

Adsorption of cesium on domestic bentonites

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Abstract Bentonite is a natural clay and one of the most promising candidates for use as a buffer material in the geological disposal systems for spent nuclear fuel and high-level nuclear waste. It is intended to isolate metal canisters with highly radioactive waste products from the surrounding rocks because of its ability to retard the movement of radionuclides by adsorption. Slovak Republic avails of many significant deposits of bentonites. Adsorption of Cs on five Slovak bentonites of deposits (Jelšový potok, Kopernica, Lieskovec, Lastovce and Dolná Ves) has been studied with the use of batch technique. In the case of Dolná Ves deposit, the mixed-layer illite–smectite has been identified as the main clay component. Natural and irradiated samples, in two different kinds of grain size: 45 and 250 μm have been used in the experiments. The adsorptions of Cs on bentonite under various experimental conditions, such as contact time, adsorbent and adsorbate concentrations have been studied. The Cation Exchange Capacity values for particular deposits drop in the following order: Jelšový potok > Kopernica > Lieskovec > Lastovce > Dolná Ves. Bentonites irradiated samples with 390 kGy have shown higher specific surface and higher values of the adsorption capacity. Distribution coefficients have been determined for bentonite-cesium solution system as a function of contact time and adsorbate and adsorbent concentration. The data have been interpreted in terms of Langmuir isotherm. The uptake of Cs has been rapid and the adsorption of cesium has increased with increasing metal concentrations. The adsorption percentage has decreased with increasing of metal

concentrations. Adsorption of Cs has been suppressed by presence of Ca^{2+} more than Na^+ cation. Sorption experiments carried out show that the most suitable materials intended for use as barriers surrounding a canister of spent nuclear fuel are bentonites of the Jelšový potok and Kopernica deposits.

Keywords Adsorption · Bentonite · Illite–smectite · Montmorillonite · Cesium · Cation exchange capacity · Deep geological repository

Introduction

Energy is a basic assumption of civilization development. However, side products of nuclear energy production include production of nuclear waste and spent nuclear fuel. Currently Slovak Republic avails of two locations (Bohunice and Mochovce) with nuclear power plant (NPP) which make up to more than a half of primary sources of energy in the Slovakia (Fig. 1). These are the most significant sources of spent nuclear fuel and high-level radioactive waste.

A significant problem of the nuclear energy production industry in Slovakia is finding the final solution to the problem of the back side of nuclear cycle—problem of definite deposition of spent nuclear fuel and high-level radioactive waste.

“The suggestion of the final part of nuclear energy strategy” (Slovak government act No. 238/2008)—a programme of deep geological repository development in Slovakia defines the development of a deep geological repository as one of the three considered alternatives. It directly supports geological research and examination of potential localities possibly suited for placement of a deep geological repository [1].

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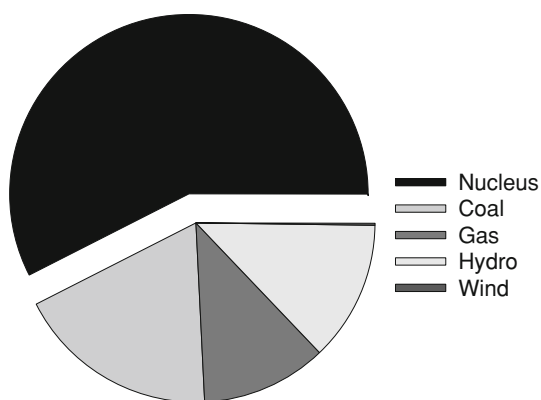


Fig. 1 Ratio of the primary energy sources in Slovakia for 2008: Nucleus 57.46%, Coal 18.31%, Gas 11.37%, Hydro 12.68%, Wind 0.19%

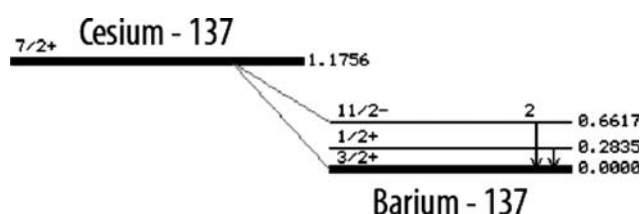


Fig. 2 Mode of decay ^{137}Cs

A safe nuclear fuel cycle depends also on the waste storage and deep geological repository that are formed by natural and engineered barriers isolating long-lived radionuclides from the biosphere are recommended. Clay is the basic component of these barriers in many cases. Bentonite is generally considered as the most appropriate clay material [2–6].

Out of the group of Cs radio-isotopes the most significant one is ^{137}Cs ($T \sim 30.1$ years, $E_{\beta} = 0.51$ (92%), 1.18 (8%) MeV, $E_{\gamma}(^{137\text{m}}\text{Ba}) = 0.6616$ MeV, Fig. 2). Fission nuclear reactions, nuclear explosions and nuclear energetic are the main sources of its existence. When dispersed in the environment ^{137}Cs migrates, sorbs onto natural matrices and also integrates in the food chain. Cesium enters human body most importantly by means of food (82–98%), drinking water (1–6%) and fallout inhalation (1–2%) and it consequently gets absorbed into the digestive tract. This element gets distributed by blood throughout the entire body and in case of continuous intake it accumulates in soft and muscle tissues. Presence of such radiotoxic metals in living organisms can lead to somatic as well as genetic changes and for this reason adsorption of that radionuclide is very carefully monitored [7–10].

Bentonites present a significant group of natural materials mostly compounded from microcrystalline particles of minerals from the group dioctahedral smectite–montmorillonite (Fig. 3) [11–17]. Crystallochemical formula of montmorillonite:

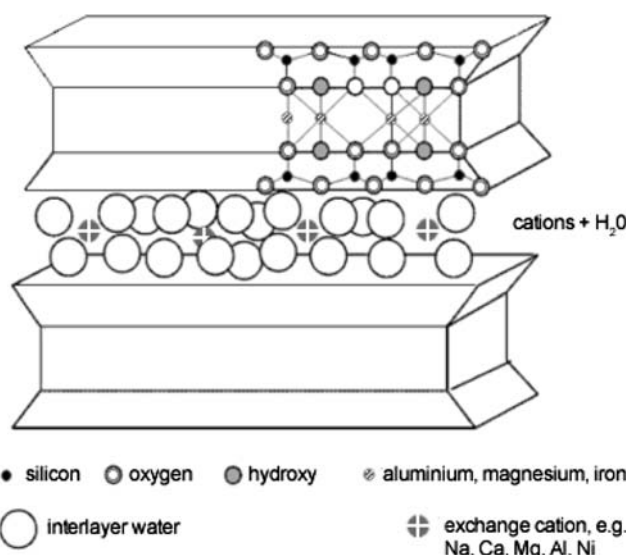
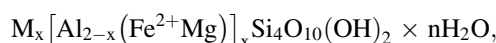


Fig. 3 Structure of smectite



where X, which represents the layer charge, can change from 0.2 till 0.6. The formula expresses the characteristic feature of montmorillonite which is the non-existence of tetrahedral charge. The entire charge is concentrated on the octahedral net. According to the presence of different octahedral cations we divide montmorillonites into:

- Al-montmorillonites,
- Mg-montmorillonites, and
- Fe-montmorillonites.

Significant characteristics of bentonites such as low permeability, high swelling ability absorption of ions pre-determine their use as sealing barriers in multi-barrier system when underground geological repository for spent nuclear fuel and high-level radioactive wastes are constructed [18–22]. An extensive study of the sorption behaviour of the bentonite barriers is an inevitable part of deep geological repository concept [23–29]. Attention is being paid to the influence of various factors on sorption processes which take place in the area of deposited spent nuclear fuel and nuclear waste. An example of such factors can be influence of radiation and residual temperature, change in pH of environment, influence of the competitive ions and organic ligands, etc. [30–37].

There are several significant bentonite deposits in the Slovak republic. The best known and in long-term mined bentonite deposit containing Al–Mg montmorillonite is in the area of Slovak upland (“Slovenské stredohorie”) in the location of Stará Kremnička: Jelšovský potok. This location features one more partly mined deposit of andesite bentonite consisting of Fe-montmorillonite: Lieskovec. In the area of East-Slovak neo-vulcanites we find two currently

mined deposits of liparite bentonites: Kuzmice and Lastovce (Ag–Mg montmorillonite). Apart from these there are currently not mined deposits Nižný Hrabovec, Fintice, Nižný Žipov and Veľaty. An interesting accumulation of basaltic bentonites was discovered in basaltic maar in the vicinity of Jelšovec in Upland of Filakovo. The deposit of mixed-layer illite–smectite of Dolná Ves is situated on the south-western rim of the stratovolcano of Kremnica.

Experimental

Solid phase

The domestic bentonite samples were taken from five Slovak deposits and technologically processed by Envigeo a.s. (Table 1). From the deposit Dolná Ves was tested the sample NDV45, modified by sedimentation technique. Natural samples of bentonite were at in two different grain sizes, samples ground under 45 μm and under 250 μm (what means, that these are fractions of bentonite with size of particles smaller than 45 and 250 μm).

Then samples of bentonites were compared from locality Jelšovský potok and Lieskovec with their non-irradiated and irradiated forms. Samples were irradiated in natural state in Slovak Institute of Metrology, using ^{60}Co source with energy of 1.17 and 1.33 MeV during cca 50 days period, with medium dose rate of $0.092 \text{ Gy}\cdot\text{s}^{-1}$. Total absorbed dose was 390 kGy. Samples of bentonite were dried in Petri dish in drying oven at the temperature 105 °C approximately 2–3 h. Then they were taken into small plastic containers and lay aside into a desiccator, where they were stored till next use.

Chemical and mineralogical analyses

Chemical composition of bentonites from deposits of Jelšovský potok, Kopernica and Lieskovec is shown in Table 2.

Lieskovec, Kopernica and Jelšovský potok have similar qualitative mineral composition (Table 3). Kopernica and Jelšovský potok are similar quantitative as well. Lieskovec has significantly lower content of smectite (about 20%

Table 2 Chemical analysis of bentonites from Jelšovský potok, Kopernica and Lieskovec

Metal oxides	Chemical analysis [%]		
	Jelšovský potok	Kopernica	Lieskovec
Silica (SiO_2)	61.25	62.19	60.56
Alumina (Al_2O_3)	22.51	19.81	19.06
Lime (CaO)	1.82	2.09	1.31
Iron oxide (Fe_2O_3)	2.53	2.47	7.22
Ferrous oxide (FeO)	0.23	0.16	0.27
Magnesia (MgO)	3.18	4.74	1.75
Manganese(II) oxide (MnO)	0.018	0.048	0.021
Sodium oxide (Na_2O)	0.11	0.32	0.48
Potassium oxide (K_2O)	1.24	0.85	1.44
Titanium oxide (TiO_2)	0.19	0.21	0.81
Phosphorus pentoxide (P_2O_5)	0.03	0.03	0.12

less), its representation is substitute mainly by kaolinite and cristobalite. Lastovce are significantly different from the other smectite deposits mainly by very low content of smectite, high content of cristobalite and not trivial presence of calcite. The sample NDV, K-bentonite from Dolná Ves represents in comparison with other used bentonites mineralogically different material. Its main clay phase is mixed-layering illite–smectite not present in other bentonites.

Aqueous phase and radiotracer

Adsorption experiments were realized in the Cs concentration range 1×10^{-5} – $5 \times 10^{-2} \text{ mol dm}^{-3}$ solutions prepared from cesium chloride (Table 4). Effect of Na^+ and Ca^{2+} ions on the adsorption of cesium was studied with its concentration $1 \times 10^{-4} \text{ mol dm}^{-3}$, while concentration of competitive ions was in the range 5×10^{-2} – $1 \times 10^{-5} \text{ mol dm}^{-3}$.

In experiments, chemicals of ordinary laboratory purity from supplies companies Slavus, s.r.o., Bratislava SR, or Lachema n.p., Brno CZ were used. Radioisotope ^{137}Cs with the volume activity 1 MBq mL^{-1} was used as a radiotracer.

Table 1 Used samples of domestic bentonites

Deposits	Jelšovský potok	Kopernica	Lieskovec	Dolná Ves	Lastovce
Type	Smectic bentonite	Smectic bentonite	Smectic bentonite	K-bentonite contains illite–smectite	Smectic bentonite
Locality	Middle Slovakia	Middle Slovakia	Middle Slovakia	Middle Slovakia	Eastern Slovakia
Sample designation	J	K	L	NDV	LA
Grain size (μm)	45, 250	45	45, 250	45	45
Irradiation form	Yes	–	Yes	–	–

Table 3 Mineralogical composition

Sample	Quartz	Albite	Biotite	Kaolinite	Smectite	K-felspar	Cristobalite	Calcite
J45	6	S	1	2.5	86	3.5	1	–
J250	2.5	0.5	2	2	82	9.5	1.5	–
K45	6.5	S	2.5	1	82	6	2	–
L45	12	1.5	0.5	6	64	9.5	6.5	–
L250	9.5	0.5	2	9.5	64	9.5	5	–
NDV45	47.5	2	–	5	–	4	5.5	–
La45	13	7.5	–	–	40.5	7	27	5

Table 4 Cesium ion concentration used in adsorption studies

c (mol dm ⁻³)
1×10^{-5}
5×10^{-5}
1×10^{-4}
5×10^{-4}
1×10^{-3}
5×10^{-3}
1×10^{-2}
5×10^{-2}

Batch method and measuring of radioactivity

Adsorption of Cs on samples of bentonites was studied through radioisotope indication using radioisotope of cesium-137 in static arrangement of experiment, in aerobic conditions at laboratory temperature. Adsorption parameters were determined after mixing in 0.05 g of adsorbent with 5 mL of water phase in plastic test tubes with internal diameter 14 mm and capacity 10 mL in laboratory extractor with constant speed of mixing both phases. After realization of adsorption and subsequent centrifugation ($t = 15$ min, 3,500 rpm) 1 mL was extracted from supernatant to measure radioactivity.

Radioactivity determination of ¹³⁷Cs solutions was done with spectrometer Modumatic using NaI(Tl) detector.

Results and discussion

Adsorption properties of bentonite were calculated by following equations:

$$\text{Distribution coefficient } K_d = \frac{a_0 - a}{a} \times \frac{V}{m} \text{ (ml g}^{-1}\text{)} \quad (1)$$

$$\text{Adsorption percentage } R = \frac{100 \times K_d}{K_d + \frac{V}{m}} \text{ (\%)} \quad (2)$$

$$\text{Adsorption capacity } \Gamma = K_d \times c_{\text{eq}} \text{ (mmol g}^{-1}\text{)} \quad (3)$$

where c_0 —initial concentration (mol dm⁻³), c_{eq} —equilibrium concentration (mol dm⁻³), V —aqueous phase volume (mL), m —adsorbent mass (g), a_0 —volume activity of

initial solution (mL s⁻¹), a —equilibrium volume activity of solution (mL s⁻¹).

Kinetics

While testing kinetics adsorption, as a liquid phase, a solution of cesium with concentration cesium cations $c_{\text{Cs}^+} = 1 \times 10^{-3}$ mol dm⁻³ was used. As a solid phase a sample of bentonite from Jelšovský potok locality J250 was used. The adsorption process was fast, maximum R was nearly reached within 1 min from the beginning of contact of solid and liquid phase. The comparable values of adsorption percentage were reached in a time interval of 1–480 min (Table 5).

A period of 2 h was chosen for the adsorption experiments.

Almost “instantaneous” capture of the cesium ions on the bentonite can be explained by adsorption and ions exchange on the surface for the cesium ions.

Adsorption experiments and cation exchange capacity

Cation exchange capacity (CEC) is defined by the ability of bentonites to adsorb the cations and keep them in the exchangeable positions—in smectites in the interlayer. During the characterisation of technological types of bentonites three methods of cation exchange determination were used. These are the Ba-method, Mg-method and

Table 5 Effect of shaking time on the adsorption of Cs on bentonite J250

t (min)	R (%)
0.17	86.8
0.50	87.3
1	88.1
5	88.3
10	88.6
15	88.9
30	89.1
60	88.7
120	89.3
240	88.7
480	89.2

Cu-triethylenetetramine method. Table 6 summarizes the CEC values [36].

The qualitative analysis of particular exchangeable cations (valid for all natural bentonite samples except the Dolná Ves sample) confirmed the dominion of Ca and Mg over Na and K elements (Table 6). The distribution of original cations indicates a match between Kopernica, Lieskovec and Jelšový potok bentonite samples, the only difference being that Lieskovec samples show a slightly higher content of Na. Dolná Ves samples are a separate group where the content of exchangeable Na and K cations is in majority (Fig. 4).

The CEC values for particular deposits drop in the following order:

Jelšový potok > Kopernica > Lieskovec > Lastovce > Dolná Ves.

NDV45 bentonites show the lowest CEC values, which were four times lower than in Jelšový potok bentonites (Fig. 4).

While a comparison of cation exchange capacity values for individual bentonite samples of Jelšový potok,

Table 6 The values of cation exchange capacity

Bentonite	Exchangeable cations			
	Na (m _{eq} /100 g)	K (m _{eq} /100 g)	Mg (m _{eq} /100 g)	Ca (m _{eq} /100 g)
J45	4.4	2.6	22.5	53.9
J250	6.1	3.1	20.8	45.8
K 45	7.2	2.5	20.5	51.1
L250	8.3	1.6	11.9	28.1
L 45	6.9	3.1	13.5	30.5
NDV 45	9.4	8.9	0.9	1.5
LA 45	10.3	0.9	13.3	94.3

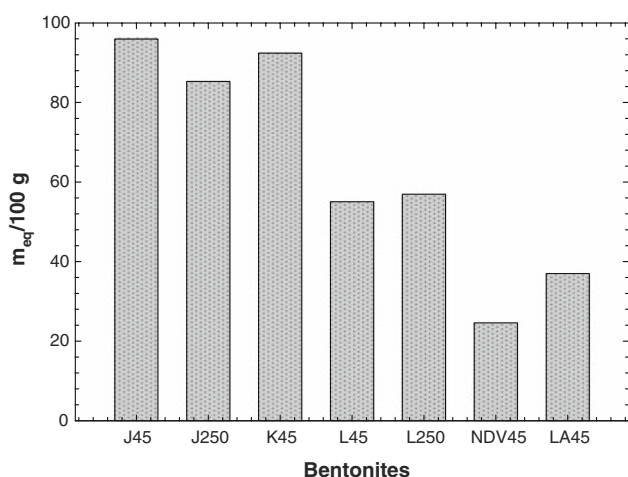


Fig. 4 The average values of CEC

Kopernica, Lieskovec, Lastovce and Dolná Ves (Fig. 4) leads to assumption that the samples of bentonite from Jelšový potok would have the highest values of adsorption capacity (Γ), the highest values of adsorbed amount of Cs were gathered in adsorption experiments with samples of the bentonite of Kopernica.

As the basic adsorption mechanism is used cation exchange. Diversity of adsorption values between individual samples could be due to different cation exchange capacity, different mineralogical structure and difference in surface area of individual samples.

The highest adsorbed amounts were reached with the bentonite sample from locality Jelšový potok J45: adsorption percentage is 97.2% (Fig. 5). The lowest values of adsorbed amount were found for the bentonite sample Dolná Ves NDV45 (adsorption percentage is 83.7%). Poor adsorption values of bentonite NDV45 could be attributed to the mineralogical structure. The sample NDV45, K-bentonite from Dolná Ves ground under 45 μ m represents in comparison with other used bentonites mineralogical different material. Its main clay phase is mixed-layering illite–smectite not present in other bentonites.

The values of adsorbed of Cs⁺ descend in line:

J45 > L45 > K45 > LA45 > NDV45.

The highest values of adsorption capacity Γ were reached in the lowest concentration of Cs in solution. The highest value of distribution coefficient ($K_d = 3,426 \text{ mL g}^{-1}$) in comparison with other bentonites was reached at adsorption of univalent cations Cs⁺, bentonite ground under 45 μ m from Jelšový potok (Table 7). The highest value of adsorption percentage ($R = 97.2\%$) in comparison with other bentonites was also observed in the bentonite sample J45.

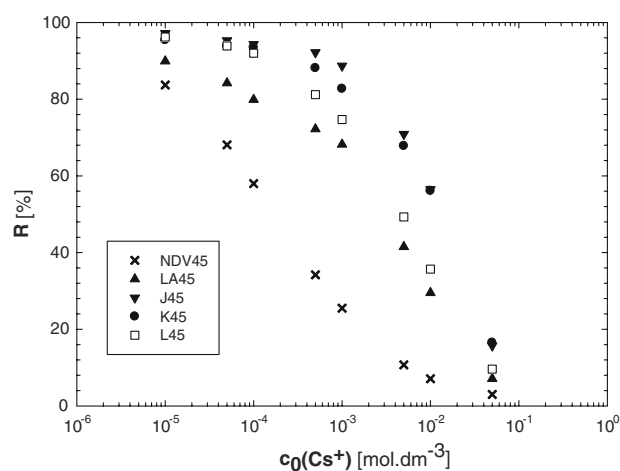


Fig. 5 Dependency of adsorption percentage on initial concentration

Table 7 Adsorption parameters of Cs on bentonite

c (mol dm ⁻³)	J45	K45	L45	LA45
	K _d (mL g ⁻¹)	K _d (mL g ⁻¹)	K _d (mL g ⁻¹)	K _d (mL g ⁻¹)
1 × 10 ⁻⁵	3,426	2,055	2,525	892
5 × 10 ⁻⁵	2,028	1,522	1,532	535
1 × 10 ⁻⁴	1,644	1,450	1,157	397
5 × 10 ⁻⁴	1,185	740	433	260
1 × 10 ⁻³	785	478	296	214
5 × 10 ⁻³	244	211	97	71
1 × 10 ⁻²	130	128	55	42
5 × 10 ⁻²	19	20	11	8

Effect of ionizing radiation

Adsorption of Cs on irradiated forms (J250-irr, L250-irr) was modelled using Langmuir isotherm (Fig. 6). Values of calculated adsorption capacity correspond with values of maximal adsorbed amount. The exposed samples of bentonites J250 and L250 showed higher values of adsorbed quantities than the forms which had not been exposed to radiation, where the J250 sample showed better adsorption qualities for Cs ions.

The studies of the radiation stability of bentonites are very important, due to their planned use as a sealing barrier in the vicinity of radioactive waste and spent nuclear fuel. The bentonite samples have been exposed to radiation in their natural form using a ⁶⁰Co source. The bentonite exposure as a result of interaction of γ -rays led to expansion of the specific surface, increasing of the adsorption capacity, and to the change in the solubility of the clay materials.

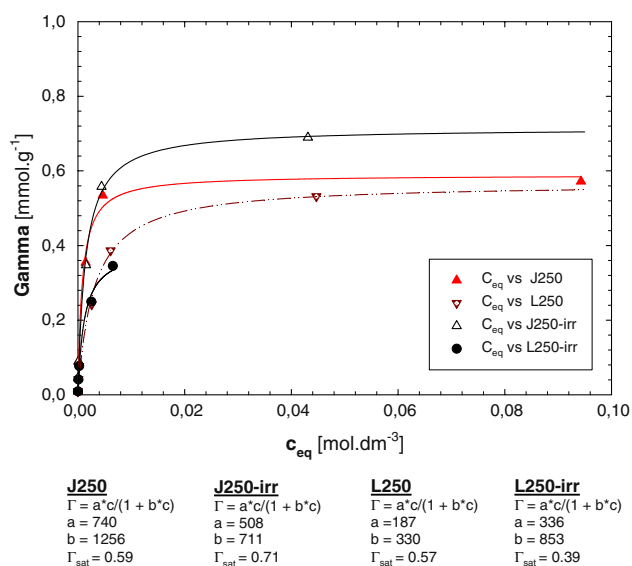


Fig. 6 Langmuir isotherm, Jelšovský potok (J250, J205-irr.) and Lieskovec (L250, L250-irr.)

Effect of particle shape

The influence of illites particle shape on adsorption of cesium was studied on Dolná Ves bentonite samples (Table 8) [38].

Different particle shape illite samples were compared, e.g., Le Puy (shape: “barrels”, France), Muloorina (shape: “barrels”, Australia), L-2A-2 (shape: “hairy”, Poland), RM30 (shape: “plates”) and SG4 (shape: “plates”; Fig. 7) as was shown in paper [37].

The K_d of illites depends strongly on their edge surface area which may be related to frayed edges. The K_d is unrelated to the total surface area of illites, which includes both basal surface area of illite fundamental particles and edge surface area. The highest K_d was found for illites with “barrel” shapes. Adsorption capacity of illites is related closely to their total surface area which is, in the most cases, equivalent to maximum expandability, which is a function of the mean thickness. The poor adsorption properties of “hairy” illite may be related to elongation of crystals along one axis, which may reduce the number of Cs specific sites.

Table 8 Characteristic of sample Dolná Ves

Sample	Shape	Fix	T _{FIX}	%S _{MAX}	TSA	A, B	r	ESA
DV	Plates	0.50	2.4	42	350	560.99	160	0.005

FIX Fixed cations content per O₁₀(OH)₂, T_{FIX} Mean particle thickness (in nm) calculated from fixed cations, %S_{MAX} Maximum expandability calculated from T_{FIX}, TSA Total surface area in m²/g calculated from T_{FIX}, A, B Particle length and width (in nm) along axis a and b, respectively, r Particle radius (in nm) ESA Edge surface area in m²/g calculated from r or A and B

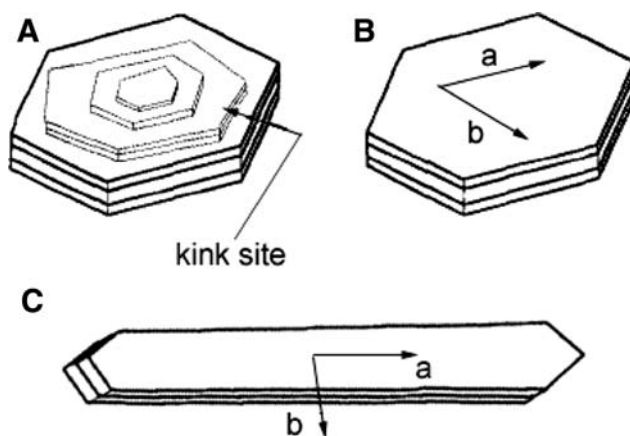


Fig. 7 Three different shapes of illite particles used in the study: A barrel-like illite, B platy illite, and C “hairy” illite

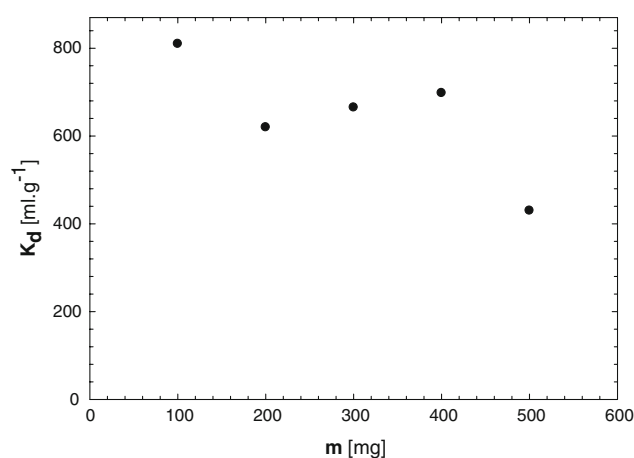


Fig. 8 Effect of adsorbate concentrations

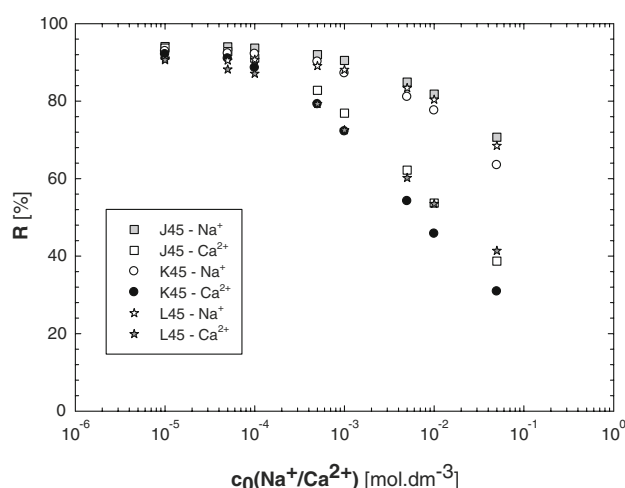


Fig. 9 Dependency of R from initial concentration of univalent and bivalent cations in solution (c_0) on the J45, K45 and L45

Effect of adsorbent concentration

A change in ratio of liquid and solid phase on the distribution coefficients in bentonite sample J250 was studied (Fig. 8). Bentonite in the range of 50–500 mg at constant liquid phase volume 5 mL of CsCl with $c = 1 \times 10^{-4} \text{ mol dm}^{-3}$ in experiments was used. A significant change of distribution coefficient in the respective phase was not observed.

Effect of cations

The Fig. 9 presents the dependency of adsorption percentage from initial concentration of univalent and bivalent cations in solution c_0 on the bentonite from Jelšovský potok, Kopernica and Lieskovec localities.

The influence of competitive cations on adsorption of Cs was observed on bentonites samples J45, K45 and L45. The higher values of adsorption percentage were achieved in

the presence of univalent competitive cations then in the presence of bivalent competitive cations. The adsorption percentage values of cesium increase when initial concentration of competitive solution cations decreases. The highest values of R were achieved at the lowest concentration of ions $1 \times 10^{-5} \text{ mol dm}^{-3}$.

The Ca^{2+} bivalent cation has the most significant influence on Cs adsorption while the univalent Na^+ cation has the least. The results indicate that adsorption of Cs can be heavily influenced by the presence of high cation concentrations of various salts, which can exist in, e.g., waste waters. The influence of cations on adsorption of Cs is possible to explain by competitive ion exchangeable reactions and by occupying of active adsorption centres of bentonites. Presence of other cations causes declining of adsorption percentage of cesium the higher concentration of competing ion.

Conclusion

Following conclusion can be drawn for the five bentonite samples of the five domestic deposits in Slovakia which are considered for use as barriers surrounding the banisters of spent nuclear fuel:

- (1) The bentonites of Jelšovský potok, Kopernica, Lieskovec and Lastovce are characteristic for their good Cs-ion sorption properties. The least suitable mineral composition concerning Cs adsorption on bentonite is that from the repository Lastovce. Potassium containing minerals are present in all samples in the same level. Calcite is present only in Lastovce bentonite. The highest adsorption percentage values and highest adsorption capacity values for cesium ions were reached for the Jelšovský potok and Kopernica bentonites. Bentonites from these two deposits had the highest CEC values.
- (2) The Dolná Ves bentonites have unsuitable sorption properties which are caused by their mineralogical content as well as CEC.
- (3) From the reason of sorption properties it is pointless to use bentonite fractions smaller than $250 \mu\text{m}$.
- (4) The change in ration of solid and liquid phase did not show any effect on Cs adsorption.
- (5) During the comparison of influence of competitive univalent and bivalent cations a higher influence of Ca^{2+} on adsorption of cesium was shown.
- (6) The ionizing radiation significantly influences the sorption properties of bentonites. Higher values of adsorption capacity and adsorption percentage have been discovered with irradiated bentonite samples. The above is of great significance considering the inevitable exposition of bentonites to ionizing radiation.

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