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ION EXCHANGE PROPERTIES OF IRRADIATED AND CHEMICALLY MODIFIED CLINOPTILOLITE REGARDING TO Cs⁺ AND Sr²⁺

L.Akhalbedashvili¹*, G. Todradze¹, N. Kekelidze², Y.Keheyan³, G.Yeritsyan⁴, R. Gevorkyan⁵

¹Tvalchrelidze Caucasian Institute of Mineral Resources, 85 Paliashvili Street, Tbilisi 0162, Georgia
 ²Iv. Javakhishvili Tbilisi State University, 1 Chavchavdze Ave., Tbilisi 0218, Georgia
 ³University of Roma "La Sapienza", 5 Aldo Moro P., Roma 00185, Italy
 ⁴Yerevan Physics Institute, 2 Alikhanyan Brothers Street, Yerevan 375036, Armenia
 ⁵Yerevan State University, 1 Alex Manoogian Street, Yerevan 0025, Armenia

The ion exchange sorption of Cs^+ and Sr^{2+} on clinoptilolite (CL) from deposit of Armenia in initial, modified by e- and γ irradiation, decationated with a hydrochloric acid, and cation-contained forms was studied. On the basis of received data the exchange capacity E for Sr^{2+} and Cs^+ , distribution factor K_d , sorption factor K_s were determined. It was shown that untreated CL(Ar), decationated CL(Ar)H and Cacontaining forms are characterized by a big ion exchange activity on strontium and cesium to compare with irradiated samples and the most perspective ion exchange sorbents for these ions are CL samples modified by a chemical way.

INTRODUCTION

Zeolite materials (porous hydrated crystalline alumosilicates) are gaining increasing importance in ion exchange, molecule sieving and catalysis. Their ion exchange capacity is one of the main parameters which characterizes sorption and technological properties of high-silicon natural zeolites. They have the attraction of low cost in addition to high thermal and chemical stability, good resistance to α , β and γ radiation and high capacity and selectivity for certain monovalent and divalent cations [1].

Zeolite-containing rocks are used in wastewater purification to remove heavy metals [2, 3], toxic and radioactive elements [4], especially radionuclides of cesium and strontium of nuclear fission products [5]. Considerable interest in the zeolites as cation exchange media has been generated in the atomic energy industry. As far back as 1960 Mercer [6] studied the use of natural zeolite, clinoptilolite, for decontamination of condensate wastes containing trace amounts of cesium and strontium radioisotopes.

MATERIALS AND METHODS

The aim of the work was to study Armenian natural zeolite clinoptilolite (ArCL), modified with irradiation (for the first time), the thermal and chemical ways, in ion exchange sorption of strontium and cesium stable nuclides. So were studied:

- the ion exchange sorption of Cs⁺ and Sr²⁺ from aqueous solutions of their salts on cation-modified samples;
- the ion exchange sorption of Sr²⁺and Cs⁺ on e- and γ-irradiated zeolite materials;
- the influence of concentration and volumes of model solutions on cesium and strontium ion exchange sorption;
- the influence of heat treatment and granulation degree;
- the ion exchange sorption of Cs⁺ and Sr²⁺ from mixture of their salts on cation-modified samples.

The exchange capacity E for Sr^{2+} and Cs^+ , distribution factor K_d , sorption factor K_s were calculated.

The clinoptilolite-type zeolite was collected from Armenia (Nor Kokhb deposit in Noyemberian region) and enriched with CL (content of CLphase increased from 70% up to 80–85%). It has been characterized as typical heulandite type III zeolite and contains minor amounts of quartz, plagioclase, montmorillonite, opal-CT, calcite, and other rock materials. Some zeolite samples were exposed in air to doses of gamma-ray irradiation, namely 200, 700 kGy at 293 K using a ⁶⁰Co γ -shell, and to different doses of e-irradiation, namely 10^{12} - 10^{15} e/cm² with a ELU-8 linear electron accelerator at 293 K.

The analysis of alkali and alkali-earth cations and impurity transition metals were carried out by atomic adsorption spectrophotometric method with a "Perkin-Elmer" AAnalyst 800 apparatus and capillary electrophoresis method with a CIA system (Milford, MA, USA). The mineralogical transformations of the acid and thermally treated materials were monitored by X-ray diffraction (XRD) using a DRON-2 operating with a graphite monochromator, using CuK α -radiation.

The materials were dried at 393 K, ground in ball mill and subsequently with pestle and mortar so as to pass from 0.25 mm sieves and stored at room temperature. The zeolites were subsequently treated with 0.25% KOH or 0.5% NH₄OH solutions at 323 K for 90 minutes using a 1:10 solid to liquid ratio. The suspensions were filtered and the solids were washed until pH 7. Acid treatment was carried out with 1N HCl at \approx 368 K using the same 1:10 solid to liquid ratio. The samples were treated twice for 40 min. After second treatment the zeolites were filtered under vacuum, washed until pH 7, dried at 333 K and ground gently with pestle and mortar so as to pass through a 0.25 mm sieves. Ca- and Na-forms were prepared from decationated CL(Ar)H by treating with corresponding salts. Finally thermal treatment involved heating of the treated zeolites at 773 K for 8 hours. Chemical composition of modified zeolite samples is presented in Table 1.

 Table 1. Chemical composition (mass %) of initial and chemically treated CL

	1	2	3	4	5	6
Sample	CL(Ar)	CL(Ar) H	CL(Ar)Ca H	CL(Ar)Na H	CL(Ar)K	CL(Ar)NH4
SiO ₂	68.16	78.88	78.3	76.4	69.50	78.00
Al ₂ O ₃	12.29	8.88	8.86	9.45	11.89	8.40
Fe ₂ O ₃	1.48	0.12	0.10	0.27	1.90	2.40
TiO ₂	0.43	0.21	_	_	_	_
SO ₃	0.12	0.04	_	_	_	_
MgO	0.30	0.22	0.24	0.18	0.28	0.36
CaO	6.33	2.38	12.04	1.87	5.60	3.69
K ₂ O	3.46	2.36	1.00	1.21	9.04	0.64
Na ₂ O	0.76	0.06	0.30	8.16	0.72	1.09
H ₂ O	1.14	7.11	_	3.44	_	5.23
MnO	0.23	_	_	_	_	_

Ion exchange sorption was carrying out in flowing (in glass column) and static regimes.

RESULTS AND DISCUSSION

Policationity of CL, the presence of iron oxides (1.5-4%) in their structure reduce exchange properties with respect to cations Cs⁺ and Sr²⁺, and so the preliminary chemical modifying is necessary for extension of ion exchange ability. The analysis on ways of modifying of zeolites with the purpose to remove iron ions and partially to enrich them at the expense of removal of some accompanying minerals shows that optimum is the acid treatment of zeolites with the subsequent exchange of hydrogen on cations of alkaline and alkali-earth metals [7, 8].

Study of ion exchange sorption of strontium and cesium on CL(Ar) modified samples. The research of exchange sorption of strontium and cesium ions in static conditions was carried out on initial CL, modified with HCl, KOH and NH₄OH solutions, calcium and sodium chlorides and for the first time on e- and γ -irradiated samples of Cl(Ar) (Table 2).

Table 2. The irradiated, thermal and chemical treatedsamples, prepared in TSU (1-6), Y-PhyI(7-10), RSU (11-14)

No	Sample type	Treatment		
1	CL(Ar)	initial, thermal treated at 773K		
2	CL(Ar)H	acid treated, thermal treated at 773K		
3		acid, CaCl ₂ treated, thermal		
	CL(AI)Call	treated at 773K		
4	$CL(\Delta r)K$	KOH treated, thermal treated		
	CL(AI)K	at 773K		
5	CI (Ar)NH	NH ₄ OH treated, thermal treated		
		at 773K		
6	CI (Ar)NaH	acid, NaCl treated, thermal treated		
	CL(II)I uII	at 773K		
7	CL(Ar) Ir12	e-irradiated with dose 10 ¹² e/cm ²		
8	CL(Ar) Ir13	e-irradiated with dose 10^{13} e/cm ²		
9	CL(Ar) Ir14	e-irradiated with dose 10 ¹⁴ e/cm ²		
10	CL(Ar) Ir15	e-irradiated with dose 10 ¹⁵ e/cm ²		
11	$CI(\Lambda r)H^*$	acid treated, γ -irradiated		
11	CL(AI)II	with dose 200 kGy		
12	CI (Ar)Ca*	acid and CaCl_2 treated, $\gamma\text{-}irradiated$		
	CL(/II)Cu	with dose 200 kGy		
13	CL(Ar)H*	acid treated, y-irradiated		
		with dose 700 kGy		
14	CL(Ar)Ca*	acid and $CaCl_2$ treated, $\gamma\text{-}irradiated$		
		with dose 700 kGy		

The obtained values of strontium exchange sorption for various samples, which are schematically represented in a Fig. 1, show that calciumcontaining CL differs with greatest ion exchange activity in relation with strontium whose E (Sr^{2+}) is equal 1.66 meq/g that more than twice exceeds the found value for initial zeolite (0.618 meq/g) and K_s is equal to 98%. The sample calcium ions were completely exchanged with passed strontium. The decationated form CL(Ar)H was also characterized with rather large ion exchange activity E (Sr^{2+}) = 0.916 meq/g and K_s = 96%.





A few other results are received for ion exchange capacity on cesium: neither e-irradiation nor chemical modifying does not influence on ion exchange sorption of cesium (Fig. 2). A possible explanation – the large size of ions (ionic radius of Cs^+ is equal to 0.169 nm) allows them to penetrate only into the large channels of CL. As the radius of the dehydrated Cs^+ ion is similar to that of the ditrigonal siloxane cavity of layer silicates, adsorption of Cs to negatively charged siloxane sites can result in innersphere complexation [9]. The reactivity of the innersphere complexation sites may differ widely. However, other relatively strong complexes may be formed at other surface sites at higher concentrations.





The samples modified by a chemical way – decationated and calcium-containing CL are most perspective ion exchange sorbents for strontium. The complete sorption on CL(Ar)CaH sample confirms the existing data [10] that strontium enters in sorbent mainly on a place of calcium and magnesium (about 84% of sorbed strontium), less (15%) on a place of sodium and almost does not replace

potassium. The sorption process for this sample is differed by equivalence as against initial and decationated. K_s for initial sample is equal to 49% but only 26% of the sum of calcium, magnesium, so-dium and potassium cations are replaced by strontium, rest – either simply physical adsorption or replacement of H⁺ that is less probable.

The ion exchange sorption of Sr^{2+} and Cs^{+} from their solutions on e- and γ irradiated samples. Exchange capacity of strontium on the e-irradiated samples with doses from 10^{12} up to 10^{14} e/cm² decreases in comparison with no irradiated and for a sample with a dose of an irradiation 10^{15} e/cm² grows a little (Fig. 3).



Fig. 3.The dependence of E (Sr^{2+}) and E (Cs^+) on the dose of e-irradiation at C₀=0.1N, size of granules is 0.20–0.25 mm

The exchange capacity E (Cs⁺) practically does not depend on the dose of an irradiation with electrons and is within 0.23–0.30 meq/g. These values are 25% lower as compared with the magnitude of adsorption E (Cs⁺) for the raw zeolite but confirm our assumption that the determining factor is the size of the cation.

So, the e-irradiation reduces the sorption characteristics of samples but at the same time the weak dependence on the dose is observed. It is possible to state that e-irradiation excites alumosilicon-oxygen skeleton increasing the electron density on it and promoting the formation of peroxide ion O_2^- . As a result of originated metastability of equilibrium, the regrouping of cations in zeolite takes place that, probably, is the reason of difficulty of access for strontium ions.

The values of E (Sr^{2+}) and E (Cs^{+}) for γ -irradiated sample are also less then 0.348 and 0.158 meq/g, accordingly, than for parent sample (0.618 and 0.38 meq/g). So, gamma rays (200 and 700 kGy) reduce the ion exchange capacity of zeo-lite more.

The information about radiation effects in zeolites, especially in natural ones, are very poor although radiation modifications of these materials can play a significant role in changing of the sorption and other physical-chemical properties [4, 11–13]. It was reported that the irradiation of US-HY zeolite by ionizing γ -rays induces contrasting effects [14, 15]. At small doses (5.50 kGy) an ordering of structure takes place whereas at higher doses the disordering of the crystal lattice. S.A. Dikanov et al. supposed that this is due to the variation of the charge states of zeolite atoms with e-irradiation but not due to structural changes.

The influence of thermal treatment, concentration, size of granules and volumes of model solutions at ion exchange sorption of Cs⁺ and Sr^{2+} on CL(Ar). The values of exchange capacity and factor of sorption for Sr²⁺ appreciably have been diminished with increase of treatment temperature and size of granules (from 0.20 to 0.50 mm) of initial zeolite. The optimal size of granules $0.2\div0.25$ mm excluded the outerdiffusion process. The thermal treatment above 673 K worsens its ion exchange sorption properties and than the higher is temperature of treatment this process makes more deep is (Table 3). It is obvious that thermal dehydration of zeolite with loss of zeolite's water initiates the migration of some alkaline and alkaline-earth cations in inaccessible position of B and C channels of CL and their strong fastening on the walls of channels. These positions become inaccessible for large, surrounded by hydrated sheath strontium ions therefore the degree of ion exchange sorption is lowered. The increase of initial concentration on the order raises the value of exchange capacity at strontium in flowing regime and does not affect the cesium sorption. Especially it is necessary to note that the constant interfusion of zeolitesolution mixes has allowed obtaining the highest values of exchange capacity, sorption and distribution factors in respect of strontium but for cesium these parameters have remained constant.

Table 3. The influence of treatment temperature on ion exchange sorption of Sr^{2+} on CL(Ar)

CI	unge sorption o		(111)
T,K	E, meq/g	K _s , %	C/C ₀ , %
293	0.32625	89.6	17.5
473	0.2700	88.5	20.2
673	0.2442	80.2	22.4
873	0.2218	69.7	32.28

Ion exchange sorption of Sr^{2+} and Cs^+ from mixture of their salts. At the last stage of this work we had prepared and examined the samples, which showed the highest ion exchange activity: CL(Ar)H, CL(Ar)HNa, and CL(Ar)HCa, to study the mutual influence of Cs⁺ and Sr²⁺ ions from their mixture of solutions on joint ion exchange sorption. It was determined the dependence of exchange capacity E, factors of sorption Ks and distribution K_d on conditions of sorption.

Table 4. The dependence of E, K_s and K_d from volume of Sr^{2+} and Cs^+ -containing solutions at $C_0 = 0.1$ N

Exchange ion	V _{sol,} ml	E, meq/g	K _s , %	K _d , ml/g
Sr^{2+}	50	0.733	97.50	402.5
Sr^{2+}	100	1.660	95.06	386.0
Sr^{2+}	150	1.501	93.20	417.0
Cs^+	30	0.363	76.66	162.8
Cs^+	50	0.419	88.57	385.0
Cs^+	100	0.630	66.98	208.8

At a ratio zeolite: solution equal 1:10 the value of exchange capacity E for all samples practically was the same with sorption coefficient of 98-100%. The similar results were obtained and in the experiences which had been carried out in dynamic conditions - value E for all samples both in static and in dynamic conditions changed in limits ~0.07 meg/g regard to Cs^+ and ~0.11–0.12 meq/g on Sr^{2+} . The dispersion of values of exchange capacity E rises and grows with increase of ratio. It is possible to conclude that at a ratio 1:10 all ions of cesium and strontium were exchanged on cations of zeolite but the equilibrium saturation was not reached else. The decationated and Ca-forms of CL have shown the least value of E concerning both ions but untreated natural CL has appeared most active at a ratio 1:50 and 1:100 on Cs^+ and in the attitude of strontium – Ca-contained CL. I.e., the ion exchange sorption of each of ions decreases at their simultaneous presence in a solution. The sorption of cesium reaches an equilibrium for modified samples at a ratio 1:50, for parent CL this level has not been reached at ratio 1:100. But the sorption value for strontium grows constantly with increase of a ratio zeolite: solution. We compared the obtained experimental results and those of ion exchange sorption from individual solutions of Cs and Sr whence follows that the values of E for sorption from these solutions are much higher (Fig. 4 and 5) than those for sorption from an intermixture of solutions of cesium and strontium for all investigated samples of CL. So, the Cs^+ and Sr^{2+} ions influence against each other, competing and interfering to each other in exchange sorption. Probably, cesium ion, close by the nature and size to potassium ion, interferes to ion exchange sorption of strontium. The diminution of values of exchange capacity for strontium is less than for cesium, i.e., strontium to a lesser degree interferes to adsorption of cesium and vice versa.

The total exchange capacity $E(Sr^{2+}+Cs^+)$ for all samples, except Ca-containing is equal to $E(Sr^{2+})$ for ion exchange sorption out of individual solution (deviation 10–20%) and twice exceed $E(Cs^+)$ out of CsNO₃.



Fig. 4. The diagram of comparative values of exchange capacity $E(Sr^{2+})$ for ion exchange sorption out of: a – individual solution of $Sr(NO_3)_2$ and b – mixture of $Sr(NO_3)_2$ and $CsNO_3$



Fig. 5. The diagram of comparative values of exchange capacity $E(Cs^+)$ for ion exchange sorption out of: a – individual solution of CsNO₃ and b – mixture of Sr(NO₃)₂ and CsNO₃

CONCLUSION

The influence of temperature of preliminary treatment of zeolites, concentration and volume of model solutions, and degree of granulation on the strontium exchange were investigated; it was calculated the sorption capacity, sorption and distribution factors. It was established that the e-irradiation decreases the ion exchange capacity not only of initial untreated CL but cation-modified too. The same decrease of ion exchange capacity was observed for γ -irradiated CL.

It is shown that hydrogen-, sodium- and calcium-containing samples of CL(Ar) are appeared the best in ion exchange sorption of cesium and strontium. So, sodium-containing CL(Ar) may be recommended for sorption of cesium nuclides from its solution and calcium-containing CL(Ar) – for sorption of strontium nuclide from its salts.

The ion exchange sorption of Cs^+ and Sr^{2+} from an intermixture of solutions of their salts is accompanied by competitive and mutually interference sorption that depresses exchange sorption in comparison with sorption from individual solutions. This distinction is especially shown for Sr^{2+} .

Tenfold and more excess of the solution over zeolite is insufficient for saturation and complete replacement of cations of zeolite with Cs^+ ions and in a greater degree with Sr^{2+} . The size of an ion plays a determining role in ion exchange sorption.

The untreated natural CL(Ar) is more active in the sorption from an intermixture of these metals' salts solutions. So, parent CL from deposit of Armenia may be recommended to purify nuclear waste water from cesium and strontium nuclides.

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Іонообмінні властивості опроміненого та хімічно модифікованого клиноптилоліту по відношенню до Cs⁺ і Sr²⁺

Л. Ахалбедашвілі, Г. Тодрадзе, Н. Кекелідзе, Е. Кехеян, Г. Єритцян, Р. Геворкян

Кавказький Інститут мінеральної сировини ім. А. Твалчрелідзе, вул. Паліашвілі 85, Тбілісі 0162, Грузія Тбіліський державний університет ім. І. Джавахішвілі, вул. Чавчавадзе 1, Тбілісі 380028, Грузія Римський університет "La Sapienza", пл. Альдо Моро 5, Рим 00185, Італія Єреванський інститут фізики, вул. Братів Аліхонян 2, Єреван 0036, Вірменія Єреванський державний університет, вул. Алека Манукяна 1, Єреван 0025 Вірменія

Вивчена іонообмінна сорбція Cs^+ і Sr^{2+} з їхніх солей на клиноптилоліті з родовищ Вірменії у вихідній, модифікованій е- і γ -опроміненням, а також у декатіоновоній і катіонній формах. Встановлено вплив типу хімічної обробки, ступеня зерніння і температури дегідратації зразків природного клиноптилоліту Вірменії, концентрації та об'єму промивних розчинів на поглинання цих іонів. За отриманими даними визначено обмінну ємність Е, коефіцієнти розподілу K_d і сорбції K_s . Показано, що необроблений CL (Ar), декатіонований і Са-вмісний вирізняються більшою іонообмінною ємністю у порівнянні з опроміненими, і найбільш перспективними сорбентами по відношенню до Sr^{2+} та $Cs^+ \epsilon$ хімічно модифіковані зразки.

Ионообменные свойства облученного и химически модифицированного клиноптилолита по отношению к Cs⁺ и Sr²⁺

Л. Ахалбедашвили, Г.Тодрадзе, Н.Кекелидзе, Е.Кехеян, Г.Еритцян, Р.Геворкян

Кавказский Институт минерального сырья им. А. Твалчрелидзе, ул. Палиашвили 85, Тбилиси 0162, Грузия Тбилисский государственный университет им. И. Джавахишвили, ул. Чавчавадзе 1, Тбилиси 380028, Грузия

Римский университет" La Sapienza", пл. Альдо Моро 5, Рим 00185, Италия

Ереванский институт физики, ул. Братьев Алихонян 2, Ереван 0036, Армения

Ереванский государственный университет, ул. Алека Манукяна 1, Ереван 0025 Армения

Изучена ионообменная сорбция Cs^+ и Sr^{2+} из их солей на клиноптилолите из месторождений Армении в исходной, модифицированной е- и γ облучением, а также в декатионированной и катионных формах. Установлено влияние типа химической обработки, степени зернения и температуры дегидратации образцов природного клиноптилолита Армении, концентрации и объема промывных растворов на поглощение этих ионов. На основании полученных данных определены обменная емкость Е, коэффициенты распределения K_d и сорбции K_s . Показано, что необработанный CL (Ar), декатионированный и Ca-содержащий отличаются более высокой ионообменной емкостью по сравнению с облученными, и наиболее перспективными сорбентами по отношению к Sr^{2+} и Cs^+ являются химически модифицированные образцы.