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ORIGINAL PAPER

Clay-salt slimes of the JSC “Belaruskali” as potential engineering barriers in the radioactive waste repositories: sorption of Cs(I), Sr(II), Eu(III) and Am(III)

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Abstract Results of sorption of caesium(I), strontium(II), europium(III) and americium(III) ions on the aluminosilicates separated from the clay-salt slimes (CSS) depending on the contact time, pH, initial metal concentration are presented. CSS 1A and CSS 1B sorbents appear to be sufficiently chemically and radiation stable for practical application in treatment of the radioactive wastewaters.

Keywords Radionuclides · Clay-salt slimes · Sorbents · Liquid radioactive waste · Waste management · Engineering barriers

Introduction

Environmental studies permanently stand in front of goal to develop novel technologies for treatment of liquid wastes. Traditional methods for removing metals from aqueous solutions comprise chemical precipitation, chemical oxidation or reduction, ion exchange, filtration, electrochemical treatment, various membrane technologies and evaporation recovery. However, these technologies may be ineffective or extremely expensive, particularly when the concentration of the metals is below 1 mg/L (Nourbakhsh et al. 1994). Besides, the conventional technologies may form big amounts of toxic, non-ecofriendly sludge which may be costly in the storage. Therefore, reduction of toxic and/or radioactive metals to an environmentally safe level by a relative cheap and environmentally friendly way is still needed.

Sorption of radionuclides on the mineral sorbents is an effective and universal method for their sequestering from aqueous solutions (Rajec et al. 1996; Galamboš 2012a, b, c, 2013; Thomson et al. 2003; Abdel Rahman et al. 2011). Among different sorbents, aluminosilicates appear to be of special interest. Because of their high chemical, thermal and radiation resistance, as well of high affinity to metal ions, they have been proposed as ideal backfill materials for engineering and geological barriers in the repositories of the radioactive wastes. The most popular include the bentonite and the montmorillonite (Legoux et al. 1992; Galamboš et al. 2011).

Present studies done by the group from two countries are continuation of the work commenced in the Institute of Nuclear Chemistry and Technology (Poland) in the frame of Task 4 “Development of spent nuclear fuel and radioactive waste management techniques and technologies” of the governmental strategic research project

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"Technologies supporting development of safe nuclear power engineering." Among others, those studies concerned sorption of selected radionuclides using domestic aluminosilicates (Grabias et al. 2015). Simultaneously, the work follows research carried out in the Republic of Belarus (Maskalchuk 2016a, b). In detail, in the present work we have checked sorptive properties of two fractions coming from the clay-salt slimes (CSS) which are the industrial wastes of the Joint Stock Company (JSC) "Belaruskali" (Soligorsk, Belarus) and contain mainly the aluminosilicates. Basic properties of CSS 1A and CSS 1B materials may be found in the papers by Maskalchuk (2016a).

In the presented work, effectuated within the last year, adsorption of mono-, di- and trivalent metals has been studied in dependence of the time applied for contact the phases, acidity of the decontaminated solution, sorbent dosage as well as impact on sorption process of the optionally present chelating agents. In addition, properties of the sorbents have been examined by the SEM method and determining their zeta potentials. The FT-IR spectra have been recorded with the aim to check the radiation stability of the sorbents. Adsorption distribution coefficients of the proposed sorbents for strontium(II) and americium(III) was compared with the corresponding values for selected sorbents found in the literature.

Materials and methods

Sorbents

The crude material had been taken from the storehouse No. 1 of the Joint Stock Company JSC "Belaruskali" (Soligorsk, Belarus) and washed five times with the distilled water to remove the soluble salts (mainly, NaCl and KCl) and dried at 50 °C to the constant mass (for about 6 h). The obtained material in further will be called 1A sorbent. Part of the 1A was treated with 0.1 M hydrochloric acid to destroy the carbonates, washed with the distilled water and dried at 50 °C for 6 h. This material will be called as 1B.

Other reagents

All chemicals used in the experiments were purchased from Sigma-Aldrich Inc (Poland). Water used was deionized.

Carrier-free radionuclides of caesium-137 ($t_{1/2} = 30.07$ y; $E_\gamma = 661.7$ keV), strontium-85 ($t_{1/2} = 64.8$ d; $E_\gamma = 514$ keV), europium-152/4 ($t_{1/2} = 13.5$ y; $E_\gamma = 344.3$ keV) and americium-241 ($t_{1/2} = 432.2$ y; $E_\gamma = 59.5$ keV) were supplied from POLATOM Swierk (Poland).

Adsorption of the radionuclides

Sorption tests were preceded by spiking solution of the desired pH with the radionuclides forming the *nca* concentration.[1] If not stated else, 50 mg of the sorbent weighted in the polyethylene test tubes was mixed with 5 cm^3 of the radioactive solutions and shaken for the required time. Except the studies of the equilibration time dependence, typical contact time was 3 h. Next, the solutions were separated by centrifuging (10 min.; 14,000 rpm) and taken for measurement of the radionuclides specific content (C) together with the initial concentration (C_0). Four radionuclides were measured simultaneously using a PerkinElmer 2480 Wizard2© Automatic Gamma Counter. All trials have been made as three independent experiments, and the concentrations have been determined as mean from at least of three samples withdrawn from the supernatant liquid.

The removal efficiency (Y_M%) of the radionuclides was calculated using Eq. 1:

$$Y_M = \frac{(C_0 - C)}{C_0} \cdot 100\% \qquad (1)$$

In the above formula, the specific concentrations of the respective radionuclides C and C_0 are expressed in Bq/dm^3.[2]

With the aim to compare the proposed sorbents with another known from the literature, radionuclide apparent distribution coefficient between solid and aqueous phase (K_d L/kg) was calculated using the given formula (Heuel-Fabianek, 2014):

$$K_d = \frac{(C_0 - C)}{C_0} \cdot \frac{V}{m} \qquad (2)$$

In the above, V denotes volume of the decontaminated solution (mL), and m is the mass of the dry sorbent used (g).

Strength of the immobilization of the radionuclides by the offered sorbents was checked in four consecutive stages. In the first, sorbents were equilibrated for 3 h with water with the aim to remove the unbound radionuclides closed in the pores. Then, slightly contaminated water was replaced by new portion of water and the systems were equilibrated again. The procedure was repeated three times. In all experiments, ratio of the sorbent to liquid phase was

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[1] No-carrier added (*nca*) radioisotope: extremely small amount of the radioactive isotope (usually in the range of 10^{-6}–10^{-7} mol) which is not stabilized with weight amount of its stable isotopes (*Nomenclature for radioanalytical chemistry* 1994).

[2] One becquerel (1 Bq) is defined as the radioactivity in which one nucleus disintegrates per one second. Because 1 Bq is a small radioactivity unit, for the practical reasons we always use the kilobecquerels (kBq) or megabecquerels (MBq).

50 g per 1 L. Quantities of the radionuclides desorbed were determined by radiometric measurements of the liquid samples.

Stability of the sorbent *in contact with the aqueous solutions*

Stability of the radionuclide-loaded sorbent contacted for the prolonged time with aqueous solutions was determined by placing samples of the sorbents (typically, 0.2 g) in the polyethylene test tube filled with 5 cm^3 of the deionized water. After the 48 h of agitating in the shaker at RT, the suspensions were centrifuged (10 min.; 14000 rpm) and the solutions were withdrawn. Main anion and cation contents were determined in the supernatant by the ion chromatography (Model DIONEX ICS-5000 DC).

Zeta potential

The charge (or zeta potential, ζ_n) of the 1A and 1B particles was determined by measuring velocity of their movement due to the electrophoresis. The speed they move is proportional to the applied electrical field strength and their zeta potential. With the aim to measure the electrophoretic velocity (Zetasizer Nano ZS instrument, Malvern, UK), the dynamic light scattering method was applied.

Fourier transform infrared spectroscopy

The vibrational (FT-IR) spectra were recorded using the JASCO FT/IR-6200 type A instrument for the pellets containing 2 mg of sorbent and 100 mg of KBr. Standard spectral region of 4000–400 cm^{-1} was recorded with the resolution of 1 cm^{-1}, and the ASCII numeric data were processed with the OPUS software version 3.1 (Bruker) with the aim to pick the peaks.

Scanning electron microscopy (SEM)

The DSM 942 Scanning Electron Microscope (Zeiss-Leo, Germany) has been used for the SEM studies of CSS 1A and CSS 1B sorbents. Surface profile was examined with the magnifications of 80, 500 and 2000 times for samples covered with a thin gold layer.

Radiation stability of the sorbents

Irradiation of ten gram samples of the 1A and 1B sorbents was carried out with Co-60 gamma rays the ambient temperature using Gamma Chamber GC 5000 using doses of 250 kGy (a dose rate of 5 kGy/h). Radiation stability of the materials has been checked by three independent methods. Infrared vibrational spectra have been registered for the irradiated sorbents and compared with these of raw materials. Furthermore, values of their ζ_n potential have been determined and their sorption properties have been compared with the values for the untreated substances.

Results and discussion

In the present work, we investigated sorption of Cs(I), Sr(II), Eu(III) and Am(III) metal ions on two CSS materials containing different amount of the carbonates. The removal efficiency (Y_M%) was studied as a function of main factors important for management of the radioactive liquid wastes.

Before starting the studies, homogeneity of bulk samples of the sorbents has been checked. For this purpose, seven randomly selected 50 mg lots of the 1A and 1B sorbents were equilibrated with equal volume of the initial solution containing four radionuclides. It has been found that the Y_M values for all samples did not differ within CSS 1A and CSS 1B series more than 0.5%. So, CSS 1A and CSS 1B have been assumed to be homogeneous and suitable for further studies.

Results of the sieve analysis (Morek Multiserw Sieves, Poland) of CSS 1A and CSS 1B sorbents show that CSS 1A and CSS 1B sorbents are rather fine materials with diameter of the main fraction being 0.2 mm. Moreover, it has been found that about 98 per cent of the particles are greater than 0.05 mm.

Further studies were performed using the no fractioned sorbents.

Sorption of the radionuclides

Removal of the radionuclides from aqueous solutions was determined in batch experiments primarily as the dependence on the contact time of sorbents with the adsorbate. Moreover, all main factors important for the design of the separation procedure have been checked.

Figure 1 shows the changes in the amount of the radionuclides sorbed as a function of time. It may be seen that Y_M reaches saturation value faster than half an hour.

In detail, mean uptake of the metals for the time of contacting phases ranging from 30 min to 180 min for the 1A sorbent has been found as 99.0 ± 0.3%, 99.0 ± 0.4%, 91.1 ± 0.6% and 97.4 ± 0.2% for Cs(I), Sr(II), Eu(III) and Am(III), respectively. For the 1B sorbent, in turn, the corresponding values are: 98.6 ± 0.2%, 98.4 ± 0.3%, 87.0 ± 0.5% and 92.3 ± 0.2%. Therefore, in all following experiments, as well as in order to design future separation procedures, the 1-h equilibration time is more than sufficient.

Acidity of the purified solution is an important factor influencing the Y_M due to the metal speciation and

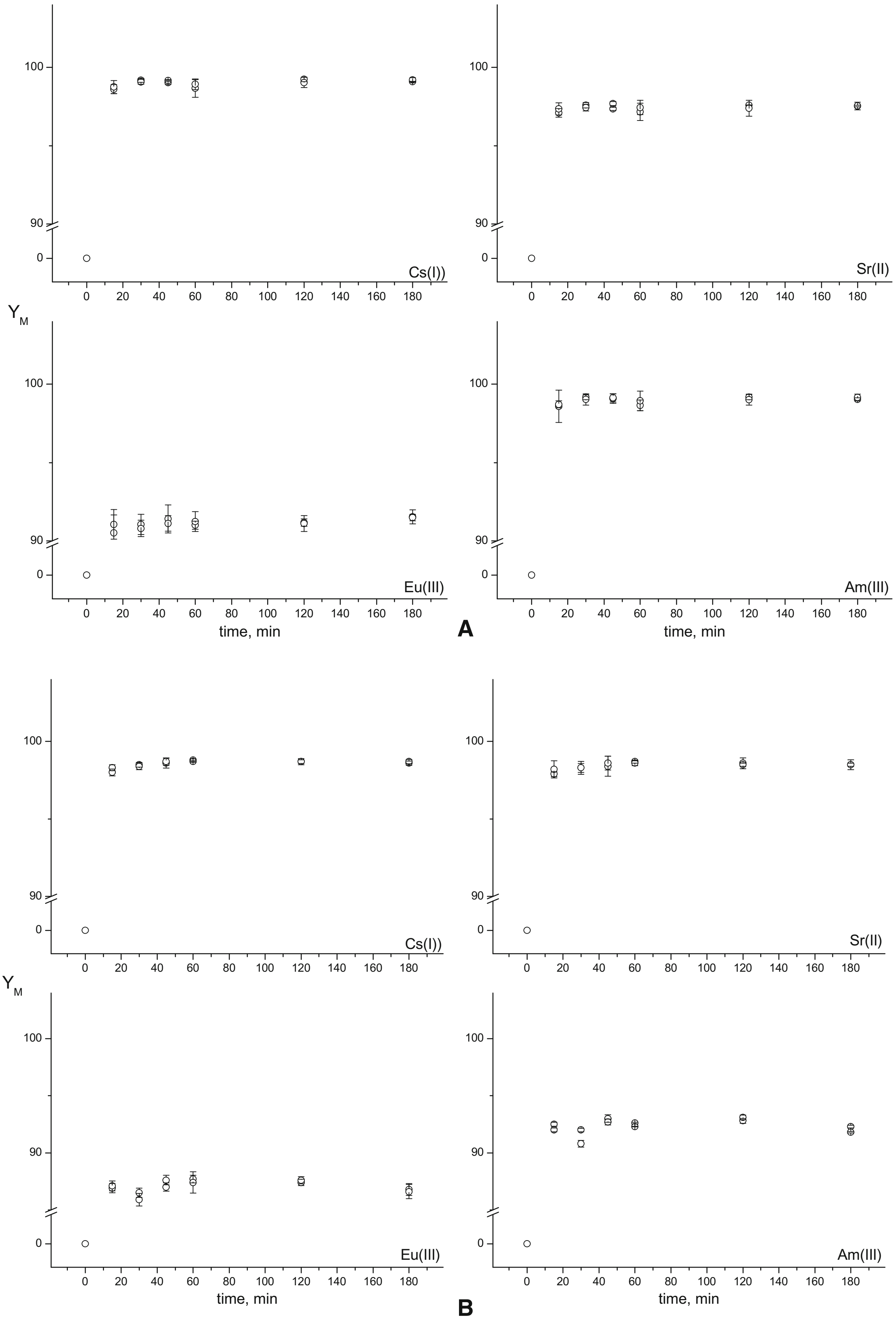

Fig. 1 Kinetics of sorption by 1A (upper) and 1B (bottom) sorbents

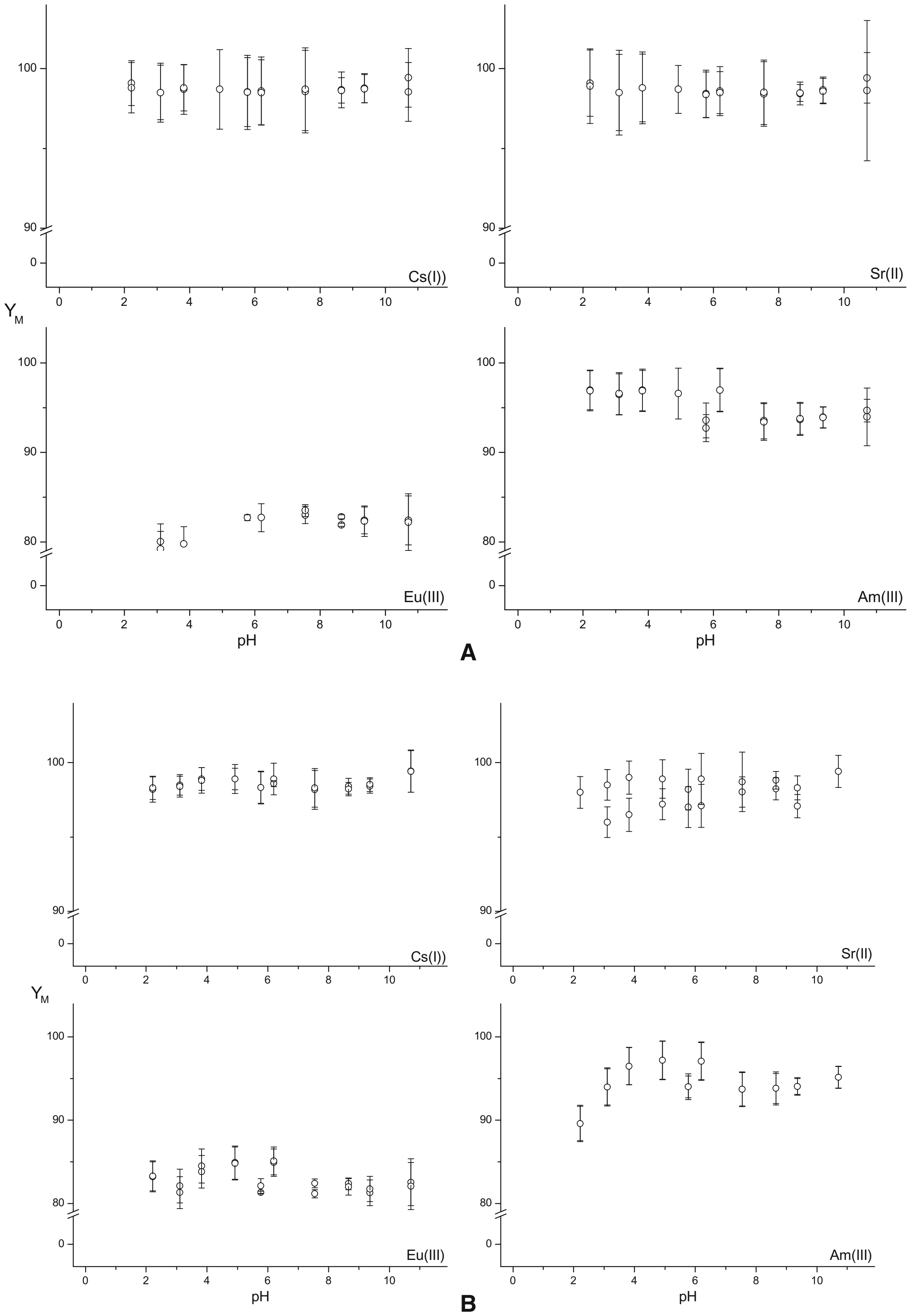

Fig. 2 Uptake of the radionuclides by 1A (upper) and 1B (bottom) sorbents in dependence of the initial acidity of the purified solution

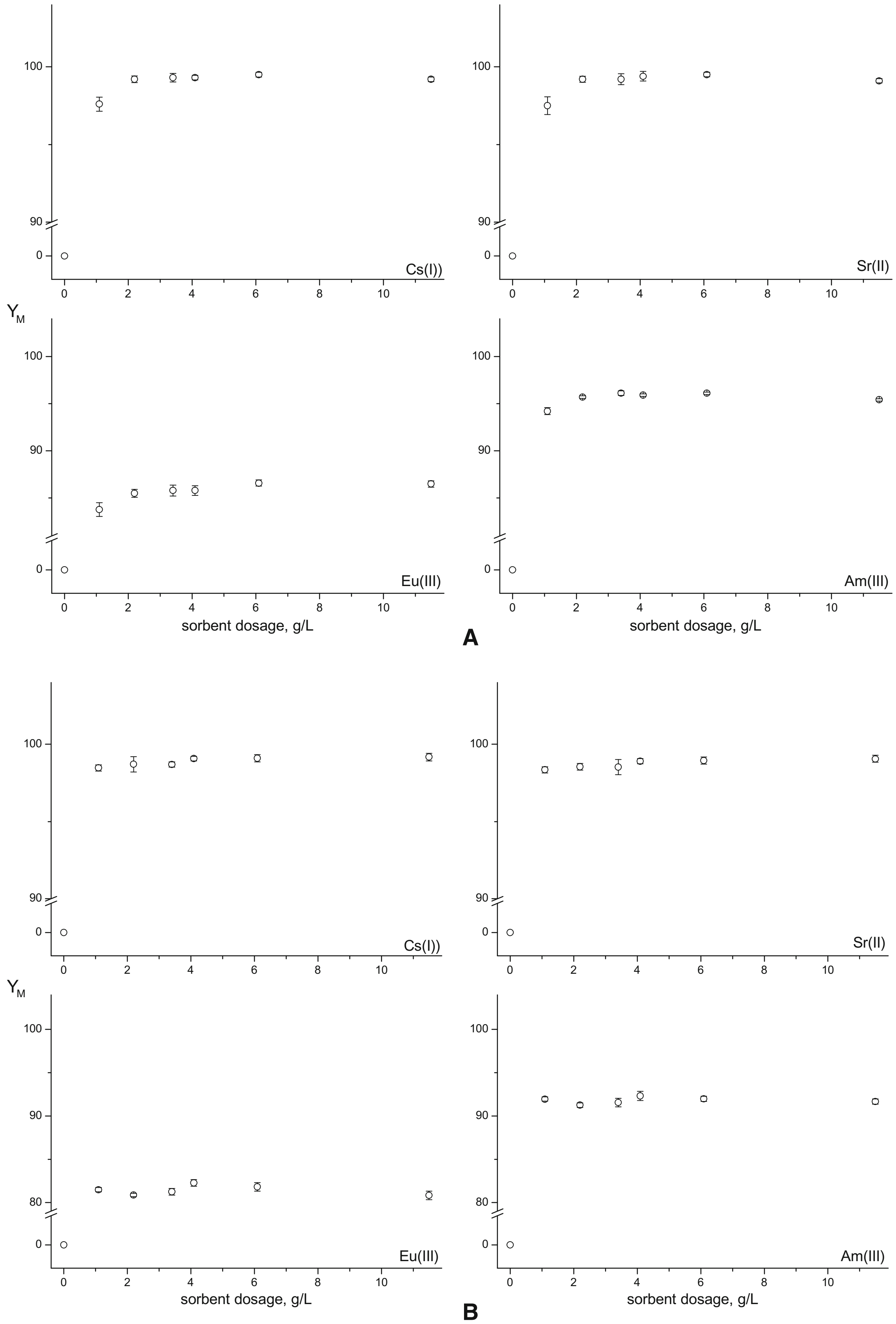

Fig. 3 Uptake of the radionuclides by 1A (upper) and 1B (bottom) sorbents in dependence of the mass of the sorbent used

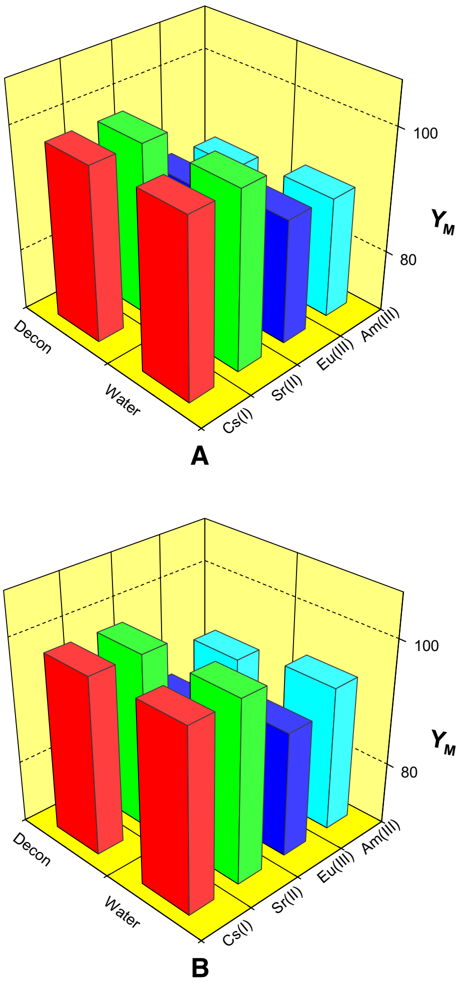

Fig. 4 Uptake of the radiometals by 1A (upper) and 1B (bottom) sorbents from water as compared from the solution containing complexing agents

dissociation of the binding sites on the sorbent surface, both determining charges of the reacting species (Akar et al. 2007). Figure 2 shows the results of sorption as a function of pH. It has been found that Y_M does not change significantly within broad range of the acidity. However, the complicated interplay of both mentioned above metal speciation and dissociation of the sorbing groups may be clearly seen.

The effect of the sorbent dosage on sorption, when all the other conditions remain constant, is presented in Fig. 3. It may be observed that the Y_M values of all metal ions increase with increasing doses of the sorbents up to the ration 2 g/L. Further increase in the sorbent doses does not provide succeeding increment of Y_M. Therefore, when application of the 1A or 1B sorbents is foreseen, 2 g/L dose of the sorbents may be proposed. The increase in the percentage of the metal ion removal with increase in sorbent dose may be related to the greater availability of the exchangeable sites or surface area at higher concentration of the sorbent. Non-significant increase observed when the sorbent doses exceed this value has been observed already, *e.g.* in the studies of Mittal (2006) or Jiao et al. (2015).

Liquid radioactive wastes often contain components originating from the decontamination of different items. The most commonly used decontamination liquids are these used in the CANDEREM (Canadian decontamination and remediation) and the CANDECON (Canadian decontamination) processes. Both solutions are mixtures of about 2×10^{-3} M oxalic acid, citric acid and EDTA (Dulama et al. 2009). So, to check possibility of application of CSS 1A and CSS 1B sorbents in the low-level radioactive waste (LLRW) management, we have tested them by sorption of the radionuclides from aqueous solutions containing a mixture of the aforementioned chelating agents and comparing the Y_M values with these for liquids which do not contain the complexing agents. As it is seen from Fig. 4, for any pair the values do not differ each other more than the experimental errors. It means that the presence of strong chelating agents, such as the citrate, oxalate or EDTA, does not reduce the radionuclides' sorption efficiency of CSS 1A and CSS 1B sorbents. So, the clay-salt slimes may be applied both for processing of the radioactively contaminated liquid wastes and as the backfillers in the engineering barriers.

Stability of the sorbent is one of the most fundamental problems to be considered when dealing with sorption. In order to make it more attractive to be used on a large scale, the sorbent must remain close to its initial properties in the whole process. For this purpose, leaching of different metals by water was performed by equilibrating of the 1 g samples with 25 mL of water for 480 min. Obtained results presented in Table 1, in comparison with the regulations of the WHO for drinking water, show that CSS 1A and CSS 1B sorbents do hardly fulfil the requirements for being applied in production of drinking water. Content of main cations and anions present in drinking water taken from the INCT tap is also given.

However, basing on our previously acquired knowledge, it is to be expected that the nanofiltration (NF) process may be effective method in the removal of the released contaminants. This problem, however, is not the object of the presented studies.

Regarding application as the novel backfill in the engineering barriers of the radioactive waste repositories, important problem for their safe operation, extremely strong fixing of the radiometals is required. As can be seen from Fig. 5, the prolonged equilibration of CSS 1A and CSS 1B radionuclide-loaded sorbents with aqueous liquids containing no radiometals shows that the radionuclides are immobilized stably.

Table 1 Chemical composition of water after equilibration with sorbents 1A and 1B in relation to the standards of the WHO

Anion/cation	WHO (2011) [mg/L]	Water INCT	1A	1B
F^-	1.5	0.047	0.58	0.79
Cl^-	250	0.144	0.99	458.46
SO_4^{2-}	250	0.471	1789.10	986.21
NO_3^-	3	–	0.60	0.26
PO_4^{3-}	–	–	–	2.66
Na(I)	*	0.247	0.94	0.66
K(I)	*	0.125	12.98	8.42
Mg(II)	*	0.196	23.43	81.06
Ca(II)	*	0.362	562.29	419.84
Sr(II)	*	–	27.7	22.24
Zn(II)	*	0.101	0.054	–
Fe(III)	0.3	–	2.40	0.169
Mn(III)	0.1	–	21.70	0.321
Ni(II)	0.07	–	–	0.016

* Not established a guideline value; not of health concern at levels found in drinking water

Approaching the end of presenting our studies on the radionuclides' sorption, we decided to compare presented before results obtained in the laboratory conditions with the uptake of the metals from the solutions of the composition resembling the real liquid radioactive waste. In the commonly available literature, data for this composition are rare and difficult to obtain. With the aim to make the comparison, we have chosen the composition published by Liu et al. (2014); all values are given in g/L: $NaNO_3$–31.560, $Al(NO_3)_3$–13.670, $Fe(NO_3)_3$–7.460, $Cr(NO_3)_2$–0.960, $Ni(NO_3)_2$–2.670, KNO_3–0.620, $Ba(NO_3)_2$–0.014, $Sr(NO_3)_2$–$0 - 0.1$ and $CsNO_3$–0.175. Our results are presented in Fig. 6. The figure shows that for small amounts of the sorbents, salts present in the solution successfully compete for the sorption sites. So, due to the co-sorption of the salts, one should use the quadruple sorbent dosage to obtain full uptake of the radiometals.

Gamma irradiation of the sorbents

Possibility of application of CSS 1A and CSS 1B materials for the liquid low-level radioactive waste management has been also tested by checking their radiation stability. With the aim of this, sorbents have been irradiated with the radiation dose of 250 kGy. Their high radiation stability, i.e. only the insignificant changes in the sorbents' structure upon irradiation, has been proven by three independent methods. In the first, sorption properties of the materials have been shown to be the same for both raw materials and the appropriate irradiated sorbents (see Fig. 7).

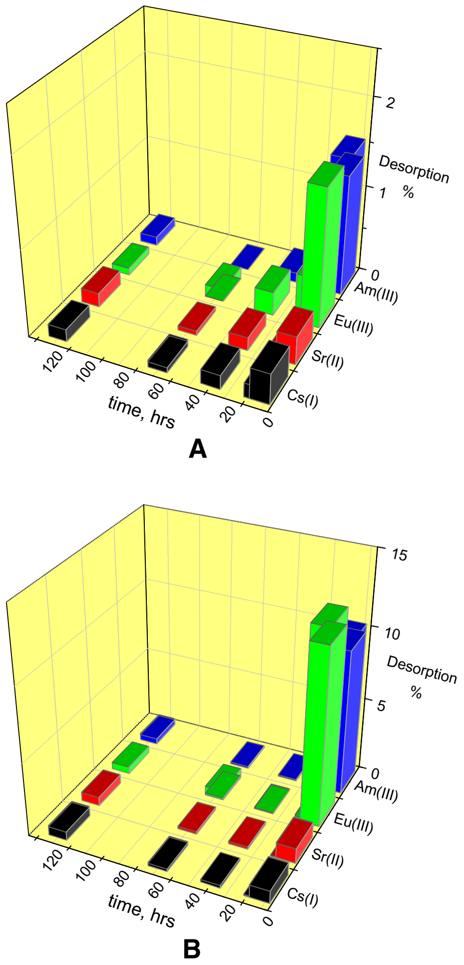

Fig. 5 Removal of the radiometals from the metal loaded 1A (upper) and 1B (bottom) sorbents

Moreover, the infrared vibrational spectra of all materials studied have been registered and pairwise compared by the numerical methods. Assignment of the vibrations in the fingerprint region of the spectra, i.e. for the 2000–400 cm^{-1} region, for both aluminosilicates may be found in the existing literature, e.g. in the papers of Bhaskar (2015) or Bendou and Amrani (2014). So, speaking only briefly, the bands around 500 cm^{-1} are commonly assigned to Si–O–Al (where Al is the octahedral cation) and Si–O–Si bending vibrations, respectively. The intensive bands in the region of about 1000 cm^{-1} in the spectra may be attributed to the Si–O stretching vibrations (in-plane) of the tetrahedral sheets. Bands at about 1500–1600 cm^{-1} and 3400–3500 cm^{-1} relate to the bending and stretching vibrations of the OH groups in the water molecules adsorbed on the clay surface. Finally, peaks around 3600 cm^{-1} characterize stretching vibrations of the hydroxyl groups coordinated to the octahedral Al^{3+} cations (Fig. 8).

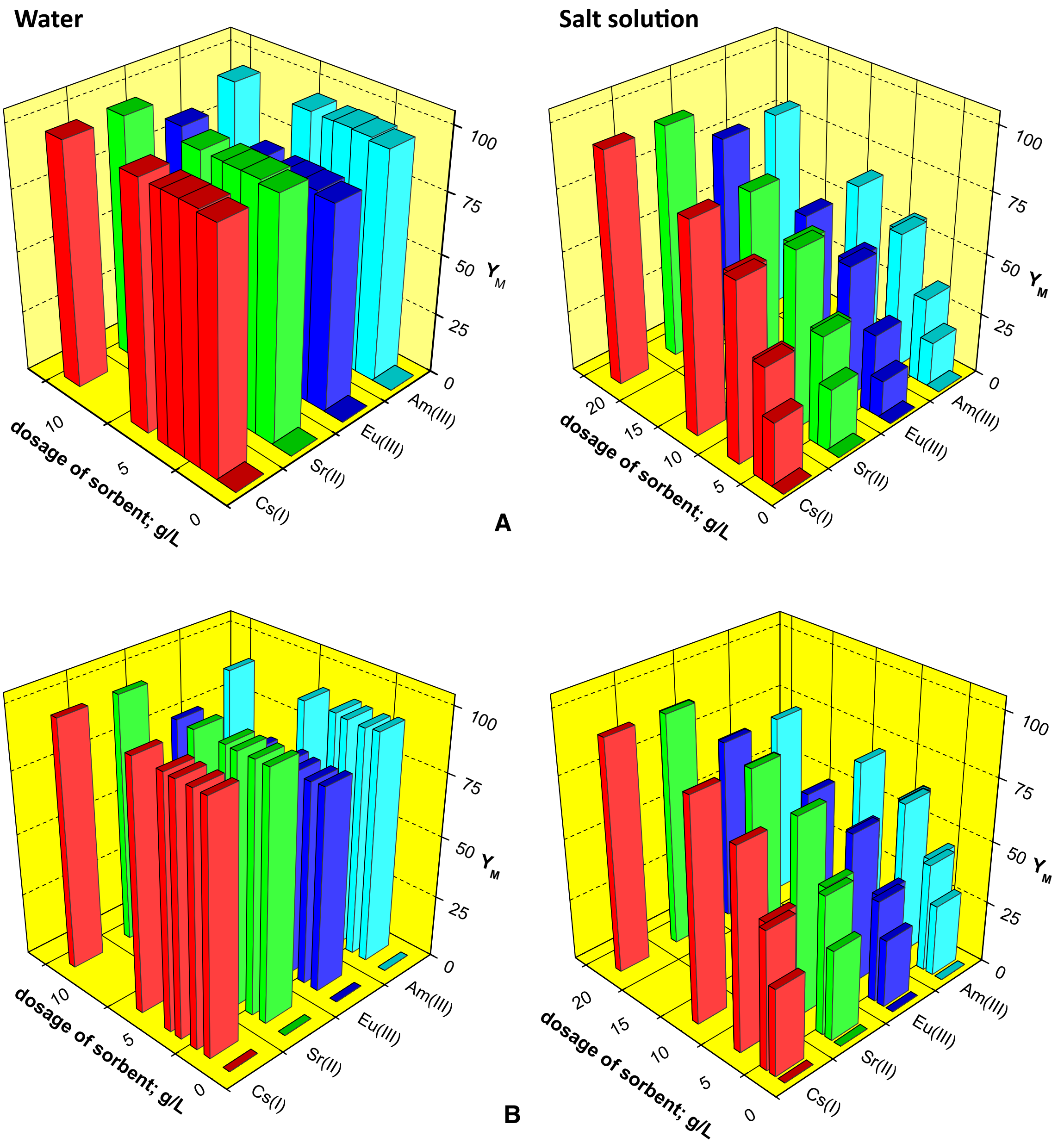

Fig. 6 Comparison of uptake of the radionuclides from water (left) with this from the simulated radioactive waste (right)

According to the proposition of Sadlej-Sosnowska et al. (2006), calculation of the Pearson's correlation coefficient (c.c.) seems to be the most powerful for comparing two spectra. The c.c. may range from the value of − 1–1. A zero value implies the lack of the correlation between both spectra, i.e. they are totally different. On the contrary, values close to ± one indicate for the similarity of the compared spectra and, consequently, of the identity of the substances being compared.

Vibrational spectra registered in the fingerprint spectral region for CSS 1A and CSS 1B sorbents, i.e. for the raw materials and the corresponding irradiated sorbents, have revealed the c.c. values of 0.97 and 0.98 for the 1A and 1B sorbents, respectively. It means that spectra of the irradiated sorbents resembled these registered for the original materials, which permits the conclusion about the sufficient radiation stability of CSS 1A and CSS 1B sorbents.

Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) is a method that informs about surface of the solid samples, including their structure, smoothness and chemical composition. In fact, the only limitation of the method is the lowest detectable particle size. Topography of the sorbent surface, raw material and the gamma ray irradiated is presented in

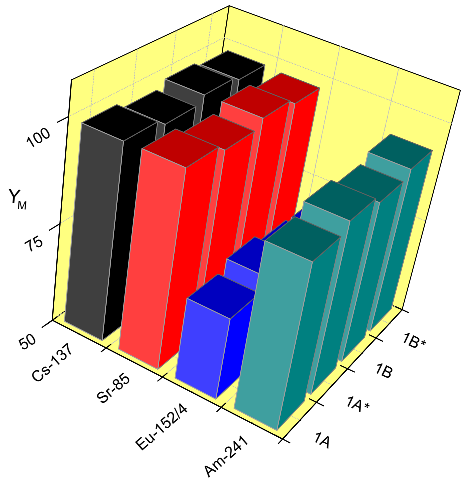

Fig. 7 Radiation stability of the sorbents studied by sorption of the radionuclides

Fig. 9. It may be seen that changes in the surface occurring upon the irradiation are negligible. Structure of CSS 1A and CSS 1B sorbents is relatively tense and aggregated. Incorporation of the solution into the sorbent inner structure (pores) also seems to be unnoticeable, which seems to be in an agreement with the chemisorption mechanism.

Zeta potential of the sorbents

Zeta potential (ζ_n), which relates to the surface charge of solid, is accounted among the crucial factors in the sorption reaction. The values for the 1A sorbent are − 20.1 and − 31 mV for pH = 4 and pH = 11, respectively. Corresponding values for the 1B sorbent are − 22.5 and − 32 mV. Values found for the materials after their gamma ray irradiation are − 20, − 30.2, − 22.6 and − 32 mV and look to be very close to the corresponding raw materials. This observation confirms the conclusion that structure of CSS 1A and CSS 1B materials remains stable under action of the ionizing radiation. So, they seem to be suitable as the additive for the barriers in the waste repositories.

Zeta potential was measured also for the 1A and 1B sorbents loaded with the natural isotope (non-radioactive) of strontium(II). Excess of strontium in the initial solution has been used as compared with the loading capacity of sorbents (ion chromatographically checked). It was found that values of the potentials are about $3 \div 4$ mV higher than in the raw materials. This observation agrees with the chemisorption mechanism of the process which depends on the chemical bonding of metals with the acidic sorbing groups present in the CSS.

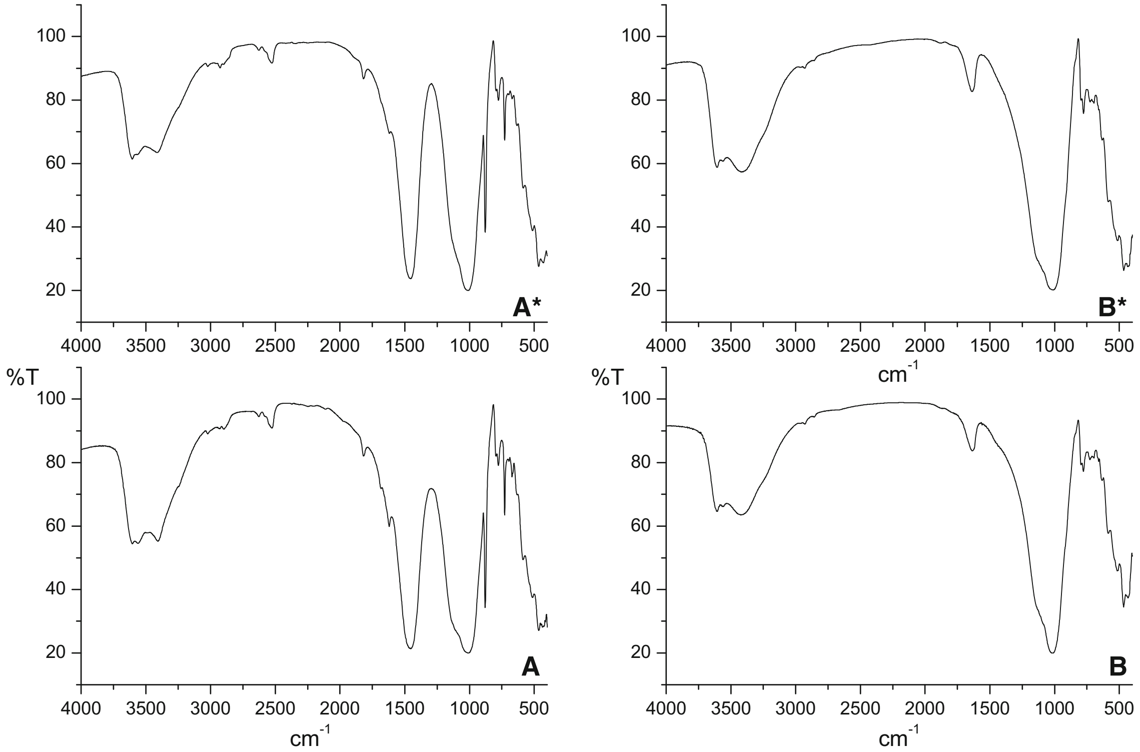

Fig. 8 IR spectra registered for CSS 1A and CSS 1B sorbents; raw materials (below) and the gamma ray irradiated (upper). Left part of the figure presents spectra of the 1A sorbent and right—of the 1B

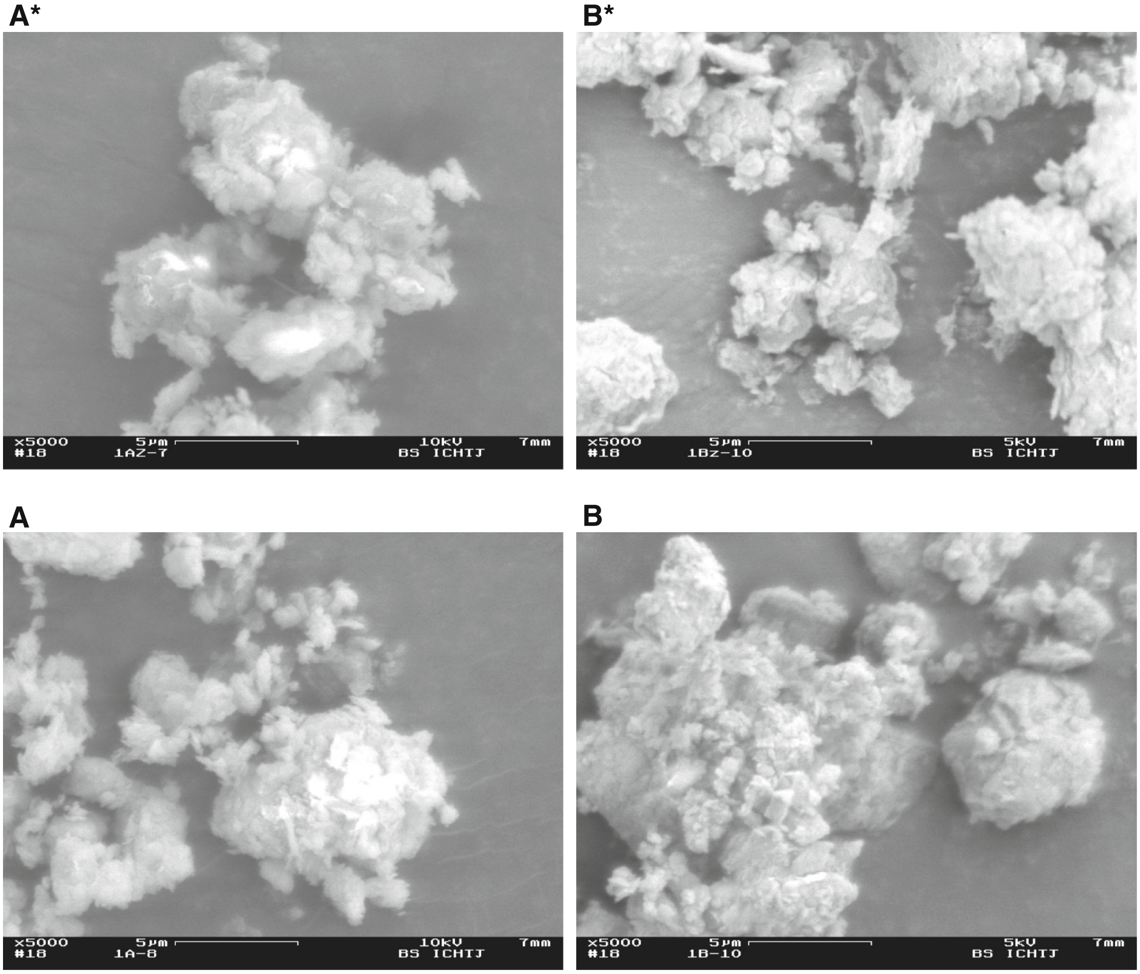

Fig. 9 Results of SEM observation of the sorbents. Magnification: 5000 times. Upper line—the irradiated materials

Clay-salt slimes (CSS) compared with other natural or waste material sorbents

Different sorption techniques with the use of natural materials (different inorganic materials, organic resins or the agricultural wastes) are accounted among the most efficient methods used for removal of metal ions from aqueous media. As a rule, they are similarly effective but cheaper than traditional chemical and physical decontamination techniques. The International Atomic Energy Agency (IAEA) literature INIS database shows for the existence of about 15.500 publications referring to the radioactive metal sorption from aqueous solutions (INIS website). Among them, about 5.500 have been found for the americium sorption and more than 9.000 for strontium. Some examples of the literature data on sorption of strontium(II) and americium(III) may be found in the paper of Fuks et al. (2016). In some cases, the original data have been recalculated according to Eq. 2.

Up today many inorganic ion exchangers, among them clays, zeolites, hydrous oxides of different metals or polyantimonic acid have been found to be useful for treating the contaminated water, soil filtrates or low-level aqueous wastes. After the metal loading, these ion exchangers may be used as the solidified liquid waste form or as the materials for incorporation into other inorganic waste matrices, such as glass, ceramics or grouts. Inorganic sorbents show greater resistance to ionizing radiation, high temperatures and harsh chemical environments than most of organic resins.

Currently, search for the novel economical, easy to use and safe methods of elimination of the heavy/radioactive metals from contaminated waters has intensified considerably. Our work fits well into this scope of research. As can be seen from the table, sorption properties of the CSS sorbents are comparable with the most common natural sorbents reviewed by Fuks et al. (2016).

Conclusion

Decontamination of the radioactive wastes with the use of clay-salt slimes, industrial waste of the JSC “Belaruskali,” was found to be effective for the solutions containing Cs(I), Sr(II), Eu(III) and Am(III) radionuclides.

CSS 1A and CSS 1B sorbents appear to be sufficiently stable for practical application in the treatment of wastewaters.

Heavy metal ions released to the water in the course of the sorption may be removed by the nanofiltration method.

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