

Sorption of ^{90}Sr and ^{137}Cs on clays used to build safety barriers in radioactive waste storage facilities*

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Academic editor: Yury Kazansky ♦ Received 23 March 2020 ♦ Accepted 20 January 2021 ♦ Published 24 June 2021

Citation: Volkov IN, Zharkova VO, Karaseva YaYu, Lysakova EI, Zakharova EV (2021) Sorption of ^{90}Sr and ^{137}Cs on clays used to build safety barriers in radioactive waste storage facilities. Nuclear Energy and Technology 7(2): 151–156. <https://doi.org/10.3897/nucet.7.69930>

Abstract

The purpose of the work was to investigate the sorptive capacity of natural clay samples with respect to ^{90}Sr and ^{137}Cs to assess the possibility of using these as components of protective barriers at radioactive waste isolation facilities. Bentonite clays of the Ziryanskoye and Desyaty Khutor deposits and high-melting clay of the Kampanovskoye deposit were selected for the investigation. The capacity of clays for sorption through ionic exchange is characterized by the value of the cation exchange capacity (CEC). In the process of sorption experiments, all of the test clays displayed a high rate of extracting strontium and cesium radionuclides from aqueous solutions. It was shown that the sorption of ^{90}Sr is affected by the content of montmorillonite in the samples: bentonite clays absorb up to 98–99% of the initial radionuclide content in the solution, while about 80% of strontium is sorbed by high-melting clay. Cesium is practically fully sorbed by the tested samples and the degree of sorption amounts to over 99%, the highest value of the distribution coefficient having been recorded for the Kampanovskoye sample ($K_d = 5.0 \times 10^3 \text{ cm}^3/\text{g}$). The method of sorbed radionuclides fixation on the clay samples were identified by selective desorption using the modified Tessier methodology. It was shown that strontium ions are more mobile than ions of cesium up to 97% of which is retained by clays.

Keywords

Sorption, cesium, strontium, clays

Literature review

In accordance with Federal Target Nuclear and Radiation Safety Program II (2030), Russia continues to decommission nuclear- and radiation-hazardous installations (Kuleshova et al. 2014), this leading to large quantities of accumulated radioactive waste (RW). According to (About the criteria 2012), RW is divided into six hazard classes depending on the aggregate state and specific activity. This classification is taken into account when selecting

the final isolation method, that is, near-surface or deep geological disposal. In the early decades of the storage facility operation, the greatest radiological hazard comes from ^{90}Sr and ^{137}Cs (Obruchikov and Tyupina 2014), which, once they enter the environment, may incorporate in the animal and human food chains (Vasilenko and Vasilenko 2001, 2002).

When in the human body, ^{90}Sr accumulates in bones (Vasilenko and Vasilenko 2002), and ^{137}Cs accumulates in organs and muscle tissues (Vasilenko and Vasilenko

* Russian text published: Izvestiya vuzov. Yadernaya Energetika (ISSN 0204-3327), 2021, n. 1, pp. 95–107.

2001). The accumulated radionuclides form a continued radiation source for a prolonged period of up to tens of half-lives: the half-life for ^{90}Sr is 28.5 years, and that for ^{137}Cs is 30.1 years (Beckman 2011).

Due to the environmental hazard posed by the radionuclides contained in RW, a multi-barrier protective system is built at storage facilities to avoid uncontrolled spread of radionuclides into the environment. The required protective properties of the barrier (buffer) materials are justified and selected at the design stage with regard for the facility's technological peculiarities and operating conditions. Sand, gravel, extracted rock, sand mix with grainy sorbent, clayey materials, and concrete grouts can be used to build safety barriers (Varlakova et al. 2013). In some countries, bentonite clays are considered predominantly as the buffer material (Oblivantsev and Shcherbakova 2007; Kuleshova et al. 2014; Cherif et al. 2017; Misana et al. 2018; Tran et al. 2018; Wissocq et al. 2018). These clays feature low water permeability, an extensive sorptive capacity with respect to most radionuclides, an increased swelling ability, and rather high heat resistance, thermal conductivity, and ductility values (Varlakova et al. 2013). Russia has an experience of using mixtures based on kaolin clays in building safety barriers with high protective properties (Ilyina et al. 2019).

The purpose of this study is therefore to assess the possibility of kaolin clays to be used as an analog of bentonite clays for the protective material at RW isolation facilities. This assessment is based on comparing the mineral and chemical compositions and the sorptive capacity with respect to ^{90}Sr and ^{137}Cs .

Materials and investigation methods

The following samples of clayey materials were tested: bentonite clays of the Zyryanskoye deposit in Kurgan region and the Desyaty Khutor deposit in the Republic of Khakassia, and high-melting clays of the Kampanovskoye deposit in the Krasnoyarsk Territory. Abbreviations used for the samples: "Z" – Zyryanskoye, "Kh" – Desyaty Khutor, "HG" – high-melting clays of the Kampanovskoye deposit.

The clayey materials under study represent polymineral mixtures the mineral composition of which is shown in Table 1. The mineral composition was analyzed using X-ray diffraction (an X-pert Pro X-ray diffractometer, PANalytical, Holland).

As can be seen from the table data, the montmorillonite mineral is the base component of bentonite clays. Kaolinite is the predominant mineral component of the Kampanovskoye clay samples, the share of which reaches up to 46 wt. %.

Structurally, montmorillonite and kaolinite are composed of two units: alumohydroxyl octahedral and silicon-oxygen tetrahedral layers. Montmorillonite has a

Table 1. Mineral composition of samples.

Mineral phase	Chemical formula	Rock		
		HG	Z	Kh
		Content, mass fraction, %		
Quartz	SiO_2	27	16	11.5
Montmorillonite	$(\text{Na,Ca})_{0.33}(\text{Al,Mg})_2(\text{Si}_4\text{O}_{10})(\text{OH})_2 \cdot n\text{H}_2\text{O}$	8	67	71
Kaolinite	$\text{Al}_2[\text{Si}_4\text{O}_{10}](\text{OH})_2$	46	6	4
Illite	$(\text{K}_{0.75}(\text{H}_3\text{O})_{0.25})\text{Al}_2(\text{Si}_3\text{Al})\text{O}_{10}(\text{H}_2\text{O})_{0.75}(\text{OH})_{0.25} \cdot 2$	–	3	1
Sericite	$\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{OH})_2$	10	–	–
Potash feldspar	$\text{K}[\text{AlSi}_3\text{O}_8]$	2	0.5	4
Plagioclases	$\text{NaAlSi}_3\text{O}_8 - \text{CaAl}_2\text{Si}_2\text{O}_8$	–	1.5	5
Anatase	TiO_2	0.5	1	–
Rutile	TiO_2	0.5	–	–
Calcite	CaCO_3	–	5	3
X-ray amorphous phase	–	2	–	0.5

structure of the 2:1 type (there are two tetrahedral silicon-oxygen layers for one octahedral alumohydroxyl layer). As a rule, the interlayer space of montmorillonite is filled with molecules of water and with cations of alkali and/or alkaline earth elements such as Na^+ , Ca^{2+} , and Mg^{2+} . These hydrated cations are capable to take part in exchange reactions with the cations of the environment and, therefore, the process of sorption by ionic exchange takes place.

Kaolinite is represented by a structure of the 1:1 type, that is, by alternating tetrahedral and octahedral layers firmly fused through hydrogen and molecular bonds (Fig. 1) (Osipov and Sokolov 2013).

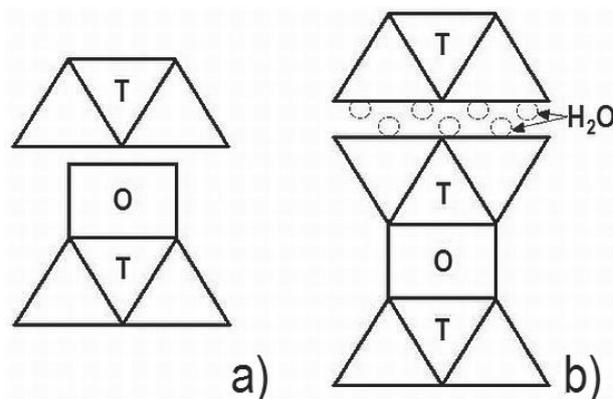


Figure 1. Schematic structures of clayey materials: a) kaolinite, b) montmorillonite (T – tetrahedral layers, O – octahedral layers).

Due to its structural features, kaolinite has much less cation-exchange sites than montmorillonite, so the process of cation sorption from an outer solution through ionic exchange is not typical of kaolinite. The quantity that characterizes ionic exchange is cation exchange capacity (CEC). The CEC can be numerically expressed as the amount of the gram equivalents of cations absorbed by 100 g of dry mineral mass (Osipov and Sokolov 2013).

The cation exchange capacity of the samples was determined by adsorbing a divalent copper complex of triethylenetetramine $[\text{Cu}(\text{Trien})]^{2+}$ (Meier and Kahr 1999). The experiments were undertaken in two series with the results averaged further for each sample after the statistical processing of data. The findings are given in Table 2.

Table 2. Clayey sample CEC determination results.

Sample	CEC, mg-eq/100 g
HG	30.5
Z	64.4
Kh	88.0

Actually, the CEC value for the Kampanovskoye high-melting clay sample is noticeably higher than the values representative of kaolin clays (3 to 15 mg-eq/100 g) (Osipov and Sokolov 2013). It stems from this that rather a high CEC value should be caused by this sample including a mineral with an extensive ion-exchange capacity. As follows from the mineral composition of the samples shown in Table 1, montmorillonite is the only mineral capable to provide high CEC values for the entire polymineral sample. This assumption is confirmed by high CEC values (64.4 to 88.0 mg-eq/100 g) for bentonite clays the base component in which is exactly the mineral in question.

Element analysis was used to confirm the mineral composition and to identify more specifically the cation-exchange complex of the clayey samples. The element analysis of ash residues (Table 3) was obtained using an Axios mAX Advanced wave X-ray fluorescent spectrometer (PANanalytical, Holland).

Table 3. Element analysis of the clayey sample ash residue.

	HG	Z	Kh
Na ₂ O, %	0.123	0.682	1.054
MgO, %	0.96	2.069	3.162
Al ₂ O ₃ , %	21.99	19.4	19.623
SiO ₂ , %	69.61	59.48	65.16
K ₂ O, %	1.575	1.124	0.985
CaO, %	0.793	4.7	2.375
TiO ₂ , %	1.184	0.957	0.799
MnO, %	0.018	0.079	0.034
Fe ₂ O ₃ , %	2.033	7.104	3.892
P ₂ O ₅ , %	0.045	0.048	0.139
S, %	0.045	0.096	0.046
Residue, %	1.626	4.261	2.730

A relatively high content of Mg and Ca in the bentonite samples (Zyryanskoye and Desyaty Khutor), as compared with Na and K, makes it possible to classify these as alkaline earth materials. Calcium and magnesium in the interlayer space of the montmorillonite mineral will be therefore the base exchange ions.

Sorption experiments

The sorption characteristics of the samples were identified based on the results of experiments to sorb tracer quantities of ⁹⁰Sr and ¹³⁷Cs on clays.

Polypropylene flasks were used for sorption from distilled water with adding a tracer quantity of radionuclides (mole/l): 3.25×10^{-10} ⁹⁰Sr and 3.41×10^{-10} ¹³⁷Cs. The experiments were conducted in aerobic conditions at a temperature of 23 ± 2 °C, and with the S:L phase ratio of 1:5.

After the preset exposure time expired, the liquid phase was separated by decanting to analyze it for the content of

⁹⁰Sr and ¹³⁷Cs with preliminary centrifugation at 6700 rpm for 10 minutes (Thermo Fisher Scientific SL 16). The content of ⁹⁰Sr was determined by liquid scintillation counting using a Tri-Carb-3180 TR/SL liquid scintillation spectrometer (Perkin-Elmer, the USA) and the Optiphase Hisafe 3 scintillation liquid (PerkinElmer, Holland). The content of ¹³⁷Cs was determined by gamma-ray spectrometry using a digital gamma-ray spectrometry suite with a coaxial GEM30 detector and a planar GLP-36360 detector from AMETEK (ORTEC). The activity of the aliquot (0.5 to 1 ml) was determined relative to the activity of the initial solution (0.5 to 1 ml) determined based on the same procedure and using the same materials.

The values of the sorption degree, S , and the interphase distribution coefficient, K_d , were calculated using the obtained data.

The sorption degree (S , %) defines the share of the radionuclide that has passed from solution to a solid phase and is calculated by the formula

$$S = [(C_0 - C)/C_0] \times 100\%,$$

where C_0 and C are respectively the initial and final concentrations of the radionuclide in the solution, Bq/cm³.

The interphase distribution coefficient (K_d , cm³/g) is equal to the ratio of the quantity of the radionuclide sorbed by rock to its content in the solution:

$$K_d = N/C = [S/(100 - S)] \times V/m,$$

where N is the quantity of the radionuclide in rock, Bq/g; C is respectively the initial and final concentrations of the radionuclide in the solution, Bq/cm³; V is the liquid phase volume, cm³; m is the rock weight, g; and S is the sorption degree, %.

The results of the experiments to determine the time the equilibrium is reached for in sorption systems are presented in Tables 4, 5.

Table 4. Kinetics of the ⁹⁰Sr sorption.

	⁹⁰ Sr sorption degree S , %							Distribution coefficient K_d at equilibrium, cm ³ /g
	1 day	3 days	7 days	10 days	14 days	30 days	45 days	
HG	74.4	78.5	79.3	79.8	80.0	78.9	80.1	7.5×10^1
Z	86.4	97.7	97.3	97.2	97.7	97.6	97.5	2.0×10^2
Kh	96.3	98.4	98.6	99.8	99.0	99.4	99.0	5.0×10^2

Table 5. Kinetics of the ¹³⁷Cs sorption.

	¹³⁷ Cs sorption degree S , %							Distribution coefficient K_d at equilibrium, cm ³ /g
	1 day	3 days	7 days	10 days	14 days	30 days	45 days	
HG	98.2	99.4	99.7	99.9	99.8	99.9	99.9	5.0×10^3
Z	85.9	97.3	99.6	99.8	98.9	99.3	99.1	5.5×10^2
Kh	98.6	97.2	99.9	99.8	98.4	99.2	99.4	8.3×10^2

As can be seen from the results of the kinetic experiments, the systems under study reach equilibrium after three days of the phase contact with the following high values of the sorption degree recorded: 80% for the kaolin clay, up

to 97.7% for the Zyryanskoye bentonite sample, and up to 99.8% for the Desyaty Khutor bentonite sample. According to (Polyakov 1970), the most widespread form of the ^{90}Sr occurrence in rock is ionic exchange, the most mobile form, as its behavior follows the ionic equilibrium law. It stems from this that the best sorption performance for strontium will be on rock with the largest content of the mineral that includes a developed interlayer and features the highest CEC values. As stated above, montmorillonite is the only mineral the interlayer space of which is capable to accommodate a strontium ion in exchange for the interlayer cations it contains. Indeed, the values of the distribution coefficients obtained for ^{90}Sr in the process of its sorption correlate with the content of montmorillonite in rock. The value $K_d = 5.0 \times 10^2$ for the montmorillonite-richest Desyaty Khutor rock sample (the content of the mineral is 71%), so strontium sorbs on the Kampanovskoye clay sample (the content of montmorillonite is 8%) with $K_d = 7.5 \times 10^1$. The sorption and rock CEC values correlate in the same way. It can be therefore assumed that montmorillonite is the mineral affecting basically the sorption of ^{90}Sr , while the retention takes place due to the ionic exchange of strontium cations for the cations in the mineral's interlayer space. In (Başçetin and Atun 2006; Missana et al. 2008), a higher absorption of strontium from water environments is shown at pH values, when the sorption mechanism of ion exchange predominates, on smectites as compared with illites and kaolinites.

The equilibrium state in the systems is reached as soon as during the first days on the Kampanovskoye high-melting clay samples (HG). In the event of the bentonite samples (Z and Kh), equilibrium is reached in the systems not less than after three days of the phase contact. This involved practically the full sorption of ^{137}Cs from the solution (the sorption degree values do not exceed 99% for all rocks). High absorption of cesium tracer quantities from aqueous solutions at neutral pH values is specific to montmorillonite clays (Wu 2009).

Comparing by the K_d values, the Kampanovskoye clay sample displays the highest sorptive capacity with respect to cesium. This can be explained by the sample containing sericite (10%), a hydromicaceous mineral, which, as shown in (Dronova et al. 2005; Kwong-Moses et al. 2020), features a high selectivity of exchange sites on lateral shears in relation to cesium cations. These sorption centers form as hydromica layers split over their cleavage surface.

Sequential extraction

Sorption experiments demonstrate the capability of rock to retain certain ions but do not provide sufficient information on the methods to fix these on rock. Selective desorption was used to identify the ^{90}Sr and ^{137}Cs occurrence forms which makes it possible to extract sequentially the radionuclides distributed by various geological forms (Konevnik et al. 2017). The authors used the selective desorption method based on the modified Tessier methodology (Table 6) (Tessier et al. 1979).

Table 6. Conditions of the ^{90}Sr and ^{137}Cs selective desorption.

Stage	Desorbent	Experiment conditions	Desorbed fraction
I	Distilled water	25 °C. S:L = 1:5. t = 2 h	Water-soluble fraction
II	1M NH_4Ac	25 °C. S:L = 1:5. t = 1 h	Exchangeable fraction
III	1M HCl	25 °C. S:L = 1:5. t = 2 h	Fraction bound by the surface complex formation mechanism; organic substances
IV	6M HCl	60 °C. S:L = 1:5. t = 4 h	Acid-soluble fraction
V	No treatment	–	Insoluble in acids. Determined from the difference between the initial content of the radionuclide sorbed on solid phase and that desorbed in four stages (sum of fractions I + II + III + IV)

The content of the radionuclides being in a mobile form (stage I), in a potentially mobile form (stages II and III), and in a firmly fixed form on rock (stages IV and V) was estimated based on the selective desorption results. Diagrams were plotted based on the experimental data for the percentage ratios of desorbed ions at different stages of the experiments (see Tables 7, 8 and in Figs 2, 3).

Table 7. ^{90}Sr desorption results.

	^{90}Sr desorption degree by stages, %				
	I	II	III	IV	V
HG	6.5	15.1	19.1	12.3	47.1
Z	2.6	21.5	15.5	8.0	52.8
Kh	2.5	23.1	16.2	7.3	51.4

Table 8. ^{137}Cs desorption results.

	^{137}Cs desorption degree by stages, %				
	I	II	III	IV	V
HG	1.2	1.2	1.1	0.8	95.7
Z	0.9	1.8	0.9	1.0	95.5
Kh	0.8	1.3	0.2	0.3	97.5

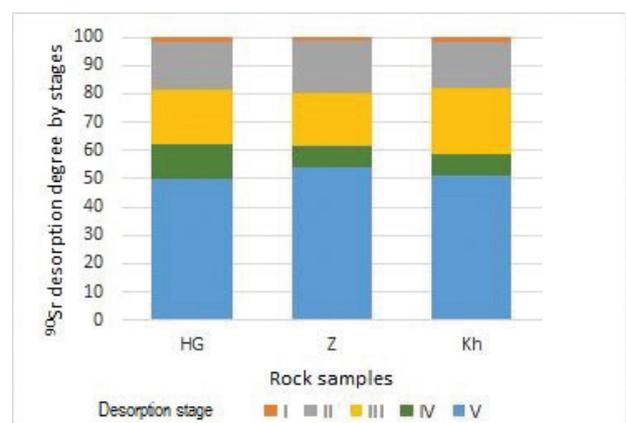


Figure 2. Results of ^{90}Sr desorption on clay samples.

As can be seen from the selective desorption result data, the quantitative distribution of ^{90}Sr by fixation forms practically take place coincides for all clay samples. The conventionally mobile ^{90}Sr , which is washed off the sam-

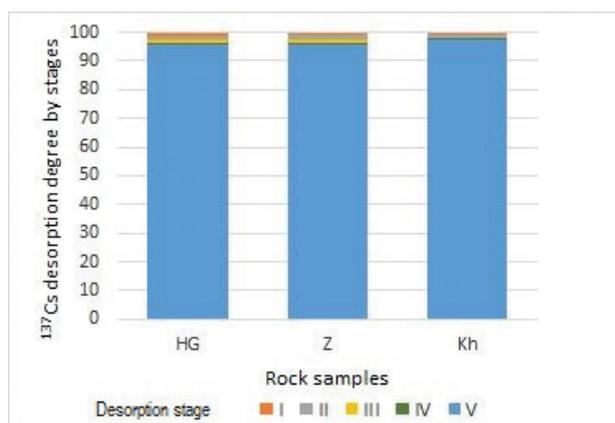


Figure 3. Results of ^{137}Cs desorption on clay samples.

ple during treatment stages I through IV, has predominantly an ion-exchange form (stage II) with about 50% of the desorbed ion aggregate quantity retained after all treatment stages (stage V). A noticeably smaller content of the ion-exchange fraction, as compared with the bentonite samples, can be seen on the high-melting clay sample, which confirms, along with the sorption experiment data, that montmorillonite contributes to the absorption of ^{90}Sr by clayey materials.

A conclusion can be made based on the experimental data that ^{137}Cs is firmly incorporated in the sorbent structure being retained there in the process of leaching even after a treatment with 6M hydrochloric acid. In (Krupskaya et al. 2016), the authors assume the following mechanisms of sorption on montmorillonite: cesium enters the interlayer space of the mineral through ionic exchange being fixed then in the mineral's structure thanks to the Cs-O bond formed in the montmorillonite's tetrahedral lattice.

High parameters of the firmly fixed phase on the high-melting clay samples can be explained both by

montmorillonite being present in the mineral composition of the clay and by the cesium ion firmly retained in the interpackage sites of sericite.

Conclusion

Studies have been undertaken to investigate the sorptive and desorptive interaction of clayey materials (bentonites of the Desyaty Khutor and Zyryanskoye deposits, and high-melting clay of the Kampanovskoye deposit) with respect to ^{90}Sr and ^{137}Cs . The bentonite clay absorption values during the sorption of strontium tracer quantities are higher than the similar values for kaolin clay, still all samples demonstrate an extensive sorptive capacity (a sorption degree of 80% and higher). The authors assume that the higher values of the strontium absorption by bentonite clays are explained by the montmorillonite mineral being predominant in their structure with the radionuclide being retained through the ionic exchange of the strontium cations for the cations in the mineral's interlayer space. Tracer quantities of ^{137}Cs are absorbed practically in full (up to 99.9%) by the clayey samples, the high-melting clay sample being the most efficient sorbent as shown by the distribution coefficient values. Unlike bentonites, the sorptive properties of high-melting clay seem to be affected not only by montmorillonite being present in its structure but also by the presence of sericite. It can be concluded based on the selective desorption results that strontium ions are more mobile (some 50% are washed away by desorbents) as up to 97% of ^{137}Cs is retained firmly on all samples. Therefore, both bentonite and kaolin clays demonstrate fairly high anti-migration properties with respect to strontium and cesium radionuclides and can be used to build safety barriers for the RW isolation.

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