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^{137}Cs, ^{85}Sr AND ^{60}Co SORPTION FROM MODEL SOLUTIONS OF LOW ACTIVITY LIQUID RADIOACTIVE WASTE BY MODIFIED OIL SHALE

Abstract. The results of the study of cesium, strontium, cobalt radionuclides sorption from model solutions simulating liquid radioactive waste on promising sorbents based on thermally modified oil shale are presented. The dependency of texture parameters obtained from the results of nitrogen adsorption-desorption on the treatment method of oil shale and their relationship to sorption parameters such as distribution coefficient and removal efficiency was discussed. Understanding how pore characteristics affect the sorption activity of such complex systems as sorbents is important for the creation of radionuclide sorption materials with improved characteristics. The efficiency of sorption of cesium, strontium and cobalt radionuclides was rather high for sorbent samples: the distribution coefficient was about 10^3–10^4 ml/g with a removal efficiency of more than 66 %. The most effective sorbent was obtained from oil shale with a heating rate of 5 deg/min without subsequent steam treatment, which is recommended to be used for low-active radioactive waste treatment.

Keywords: sorbent; oil shale; cesium, strontium, cobalt, radionuclides; liquid radioactive waste treatment

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СОРБЦИЯ РАДИОНУКЛИДОВ ^{137}Cs, ^{85}Sr И ^{60}Co ИЗ МОДЕЛЬНЫХ РАСТВОРОВ ЖИДКИХ РАДИОАКТИВНЫХ ОТХОДОВ НИЗКОЙ АКТИВНОСТИ МОДИФИЦИРОВАННЫМИ ГОРЮЧИМИ СЛАНЦАМИ

Аннотация. Представлены результаты исследования сорбции радионуклидов цезия, стронция и кобальта перспективными сорбентами на основе термически модифицированных горючих сланцев из растворов, моделирующих жидкие радиоактивные отходы. На основании результатов низкотемпературной адсорбции–десорбции азота установлена зависимость текстурных параметров исследуемых сорбентов от метода обработки горючего сланца, а также описана зависимость эффективности сорбции (по величинам сорбции и коэффициента распределения) радионуклидов от текстурных параметров сорбентов. Понимание того, каким образом характеристики пор оказывают влияние на сорбционную активность таких сложных систем, как сорбенты, важно для создания высокоэффективных сорбционных материалов с усовершенствованными характеристиками. Установлено, что материалы на основе модифицированного горючего сланца сорбируют радионуклиды цезия, стронция, кобальта с высокой эффективностью: коэффициенты распределения каждого радионуклида составляют 10^3–10^4 мл/г при степени извлечения более 66 %. Наибольшую эффективность проявляет сорбент, полученный из горючего сланца при нагреве со скоростью 5 град/мин без последующей обработки паром. Данный сорбент рекомендован к применению для очистки жидких радиоактивных отходов низкой активности от радионуклидов цезия, стронция, кобальта.

Ключевые слова: сорбент, горючие сланцы, радионуклиды цезия, стронция, кобальта, очистка жидких радиоактивных отходов

Для цитирования. Сорбция радионуклидов ^{137}Cs, ^{85}Sr и ^{60}Co из модельных растворов жидких радиоактивных отходов низкой активности модифицированными горючими сланцами / Е. Э. Зарубо-Венглинская [и др.] // Вес. Нац. акад. навук Беларусі. Сер. хім. навук. – 2023. – Т. 59, № 1. – С. 49–55. https://doi.org/10.29235/1561-8331-2023-59-1-49-55

Introduction. The significant volumes of radioactive waste are generated as the result of nuclear and radiation facilities operation. Depending on facility type (e.g. nuclear power plant, research reactor, isotope manufacturing etc.), various technological processes contribute to formation of liquid radioactive waste (LRW) but the principal are: equipment and premises decontamination, treatment of technological water with radionuclide impurities, regeneration of ion-exchangers [1].The main radiation hazard of such waste is determined by long-lived radionuclides, which are either fission products such as $^{134,\,137}$Cs, ^{90}Sr or activation products such as ^{60}Co [2, 3]. Therefore, the development of novel effective methods and materials for removal of indicated radionuclides from LRW is a promising challenge.

From the point of view of technological and economic benefits, rather simple implementation and high efficiency, sorption treatment of LRW is widespread [1]. Depending on activity level and chemical composition of LRW, various types of sorption materials are used. A group of natural based materials can be distinguished among them due to additional advantages, such as abundance, local availability and relatively low cost [4].

Oil shale is a promising raw material in this respect. This is a material that, on the one hand, can be used as energy source (due to the organic component called kerogen), and on the other hand – as sorbent (due to the presence of a mineral component) [5]. The modification of oil shale by heating in different conditions leads to partial or full removal/carbonization of organic component and is essential structural transformation of mineral base [6]. The inorganic part is 60–80 % of the total mass of the oil shale and consists mainly of silicon, aluminum, iron, calcium and magnesium oxides. Such composition is close to natural aluminosilicates (clay minerals) composition, which allows to expect high sorption activity of modified oil shale toward cesium, strontium and cobalt radionuclides [7].

In Belarus, oil shale deposits are located in the south of the republic (Luban and Turov deposits) [8]. This oil shale is inappropriate to use only as an energy raw material due to its low energy efficiency, so this material demands complex use [9]. The efficiency can be increased by using oil shale as a sorbent for LRW treatment as well, which is quite justified due to the effective sorption of radionuclides on mineral part of oil shale [10]. The main advantages of modified oil shale to be used as sorbents are high radiation, thermal and chemical stability [11]. In addition, low leaching rate of radionuclides, compatibility with inorganic matrices for immobilization, alongside with low sorbent cost makes it possible to dispose it immediately as solid radioactive waste [12].

The objective of this work was to study the possibility of using modified oil shale as sorbents for cesium, strontium and cobalt radionuclides in complex LRW treatment and to determine the effects of modification conditions on sorption efficiency of material.

Experimental. The sorbents were prepared by thermal destruction of natural oil shale from Turov deposit, Belarus. At the first stage, oil shale samples with grains of 1–3 mm size were heated at different rates (2, 5, and 10 deg/min) to 420 °C and kept at this temperature for 40 min. At the second stage, heating was carried out without or with live steam at 450 °C for 30 min.

The texture and surface properties of the modified oil shale samples were evaluated from the isotherms of low-temperature nitrogen adsorption-desorption, measured by the volumetric method on an ASAP 2020 MP surface area and porosity analyzer (Micromeritics, USA). The error of measuring the specific surface of the samples was 15%, and of the volume and diameter of the pores was 1 %. The activity of each radionuclide in solution before and after sorption was measured by gamma-beta spectrometer MKS AT-1315 (Atomtex, Belarus).

Efficiency of sorption of radionuclides by modified oil shale was studied for model solutions which simulates LRW and contains cesium, strontium and cobalt radionuclides. Model solutions were prepared on the basis of distilled water by adding ^{137}Cs, ^{85}Sr, and ^{60}Co with initial specific activity of each radionuclide of ~100 kBq/L. The pH of the solutions was adjusted with 0.1 M NaOH solution to ~7.0.

Sorption of radionuclides on the sorbents was carried out under static conditions by batch sorption in limited volume. The ratio of volume of solution to mass of sorbent was constant for every experiment –1000 ml/g. Sorption equilibrium was established within 24 h with constant stirring. After the end of sorption, the solution was separated from the sorbent by filtration through paper filter with 3–10 μm pore size.

The efficiency of the sorbent was estimated by the value of sorption S, % and distribution coefficient K_d, ml/g of each radionuclide, which were calculated according to the following equations:

$$S = \frac{A_0 - A}{A_0}, \tag{1}$$

$$K_d = \frac{A_0 - A}{A} \cdot \frac{V}{m}, \tag{2}$$

where A_0 – is initial activity of radionuclide in solution before sorption, Bq; A – equilibrium activity of radionuclide in solution after sorption, Bq; V – volume of solution, ml; m – mass of sorbent, g.

Results and Discussion. Figures 1–6 (left) show the isotherms of low-temperature nitrogen adsorption-desorption on investigated oil shale samples. All isotherms are irreversible and have hysteresis loops. This fact indicates the presence of mesopores (2–50 nm) in the samples [13], in which, along with multilayer adsorption, capillary condensation of nitrogen occurs [14]. The isothermal curves of nitrogen adsorption-desorption by oil shale belong to Type IV according to the IUPAC classification [13]. The shape of the hysteresis loop is close to the Type H3, which is inherent to sorbents having disordered, lamellar pore structure, slit- and wedge-shaped pores [15]. The gentle slope of the adsorption curve within the p/p_0 range of ~ 0.05–0.30 indicates an appreciable overlap of monolayer and multilayer adsorption, when the precise interpretation of the specific monolayer capacity is questionable [13].

It should be noted that neither the heating rate of oil shale, nor their steam treatment affect the shape of low-temperature nitrogen adsorption-desorption isotherms.

Fig. 1–6 (right) show pore size distribution (PSD-plots) for all the samples of modified oil shale studied in the current work.

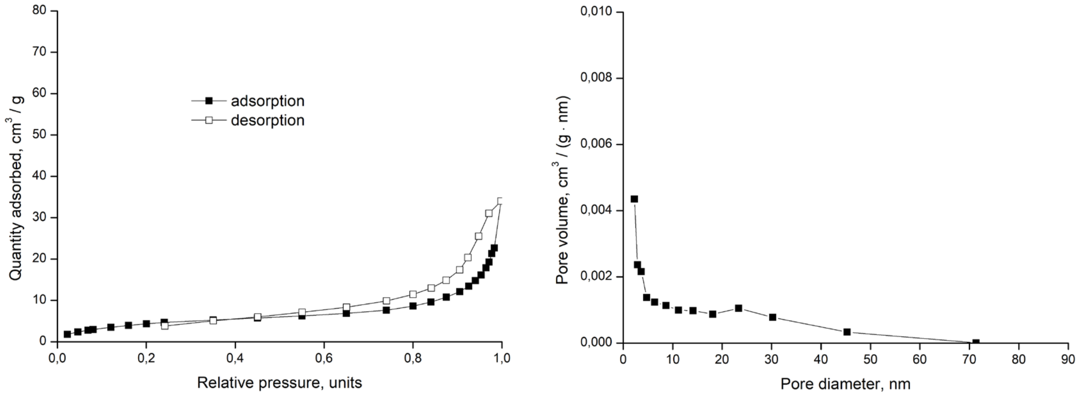

Fig. 1. Isotherm of low-temperature nitrogen adsorption-desorption (left) and PSD-plots (right) for a sample of oil shale heated at a rate of 2 deg/min at the first stage without subsequent steam treatment at the second stage

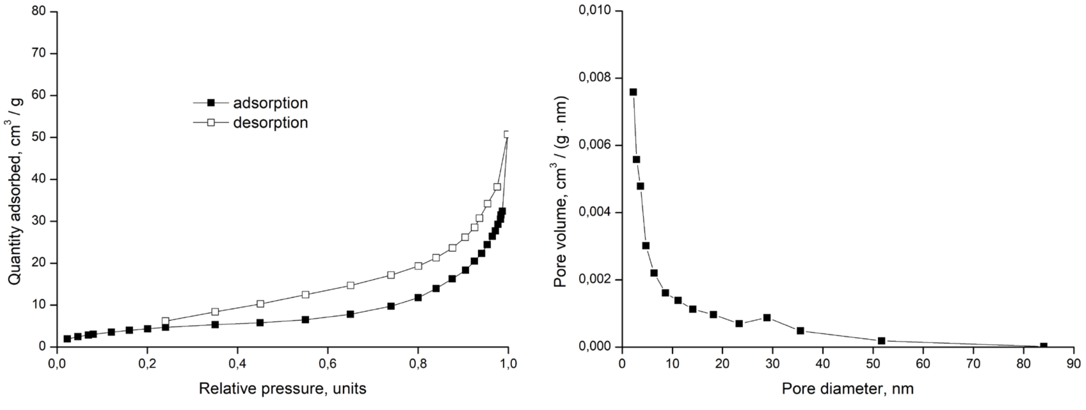

Fig. 2. Isotherm of low-temperature nitrogen adsorption-desorption (left) and PSD-plots (right) for a sample of oil shale heated at a rate of 5 deg/min at the first stage without subsequent steam treatment at the second stage

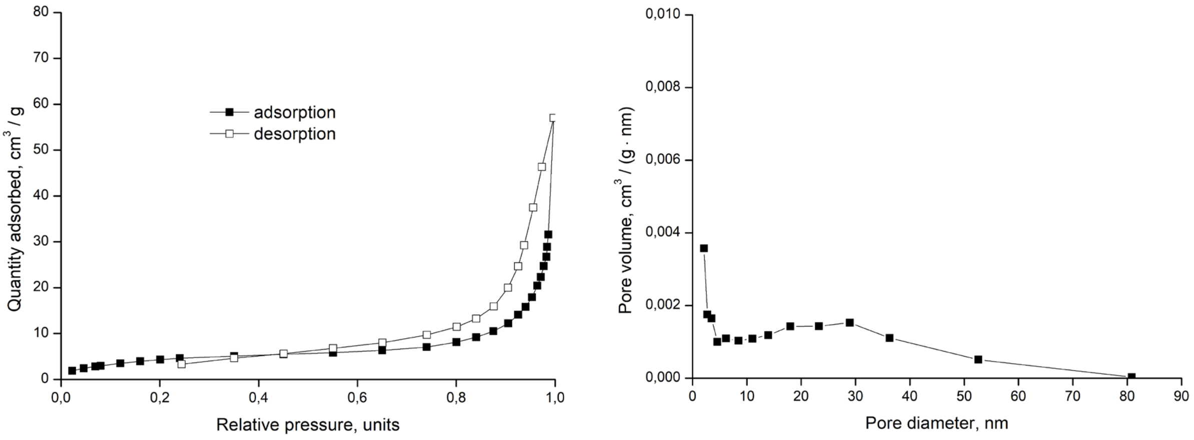

Fig. 3. Isotherm of low-temperature nitrogen adsorption-desorption (left) and PSD-plots (right) for a sample of oil shale heated at a rate of 10 deg/min at the first stage without subsequent steam treatment at the second stage

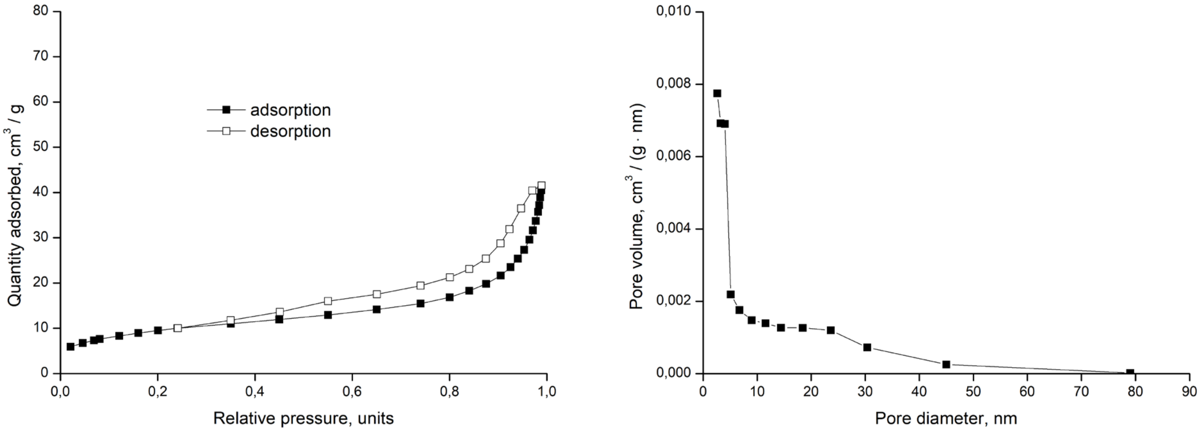

Fig. 4. Isotherm of low-temperature nitrogen adsorption-desorption (left) and PS-plots D (right) for a sample of oil shale heated at a rate of 2 deg/min at the first stage with the subsequent steam treatment at the second stage

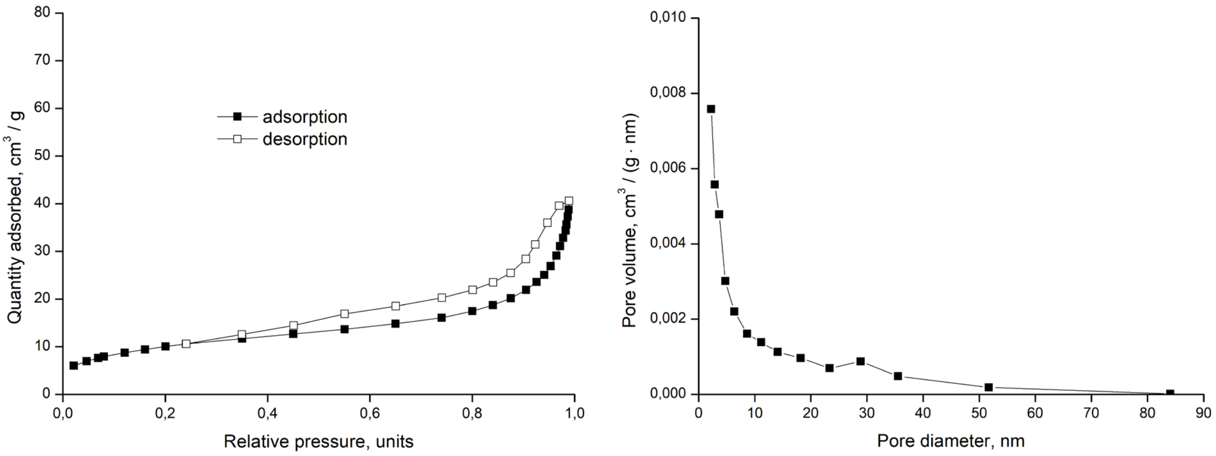

Fig. 5. Isotherm of low-temperature nitrogen adsorption-desorption (left) and PSD-plots (right) for a sample of oil shale heated at a rate of 5 deg/min at the first stage with the subsequent steam treatment at the second stage

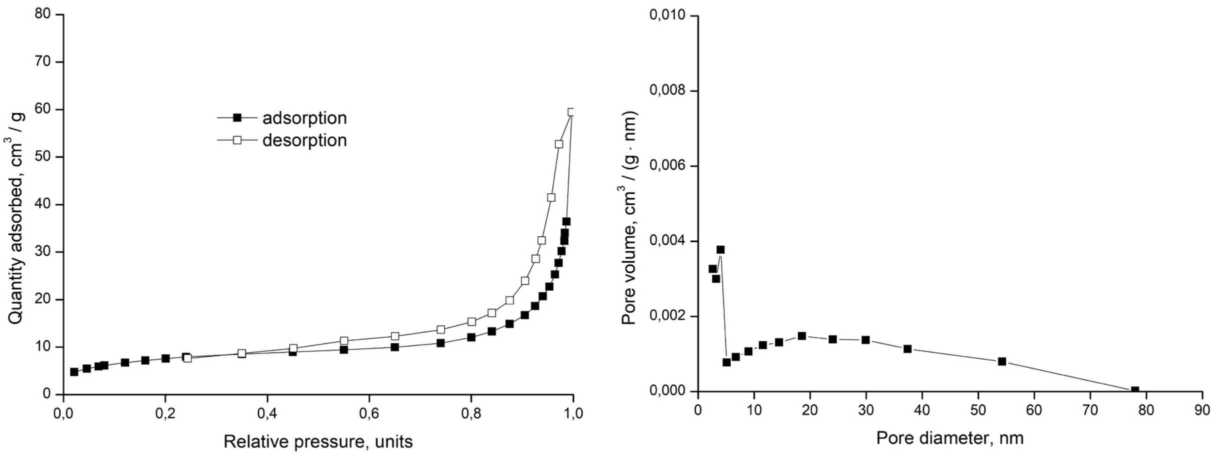

Fig. 6. Isotherm of low-temperature nitrogen adsorption-desorption (left) and PSD-plots (right) for a sample of oil shale heated at a rate of 10 deg/min at the first stage with the subsequent steam treatment at the second stage

Fig. 1–6 show that most of the pores of modified oil shale have sizes corresponding to mesopores. The treatment of sorbents with steam does not significantly affect the shape of the PSD-curves, although it does affect the total pore volume and, consequently, the intensity of the mentioned curves.

It should also be noted that heating the oil shale at a rate of 10 deg/min leads to a decrease in the total pore volume in comparison with the samples heated at rates of 2 and 5 deg/min. Such changes are probably associated with the partial destruction of oil shale particles at a higher heating rate. In addition, for samples with a heating rate of 10 deg/s, there is an increase in the number of pores with an average pore size of 20–30 nm comparing with pores of other size, which is due to the more intense carbonization of the organic component of the oil shale and its removal from the pores.

The values of the surface area and pore parameters of modified oil shale sorbents before and after steam treatment are presented in Table 1. The data are obtained on the basis of experiments on low temperature nitrogen adsorption-desorption by modified oil shale samples.

Table 1. **Values of specific surface area and pore parameters of the modified oil shale samples**

Heating rate, deg/min	Specific surface area, m^2/g				Pore volume, cm^3/g				Average pore size, nm			
	without steam treatment		steam treatment		without steam treatment		steam treatment		without steam treatment		steam treatment	
	A_{sp}	A_{BET}	A_{sp}	A_{BET}	$V_{\text{sp.ads}}$	$V_{\text{sp.des}}$	$V_{\text{sp.ads}}$	$V_{\text{sp.des}}$	D_{ads}	D_{des}	D_{ads}	D_{des}
2	15	18	33	35	0.053	0.048	0.058	0.063	14	13	7	8
5	15	18	35	37	0.049	0.059	0.055	0.061	13	16	6	7
10	15	17	26	28	0.045	0.072	0.053	0.082	12	19	8	12

The largest specific surface area corresponds to sorbents which are heated at a rate of 2 (33–35 m^2/g) and 5 deg/min with the subsequent steaming (35–37 m^2/g). For the other studied samples, the specific surface area is about 15–28 m^2/g. The specific surface area increases by ~ 2 after steam treatment.

The real volume and average pore size of the sorbents, due to the presence of hysteresis on the nitrogen adsorption-desorption isotherms, is recommended to be estimated by the values obtained from desorption curve. The reason is that desorption conditions (due to the lower pressure of the saturated vapor above the meniscus of the liquid in the pore) are closer to equilibrium than adsorption conditions [16]. Among samples without steaming, the largest pore volume corresponds to the samples which are heated at a rate of 5 and 10 deg/min (0.059 and 0.072 cm^3/g respectively). Among the oil shale treated with live steam, a sample with a heating rate of 10 deg/min at the first stage has the largest pore volume (0.082 cm^3/g). It can be seen that higher heating rate of oil shale, as well as its treatment with live steam, increases the total pore volume of the sorbent.

According to Table 1, the steam treatment of oil shale leads to a decrease in pore diameter of the sorbent by a factor of ~2. This is due to appearance of a large number of new small pores, which is associated with the removal of shale organic component. Presumably, the size and shape of existing pores increases in length, although the pores' diameter does not change. This assumption also explains the increase in total pore volume of oil shale after steam treatment. The heating rate of oil shale at the first stage affects the average pore diameter insignificantly, although when the oil shale is heated at a rate of 10 deg/min, there is a slight increase in the average pore size, the reason for which is explained above.

The general regularities of the influence of different processing regimes on the parameters of pores and surface area of oil shale are as follows: steam treatment increases the surface area of the samples, reduces the average pore size and increases the total pore volume significantly. At the same time, the heating rate of oil shale at the first stage of treatment has an insignificant effect on the values of pore size and surface area. The values of sorption and distribution coefficient for each radionuclide by all the studied samples of oil shale are presented in Table 2.

Table 2. **Sorption and distribution coefficients of cesium, strontium and cobalt radionuclides on the samples of modified oil shale**

Heating rate, deg/min	^{137}Cs				^{85}Sr				^{60}Co			
	without steam treatment		steam treatment		without steam treatment		steam treatment		without steam treatment		steam treatment	
	S, %	K_d·10^{-3}, ml/g	S, %	K_d·10^{-3}, ml/g	S, %	K_d·10^{-3}, ml/g	S, %	K_d·10^{-3}, ml/g	S, %	K_d·10^{-3}, ml/g	S, %	K_d·10^{-3}, ml/g
2	76±2	3.2±0.3	82±1	4.7±0.4	69±1	2.8±0.2	79±1	3.7±0.2	73±2	3.7±0.5	88±1	7.2±0.8
5	91±1	10.4±0.5	82±1	4.5±0.2	72±1	2.6±0.1	75±1	3.0±0.1	94±1	15.6±3.4	94±2	16.6±4.8
10	81±1	4.5±0.3	80±1	4.2±0.3	66±1	2.0±0.1	69±1	2.4±0.1	74±1	3.0±0.3	81±2	4.3±0.4

All the sorbent samples studied in this work show high efficiency in sorption of ^{137}Cs, ^{85}Sr, and ^{60}Co from solutions simulating LRW: the values of sorption and distribution coefficient were 76–91 % and (3.2–10.4)·10^3 ml/g, 66–75 % and (2.0–3.7)·10^3 ml/g, 73–94 % and (3.0–16.6)·10^3 ml/g, respectively.

The most effective sorbent for complex purification of model LRW solution via sorption is modified oil shale obtained by heating rate of 5°C/min at the first stage and without steam treatment at the second stage. The values of the distribution coefficient for ^{137}Cs, ^{85}Sr, and ^{60}Co were 4.4·10^3, 3.0·10^3, 16.6·10^3 ml/g for this sorbent, respectively.

Although the increase is insignificant, it should be noted that, in general, the steam treatment of oil shale increases the efficiency of sorption of radionuclides on sorbents. Among all the samples of oil shale sorbents, the greatest increase in efficiency of radionuclide sorption is noticed for the sample with a heating rate of 2 deg/min. After the live steam treatment, the distribution coefficients of radionuclides on the sorbent increase as follows: by 1.5 times (from 3.2·10^3 to 4.7·10^3 ml/g) for ^{137}Cs, by 1.7 times (from 2.2·10^3 to 3.7·10^3 ml/g) for ^{85}Sr and by 2.7 times (from 2.6·10^3 to 7.1·10^3 ml/g) for ^{60}Co.

Obviously, an increase in the efficiency of sorption of radionuclides on steam-treated oil shale occurs due to an increase of the available surface area of the sorbent, as well as an increase of the total pore volume.

Conclusions. Modified oil shale is a promising sorbent for treatment of LRW containing ^{137}Cs, ^{85}Sr, and ^{60}Co. The distribution coefficients of these radionuclides on the indicated sorbents are ~10^3–10^4 ml/g and sorption values are above 66 %. Due to its low cost, chemical, thermal and radiation resistance, low rate of leaching of radionuclides from contaminated sorbents, as well as the possibility of disposal spent sorbents with minimal conditioning, oil shale is a very attractive material for LRW treatment.

Undoubtedly, understanding and testing the effects of the pore system contributes to a better control of the adsorption activity of complex systems, so that radionuclide adsorbents with improved designs could become accessible. The live steam treatment of oil shale increases its efficiency as a sorbent only slightly. To obtain the most effective sorbents for the sorption of ^{137}Cs, ^{85}Sr, and ^{60}Co from LRW, it is recommended to process oil shale with the method indicated in the work at a heating rate of 5 deg/min without or with subsequent steam treatment. Steaming intensifies the removal of the organic component of oil shale, which leads to the increase of available surface area of the sorbent, as well as total volume of its pores.

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