
 CHEMICAL KINETICS
AND CATALYSIS

Kinetic and Diffusion Parameters of the Sorption of Co^{2+} Ions by a Sorbent Based on Industrial Waste

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Abstract—A sorbent based on industrial waste (borogypsum), obtained via autoclave treatment, is used to study the kinetic and diffusion parameters of the sorption of Co^{2+} ions from aqueous solutions of cobalt chloride with no salt background. Kinetic curves of the sorption of Co^{2+} ions are obtained and analyzed using models of chemical kinetics at temperatures 20, 40, and 60°C. Such thermodynamic parameters as standard enthalpy (ΔH°), standard entropy (ΔS°), standard free energy (ΔG°), and coefficients of Co^{2+} ions diffusion are determined at selected temperature conditions.

Keywords: industrial waste, sorbent, cobalt ions, sorption kinetics, coefficients of diffusion

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INTRODUCTION

Cobalt is the most radiation-hazardous element. It is activated to form isotope cobalt-60, which is distinguished by both the high energy of its gamma quanta (~1.2 MeV) and its long half-life ($T_{1/2} = 5.272$ years). Radioactive cobalt ^{60}Co is a component of ionic corrosion products of the primary coolant circuits of nuclear power plants. Having a long half-life, ^{60}Co in combination with isotope ^{137}Cs determines the radiation background when dismantling decommissioned nuclear power plant units after their long-term exposure and final shutdown [1–4].

Emergency situations in which cobalt gets into the environment are also known. An example is the accident on nuclear submarine K-431 in Chazhma Bay (Russia, Far East) in 1985. Mostly cobalt-60 was released into the environment, while the proportion of strontium-90 and cesium-137 was small. Experts are currently monitoring the radiation–hygienic conditions in the area of this radiation accident and emphasize the need for periodic monitoring of the radiation parameters of environmental features and biota [5].

Natural and synthetic silicates, and materials based on them, occupy a special place in the range of materials used for the sorption and immobilization of cobalt. The scientific literature contains information on such compounds as bentonite, montmorillonite, diatomite, sepiolite [6], a mixture of natural zeolite phillipsite ($\text{K}_6(\text{Si}_{10}\text{Al}_6)\text{O}_{32} \cdot 12\text{H}_2\text{O}$) and bentonite [7],

magnesium hydrosilicate [8], synthetic aluminosilicate gel [9], amorphous and crystalline silicates and calcium hydrosilicates [10–13]. Modified silicate-containing materials include, e.g., aluminum silicate modified with magnesia [14], copper ferrocyanide and silica nanoparticles ($\text{SiO}_2\text{—CuFe—CN}$) [15], bentonite modified with formaldehyde [16], and montmorillonite clay modified with thoron [17].

It was established in a number of works [10, 11, 18, 19] that the maximum sorption capacity of amorphous and crystalline calcium silicates $n\text{CaO} \cdot m\text{SiO}_2$ relative to Co^{2+} ions varies from 0.18 to 4.1 mmol g^{−1}. Using these data, the authors [13, 20] developed compositions based on $\text{CaCoSi}_2\text{O}_6$ for obtaining solid-state ceramic matrices for cobalt immobilization using spark plasma sintering with a high relative density (up to 99.9%), compressive strength (up to 637.3 MPa) and Vickers microhardness (up to 8.34 GPa), a rate of cobalt leaching (R_c) no faster than 10^{−7} g cm^{−2} day^{−1}, and a coefficient of diffusion (D_c) of 10^{−12}–10^{−17} cm² s^{−1}, which meets the requirements of GOST R 50926-96 for solidified high-level waste.

The authors of [20] described the sorption properties of the initial raw material for ceramic matrices. It is based on calcium silicate synthesized from waste from producing boron (borogypsum) under conditions of hydrothermal treatment. Comparative data on the composition, morphology, thermal behavior, specific surface area, and porosity of original and cobalt-

saturated powders were presented. The isotherm of cobalt sorption was presented and analyzed as well. The maximum sorption capacity of the material with respect to Co^{2+} ions was 3.7 mmol g^{-1} . However, the kinetic and diffusion parameters of cobalt sorption by the initial material were not studied.

The aim of this work was to study the diffusion and kinetic parameters of cobalt sorption by a material based on calcium silicate obtained under hydrothermal conditions from the waste of boron production.

EXPERIMENTAL

Waste from the production of boric acid (borogypsum) was used to obtain a sorbent based on calcium silicate. The main components of borogypsum are calcium sulfate dihydrate and amorphous silica. The contents of the main components were, in wt %: SiO_2 , 32.2; CaO , 28.4; SO_3 , 31.3; and Fe_2O_3 , 2.7. The specific surface area of the borogypsum was $12.9 \text{ m}^2 \text{ g}^{-1}$. The borogypsum was mixed with a solution of potassium hydroxide of analytical grade in a stoichiometric ratio. Synthesis was conducted for 2 h inside an autoclave at a pressure of 1.7 atm. The degree to which the reaction was complete under the specified conditions of preparation was 81.2% [21].

X-ray diffraction patterns of precipitates were recorded on an automatic D8 ADVANCE diffractometer (Germany) with samples rotated in CuK_β radiation. X-ray diffraction analysis (XRD) was performed using the EVA search program with the PDF-2 powder data bank.

The specific surface area of the material was determined via low-temperature nitrogen adsorption according to the BET (Brunauer–Emmett–Teller) procedure using a Sorbtometer-M device (Russia). Before measuring the specific surface area, the samples were vacuum dried for 0.5–2 h at 100–110°C (depending on humidity) to remove any water.

The density of the sorbent was determined using a pycnometer.

The pH of the solution was measured using a MULTITEST IPL-102 pH meter/ion meter with an ESC-10601/7 glass electrode (Russia), standardized with buffer solutions.

Experiments on the kinetics of sorption were performed under static conditions at a solid-to-liquid ratio of 1 : 400 and temperatures 20, 40, and 60°C using aqueous solutions of cobalt chloride $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ with no salt background. The initial concentration of Co^{2+} ions was 1.35 mmol/L (pH 6.2). Samples of the sorbent were placed in a series of test tubes, filled with an aqueous solution of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, and shaken for 1 to 180 min on an RT 15 magnetic stirrer (IKA WERKE, Germany).

The concentration of Co^{2+} ions was determined via inductively coupled plasma atomic emission spec-

trometry on an iCAP 7600 Duo spectrometer (Thermo Scientific, United States) in the radial plasma observation mode ($\lambda = 228.6$). The limit of detection for cobalt ions in aqueous solutions during radial plasma observation is $0.001 \mu\text{g mL}^{-1}$. The error in determining the concentration of cobalt in solutions in the range of 0.001 – $10 \mu\text{g mL}^{-1}$ is 15%.

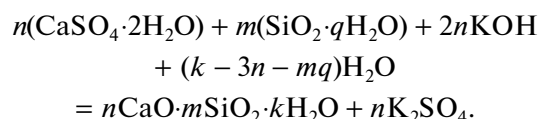
The sorption capacities (A_c , mmol g^{-1}) of the studied samples were calculated with the formula

$$A_c = \frac{(C_{\text{init}} - C_{\text{eq}})}{m} V, \quad (1)$$

where C_{init} is the initial concentration of Co^{2+} ions in the solution, mmol L^{-1} ; C_{eq} is the equilibrium concentration of Co^{2+} ions in the solution, mmol L^{-1} ; V is the volume of the solution, L; and m is the mass of the sorbent, g.

RESULTS AND DISCUSSION

The process for obtaining calcium hydrosilicates from borogypsum using an alkaline agent (potassium hydroxide) is described by the summary equation



In this work, borogypsum was treated in an autoclave. This allowed us to raise the intensity of the reaction, relative to normal conditions and ultrasonic treatment [22].

According to X-ray phase analysis (XPA), each sample contained crystalline phases of calcium carbonate CaCO_3 in modifications of calcite (PDF-2, 01-072-1937) and vaterite (PDF-2, 01-072-0506), quartz SiO_2 (PDF-2, 01-086-1560), tricalcium orthosilicate $\text{Ca}_3\text{SiO}_4\text{O}$ (PDF-2, 01-070-1846). The specific surface area of the obtained material was $22.0 \text{ m}^2 \text{ g}^{-1}$, and the density was 2.25 g cm^{-3} . Firing the sample at 850°C produced the crystalline phase of wollastonite in a triclinic modification, according to data from X-ray diffraction and thermogravimetric analysis [21].

Figure 1 shows kinetic curves of the sorption of Co^{2+} ions at temperatures 20, 40, and 60°C.

We can see from the kinetic curves that the sorption capacity rose along with temperature, from 0.336 to 0.54 mmol g^{-1} .

The mechanism of sorption by calcium silicate-based compounds from this type of technogenic raw material (borogypsum), which is a multicomponent system $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ – SiO_2 – H_2O , was studied in [18, 20, 22]. Because of the solubility of CO_2 in water, we must consider the interaction between carbonic acid, which is much stronger than polysilicic acids, and calcium silicate formed in the alkaline treatment of borogypsum, which actually results in the decomposition

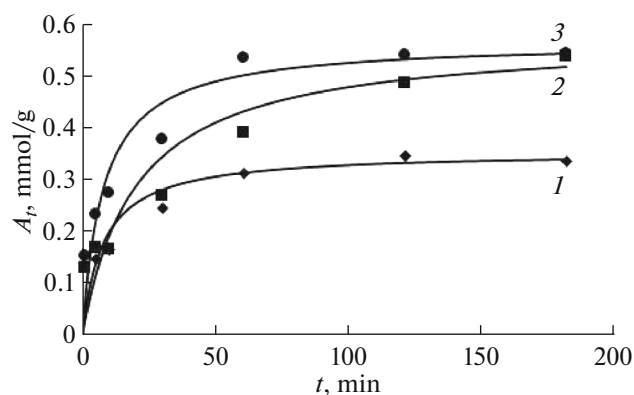


Fig. 1. Kinetic dependences of the sorption of Co^{2+} ions at temperatures (1) 20, (2) 40, and (3) 60°C.

of silicate with the formation of calcium carbonate and amorphous forms of silicon dioxide. Due to their chemical nature, both the original calcium silicates and the products of their interaction with carbonic acid interact with all salts of heavy metals stronger than polysilicic and carbonic acids, according to the type of cation exchange. To explain the mechanism of Co^{2+} ions sorption, we calculated the changes in Gibbs

free energy ΔG_{298}° for reactions during the interaction between an aqueous solution of cobalt chloride and silicate and calcium carbonate that were possible in the considered system [18, 22]. We also analyzed the dependence of the concentration of Ca^{2+} ions in filtrates after sorption and in solid sediments on the amount of sorbed cobalt [20]. Thermodynamic calculations and data on the change in the content of Ca^{2+} ions in solid sediments and filtrates after sorption confirmed that the main mechanism of sorption was the cation exchange of Ca^{2+} and Co^{2+} ions in the components of the sorbent, in combination with surface complexation.

We may assume the dependences of the sorption capacity on the temperature and period of sorption

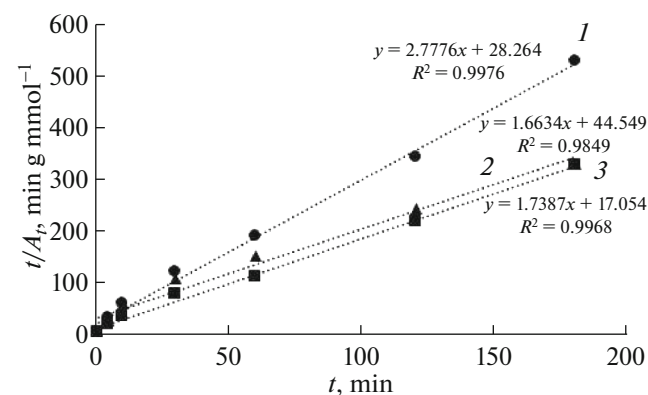


Fig. 2. Dependences of relative values of sorption capacity at t from time to time, according to experimental data on the sorption of Co^{2+} ions at (1) 20, (2) 40, and (3) 60°C.

shown in Fig. 1 were determined by both cation exchange and diffusion in the volume of sorbent particles, and the coefficients of diffusion grew along with temperature.

The data on sorption capacity based on temperature and period of sorption were processed using pseudo-first and pseudo-second order models:

$$\log(A_e - A_t) = \log A_e - \frac{k_1}{2.303} t, \quad (2)$$

$$\frac{t}{A_t} = \frac{1}{k_2 A_e^2} + \frac{1}{A_e} t, \quad (3)$$

where k_1 is the sorption rate constant of the pseudo-first order model, k_2 is the sorption rate constant of the pseudo-second order model. A_e and A_t are the sorption capacities at equilibrium and time t , respectively.

Apparent pseudo-rate constants k_1 and k_2 , and the corresponding squares of coefficients R^2 showing the correctness of the correlation with the kinetic models of pseudo-first and pseudo-second orders, are given in Table 1.

The data presented in Table 1 show that sorption in the analyzed time interval is best described by a pseudo-second order model, as indicated by the corresponding coefficients of correlation.

Linear dependences obtained from experimental data using the pseudo-second order equation are shown in Fig. 2.

We used the equation substantiated in [23] to describe the kinetics of topochemical reactions, which include the process now under study:

$$A_t = A_m k t \frac{1}{1 + k t}, \quad (4)$$

where k is a constant with the dimension $(\text{time})^{-1}$; t is the period of sorption; A_t and A_m are the current and maximum values of sorption capacity.

Table 1. Results from processing the kinetic curves of sorption of Co^{2+} ions by chemical kinetics models

$T, ^\circ\text{C}$	Kinetic model			
	pseudo-first order		pseudo-second order	
	$k_1 \times 10^{-2}, \text{min}^{-1}$	R^2	$k_2, \text{g mmol}^{-1} \text{min}^{-1}$	R^2
20	0.038	0.988	0.27	0.9976
40	0.018	0.9972	0.061	0.9849
60	0.053	0.9388	0.18	0.9968

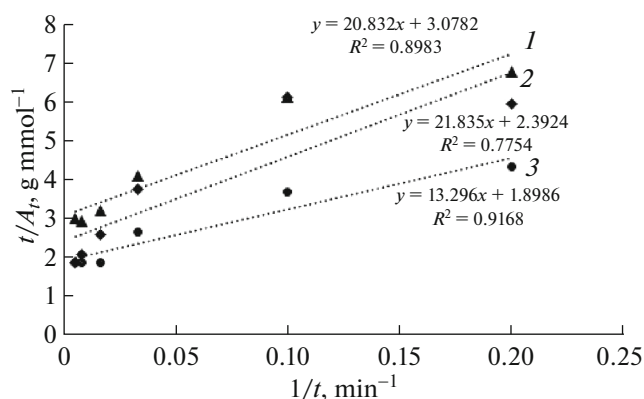


Fig. 3. Dependences of the reciprocal value of the sorption capacity of calcium silicate $1/A_t$ from $1/t$ at different temperatures: (1) 20, (2) 40, and (3) 60°C.

To determine constants k and A_m , Eq. (4) is transformed into a linear equation of the form ($y = a + bx$):

$$\frac{1}{A_t} = \frac{1}{A_m} + \frac{1}{ktA_m} \quad (a = 1/A_m; b = 1/(A_mk)). \quad (5)$$

Linear kinetic equations were obtained for different sorption temperatures (Fig. 3): $y = 20.832x + 3.0782$, $R^2 = 0.8983$ (20°C); $y = 21.835x + 2.3924$, $R^2 = 0.7754$ (40°C); $y = 13.296x + 1.8986$, $R^2 = 0.9168$ (60°C). The values were determined using equations k equal to 0.15, 0.11, and 0.14 min^{-1} for the corresponding temperatures. Calculated values of maximum sorption capacity A_m were 0.32 mmol g^{-1} for 20°C, 0.42 mmol g^{-1} for 40°C, and 0.53 mmol g^{-1} for 60°C.

Data on Co^{2+} ions sorption silicate sorbent, measured at different temperatures (293, 313, and 333 K), were used to calculate the change in Gibbs free energy (ΔG), enthalpy changes (ΔH°), and changes in entropy (ΔS°) according to the formulas

$$\Delta G = -RT \ln k_c, \quad (6)$$

$$\ln k_c = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT}, \quad (7)$$

$$\Delta G = \Delta H^\circ - T\Delta S^\circ, \quad (8)$$

where k_c is the constant of equilibrium, T (K) is temperature, and R (8.314 $\text{J mol}^{-1} \text{K}^{-1}$) is the universal gas constant.

Table 2. Thermodynamic parameters of sorption of Co^{2+} ions by silicate sorbent

ΔH° , kJ/mol	ΔS° , J/(mol K)	ΔG° , kJ/mol		
		293 K	313 K	333 K
−9.91	30.4	−18.82	−19.4	−20.03

The thermodynamic parameters of Co^{2+} ions adsorption are summarized in Table 2. The negative values of ΔG° given in Table 2 confirm that sorption is spontaneous. The drop in ΔG° at higher temperatures indicates they favor adsorption.

To reach a conclusion about the diffusion parameters of the sorption of Co^{2+} ions, the equations given by Timofeev in [24] were used for highly dispersed materials:

$$\Upsilon_t = \Upsilon_0 + (2S/V)(\sqrt{t\sqrt{D}})/\sqrt{\pi}, \quad (9)$$

where Υ_t , A_t/A_m is the relative sorption; S is the specific surface area of the sorbent, $\text{cm}^2 \text{g}^{-1}$; V is the volume of the sorbent sample, cm^3 ($V = m/\rho$, where m is the mass of the sorbent, g; and ρ is the density of the sorbent, g cm^{-3}); t is time, s; D is the coefficient of diffusion, $\text{cm}^2 \text{s}^{-1}$; and $\pi = 3.14$.

Experimental data are required when using this approach. According to Eq. (4), Υ_t is less than 0.5, but such quantities of relative sorption are possible at periods of sorption $t < 1/K_t$. It is difficult to obtain them experimentally, so values A_m and k for the selected temperatures were determined by calculating $\Upsilon_t = A_t/A_m$ at times $t < 1/K_t$ using Eq. (5) (Table 3).

We constructed dependences $\Upsilon_t = f(\sqrt{t})$ and the linear equations presented in Fig. 4 using the data presented in Table 3 for temperatures 20, 40, and 60°C, respectively:

$$y = 0.0282x - 0.0725,$$

$$R^2 = 0.9922;$$

$$y = 0.0234x - 0.071,$$

$$R^2 = 0.9755;$$

$$y = 0.0272x - 0.0718,$$

$$R^2 = 0.9958.$$

Slope coefficients at x according to Eq. (9) were $(2S/V)\sqrt{D}/\sqrt{\pi} = 0.0282$ for a temperature of 20°C; 0.0234 for a temperature of 40°C; and 0.0272 for a temperature of 60°C. All data for calculating the coefficient of diffusion were determined: volume V of the sorbent ($m = 0.05$ g; density, 2.25 g cm^{-3}) and specific surface area ($22 \text{ m}^2 \text{g}^{-1}$). The coefficients of ion diffusion calculated using the described procedure were 0.25×10^{-14} , 0.16×10^{-14} , $0.23 \times 10^{-14} \text{ cm}^2 \text{s}^{-1}$ for temperatures 20, 40, and 60°C, respectively.

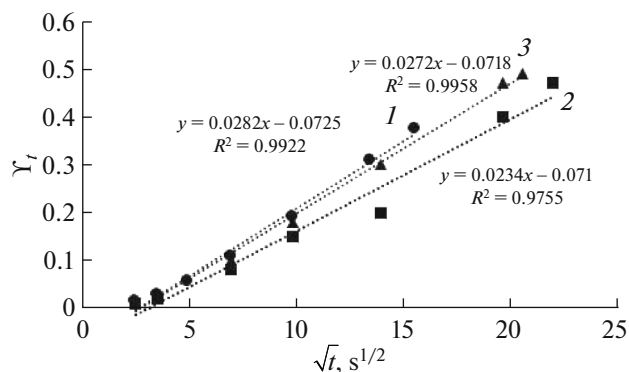
CONCLUSIONS

The kinetic and diffusion parameters of Co^{2+} ions sorption were studied under static conditions with a sorbent based on industrial waste (borogypsum) using aqueous solutions of cobalt chloride with no salt background. The hydrothermal treatment of borogypsum

Table 3. Data for calculating coefficients of diffusion

$t = 20^{\circ}\text{C}, A_m = 0.32 \text{ mmol g}^{-1}$				$t = 40^{\circ}\text{C}, A_m = 0.42 \text{ mmol g}^{-1}$				$t = 60^{\circ}\text{C}, A_m = 0.53 \text{ mmol g}^{-1}$			
$t, \text{ s}$	$A_t, \text{ mmol/g}$	$\sqrt{t}, \text{ s}^{1/2}$	$\Upsilon_t = A_t/A_m$	$t, \text{ s}$	$A_t, \text{ mmol/g}$	$\sqrt{t}, \text{ s}^{1/2}$	$\Upsilon_t = A_t/A_m$	$t, \text{ s}$	$A_t, \text{ mmol/g}$	$\sqrt{t}, \text{ s}^{1/2}$	$\Upsilon_t = A_t/A_m$
6	0.0048	2.45	0.015	6	0.0046	2.45	0.011	6	0.0073	2.45	0.014
12	0.0093	3.46	0.029	12	0.009	3.46	0.021	12	0.014	3.46	0.026
24	0.018	4.9	0.056	48	0.034	6.93	0.081	48	0.053	6.93	0.1
48	0.034	6.93	0.11	96	0.063	9.8	0.15	96	0.097	9.8	0.18
96	0.062	9.8	0.19	192	0.11	13.9	0.2	192	0.16	13.9	0.3
180	0.099	13.4	0.31	384	0.17	19.6	0.4	384	0.25	19.6	0.47
240	0.12	15.49	0.375	480	0.197	21.9	0.47	420	0.26	20.5	0.49

(2 h at a pressure of 1.7 atm) produced a material with density 2.25 g cm^{-3} and specific surface area $22 \text{ m}^2 \text{ g}^{-1}$. The sorption capacity increase from 0.336 to 0.54 mmol g^{-1} when the temperature was raised from 20 to 60°C . The sorption in the analyzed time interval was described best by a pseudo-second order model with such thermodynamic parameters as standard enthalpy (ΔH°), standard entropy (ΔS°), and standard free energy (ΔG°). The reduction of ΔG° at higher temperatures indicates that higher temperatures favor adsorption. The coefficients of ion diffusion, calculated using the described procedure, were 0.25×10^{-14} , 0.16×10^{-14} , and $0.23 \times 10^{-14} \text{ cm}^2 \text{ s}^{-1}$ for temperatures 20 , 40 , and 60°C , respectively. If we assume the experimental data on sorption and kinetics were determined with errors of at least $10\text{--}15\%$, the resulting calculated data on coefficients of diffusion must be considered approximate, and similar data on other sorbents of similar composition are needed for a comparative analysis.

**Fig. 4.** Dependences of relative sorption on \sqrt{t} at: (1) 20°C , (2) 40°C , and (3) 60°C .

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CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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