Effect of gamma-irradiation on adsorption properties of Slovak bentonites

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Abstract One of the basic prerequisites for the use of bentonite as engineering barrier in deep geological repositories for radioactive waste and spent nuclear fuel is their stability against ionizing radiation stemming from radionuclides present in radioactive waste and spent nuclear fuel. The aim of this study was to compare the changes in the adsorption properties of selected Slovak bentonites in relation to uranium fission products (^{137}Cs and ^{90}Sr), prior to and after irradiation of bentonites with a ^{60}Co γ-source and specifying the changes in the structure of Slovak bentonites induced by γ-radiation. The changes in irradiated natural forms of Slovak bentonites and the changes in their natrified analogues and fractions with different grain sizes were studied from five Slovak deposits: Jelšový potok, Kopernica, Lastovce, Lieskovec and Dolná Ves. The EPR spectra of bentonites from deposits Jelšový potok and Lieskovec with absorbed doses of 10^4 and 10^5 Gy γ-rays showed no changes in the structure of the studied Slovak bentonites. The changes, which in terms of structure destabilization can be considered insignificant, occurred only in bentonites with absorbed doses of γ-radiation as much as 1 MGy. The absorbed dose of 1 MGy γ-radiation did not have an effect on the adsorption of cesium on every studied bentonite. Changes that can also be regarded as insignificant occurred only during strontium adsorption, especially on Fe–bentonite from deposit Lieskovec and Ca–Mg–bentonite from deposit Jelšový potok, when an increase in the adsorption capacity occurred. Attention should be paid in further research of this topic which would require carrying out experiments on bentonite samples with absorbed doses higher by several orders of magnitude.

Keywords Sorption · Strontium · Cesium · Radiation stability · Slovak bentonites · Illite/smectite · Radioactive waste · Spent nuclear fuel · Deep geological repository

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Introduction

Bentonite and other clay rocks are of paramount importance in the field of environmental and waste management [1–6]. Bentonites from Slovak deposits should be used as part of multi-barrier system in deep geological repositories for spent nuclear fuel and radioactive waste [7–10]. This requires extensive multidisciplinary research a comprehensive and detailed characterization of bentonites [11–17]. The use of bentonite rocks, or bentonite backfills in the repository is destined to their mineralogical, erosion and rheological properties [18–22], and their favorable adsorption and retardation behavior to the activation and corrosion products and the fission products [23–28].

The principal sources of radiation in HLW are β-decay of the fission products (e.g., ^{137}Cs and ^{90}Sr) and α-decay of

the actinide elements (e.g., U, Np, Pu, Am, and Cm). The ^{90}Sr and ^{137}Cs are ecotoxically significant radioisotopes. [137]Cs decay has a half-life of ∼30.07 years and proceeds by both beta decay and gamma emission from an intermediate state. Both the electron and gamma emissions are highly ionizing radiation. The gamma radiation is very penetrating, and the beta radiation, though very short range, is very dangerous when ingested because it deposits all that energy in a very short distance in tissue. [90]Sr undergoes beta decay, emitting electrons with energy 0.546 MeV with a half-life of ∼28.8 years. When dispersed in the environment migrates, adsorbs onto natural matrices and also integrates in the food chain. Presence of such radiotoxic metals in living organisms can lead to somatic as well as genetic changes and for this reason adsorption of these radionuclides is very carefully monitored [11, 29–33].

Adsorption properties of bentonites are determined by their chemical and mineralogical composition, value of cation exchange capacity and specific surface [34–42]. The purpose of the multidisciplinary study of bentonites is to assess comprehensively in the long term the geotechnical properties and to simulate conditions expected in deep geological repository for spent nuclear fuel and radioactive waste [43–55]. One of the basic prerequisites for the use of bentonites as engineering barriers is their stability against exposure to ionizing radiation, which comes from radionuclides present in irradiated fuel and radioactive waste [56–59].

The vast activity during the first 100 years falls on the activity of ^{137}Cs and [90]Sr. Gamma radiation levels during the containment period and α-radiation levels in the packing material during the controlled release period were calculated based on the conceptual design of the waste package for a high-level nuclear waste repository in basalt. The maximum gamma dose rate was 250 rad/h and the maximum absorbed dose during the containment period was 66 Mrad. The alpha radiation levels calculated were strongly dependent on the assumed solubility and sorption of the radionuclides considered. The total absorbed alpha dose was 1.4×10^{10} rad at the packing/container interface, and 1.2×10^{10} and 1.1×10^{10} rad at 7 and 19 cm from the container wall, respectively [60]. According to Weber et al. [11] the total radiation dose originating of mainly from β-radiation, may reach a value of 10^{4} MGy over 100 years. Previous studies concerning γ-irradiation of bentonite and various clay minerals (kaolinite, montmorillonite, palygorskite, muscovite), have only found small or insignificant effects on both the physical and chemical properties of bentonite and montmorillonite, even at very high doses (several MGy) [61–65]. After γ-irradiation of bentonite, there have been no changes detected in the structure [66–71]. There were seen only small systematic changes in water content of smectite. High doses of γ-radiation have a insignificant impact on the destabilization of the structure of clay minerals [72–75].

However, in previous studies of Holmboe et al. [76–78] radiation induced effects manifested as increased structural Fe(II)/FeTot ratio and increased colloid stability, respectively, were found. The effects of γ-radiation on the apparent diffusivity values and sorption coefficients in bentonite for Cs^{+} and Co^{2+} were tested under different experimental conditions. Radiation induced effects on sorption were in general more noticeable for Co^{2+} than for Cs^{+}, which generally showed no significant differences between irradiated and unirradiated clay samples. For Co^{2+} however, the sorption to irradiated MX80 was significantly lower than to the unirradiated clay samples regardless of the experimental conditions. This implies that γ-radiation may alter the surface characteristics contributing to surface complexation of Co^{2+}.

This article builds on previous work by our department that dealt with the study of adsorption properties of bentonite barriers and factors which would affect these characteristics [79–83]. The aim of this research is to compare the changes in the adsorption properties of Slovak bentonites in relation to the fission products of uranium, i.e. [137]Cs and ^{90}Sr, prior to and after irradiation of bentonites with a [60]Co γ-source; to specify the changes in the structure of Slovak bentonites induced by γ-radiation, by means of EPR method.

Experimental

Solid phase

Bentonite samples, containing mainly dioctahedral smectites of various chemical and mineralogical compositions were taken from five Slovak deposits: Jelšový potok, Kopernica, Lieskovec, Lastovce and Dolná Ves (Table 1).

Genetically they are partially different deposits, even when their common feature is that they were all formed as a product of volcanic activity and subsequent effect of alteration processes. Two groups of deposits from the bentonite on the base of qualitative point were chosen:

1. Smectic deposits—a basic component forming clay part of a rock are smectites:

   - high smectic—contains more that 60% (Kopernica, Jelšový potok, Lieskovec);
   - low smectic—contains about 40% (Lastovce).

2. Illite/smectite deposit—a basic component comprising clay part of a rock is mixture layered mineral illite/smectite (Dolná Ves).

From the view of crystallochemical characteristics of the main clay component deposits structure can be divided this way:

Table 1 Used samples of Slovak bentonites

Deposits	Jelšový 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
Natural forms	J	K	L	NDV	LA
Natrified forms	BJ	BK	BL	–	–
Sr-saturated forms	J-Sr	–	L-Sr		
Irradiated forms	J-irr	K-irr	L-irr	NDV-irr	LA-irr
Grain size (μm)	15, 45, 250	15, 45	15, 45, 250	45	45

Table 2 Chemical composition

Sample	SiO_2 (%)	Al_2O_3 (%)	CaO (%)	Fe_2O_3 (%)	MgO (%)	MnO (%)	Na_2O (%)	K_2O (%)	TiO_2 (%)	P_2O_5 (%)	SO_3 (%)
J15	67.90	21.12	1.61	2.88	3.44	0.08	0.64	1.67	0.16	0.32	0.21
J45	67.41	21.32	1.78	2.81	4.22	0.09	0.40	1.01	0.09	0.71	0.21
J250	68.33	20.50	2.03	2.52	4.37	0.07	0.64	1.23	0.18	0.06	0.07
BJ15	65.25	21.51	2.62	2.65	2.84	0.07	3.09	1.00	0.08	0.73	0.16
BJ45	66.00	22.67	1.55	2.62	2.84	0.07	3.02	1.04	0.12	0.01	0.07
K15	66.09	23.96	1.90	2.69	2.93	0.05	0.43	1.68	0.15	0.01	0.11
K45	66.05	23.93	2.00	2.61	2.78	0.05	0.26	1.21	0.09	0.98	0.06
LA45	74.59	14.46	4.35	2.51	1.55	0.05	1.27	0.87	0.24	0.05	0.05
L15	65.22	21.00	1.43	7.02	1.82	0.08	0.59	1.87	0.70	0.08	0.20
L45	67.26	19.54	1.31	6.98	1.68	0.09	0.51	1.70	0.75	0.09	0.11
L250	63.40	22.29	1.17	8.19	1.59	0.04	0.46	1.73	0.87	0.20	0.07
DV45	66.53	25.04	0.14	2.43	0.62	0.02	0.05	4.97	0.15	0.03	0.03

- Al–Mg montmorillonite—smectite is identified as a montmorillonite and its octaedric structure is formed mainly by Al and Mg (Kopernica, Jelšový potok, Lastovce),
- Fe montmorillonite—smectite is identified as a montomorillonite and his octaedric structure is formed mainly by Fe (Lieskovec),
- Illitic-smectic—main clay component is mixture layered illite/smectite (Dolná Ves).

The chemical and mineralogical composition of the bentonite samples are in Tables 2 and 3.

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 desiccator, where they were stored till next use.

Aqueous phase and radiotracer

Adsorption experiments were realized in the Cs and Sr concentration range 1×10^{-5}–5×10^{-1} mol L^{-1} solutions prepared from CsCl and $Sr(NO_3)_2$. In experiments chemicals of ordinary laboratory purity from supplier Slavus, s.r.o., Bratislava SR, or Lachema n.p., Brno CZ were used. Radio-isotope ^{137}Cs with the volume activity 1 MBq mL^{-1} and ^{85}Sr with the volume activity 3.7 MBq mL^{-1} were used as radiotracers.

Irradiation of bentonite samples

Irradiating the samples was a time consuming and technically demanding process that aimed to achieve the highest absorbed radiation dose. The interval of radiation doses (10^4, 10^5 and 10^6 Gy) was chosen based on studies published so far which dealt with radiation stability of clay minerals and the options of irradiation facility. As a source of ionizing radiation a ^{60}Co γ-source was chosen.

The bentonite samples were not modified before irradiation; they were irradiated dry and under aerobic condition.

For capacity reasons of the irradiation facility the highest absorbed radiation dose was ∼1 MGy. The adsorption experiments with those samples which were irradiated with absorbed doses lower than 1 MGy are not

Table 3 Mineralogical composition

Sample	Quartz	Albite	Biotite	Kaolinite	Smectite	K-felspar	Cristobalite
J15	9.5	–	1.5	3	76	8.5	1.5
J45	6	S	1	2.5	86	3.5	1
J250	2.5	0.5	2	2	82	9.5	1.5
K15	6	S	2.5	2	80	8	1.5
K45	6.5	S	2.5	1	82	6	2
L15	5.5	1	1.5	6.5	67.5	12	6
L45	12	1.5	0.5	6	64	9.5	6.5
L250	9.5	0.5	2	9.5	64	9.5	5
LA45	13	7.5	–	–	40.5	7	27
BJ15	2	S	0.5	–	94	3.5	–
BJ45	11	1	1.5	–	74	11	1.5
BJ250	6	S	1	–	84.5	8.5	–
DV45	47.5	2	–	–	40.5	7	27

mentioned in the article. Their analyses showed no changes compared to the non-irradiated samples. Table 4 shows the conditions of irradiating the bentonite samples with absorbed dose 1 MGy.

Batch method and measuring of radioactivity

Adsorption of Cs and Sr on the samples of bentonites was studied through radioisotope indication using radioisotope of ^{137}Cs and ^{85}Sr in static arrangement of experiment, in aerobic conditions at laboratory temperature. Adsorption parameters were determined after mixing in 0.05 g of sorbent 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, 3500 rot/min.) was from supernatant extracted 1 mL to measure radioactivity. Radioactivity determination of ^{137}Cs and ^{85}Sr solutions was done with spectrometer using NaI(Tl) detector. The statistical error of the measurement was below 1%.

Table 4 Conditions of irradiating the bentonite samples

Maximal absorbed dose (MGy)	1
Irradiation source	^{60}Co
Energies of γ-rays	1.17 MeV
	1.33 MeV
Max absorbed dose	
External side	2 kGy h^{-1}
Reversal side	1 kGy h^{-1}
Depth	5 cm
Irradiation time	1 month

Electron paramagnetic resonance

Electron paramagnetic resonance (EPR) or spin magnetic resonance is used only in systems of paramagnetic nature. Defects caused by γ-radiation are paramagnetic in nature, and therefore EPR is suitable for their characterization. 20–25 mg of bentonite of about 2 mm in diameter was poured into thin 10 cm cuvettes. They were placed into ERS 230 EPR spectrometer (ZWG Akad. Wiss. Berlin, Germany), which operates in X-band (~ 9.3 GHz) with modulation amplitude 0.1 mT and microwave power 5 mW. The EPR spectra were obtained by magnetic field modulation to obtain, which are the first derivatives of absorption spectra.

Results and discussion

Adsorption properties of bentonite were calculated by following equations:

Distribution coefficient $K_d = (a_0 - a/a) \times V/m \, (\text{mL g}^{-1})$ (1)

Adsorption percentage $R = (100 \times K_d)/(K_d + V/m) \, (\%)$ (2)

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

where c_{eq}—equilibrium concentration (mol L^{-1}), V—aqueous phase volume (mL), m—sorbent mass (g), a_0—volume activity of initial solution (mL s^{-1}), a—equilibrium volume activity of solution (mL s^{-1}).

Shaking time

The adsorption of Cs and Sr was investigated as a function of shaking time in previous experiments and the results

Table 5 Values of the Sr-adsorption on non-irradiated and irradiated bentonites from Jelšový potok and Lieskovec

Samples:	J250		J250irr		L250		L250irr	
c_{Sr2+} (mol L^{-1})	R (%)	K_d (mL g^{-1})	R (%)	K_d (mL g^{-1})	R (%)	K_d (mL g^{-1})	R (%)	K_d (mL g^{-1})
5×10^{-1}	1.3	1.4	1.2	1.4	0.7	0.7	0.6	0.6
1×10^{-1}	5.1	5.4	6.5	6.9	2.2	2.2	2.7	2.7
5×10^{-2}	6.7	7.2	7.2	7.8	4.1	4.3	4.8	5
1×10^{-1}	26.1	35.2	28.6	40.0	21.6	27.5	25.9	34.9
5×10^{-3}	43.9	78.3	46.7	88	29.9	42.7	34.3	52.2
1×10^{-3}	75.0	299	81.0	425	70.0	233	77.2	339
5×10^{-4}	80.7	419	84.1	529	78.9	373	86.3	630
1×10^{-4}	90.2	916	95.6	2176	84.6	549	91.2	1038
5×10^{-5}	94.3	1658	97.7	4173	90.0	900	97.3	3631
1×10^{-5}	95.5	2139	98.7	7374	90.4	942	98.1	5076

show that the adsorption is very fast and equilibrium is reached almost instantaneously after mixing [23, 79–83]. 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. A period of 2 h was chosen for the further adsorption experiments.

Adsorption of Cs comparing to Sr cations reach higher R values.

Almost "instantaneous" capture of the Cs and Sr ions on the bentonite can be explained by adsorption and/or ions exchange with some ions on the surface of the bentonite. The sharp increase in the sorption percentage at pH 6 can be due to hydrolytic adsorption by ion exchange in consequence of the reaction between the $Sr(OH)^+$ and OH^- groups on the surface of the adsorbent and lower concentration of competitive H^+ ions for adsorption sites as well. In the systems open atmosphere and above pH 7, some of the Sr^{2+} uptake could also be due to the precipitation of $SrCO_3$ on the bentonite surface [23].

Adsorption of Cs(I) and Sr(II)

Adsorption of cesium and strontium (Table 1) was studied in the whole concentration range of Cs and Sr (from od 1×10^{-5} do 5×10^{-1} mol L^{-1}), for all non-irradiated and irradiated samples of the Slovak bentonites. Adsorption parameters for non-irradiated forms of bentonites, on their natrified analogues and samples with different grain size are consistent with data published by Galambos et al. [79–83]. When comparing the adsorption parameters for unirradiated and irradiated forms, the differences were not observed in values for Γ, R and K_d.

Adsorption parameters for non-irradiated strontium forms bentonites, their natrified analogues and samples with different grain size are consistent with the results of

experiments published by Galambos et al. [79, 80, 82].Therefore, the results of the experiments with non-irradiated samples will be discussed further. In Figs. 1 and 2 is presented dependance of the strontium adsorption percentage from its initial concentration in solution for samples of bentonites from Jelšový potok and Lieskovec deposit.

When comparing non-irradiated and irradiated samples of Jelšový potok deposit, in the irradiated samples a slight increase in strontium adsorption has been observed (Fig. 1). Differences in values started already at the highest initial concentration of strontium $c_0 = 5 \times 10^{-1}$ mol L^{-1} and continued throughout the studied concentration range of strontium. The most significant difference in values was at the initial concentration of strontium $c_0 = 5 \times 10^{-3}$ mol L^{-1}, where the $R_{J45} = 49\%$ and $R_{J45irr} = 55\%$.

When comparing non-irradiated and irradiated samples of the bentonite from deposits Lieskovec, an increased strontium adsorption (Fig. 2) has been observed in the irradiated samples. Differences have been seen already at the highest initial concentration of strontium $c_0 = 5 \times 10^{-1}$ mol L^{-1} and continued throughout the studied concentration range of strontium. After irradiation of the sample L45, at the highest initial concentration of strontium $c_0 = 5 \times 10^{-1}$ mol L^{-1}, value of the adsorption (%) increased from 0.5 to 1%. After irradiation of the sample L45, at low initial strontium concentration $c_0 = 1 \times 10^{-5}$ mol L^{-1}, value of the adsorption (%) increased from 90 to 97%.

When comparing non-irradiated and irradiated samples of the bentonites from the deposits in Kopernica, the situation is similar to bentonite deposits of Jelšový potok and Lieskovec. For the irradiated samples was observed an increase of adsorbed strontium. The differences started at the highest initial concentration of strontium $c_0 = 5 \times 10^{-1}$ mol L^{-1} and continued throughout the studied concentration range of strontium. After irradiation of the sample K45, at the highest initial concentration of strontium $c_0 = 5 \times 10^{-1}$ mol L^{-1},
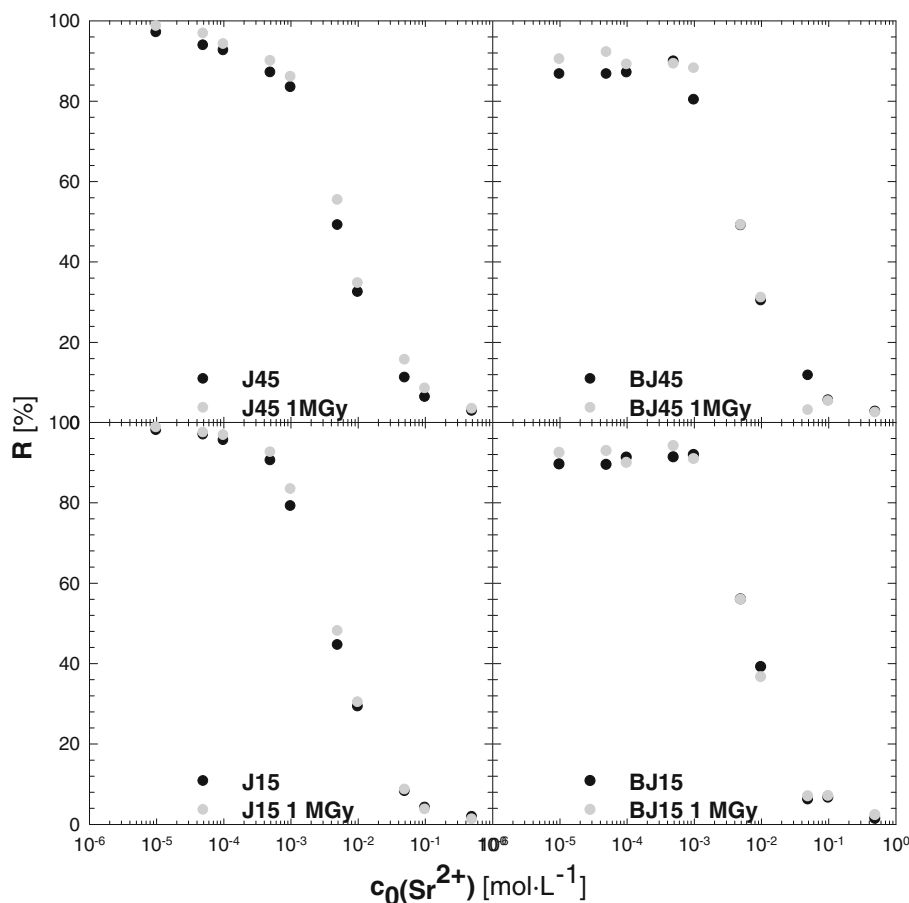
value of the adsorption (%) increased from 1 to 1.5%. After irradiation of the sample K45, at low initial strontium concentration $c_0 = 1 \times 10^{-5}$ mol L^{-1}, no increase of the value of adsorption (%) has been observed.

When comparing non-irradiated and irradiated samples of the bentonite from deposits in Lastovce and Dolná Ves, the situation is identical to that seen with samples from Jelšový potok, Lieskovec and Kopernica. For the irradiated samples were recorded increased adsorption capacity and adsorption percentage values of strontium. The differences started at the highest initial concentration of strontium $c_0 = 5 \times 10^{-1}$ mol L^{-1} and continued throughout the studied concentration range of strontium.

After irradiation of the sample LA45, at the highest initial concentration of strontium $c_0 = 5 \times 10^{-1}$ mol L^{-1}, value of the adsorption (%) has not changed. An increase has been possible to record from the Sr-concentration $c = 1 \times 10^{-1}$ mol L^{-1} from 1.4 to 2.4%. After irradiation of the sample K45, at low initial strontium concentration $c_0 = 1 \times 10^{-5}$ mol L^{-1}, an increase of the value of adsorption (%) from 69 to 72% has been observed.

After irradiation of the sample DV45, at the highest initial concentration of strontium $c_0 = 5 \times 10^{-1}$ mol L^{-1}, value of the adsorption (%) has not changed. An increase

has been possible to record from the Sr-concentration $c = 1 \times 10^{-2}$ mol L^{-1} from 0.3 to 4.6%. After irradiation of the sample K45, at low initial strontium concentration $c_0 = 1 \times 10^{-5}$ mol L^{-1}, an increase of the value of adsorption (%) from 40 to 44% has been recorded.

Langmuir isotherms for adsorption of Cs and Sr on bentonites Jelšový potok and Lieskovec

Langmuir's adsorption isotherms for Cs and Sr on bentonites from deposits Jelšový potok and Lieskovec for their non-irradiated and irradiated forms are shown in Fig. 3. The Langmuir adsorption of Cs compared to the adsorption of Sr achieved higher levels of adsorption capacity Γ. The Langmuir adsorption of Cs at 0.5 mol L^{-1}, the non-irradiated sample J250 achieved higher levels of adsorption capacity ($\Gamma = 0.95$ mmol g^{-1}) compared to the sample L250 ($\Gamma = 0.50$ mmol g^{-1}). That difference in the values of Γ was observed until concentrations of Cs$^+$ $c_0 = 1 \times 10^{-3}$ mol L^{-1}. The value Γ were approximately identical within the Cs$^+$ concentration ranging from 5×10^{-4} to 1×10^{-5} mol L^{-1}. For adsorption of Sr at 0.5 mol L^{-1}, the non-irradiated sample J250 achieved higher levels of

Fig. 2 The dependency of the adsorption (R; %) on the initial Sr-concentration in the solution (c_0) on the non-irradiated and irradiated bentonites Lieskovec
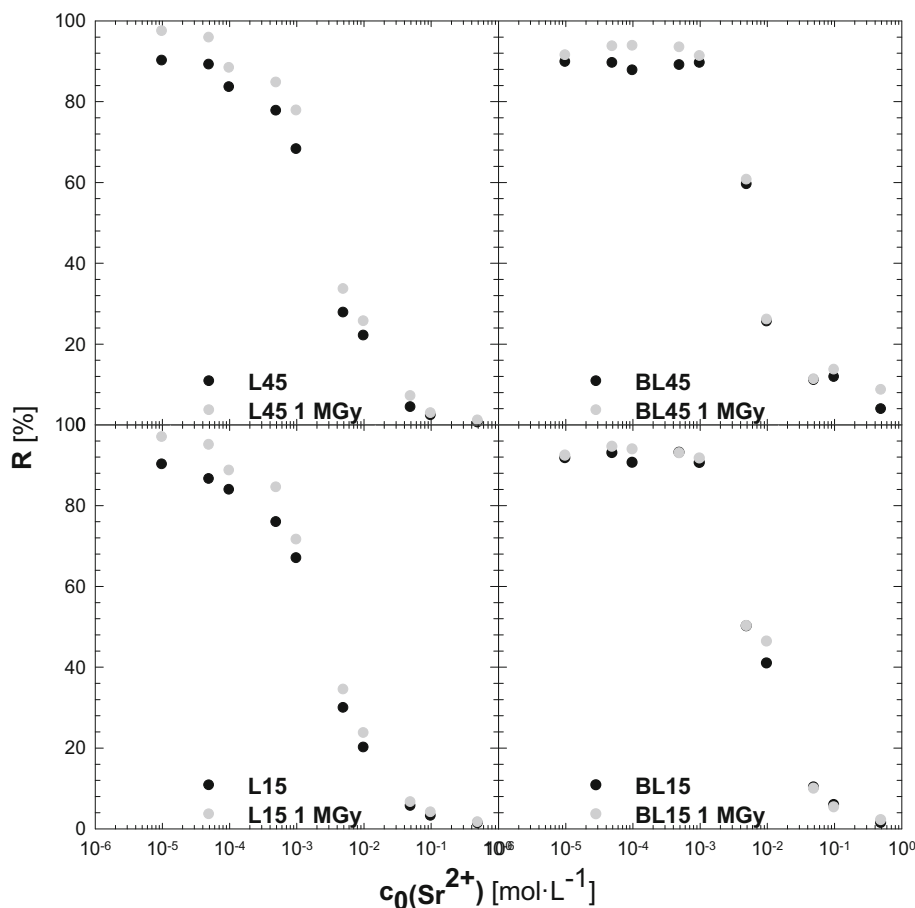
adsorption capacity ($\Gamma = 0.61$ mmol g^{-1}) ain comparison to the sample L250 ($\Gamma = 0.27$ mmol g^{-1}). That difference in the values of Γ was observed throughout the studied range of concentrations of strontium.

A cation-exchange mechanism on the basal surface and edge sites are the main chemical process during adsorption. Differences in studied samples could arise from differences of CEC, differences in the mineralogical compositions and differences in the specific surface area of the individual samples.

When comparing the adsorption of Cs on the non-irradiated and irradiated form, as for J250 and also for L250 there were not observed disparities in the values of Γ. For compared Sr adsorption on irradiated and non-irradiated specimens, as for J250 and J250irr and also L250 and was L250irr, there were observed disparities in the values of Γ. On non-irradiated J250 specimen, at the lowest concentration of Sr, the value of Γ equalled to 9.55×10^{-4} mmol g^{-1}, while for irradiated specimen J250irr the value was higher, $\Gamma = 9.87 \times 10^{-4}$ mmol g^{-1}. For adsorption of Sr the differences of Γ values are more significant between L250 and J250 specimens. At the lowest concentration of Sr was the Γ value equal to 9.04×10^{-4} mmol g^{-1} in non-irradiated L250 specimen, whereas

in irradiated specimen L250irr was its value higher; $\Gamma = 9.80 \times 10^{-4}$ mmol g^{-1}.

Values of distribution coefficients and of percentage of adsorption for strontium on J250 and J250irr; L250 and L250irr are listed in Table 5. Similar as to Γ values also the values of R and K_d differ in non-irradiated and irradiated specimens. Larger differences were observed in L250, where R and K_d values at the lowest Sr concentration in non-irradiated/irradiated specimens were as follows: $R = 90.4\%$ (L250) and $R = 98.0\%$ (L250irr) and $K_d = 942$ mL g^{-1} (L250) and $K_d = 5076$ mL g^{-1} (L250irr).

EPR study

In the adsorption experiments of cesium there was no effect of γ-radiation on the adsorption processes of bentonite found. As for strontium, there was a slight increase in the adsorption capacity observed, which can however be attributed to statistical uncertainty. As a direct method allowing the identification of changes in the structure of the studied clay minerals, electron paramagnetic resonance spectroscopy (EPR) was chosen. The advantage of EPR is high sensitivity; under favourable conditions it is possible to measure paramagnetic systems with spin concentration

Fig. 3 Langmuir isotherms for Cs^+ and Sr^{2+} adsorption on the non-irradiated and irradiated bentonites Jelšový potok (J250) and Lieskovec (L250)
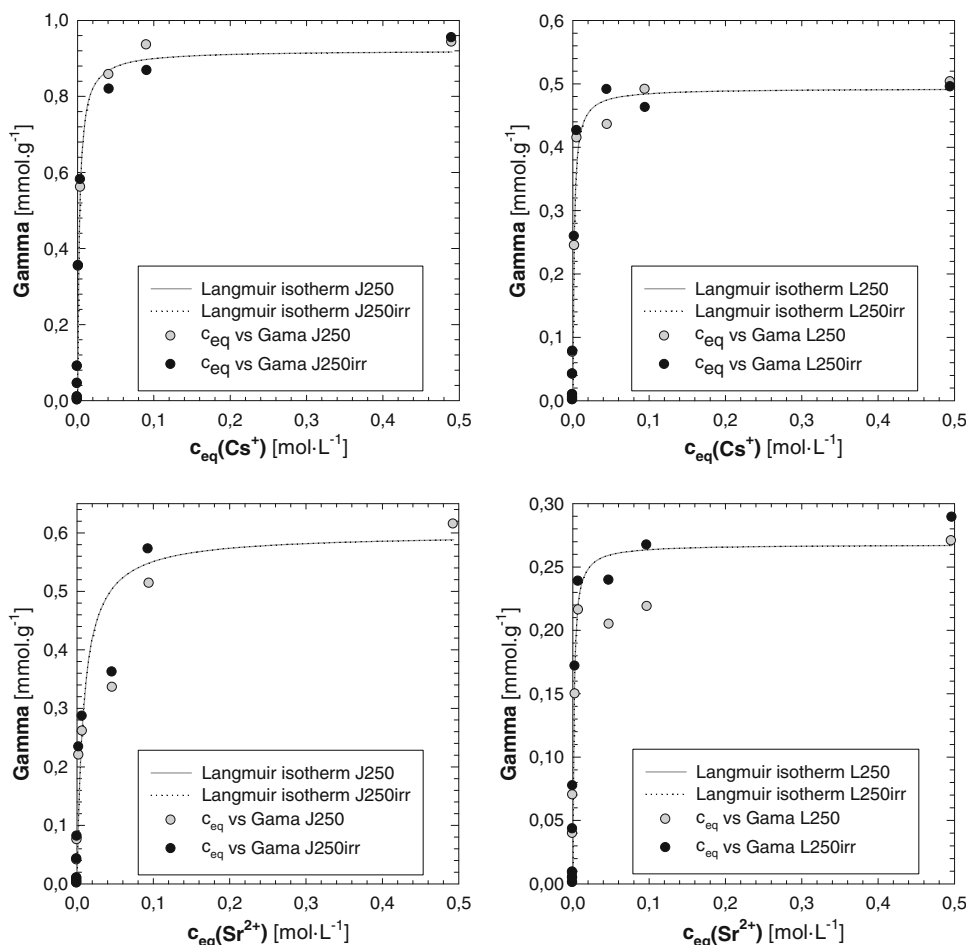
of 10^{-9} mol L^{-1}. Most smectites provide Fe^{3+} EPR spectrum in the area of $g = 4.3$ and $g = 2.0$.

EPR spectroscopy was used to study the bentonites from deposits Jelšový potok and Lieskovec, the samples prior to and after irradiation, and the samples which were saturated by $Sr(NO_3)_2$ of three different concentrations: 10^{-1}, 10^{-3}, 10^{-5} mol L^{-1}; and the samples which were irradiated with three different doses of γ-rays 10^4, 10^5 and 10^6 Gy:

1. J15, J15irr, J15-Sr, J15irr-Sr,
2. J250, J250irr, J250-Sr, J250irr-Sr,
3. L15, L15irr, L15-Sr, L15irr-Sr,
4. L250, L250irr, L250-Sr, L250irr-Sr,
5. L250 10^4 Gy, L250 10^5 Gy, L250 10^6 Gy.

Figure 4a displays the EPR spectrum of bentonite from deposit Jelšový potok J15irr and J15. In 156 to173 mT magnetic field there is a peak located whose calculated values of the effective Zeeman g-factors are (J15) $g = 4.11$ for the non-irradiated bentonite and (J15irr) $g = 4.13$ for the irradiated bentonite. They correspond to Fe^{3+} incorporated into the smectite structure, which are exchanged for Al^{3+}. Due to the absence of a frequency meter in the spectrometer, the g-factors were calculated by means of

comparison to known values of the g-factor $g(B_{Dpp}) = 2.0036$ and the magnetic field for the electron $B_{Dpp} = 335.7$ mT as follows:

$$hv = g(B_{Dpp})\beta B_{Dpp} \tag{4}$$

$$g(B_{Dpp})\beta B_{Dpp} = g_1 \beta B_1 \tag{5}$$

$$g_1 = \frac{g(B_{Dpp})B_{Dpp}}{B_1} \tag{6}$$

This way all the other values of the effective Zeeman g-factor were calculated. Other peaks for the non-irradiated and irradiated samples are broad signals at 278–335 mT. This is Fe^{3+} in the interlayer space, in clusters or FeO(OH). This signal is characterized by a strong spin–spin interaction. The values of g-factors are $g = 2.02$ for J15 and $g = 2.01$ for J15irr. The narrow signal in the 334–336 mT area with $g = 2.01$ belongs to the trapped electron in the axial symmetry, which is typical for smectites.

Figure 4b shows the EPR spectrum of bentonite from deposit Jelšový potok J250 and J250irr. The peaks in the 156–173 mT area with $g \approx 4.1$ correspond to Fe^{3+} in the structure. In the non-irradiated sample (J250) the presence of manganese was proved with $g = 2.04$, in the range of

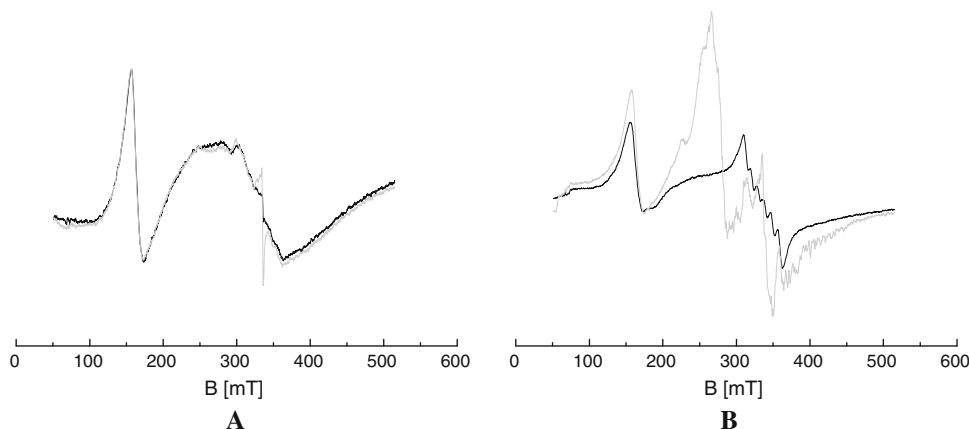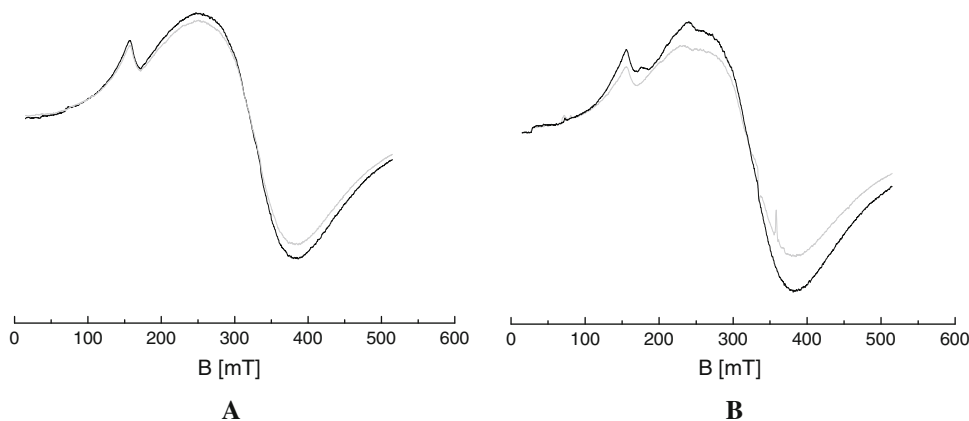Fig. 4 EPR spectrum of bentonite from deposit Jelšový potok **a** J15 and J15irr, **b** J250 and J250irr

A

B

Fig. 5 EPR spectrum of bentonite from deposit Lieskovec **a** L15 a L15irr, **b** L250 a L250irr

A

B

magnetic field from 310 to 356 mT. Manganese is one of those elements that are normally not found in the bentonites from deposit Jelšový potok. The presence of manganese can be explained by the contamination of the sample during mechanical modification—grinding—of the bentonite mineral. The 313–349 mT area of the irradiated sample (J250irr) was identified as the A-center, i.e. a structural defect of –Si–O$^-$ type with values of $g_{\parallel} = 2.15$, $g_{\perp} = 1.93$ and mean $g = 1.99$. So far there has been no structural defect identified in the 266–288 mT area with $g = 2.4$.

In Fig. 5a the first peak from the left with $g = 4.14$ is for L15 and 4.13 for L15irr which are assigned to Fe^{3+} in the structure. The broad peak with $g = 2.06$ for L15 and 2.07 for L15irr account for Fe^{3+} in the interlayer space. It follows that the γ-radiation dose of 1 MGy had no effect on the structure of the L15 sample. The same situation was observed for sample L250 (Fig. 5b). At a magnetic field of ~ 333 mT there is a visible sign of a trapped electron for the irradiated L250irr sample.

The saturation with $Sr(NO_3)_2$ proved an effect on the spectrum of the irradiated sample (J250irr-Sr). For the saturated samples of bentonites with $Sr(NO_3)_2$ there is the peak missing for the A-center and the peak of an unidentified defect. These findings can be explained by

recombining the paramagnetic defects with strontium cations. The trapped electrons were not affected by this modification. The EPR spectra of bentonite from deposit Lieskovec L250 saturated with $Sr(NO_3)_2$ at concentrations of 10^{-1}, 10^{-3} and 10^{-5} mol L^{-1} showed that all the peaks correspond to Fe^{3+} in the smectite structure. The irradiated samples have the same course as the non-irradiated ones. Only at concentration of 10^{-5} mol L^{-1} $Sr(NO_3)_2$ the signal strength is less, which is caused by the conversion of weight to 20 mg.

The EPR spectra of the bentonite samples from deposits Jelšový potok and Lieskovec, which were irradiated at three different doses of γ-radiation 10^4, 10^5 and 10^6 Gy, are shown in Fig. 6. No significant changes occurred in the L250irr sample. In the J250irr bentonite the 10^4 and 10^5 doses did not elicit any changes either. Their course is similar to the one of the non-irradiated bentonite. Changes occurred only at a dose of 10^6 Gy; they are discussed above.

Stríček et al. [58, 59] studied the effect of ^{60}Co irradiation on Slovak bentonites. The changes on clay fractions (>2 μm) from deposits Jelšový potok and Lieskovec were studied in three different forms: dried at 105 °C, hydrated, and hydrated with the addition of 10% powdered metal Fe^0 (after irradiation of the samples, the Fe^0 admixture was

Fig. 6 EPR spectra of bentonite **a** J250irr and **b** L250irr—irradiated with doses of 10^4, 10^5 and 10^6 Gy
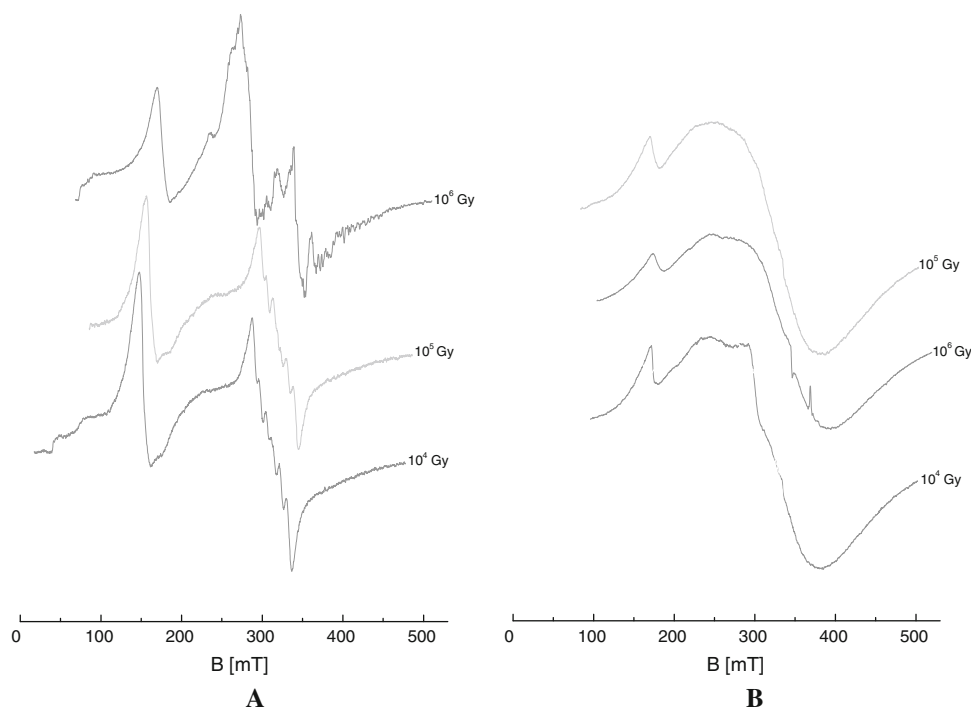
removed by permanent magnet). The total absorbed radiation dose was 0.2, 0.7, 1 and 1.5 MGy; the dose rate was at 0.21 kGy h^{-1}. In order to determine changes in irradiated bentonites, as the basic method X-ray diffraction analysis of powdered non-oriented and powdered oriented samples was used. To verify changes in the structure of smectite, IR spectroscopy was used, which provides information on the occupation of the tetrahedral and octaedric site of smectite and at the same time, it allows to identify newly formed mineral phases, and also to check the presence of newly formed amorphous form of SiO_2, which could indicate the decomposition/degradation of smectite structure. The mineral composition, IR spectra, CEC as well as the specific surface area of the original and irradiated samples is the same. The thickness distribution of smectite crystals and their average thickness confirmed the mineral stability of the irradiated smectite. Measuring the charge in smectites proved a difference in the hydrated sample from Jelšový potok, where the charge fell slightly after absorbing a 1.5 MGy dose. The minor deviations compared to the baseline non-irradiated material, which occurred in some cases after conducting the experiments, have no significant effect on the properties of the studied Slovak bentonite samples.

Conclusion

The EPR spectra of bentonites from deposits Jelšový potok and Lieskovec with absorbed doses of 10^4 and 10^5 Gy

γ-radiation showed no changes in the structure of the studied Slovak bentonites. The changes, which in terms of structure destabilization can be considered insignificant, occurred only in bentonites with absorbed doses of γ-radiation as much as 1 MGy. The absorbed dose of 1 MGy γ-radiation did not have an effect on the adsorption of cesium in every studied bentonite. The changes, which can also be regarded as insignificant, occurred only during strontium adsorption, especially in Fe-bentonite from deposit Lieskovec and Ca–Mg–bentonite from deposit Jelšový potok, when an increase in the adsorption capacity occurred. Attention should be paid in further research of this topic which would require carrying out experiments on bentonite samples with absorbed doses higher by several orders of magnitude.

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