm) with various pore shapes.

(2) It was found that uranium compounds can be 85–100% recovered from sulfate and carbonate solutions under the optimal sorption process conditions.

(3) It was found that the nature of the influence exerted by pH on the degree of uranium sorption from sulfate and carbonate solutions by the sorbents under study and the optimal pH values are independent of the specific surface area and zirconium oxide content of a sorbent.

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