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SYNTHESIS AND PROPERTIES OF INORGANIC COMPOUNDS

Synthesis of Calcium Aluminosilicates from Nanostructured Synthetic Na Zeolites and Study of Their Sorption Properties

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Abstract—Calcium aluminosilicates synthesized by chemical modification of nanostructured synthetic Na zeolites were characterized. The sorption properties were studied for calcium aluminosilicates with $SiO_2 : Al_2O_3$ ratios of 2 : 1, 4 : 1, 6 : 1, 8 : 1, and 10 : 1. The maximum capacity of these compounds to sorb Cs^+ ions under static conditions from solutions without salt background was shown to reach 1.45 mmol/g (192.7 mg/g). The results of this work allow one to consider these compounds as promising materials for the sorption and immobilization of long-lived radionuclides.

Keywords: calcium aluminosilicates, chemical modification, sorption, cesium, maximum sorption capacity

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INTRODUCTION

Long-lived radionuclides are dangerous to living organisms when accumulated above the permissible limits, and enough data has been accumulated on many elements that prove their negative impact on biological objects [1, 2]. To eliminate their negative impact on biospheric biological objects, various methods and materials are used to concentrate these radionuclides as highly stable compounds with subsequent processing and disposal. In this context, it was of interest to obtain efficient materials for the sorption and immobilization of long-lived radionuclides, in particular, ^{137}Cs.

As such materials, various natural and synthetic aluminosilicates (zeolites) are widely used [3–10].

Previously [11–13], we studied the sorption of Cs^+ ions by X-ray amorphous nanostructured potassium aluminosilicates ($KAlSi_xO_y \cdot nH_2O$, where $x = 1–5$, $y = 2(x + 1)$) under static conditions, including the sorption from solutions with different salt backgrounds. The kinetic parameters of the sorption were investigated, and the activation energy of sorption was found. It was determined that the maximum capacity of $KAlSi_3O_8 \cdot 1.5H_2O$ to sorb Cs^+ ions under static conditions from solutions without salt background reaches 3.7 mmol/g, and the interphase distribution coefficient K_d of ions at a solid : liquid ratio of 1 : 4000 reaches 1×10^5 mL/g. Aluminosilicates of a similar composition can be obtained from various silicon-containing waste, e.g., phytogenic waste [14].

Earlier [15, 16], we also investigated the capacity of synthetic calcium aluminosilicates to sorb Sr^{2+} and Cs^+ from aqueous solutions of various ionic compositions under static conditions. To obtain synthetic calcium aluminosilicates with Al : Si ratios of 2 : 2, 2 : 6, and 2 : 10, salts of polysilicic acid with a given $SiO_2 : M_2O$ ratio (M is an alkali metal (potassium)) were initially synthesized. The obtained solution of liquid potassium glass was mixed with a solution of calcium and aluminum chlorides according to the stoichiometric coefficients of the equation

$$8KOH + nSiO_2 \cdot kH_2O + CaCl_2 + 2AlCl_3 \cdot 6H_2O + H_2O = CaAl_2Si_nO_{(n+2)2} \cdot mH_2O + 8KCl + H_2O, \text{ where } n = 2; 6; 10. \quad (1)$$

We detected that the $SiO_2 : Al_2O_3$ ratio in the aluminosilicates produced by the described method is within the error of measuring the specified molar ratios of silicon and aluminum oxides, but if the total charge of cations exceeds the aluminum content in the synthesized aluminosilicate, then it follows that the synthesis product may contain other phases: hydroxides or carbonates. If aluminum during the replacement of silicon is in the tetrahedral environment of oxygen as an anionic complex, then the NMR spectra contain peaks of aluminum in the octahedral environ-

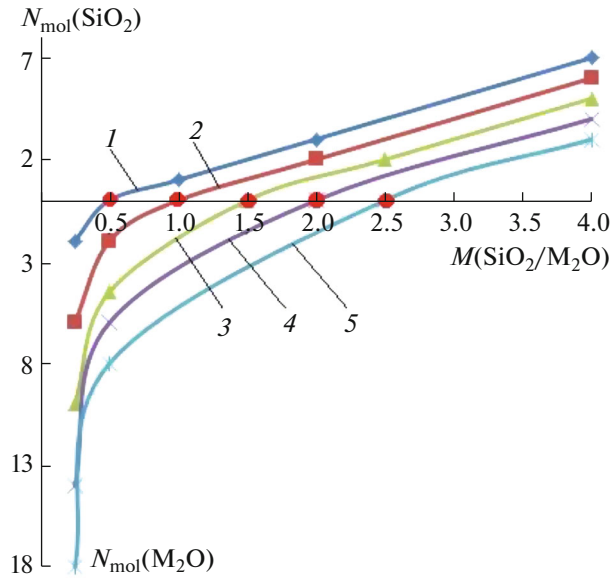

Fig. 1. Dependence of the composition of aluminosilicates with given k on M_{liq} of the liquid glass in the system $M_2O–SiO_2–AlCl_3–H_2O$: (*1*) $MAlSiO_4$, (*2*) $MAlSi_2O_6$, (*3*) $MAlSi_3O_8$, (*4*) $MAlSi_4O_{10}$, and (*5*) $MAlSi_5O_{12}$.

ment of oxygen, which confirms the presence of aluminum hydroxide in the studied composition. The aluminosilicates obtained by this method are not single-phase; the end products also contain insoluble calcium carbonates, which interact with the sorbate.

These compounds, which are obtained from aqueous solutions by a low-temperature and rapid method, are promising as a sorption material for removing ^{137}Cs from various aqueous media and can also be used as an inorganic base for obtaining solid-state matrices to reliably immobilize this radionuclide, e.g., when conditioning spent sorbent [17–20]. Silicates as raw materials for such matrices meet the requirements imposed on them (high chemical and radiation stability and mechanical strength) [21].

It is known that salts of polysilicic (aluminosilicic) acids, which include aluminosilicates, are easily subjected to chemical modification, namely, the replacement of cations that compensate for the excess charge of the aluminum–oxygen tetrahedron with cations of salts of stronger acids.

We synthesized Na aluminosilicates with given $SiO_2 : Al_2O_3$ ratios of 2 : 1, 4 : 1, 6 : 1, 8 : 1, and 10 : 1. They were then chemically modified with calcium ions by treatment in a calcium salt solution at a temperature of no more than 100°C. After the modification, the degrees of replacement of Na^+ ions with calcium were compared, and under the same treatment conditions, data were obtained on the composition, morphology, and capacity of calcium aluminosilicates with different $SiO_2 : Al_2O_3$ ratios to sorb Cs^+ ions.

EXPERIMENTAL

Synthesis and chemical modification of samples. The initial substances for the synthesis of aluminosilicates with given $SiO_2 : Al_2O_3$ ratios of 2 : 1, 4 : 1, 6 : 1, 8 : 1, and 10 : 1 were pure $AlCl_3·6H_2O$ (GOST 2463-80), aqueous silicic acid $SiO_2·nH_2O$ (GOST 4214-78), analytically pure NaOH, and pure $CaCl_2$.

Samples of aluminosilicates $M_2Al_2Si_{2k}O_{2(2k+2)}·nH_2O$ (M = Na; k = 1, 2, 3, 4, 5) with varying $SiO_2 : Al_2O_3$ ratios (2 : 1, 4 : 1, 6 : 1, 8 : 1, and 10 : 1) were synthesized by dissolving silica in sodium hydroxide to obtain liquid glass with given silicate modulus $M_{liq} = SiO_2 : M_2O$ (M = Na) and then mixing the resulting solution with an aluminum chloride solution. The ratios of the components were calculated in such a way as to obtain aluminosilicates with a given value of k = Si : Al and a final pH value of the solution of 7. Figure 1 presents the dependence of the composition of aluminosilicate (point of intersection with the abscissa axis) on the modulus of the liquid glass when obtained from solutions with a final pH of the solution of 7.

The selected conditions are met if the prepared liquid glass solution has a modulus of $M_{liq} = 1/2k$, and the components are taken according to the stoichiometric coefficients of the equation

$$4M_2O + 2kSiO_2 + 2AlCl_3 = M_2Al_2Si_{2k}O_{2(2k+2)} + 6MCl. \quad (2)$$

The formed precipitate was separated from the solution through a Blue Ribbon filter; during washing in the filtrate, the presence of chloride ions was monitored. Precipitates were dried until constant weight at a temperature of 100–105°C.

Table 1. Elemental composition, content of water of crystallization, and specific surface area of Na aluminosilicates

Formula	H_2O_{cryst}, mol	Elemental composition, wt %			Specific surface area S_{sp}, m^2/g
		Na	Si	Al	
$NaAl_2Si_2O_8$	3.0	18.7	42.1	39.0	254.5
$NaAl_2Si_4O_{12}$	3.4	13.3	56.8	29.8	143.9
$NaAl_2Si_6O_{16}$	4.5	13.7	63.5	22.6	225.6
$NaAl_2Si_8O_{20}$	5.5	9.5	70.9	19.5	154.0
$NaAl_2Si_{10}O_{24}$	6.0	6.9	77.0	16.1	304.9

To obtain Ca zeolites, the initial Na aluminosilicates were kept in a saturated solution of calcium chloride for 2 h at low boiling. Then, the precipitate was separated from the solution through a Blue Ribbon filter, washed with distilled water until the test for chloride ions was negative, and dried at 105°C.

Sorption experiments. Experiments on the sorption of Cs^+ ions were carried out under static conditions at a solid : liquid ratio of 1 : 100 and a temperature of 20°C from aqueous solutions of cesium chloride without salt background with various initial concentrations of Cs^+ ions up to 75 mmol/L while stirring on an RT 15 Power magnetic stirrer (Ika Werke, Germany) for 3 h.

Analysis methods. The amount of water of crystallization in the obtained zeolites (Table 1) was determined from the difference in weight between the sample dried at 100–105°C and the sample annealed at 800°C. The amount of water of crystallization was also calculated from the thermal curves recorded with a MOM Q-1500 D Paulik-Paulik-Erdey derivatograph (Hungary) with a temperature determination accuracy of ±5°C during annealing samples to 800°C at a rate of 5 deg/min in an open platinum crucible in air.

The elemental composition of the synthesized aluminosilicates was quantitatively determined by energy-dispersive X-ray fluorescence spectroscopy with a Shimadzu EDX 800 HS spectrometer (Japan). A portion of the sample was ground in an agate mortar with boric acid (2 : 1 by weight) and placed in a mold 20 mm in diameter. The emitter tablet was pressed for 2 min at a pressure of 5000 kg, after which it was placed in a spectrometer and measurements were taken. The exposure time was 100 s in each energy channel, the radiation source was an X-ray tube with an Rh anode, the concentration of elements was calculated by the method of fundamental parameters using the software package of the spectrometer without taking light elements into account.

The specific surface area of the samples was determined by low-temperature nitrogen adsorption with a Sorbtometer-M instrument (Russia).

The X-ray powder diffraction patterns of the precipitates were recorded with a Bruker D8 ADVANCE automatic diffractometer (Germany; CuK_α radiation). The X-ray powder diffraction patterns were identified using the EVA software with the PDF-2 database.

The study of the morphological characteristics and the confirmation of the local elemental composition were carried out with a Hitachi S5500 high-resolution electron microscope (Japan).

The ^{27}Al, ^{29}Si, and ^1H 12-kHz MAS NMR spectra were recorded with a Bruker ADVANCE AV-300 (Germany; B_0 = 7 T). The external standards in the ^{27}Al spin-echo NMR spectra were diluted aqueous solutions of $AlCl_3 \cdot 6H_2O$ ($[Al(H_2O)_6]^{3+}$ ion) and NaCl. The ^{29}Si NMR spectra were recorded using the ^{29}Si–{^1H} cross correlation with the suppression of ^{29}Si–^1H interactions, and the chemical shifts of ^{29}Si and ^1H were measured from the signal of tetramethylsilane. The accuracy of determining the chemical shifts and integral intensities of signals were 1 ppm and 10%, respectively.

The contents of Cs^+ ions in the initial solutions and the filtrates after the sorption were determined by atomic absorption spectrometry on a Solaar M6 double-beam spectrometer (Thermo Scientific, USA) using the 852.1 nm analytical line. The detection limit for cesium ions in aqueous solutions is 0.01 μg/mL.

The sorption capacity A_a (mmol/g) of the studied samples was calculated as

$$A_c = \frac{(C_{in} - C_e)}{m} V, \quad (3)$$

where C_{in} is the initial concentration of Cs^+ ions in the solution, mmol/L; C_e is the equilibrium concentration of Cs^+ ions in the solution, mmol/L; V is the volume of the solution, L; and m is the weight of the sorbent, g.

RESULTS AND DISCUSSION

Characterization of the Initial and Modified Aluminosilicates

All the X-ray powder diffraction patterns of the samples of Na aluminosilicates with SiO_2 : Al_2O_3 ratios from 2 to 10 show broad maxima at small 2θ. The absence of clear Bragg peaks confirms the X-ray amorphism of the samples, which is determined by their low degree of crystallinity and the nanosize of

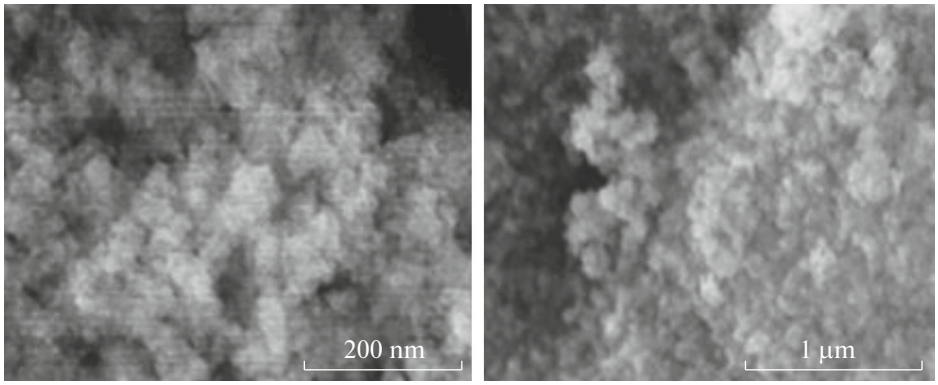

Fig. 2. Scanning electron microscopy images of sodium aluminosilicate.

crystallites. It was found that, with an increase in the $SiO_2 : Al_2O_3$ ratio in the series 2 : 1, 4 : 1, 6 : 1, 8 : 1, 10 : 1, the interplanar spacings increase sequentially from 3.359 to 3.711 Å.

Table 1 presents the composition, specific surface area, and content of water of crystallization in the obtained samples,

The produced samples of the composition $M_2Al_2Si_xO_{2(x+4)}\cdot nH_2O$ (M = Na) are nanodispersed powders with a developed surface, which consist of 10–20-nm particles coalesced into agglomerates. Figure 2 shows the scanning electron microscopy images of the sample of nanostructured sodium aluminosilicate with a $SiO_2 : Al_2O_3$ ratio of 2 : 1. No fundamental differences were detected in the morphology of samples with different $SiO_2 : Al_2O_3$ ratios.

The studied sodium aluminosilicates were found to be characterized by an increase in the specific surface area with an increase in the $SiO_2 : Al_2O_3$ ratio, which reaches more than 300 m^2/g (Table 1).

Table 2 presents the elemental composition of Ca zeolites obtained by the modification of Na aluminosilicates with various $SiO_2 : Al_2O_3$ ratios.

In the samples of calcium aluminosilicates obtained by this method of chemical modification of the initial sodium aluminosilicates, no sodium was found, and the specific surface area of the samples is lower by a factor of 2–2.5. The complete replacement of sodium cations by calcium in Na aluminosilicates should be explained by the closeness of the ionic radii (0.104 and 0.116 nm for calcium and sodium ions, respectively). The morphology of calcium aluminosilicates after the modification does not differ from that of the initial Na-form aluminosilicates.

NMR Study of Aluminosilicates

The ^{27}Al NMR spectra (Fig. 3a; Table 3) show signals near ~0 and ~50 ppm, which can be assigned to octa- and tetracoordinated aluminum atoms, respectively [22, 23]. The signals of the tetracoordinated atoms are more intense and more noticeable because of their smaller width. The intensity of these signals increases with increasing silicon content in the sample, and for $CaAl_2Si_{10}O_{24}$, it becomes difficult to determine the presence of a signal near 0 ppm.

The ^{29}Si MAS NMR spectra of the studied compounds (Fig. 3b) each contain one broad signal, the position of which regularly depends on the composition of the sample and is determined by the ratio of the Si–O–Si and Si–O–Al structural fragments. Whereas the signal of $CaAl_2Si_{10}O_{24}$ has a shift of –98.5 ppm, the signal of $CaAl_2Si_2O_8$, the signal position is –86.5 ppm.

The ^1H MAS NMR spectra show signals at 4.6–4.8 ppm, which are composite signals of the samples $CaAl_2Si_6O_{16}$ and $CaAl_2Si_8O_{20}$.

Table 2. Elemental composition of Ca zeolites obtained by modification of Na aluminosilicates

$SiO_2 : Al_2O_3$ ratio in initial Na aluminosilicate	Assumed formula	Elemental composition, wt % (mol)			
		Na	Ca	Si	Al
2 : 1	$CaAl_2Si_2O_8$	–	27.0 (0.67)	28.5 (1.0)	32.6 (1.2)
4 : 1	$CaAl_2Si_4O_{12}$	–	24.4 (0.61)	50.9 (1.8)	24.1 (0.89)
6 : 1	$CaAl_2Si_6O_{16}$	–	22.0 (0.55)	53.1 (1.9)	21.2 (0.78)
8 : 1	$CaAl_2Si_8O_{20}$	–	19.6 (0.49)	63.8 (2.3)	15.9 (0.58)
10 : 1	$CaAl_2Si_{10}O_{24}$	–	14.4 (0.36)	68.5 (2.5)	17.1 (0.63)

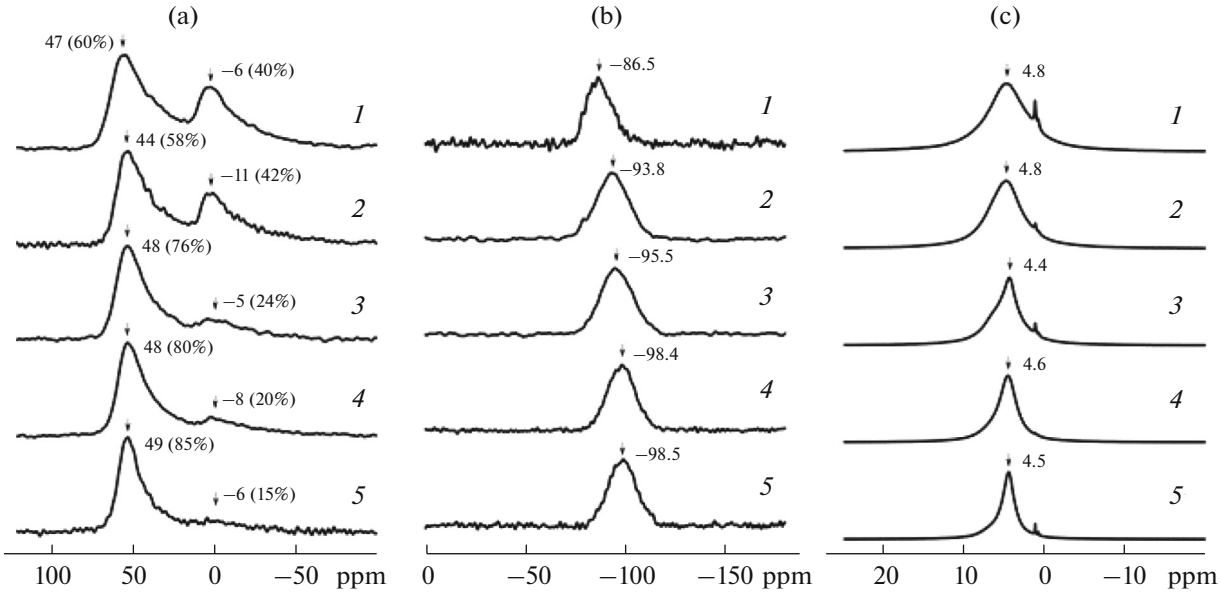

Fig. 3. (a) ^{27}Al, (b) ^{29}Si, and (c) ^{1}H MAS NMR spectra of the studied calcium aluminosilicates with $SiO_2 : Al_2O_3$ ratios of (*1*) 2 : 1, (*2*) 4 : 1, (*3*) 6 : 1, (*4*) 8 : 1, and (*5*) 10 : 1.

Based on the data on the elemental composition and NMR spectra, Ca^{2+} ions in the samples are in excess. If it is taken into account that some (up to 60–80%) of the Al atoms are in the tetrahedral coordination and belong to the zeolite, then the same number of moles of charges should be compensated by the charges of cations, in this case, calcium ions, and the excess part of calcium should be assigned to carbonate calcium obtained during the synthesis, which is confirmed by experiments. The rest of the Al atoms (20–40%), which are in the octahedral coordination with oxygen, should be attributed to hydroxide $Al(OH)_3$.

Thus, it should be concluded that the compounds obtained have the phase and elemental compositions presented in Table 4.

Sorption of Cs^+ Ions

Figure 4 illustrates the dependences of the sorption capacity of the samples of calcium aluminosilicates on the initial concentration of Cs^+ ions in the solution.

To determine the parameters of the isotherms, the experimental data on the sorption were processed in the coordinates of the linear Langmuir equation

$$1/A_s = [1 / (A_m K_L C_e)] + 1/A_m, \quad (y = ax + b), \qquad (4)$$

where A_s and A_m are the sorption capacity and the maximum sorption capacity, respectively, mmol/kg; K_L is the Langmuir constant, L/mmol; and C_e is the equilibrium concentration, mmol/L.

Table 5 presents the graphically determined parameters of the Langmuir equations. The Langmuir model is seen to be suitable for describing the sorption of Cs^+ ions by the studied compounds, as evidenced by the corresponding correlation coefficients.

The data obtained show certain differences in maximum sorption capacity between the samples with different $SiO_2 : Al_2O_3$ ratios. The capacity of aluminosilicates 3–5 to sorb Cs^+ ions is higher than that of samples 1 and 2. The maximum sorption capacity A_m was found for sample 3 and reaches 1.45 mmol/g (192.7 mg/g) (Fig. 4).

Table 3. Parameters of the ^{27}Al NMR spectra

No. of compound	Compound	Component 1 (tetrahedron)		Component 2 (octahedron)	
		CS*, ppm	area, %	CS, ppm	area, %
1	$CaAl_2Si_2O_8$	47	60	−6	40
2	$CaAl_2Si_4O_{12}$	44	58	−11	42
3	$CaAl_2Si_6O_{16}$	48	76	−5	24
4	$CaAl_2Si_8O_{20}$	48	80	−8	20
5	$CaAl_2Si_{10}O_{24}$	49	85	−6	15

*CS stands for Chemical shift.

Table 4. Phase and elemental compositions and specific surface area of calcium aluminosilicates (samples after drying at 105°C)

Overall formula	H_2O_{cryst}, mol	Elemental composition, wt % (mol)			Phase composition, %	Specific surface area S_{sp}, m^2/g (relative density d, g/cm^3)
		Si	Al	Ca		
$Ca_{0.67}Al_{1.20}Si_{1.0}O_{3.44}(OH)_{1.44}(CO_3)_{0.31}$	4	28.5 (1.0)	32.6 (1.2)	27.0 (0.67)	Amorphous phase (68.5%), hydroxide $Al(OH)_3$ (23.8%), calcite $CaCO_3$ (7.7%)	81.0 (2.08)
$Ca_{0.61}Al_{0.89}Si_{1.8}O_{4.63}(OH)_{1.12}(CO_3)_{0.35}$	5	50.9 (1.8)	24.1 (0.89)	24.4 (0.61)	Amorphous phase (77%), hydroxide $Al(OH)_3$ (15.5%), calcite $CaCO_3$ (7.5%)	67.5 (2.2)
$Ca_{0.55}Al_{0.78}Si_{1.9}O_{4.98}(OH)_{0.56}(CO_3)_{0.25}$	6	53.1 (1.9)	21.2 (0.78)	22.0 (0.55)	Amorphous phase (86.1%), hydroxide $Al(OH)_3$ (8.3%), calcite $CaCO_3$ (5.6%)	71.8 (2.03)
$Ca_{0.49}Al_{0.58}Si_{2.3}O_{5.53}(OH)_{0.35}(CO_3)_{0.26}$	7	63.8 (2.3)	15.9 (0.58)	19.6 (0.49)	Amorphous phase (89.6%), hydroxide $Al(OH)_3$ (5%), calcite $CaCO_3$ (5.4%)	123.3 (1.99)
$Ca_{0.36}Al_{0.63}Si_{2.5}O_{6.07}(OH)_{0.28}(CO_3)_{0.09}$	8	68.5 (2.5)	17.1 (0.63)	14.4 (0.36)	Amorphous phase (94.4%), hydroxide $Al(OH)_3$ (3.8%), calcite $CaCO_3$ (1.8%)	134.3 (2.0)

Table 5. Parameters of the Langmuir equations in the sorption of Cs^+ ions by aluminosilicates

Compound	Parameters of Langmuir equation		
	A_m, mmol/g	k, L/mmol	R^2
$CaAl_2Si_2O_8$	0.841	0.11	0.9842
$CaAl_2Si_4O_{12}$	0.844	0.21	0.9900
$CaAl_2Si_6O_{16}$	1.45	1.18	0.9924
$CaAl_2Si_8O_{20}$	1.37	0.55	0.9776
$CaAl_2Si_{10}O_{24}$	1.34	0.39	0.9772

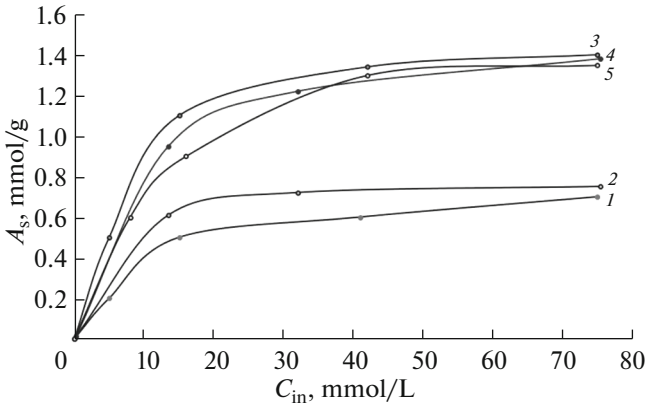

Fig. 4. Isotherms of sorption of Cs^+ ions by the samples of calcium aluminosilicates with $SiO_2 : Al_2O_3$ ratios of (*1*) 2 : 1, (*2*) 4 : 1, (*3*) 6 : 1, (*4*) 8 : 1, and (*5*) 10 : 1.

CONCLUSIONS

A number of nanostructured calcium aluminosilicates obtained from Na aluminosilicates with $SiO_2 : Al_2O_3$ ratios of 2 : 1, 4 : 1, 6 : 1, 8 : 1, and 10 : 1 were synthesized and studied. It was determined that calcium aluminosilicates are X-ray amorphous and have a specific surface area from 67 to 134 m^2/g, which increases with increasing $SiO_2 : Al_2O_3$ ratio. The phase and elemental composition was determined, and the morphology of the obtained compounds was studied.

The sorption of Cs^+ ions under the considered conditions is described by the Langmuir equation. The maximum sorption capacity is observed for the synthesized calcium aluminosilicate with a $SiO_2 : Al_2O_3$ ratio of 6 : 1 and is 1.45 mmol/g (192.7 mg/g).

It was shown that the sorbents based on calcium aluminosilicates obtained by chemical modification of Na aluminosilicates by their treatment in a calcium chloride solution can be recommended as efficient sorbents for removing Cs^+ ions from aqueous solutions under static conditions.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

AUTHOR CONTRIBUTIONS

P.S. Gordienko and S.B. Yarusova conceived and designed the experiment. I.A. Shabalin and E.A. Nekhlyudova synthesized the samples. A.B. Slobodyuk made the NMR study of the samples. O.O. Shichalin and E.K. Papynov co-processed data and co-wrote the paper. V.G. Kuryavyi studied the morphological characteristics of the samples. N.V. Polyakova analyzed the elemental composition of the samples by energy-dispersive X-ray fluorescence spectroscopy. Yu.A. Parot'kina carried out atomic absorption spectrometry studies. All authors discussed the results.

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