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WATER RESISTANCE OF SOLIDIFIED RADIOACTIVE WASTE

В.І. Ковальчук, І.Л. Козлов, О.А. Дорож, М.Д. Богданов, Н.О. Мелетенко. **Водостійкість затверділих радіоактивних відходів.** Розглянуто результати експериментальних досліджень вилуговування, які характерні для рідких радіоактивних відходів радіонуклідів, з цементних матриць для тривалого зберігання. Показано, що вилуговування є характеристикою хімічної стійкості матриць, що свідчить про здатність матеріалу матриць перешкоджати поширенню локалізованих в них радіонуклідів в навколишнє середовище. Відзначено, що швидкості вилуговування радіонуклідів з цементних матриць змінюються з плином часу їх контакту з водомісткими середовищами. Наведено і проаналізовано хронометричні залежності швидкостей вилуговування. Показано, що вони складаються з двох ділянок різної тривалості. Початкова ділянка, тривалістю до 250 годин, відрізняється більш високою крутизною зі зменшенням абсолютного значення швидкості до 2 порядків. Наступна ділянка, тривалістю до 2500 і більше годин, відрізняється асимптотичним зниженням швидкості до постійного мінімального значення. Отримано апроксимуючі функції експериментальних хронометричних залежностей вилуговування радіонуклідів у вигляді логарифмостепеневих виразів, справедливих в інтервалах тривалості експериментів, з достовірністю не менше 0,9. Показано, що найбільш інтенсивно піддаються вилуговуванню одновалентні іони натрію і цезію. Абсолютні значення швидкостей вилуговування одновалентних нуклідів на два-три порядки більші, ніж двовалентних, за інших рівних умов. Значний вплив на швидкість вилуговування надає зміст нукліду в складі матеріалу компаунда. Підвищення температури матриці сприяє збільшенню швидкостей вилуговування, що швидше за все обумовлено позитивним температурним коефіцієнтом дифузійної характеристики. Опромінення матриць зменшує швидкість вилуговування в результаті зниження пористості тіла матриці і утворення малорозчинних гідратів. Окислювально-відновні показники розчинів, пов'язаних матрицями, не робить істотного впливу на швидкість вилуговування.
Ключові слова: водостійкість, затвердіння, вилуговування, радіонуклід, цемент, матриця, швидкість, іони

V. Kovalchuk, I. Kozlov, O. Dorozh, N. Bogdanov, N. Meletenko. **Water resistance of solidified radioactive waste.** The results of experimental studies of leaching characteristic of liquid radioactive waste of radionuclides from cement matrices for long-term storage are considered. It is shown that leach ability is a characteristic of the chemical resistance of matrices, indicating the ability of the matrix material to prevent the spread of radionuclides localized in them into the environment. It is noted that the rate of leaching of radionuclides from cement matrices changes with the time of their contact with aqueous media. Chronometric dependences of leaching rates are presented and analysed. It is shown that they consist of two sections of different duration. The initial section, lasting up to 250 hours, is distinguished by a higher steepness with a decrease in the absolute value of the speed to 2 orders of magnitude. The subsequent section, lasting up to 2500 hours or more, is characterized by an asymptotic decrease in speed to a constant minimum value. Approximating functions of the experimental chronometric dependences of radionuclide leaching were obtained in the form of power-logarithm expressions, valid in the intervals of the duration of the experiments, with a reliability of at least 0.9. It is shown that monovalent sodium and cesium ions are most intensively subject to leaching. The absolute values of the rates of leaching of monovalent nuclides are two to three orders of magnitude higher than those of divalent ones, all other things being equal. The content of the nuclide in the composition of the compound material has a significant effect on the leaching rate. An increase in matrix temperature promotes an increase in leaching rates, which is most likely due to a positive temperature coefficient of diffusion characteristics. Irradiation of the matrices decreases the leaching rate as a result of a decrease in the porosity of the matrix body and the formation of poorly soluble hydrates. The redox values of matrix-bound solutions have no significant effect on the leaching rate.
Keywords: water resistance, curing, leaching, radionuclide, cement, matrix, velocity, ions

Introduction

The development and application of nuclear technologies is accompanied by the inevitable formation of waste of a high degree of potential danger of causing damage to the environment. In developed countries, the reduced amount of accumulated radioactive waste (RW) exceeds 10^{11} Bq/person [1]. The variety of forms of such wastes did not allow building a coherent system of these interactions with the environment.

The prospect of decommissioning nuclear power units that have exhausted their resources entails an increase in the amount of waste, and, accordingly, the actualization of the problem of their interaction with the environment.

In this work, an attempt is made to analyse the chemical stability of solidified liquid radioactive waste in contact with an aqueous medium, in order to justify the requirements for protective barriers for long-term storage of radioactive waste.

Analysis of recent publications. Current state of the art in research on the water resistance of solidified radioactive waste

There are numerous studies of the interaction of various forms of radioactive waste (RW) with the environment.

Studies of the properties of various forms of radioactive waste (RW) subject to long-term storage contain information about their mechanical, thermal and chemical interactions with the environment [1 – 9]. Mainly chemical interactions have been studied experimentally. The methodological support for such problems solving is presented in sufficient detail in [2].

The chemical stability of cement-based matrices is considered in [3, 5 – 9]. The main factor, which in all cases is attributed to the properties of matrices, is their physical and chemical nature.

This makes it difficult to generalize the results obtained in order to substantiate the effectiveness of the matrix type.

Formulation of the problem

Localization of solidified waste with low and medium levels of activity is carried out by their burial in near-surface storage facilities of different levels of engineering support [1]. Increasing the reliability of ground repositories is achieved by creating barriers that ensure the retention of radionuclides in the repository.

It is advisable to place chemically stable solidified radioactive waste in the ground without special reinforcement of the walls and bottom of the storage facilities. The chemical stability of solidified radioactive waste and their simulators is estimated by the intensity of leaching of radionuclides from them during prolonged contact with water and aqueous solutions [2]. The existing standards allow for burial of solidified waste in concrete repositories at a leaching rate of radionuclides of no more than 10^{-4} g/cm^2·day [3].

The purpose and objectives of the study

The leaching rate determined experimentally is taken as the regulatory rate of radionuclide release for specific forms of RW [4].

RW cementation technology is spreading at the stations. Therefore, generalization of information on the resistance to leaching (water resistance) of such products seems relevant. The aim of this work is to assess the chronometric patterns of leaching of typical radionuclides.

Methods for studying the rate of leaching of radionuclides from solidified radioactive waste

The rate of leaching of radionuclides from solidified waste depends on many factors, among which the most important are the nature of the radionuclide and the duration of contact of the waste with an aqueous medium [5]. The elements of matrix structures (sodium, calcium) and carriers of radioactivity (cesium, cobalt, strontium) are of the greatest interest.

The mechanism of the transfer of radionuclides from calcined materials to water is determined by the solubility of the compounds entering the product and their distribution in the product space. The leaching rate from such materials is variable. In the initial period, radionuclides are leached on the surface of contact with the aqueous medium. As the surface becomes depleted in nuclides, the rate decreases and is further determined by the diffusion of radionuclides over the surface of micropores and the rate of exchange of the solution formed in the pores with the solvent.

The leaching rate of individual radionuclides (or their mixtures) is expressed by the ratio of the activity (or mass) of radionuclides A_τ that have passed into the water-containing medium during the time of contact of the sample with the medium τ of its surface S, to the specific activity (or mass) of the solid product A_0:

$$R = \frac{A_\tau}{A_0 \cdot S \cdot \tau}. \tag{1}$$

The analysis of the chronometric dependences of the leaching rates and the assessment of the influence of individual factors on their intensity was carried out on the basis of experimental data [3, 6 – 8].

Compounds, the rate of leaching of some components of which are discussed below, are characterized by the following indicators (Table 1).

Table 1

Quality indicators of compounds

Index	Compound by			
	[3]	[6]	[7]	[8]
Cement	400	M-500	Contact hardening binders	47.6 % by mass
Mortar / cement ratio	0.85	0.6	0.4	0.1
Water / cement	0.6	0.4	–	0.5
Radionuclide fixer	Iron oxide exchangers, with an exchange capacity of 7 meq/g	–	–	Bentonite, 4.8 % by mass
Liquid radioactive waste or imitation	$C_2H_2O_4$; $KOH+KMnO_4$	$NaNO_3$	vat residue	vat residue 23.8 % by mass

Research results, analysis of the features of radionuclide leaching

Matrices formed on the basis of Portland M-500 cement with mixing with $NaNO_3$ solution are characterized by a significant sodium content and high intensity of its leaching, which is most likely due to the washing out of the $NaNO_3$ salt [6].

The leaching rate within 55 hours reaches the minimum value ($2 \cdot 10^2$ g/(cm$^2 \cdot$day)), which is two orders of magnitude higher than the standard value. This area with a confidence of 0.99 is displayed as a power expression:

$$R = 10^{-0.3888Ln(t) - 1.3954} , \qquad (2)$$

Further, for 20 days the intensity increases monotonically more than twofold, reaching a maximum of $5.3 \cdot 10^2$ g/(cm$^2 \cdot$day) and then asymptotically decreases to the previously reached minimum value (Fig. 1).
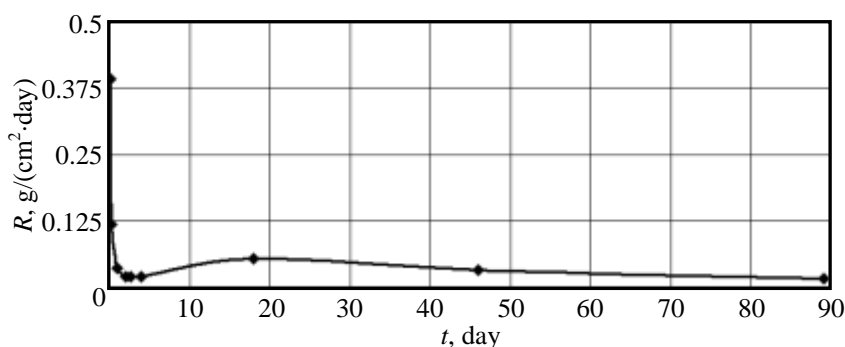

Fig. 1. Dependence of the rate of sodium leaching from the cement matrix on time

In general, the dependence of the leaching rate on the duration of contact within the observation period, with a confidence level of 0.99, is represented by the function:

$$\lg R = -0.2594(\lg t)^4 + 0.2588(\lg t)^3 + 0.7926(\lg t)^2 - 0.7847(\lg t) - 1.5007 . \qquad (3)$$

Piecewise extrapolation of the above dependence shows that 100 days after the last measurement, the decrease in speed reaches 40 % of its value, and after a year – about 70 %, which is almost 50 times higher than the standard value.

It can be assumed that this character is explained by the intense dissolution of the salt of the surface layers of the matrix and the slow diffusion of ions from the deep layers of the matrix. The effect of irradiation on the sodium leaching rate is not detected.

Leaching of calcium, in contrast to sodium, from matrices of the same composition (Fig. 2), does not exceed the standard values from the initial moment of observation. For an unirradiated sample, the rate decreases within 120 days from $3.3 \cdot 10^{-4}$ to $7.0 \cdot 10^{-6}$ g/(cm^2·day), and for an irradiated sample with a dose of 195 kGy it decreases from about $9.2 \cdot 10^{-5}$ to $6.2 \cdot 10^{-7}$ g/(cm^2·day). The tendency of a decrease in the rate of calcium leaching with an increase in the absorbed dose is most likely associated with a decrease in the porosity of the samples and pore sizes under prolonged irradiation [6]. A more pronounced effect for calcium is possibly caused by the formation of a poorly soluble calcium peroxide hydrate $CaO_2 \cdot 8H_2O$ during irradiation. Excretion of calcium in solution after 5 months is almost an order of magnitude less than sodium.
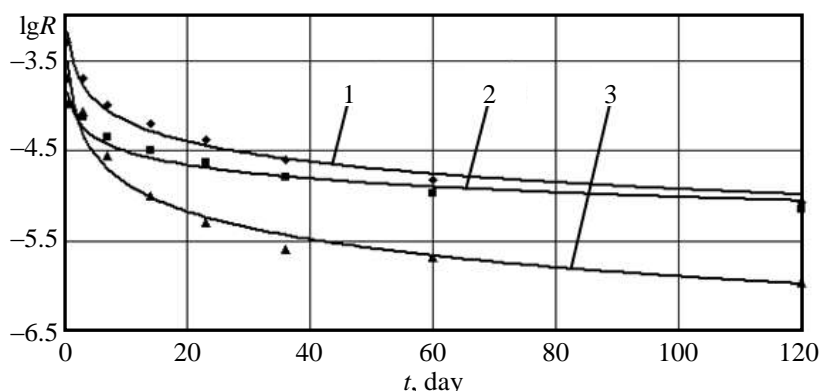

Fig. 2. Influence of radiation dose and observation duration on the rate of calcium leaching: 1 – dose 0 kGy; 2 – dose 55 kGy; 3 – dose 195 kGy

The rate of calcium leaching with a confidence of 0.95 is approximated by a power-law relationship, in which the radiation dose and the natural logarithm of time appear:

$$R = 10^{(-2 \cdot 10^{-5} \cdot D^2 + 3 \cdot 10^{-3} \cdot D - 0.33) \cdot Lnt - (6 \cdot 10^{-5} \cdot D^2 - 14 \cdot 10^{-3} \cdot D - 3.41)} . \tag{4}$$

The behaviour of corrosion products (Fe, Ni, Cr, etc.) in the cement compound characterizes the leachability of cobalt [3]. The study was carried out with cement compounds filled with fine-grained (50…70 μm) iron oxide foreign exchangers during the cementing of reducing and oxidizing spent decontamination solutions containing, respectively, oxalic acid and hydroxide with potassium permanganate, made it possible to reveal the exponential nature of the dependence of the intensity of radionuclide release on time.

The rate of leaching of cobalt from compounds (Fig. 3), which have bound oxidizing and reducing solutions with a reliability of 0.91…0.98, are presented in logarithmic coordinates, where the time is calculated in days, and the rate is g/(cm^2·day), by the ratios:

– with oxidizing solutions $R = 10^{-0.4233 Ln(x) - 4.8786}$;

– with reducing solutions $R = 10^{-0.4027 Ln(x) - 5.0398}$. $\tag{5}$

Oxidizing solutions provide a slightly higher leaching rate compared to reducing solutions.

As a rule, cement matrices bind mortars, the salt content of which does not exceed 160 g/dm^3. The technology tested at the South-Ukrainian NPP during the curing of bottoms with a salt content of 400…600 g/dm^3, based on the use of contact hardening mineral binders, made it possible to obtain compounds with a 5…7 fold reduction in volume and sufficient strength [7].
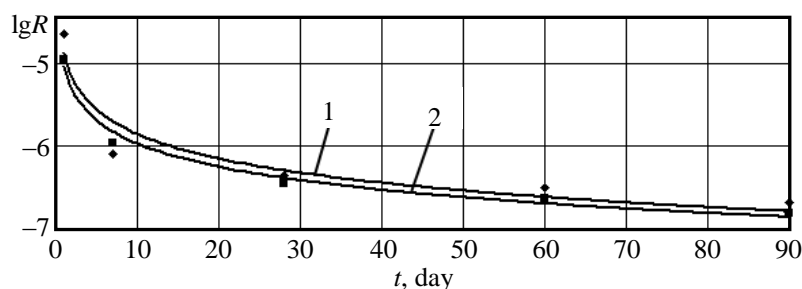
Fig. 3. Dependence of the rate of leaching of cobalt from the cement matrix on time
for oxidizing and reducing solutions: 1 – oxidative; 2 – reductive

The results obtained in the study of the leaching of these compounds note significantly lower values of the leaching rates. While a higher concentration of the bottoms residue corresponds to a lower value of the rate.

The absolute values of the rates are close in magnitude to the rates of leaching from vitrified matrices. The leaching rate from the compounds with a confidence level of 0.97…0.99 is approximated by the ratios:

Samples with the salinity of the vat residue

$$
\begin{array}{ll}
400 \text{ g/dm}^3 & R = \exp(-1.944 Ln(t) - 6.0127) \\
600 \text{ g/dm}^3 & R = \exp(-1.592 Ln(t) - 7.3099)
\end{array}
\tag{6}
$$

Leachability of strontium characterizes the chemical resistance of the cement stone itself, in the structure of which it is included along with calcium [3].

The high initial velocity is due to the dissolution of salts on the block surface, and the subsequent decrease and stabilization is due to the diffusion process of radionuclide movement from the depth of the block to its surface (Fig. 4).
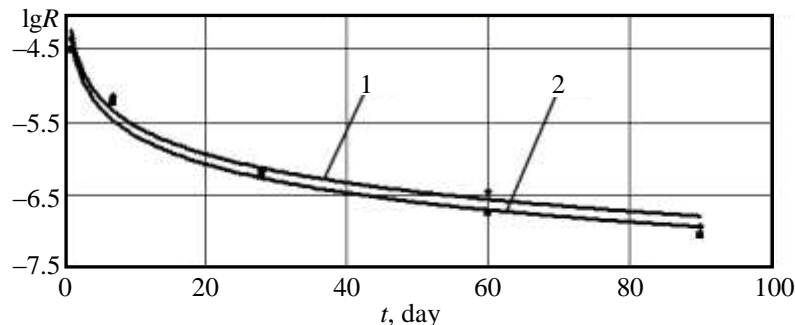

Fig. 4. Dependence of the rate of leaching of strontium from the cement matrix on time for oxidizing and
reducing solutions: 1 – oxidative; 2 – reductive

The leaching rate from the compounds with a certainty of 0.97…0.98 is approximated by the ratios:

– with oxidizing solutions $\qquad R = 10^{-0.5655 Ln(t) - 4.2436}$;

– with reducing solutions $\qquad R = 10^{-0.5724 Ln(t) - 4.3604}$.

$$
\tag{7}
$$

The absolute values of the rates of leaching of calcium, cobalt and strontium are displayed by the same regularities and in the same range of values.

Leachability of cesium (Fig. 5), which is normalized at the level of 10^{-3} g/(cm^2·day), characterizes the sorption capacity of iron oxide ion exchangers [3].
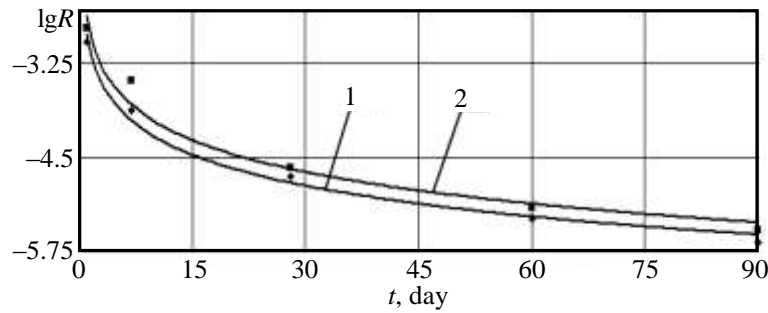
Fig. 5. Dependence of the rate of leaching of cesium from the matrix with oxidizing and reducing liquid radioactive waste on time: 1 – oxidative; 2 – reductive

The intensity of the release of cesium from the compound is almost an order of magnitude lower than the rate of sodium leaching and does not exceed the standards permissible for long-term storage. With a confidence of 0.97…0.99, it is approximated by the ratios:

– with oxidizing solutions $\quad\quad\quad\quad\quad\quad\quad R = 10^{-0.5946 Ln(x) - 2.8555}$;

– with reducing solutions $\quad\quad\quad\quad\quad\quad\quad R = 10^{-0.6138 Ln(x) - 2.6067}$. $\quad\quad\quad\quad$ (8)

Tests of the leachability of compounds (Fig. 6) obtained during the curing of bottoms with a salinity of 400 g/dm^3 showed that at an absorbed dose of more than 60 Gy, the cesium yield rate decreases by 12 % for cesium 134 and by 24 % for cesium 137 [7].
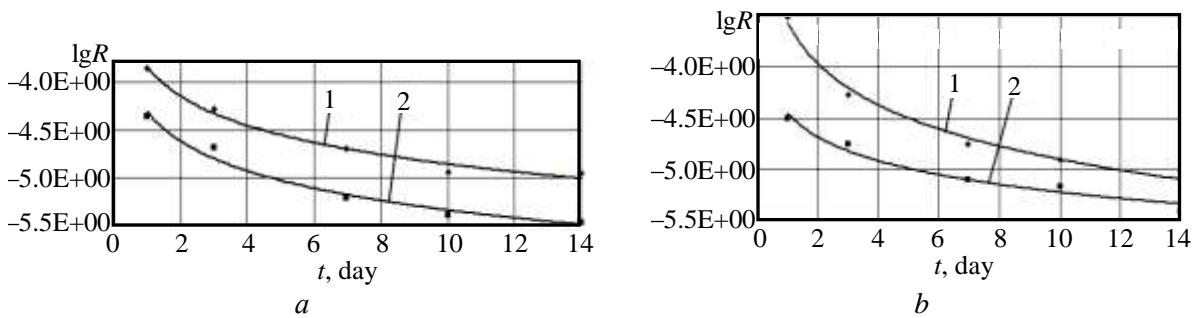

Fig. 6. Dependences of the leaching rate on time from the matrices of nuclides 134Cs (a) and 137Cs (b): 1 – salt content 400 g/dm^3; 2 – salt content 600 g/dm^3

The intensity of the release of cesium from the compound does not exceed the permissible standards for long-term storage and with a reliability of 0.97…0.98 is approximated by the ratios (Table 2).

Table 2

The rate of leaching of cesium from the compound depending on the radiation dose

Absorbed dose, Gy	Radionuclide			
	^{134}Cs		^{137}Cs	
0	$R = 10^{-0.4455 Ln(x) - 3.836}$		$R = 10^{-0.5799 Ln(x) - 3.5674}$	
66	$R = 10^{-0.4526 Ln(x) - 4.3048}$	(9.1)	$R = 10^{-0.3336 Ln(x) - 4.4537}$	(9)

The effect of temperature on the leaching of cesium ions from the irradiated and unirradiated cement matrix is practically not manifested [6]. Fig. 7 shows the dependence of the fraction washed into the solution at temperatures of 20 and 90 °C. The leaching rate noticeably decreases with the holding time and reaches approximately constant values already after about 40 hours.
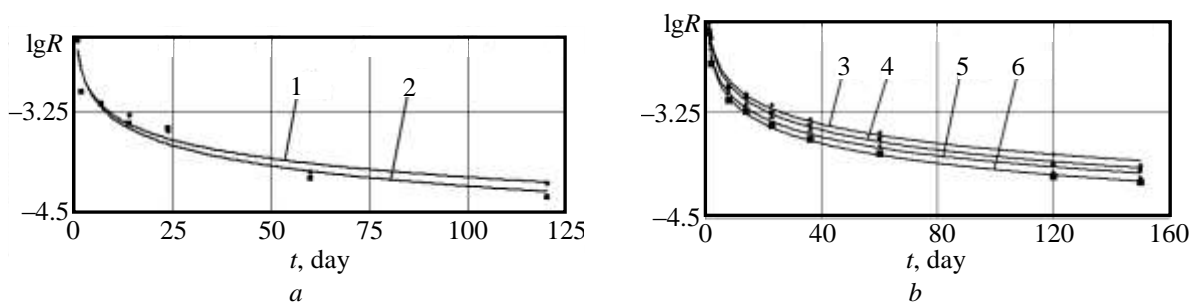
Fig. 7. Dependence of ^{133}Cs leaching rate on time at different temperatures: at 20 °C (a); at 90 °C (b); 1 – unirradiated sample; 2 – irradiated sample, dose 2200 kGy; 3 – unirradiated sample; 4 – irradiated sample, dose 55 kGy; 5 – irradiated, dose 195 kGy; 6 – a irradiated sample, dose 2840 kGy

Immobilization of high-level waste into a cement matrix (Fig. 8) does not significantly affect the leaching rate [6] and is approximated by $R = 10^{-0.1939 Ln(x) - 3.3002}$ a ratio with a reliability of 0.99.
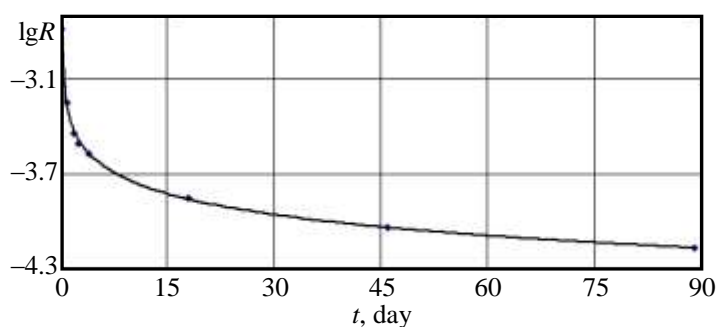

Fig. 8. Dependence of ^{137}Cs leaching rate from cement matrices with high-level waste on time

Leaching of boron-containing compounds is characterized by a decrease in the rate of release of nuclides over time [8]. The time it takes for the leaching rate to reach a stable value is described by the equation:

$$\tau = 1867 + 0.027 m_c - 2.96 m_b - 0.51 m_{cm}, \tag{10}$$

where m_{cs} is the mass of the salts of the imitate in the compound, g; m_{bw} is the mass of water in the compound, g; m_{cm} is the mass of cement in the compound, g.

Correlation coefficient $R^2 = 0.96$.

From the equation it can be seen that the amount of water in the compound most of all affects the time to reach the standard indicator, with an increase in which the yield period decreases.

Conclusions

1. Rates of leaching of radionuclides from cement matrices change with the course of time of contact of the latter with water-containing boundaries.

2. Chronometric dependences of leaching rates consist of two sections. The initial section, which lasts up to 250 hours, is characterized by a higher steepness. The subsequent section, lasting up to 2500 hours or more, is characterized by an asymptotic decrease in speed to a constant minimum value.

3. Chronometric dependences of leaching of radionuclides are reliably approximated by logarithmic-power functions in the intervals of the duration of the experiments.

4. Monovalent ions of sodium and cesium are most intensively exposed to leaching. The exit speed is proportional to the content of the component in the matrix structure and the exposure duration. Within 30…40 days, the rate decreases to the minimum stable value determined by the rate of diffusion of the nuclide in the matrix.

5. The absolute values of the rates of leaching of monovalent nuclides are two to three orders of magnitude higher than those of divalent ones, all other things being equal.

6. An increase in the temperature of the matrix promotes an increase in the rates of leaching.

7. Irradiation of the matrices reduces the leaching rate.

8. Redox values of matrix-bound solutions do not significantly affect the leaching rate.

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