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REMOVAL OF RESIDUAL CONCENTRATIONS OF CESIUM IONS FROM LOW-LEVEL RADIOACTIVE SOLUTIONS

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Because of mathematical modelling, the region of existence of the component composition of a complex inorganic sorbent designed to remove residual concentrations of cesium ions from low-level radioactive aqueous solutions was determined. The olay they limited by x1 – the number of nanoparticles of the iron oxide component from 18 to 19.5 ml, by x2 – the amount of copper ferrocyanide from 0.9 to 1.05 g, by x3 – the amount of Portland cement from 0.88 to 0.95 g. The determined concentrations of the complex inorganic sorbent provide for the removal of cesium ions from 98 to 100% and the capacity of the complex inorganic sorbent from 9 to 9.5 mg/g. According to the data on the frequencies of occurrence of the variation factors and their products in terms of their influence on the degree of cesium ion extraction from solution and the sorption capacity of the complex inorganic sorbent, they ranked in the following sequence, namely: x1x2x3>x1x2>x2x3>x1x3>x3>x2>x1. The influence of variation factors on other properties of the initial parameters is less relevant.

INTRODUCTION

The modern development of nuclear energy it has aimed at achieving sustainable development through the production of clean energy. At the same time, energy production is accompanied by the accumulation of man-made radioactive waste generated during the extraction and processing of uranium ores, uranium enrichment, manufacture of nuclear reactors, operation of nuclear power plants, reprocessing and regeneration of spent nuclear fuel, leaks during transportation and storage of radioactive waste, etc. [1, 2]. At the same time, it is inevitable that liquid radioactive waste and wastewater containing cesium, strontium and other natural radionuclides will be generate. Particular attention should be paid to ^{137}Cs, which is a fission product of plutonium and uranium, is one of the main dose-forming radionuclides, has a long half-life (more than thirty years), and is characterized by high volatility, solubility and activity [3].

As you know, cesium is in the first group of the periodic table of D.I. Mendeleev, and therefore has similar chemical and physical properties to sodium and potassium. ^{137}Cs is a source of γ-radiation, and as one of the most widespread radionuclides, it has a significant harmful effect on the environment. When released into the environment, even in small amounts, cesium they be deposited and accumulated in the soft tissues of aquatic and terrestrial organisms [2, 4]. When ^{137}Cs enters the human body, it causes disorders of the reproductive system, kidneys, liver and other organs [5–7].

An urgent task of environmental safety is to solve the problem of removing residual concentrations of radioactive cesium-137 from low-level aqueous solutions. To date, many methods of cesium ion extraction they known, but practice shows that the most effective is the combination of several methods and the achievement of selective extraction of ^{137}Cs.

The most common methods are: adsorption, ion exchange, chemical precipitation, chemical reduction, membrane technologies, coagulation, extraction, ion flotation [2, 4, 8–11]. Adsorption methods are the most widely used in liquid radioactive waste decontamination technologies due to a wide range of adsorbents, process efficiency, simple technology and a wide range of applications [12]. The most effective sorbents for cesium extraction are ferrocyanide sorbents (synthetic materials based on iron (III), nickel (II), copper (II), and other metals or their mixtures) [13, 14]. These materials, due to their stability, cost, and sorption parameters, are superior to the known synthetic sorbents based on silica gel and silica. Therefore, the issue of obtaining effective sorption materials for the extraction of ^{137}Cs ions from low-level liquid radioactive waste is relevant.

MATERIALS

The authors have developed a complex inorganic sorbent (CIS) based on three components:

1) The first component: preparation of powdered copper ferrocyanide by solid-phase reaction:

$$2CuSO_4 \cdot 5H_2O + K_4[Fe(CN)_6] \cdot 3H_2O =$$
$$Cu_2[Fe(CN)_6] \cdot 7H_2O + 2K_2SO_4 + 6H_2O.$$

For this purpose, potassium ferrocyanide $K_4[Fe(CN)_6] \cdot 3H_2O$ and copper sulfate $CuSO_4 \cdot 5H_2O$ in equal proportions by weight were ground in a ceramic mortar for 30 min to obtain powdered $Cu_2[Fe(CN)_6] \cdot 7H_2O$ (CFC) (Fig. 1).

According to the data (see Fig. 1), copper sulphate is characterised by the following wavenumbers, cm^{-1}: 520, 577, 610, 670, 965, 1005, 1165, 1205, 1650. For the valence vibrations of OH-groups, they are 3430 and 3750 cm^{-1}. Iron ferrocyanide is characterised by the following wavenumbers, cm^{-1}: 586, 1623, 1645, and for the OH-group – 3470 and 3530.

Copper ferrocyanide has the following wavenumbers, cm^{-1}: 589, 619, 655, 1120, 1200, 1620.

2) The second component: nanoparticles of the iron amorphous component. To obtain this component, tap water they passed through a reactor operating at direct current using the PLASMA-SORB technology [15] (Fig. 2).

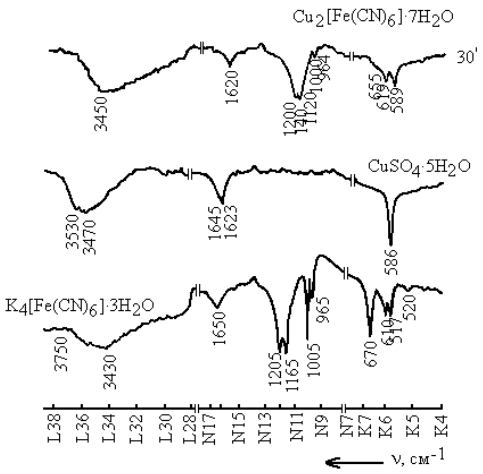


Fig. 1. IR spectrum of $Cu_2[Fe(CN)_6]\cdot 7H_2O$

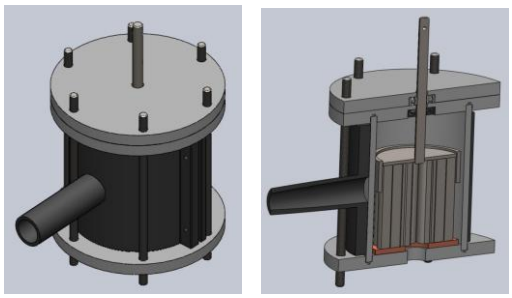


Fig. 2. The scheme of a DC reactor: U=40 V; I=15 A; P=600 W

This component we developed under plasma and electrolysis conditions. According to XRD and IR spectrum, the second component of the complex inorganic sorbent consists of 3 Crystalline phases: FeO – 89%; Fe-fcc 3.8%; Fe-bcc 6.7% (Fig. 3) and contains an X-ray amorphous component in the form of FeCuOHCl (Fig. 4).

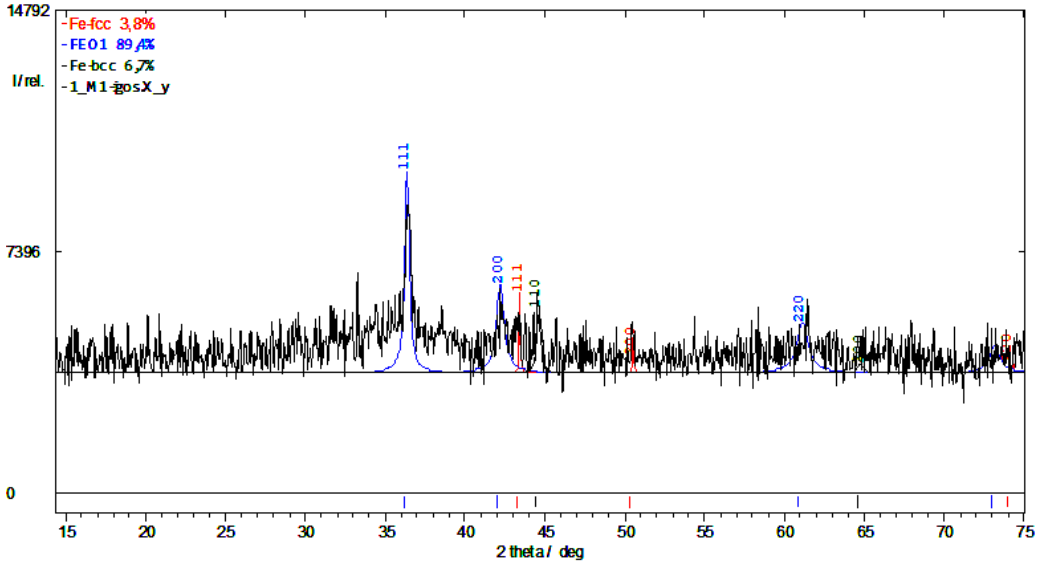


Fig. 3. X-ray phase analysis of iron nanoparticles

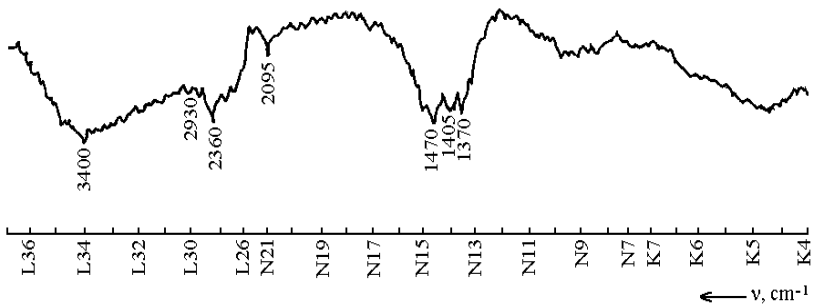


Fig. 4. IR spectrum of iron nanoparticles

For FeCuOHCl we characterized by the following wave numbers, cm^{-1}: 1370; 1405; 1470; 2095; 2360, 2930 and for the valence vibrations of OH-groups – 3400.

3) Third component: Portland cement PC I-500 (Dickergoff, Ukraine).

Low-level radioactive water we prepared by the acid method from soil collected in the suburbs of Fokushima approximately the NPP. A 1 g soil sample we dissolved in 10 ml of 40% HF overnight, 700 ml of tap water we added while stirring, and the pH we adjusted to a value not exceeding 8.5 with NaOH solution. The concentration of sodium ions was 0.09 mol/l. This concentration of sodium ions does not affect the sorption of ^{137}Cs ions by CIS.

According to X-ray fluorescence analysis, the Fukushima soil contains the following chemical elements, %: O – 0.077; Al – 0.129; Si – 0.08; P –

0.017; S – 0.008; K – 0.051; Ca – 0.034; Ti – 0.011; V – ppm 446±46; Cr – ppm 163±20; Mn – 0. 003; Fe – 0.029; Ni – ppm 168±7; Cu – ppm 201±5; Zn – 0.001; Ga – ppm 93±4; As – ppm 98±6; Rb – ppm 369±6; Sr – 0.001; Y – ppm 108±7; Zr – 0.001; Nb – ppm 37±5; Ba – ppm 323±68; Pb – 0.002. In terms of oxides, wt.%: Al_2O_3 – 10.436; SiO_2 – 46.861; ZrO_2 – 0.11; Nb_2O_5 – ppm 52; P_2O_5 – 1.492; SO_3 – 2.514; K_2O – 5.464; CaO – 8.095; Cr_2O_3 – ppm 238; Ni_2O_3 – ppm 236; MnO_2 – 0.546; Fe_2O_3 – 21.318; CuO – ppm 252; ZnO – 0.22; TiO_2 – 2.508; V_2O_5 – 0.08; Ga_2O_3 – ppm 125; As – ppm 130; Rb_2O – ppm 404; SrO – 0.069; Y_2O_3 – ppm 138; BaO – ppm 361; PbO – 0.094.

METHODS

Physical and chemical methods of analysis we used to determine the phase composition of the components of the CIS. X-ray fluorescence (Expert 3L spectrometer of LLC "SPE Institute of Analytical Control Methods", Kyiv, Ukraine), X-ray phase (Ultima IV diffractometer, Rigaku, Japan) in the range 2Θ=15...75° with Graphite monochromator Cu. Infrared spectroscopy method – Spectord 125 (Germany) in the range 400…4000 cm^{-1}. The ICDD PDF-2 and PDF-4 databases we used for data interpretation.

The pH value was determined using a HORIBA LAQUA-PH1500-SS pH meter (with a 9651-10D electrode).

The Cs activity of the studied water was determined using an Atol-3M radiometer (LLC "SPE Institute of Analytical Control Methods", Kyiv, Ukraine) with Spectroline software.

Method of purification of low-level aqueous solutions using CIS.

A 100 ml of radioactive water was poured into a chemical beaker (V=300 ml) using a measuring cylinder, the calculated amount of the first component of the CIS was added and kept in a laboratory mixer with constant stirring for 5 min at a speed of 120 rpm. After 5 min, the second and third components of the CIS we added to the mixture and mixed for 10 min. The resulting mixture we filtered on blue ribbon filter paper. Purified water we tested for activity using the Atoll-3M radiometer.

The optimization of CIS compositions was carried out using a three-factor simplex central design of the experiment in the STATISTICA 12 mathematical environment with the implementation of a special cubic model that takes into account the non-linearity of the influence of factors on the properties of the output parameters [16–18].

The following factors were chosen as varying factors: X1 – the first component of the CIS in the amount of 10 to 30 ml (C=1.03 mg/l); X2 – the second component of the CIS in the amount of 0.5 to 2 g; X3 – the third component of the CIS in the amount of 0.5 to 2 g.

The factors of variation and the planning matrix of the experiment they shown in Tables 1 and 2.

Table 1

Variation factors

Factors, view	Natu-ral	Codes	Varying levels		Interval of variation
			0	1	
Fe-FeO	ml	X1	10	30	20
CFC	g	X2	0.5	2	1.5
PC	g	X3	0.5	2	1.5

Table 2

Planning matrix experiment

Matrix plan codes			Matrix plan in full size		
X1	X2	X3	Fe-FeO	CFC	PC
0	1	0	10	2	0.5
0.33	0.33	0.33	16.7	1	1
1	0	0	30	0.5	0.5
0.5	0.5	0	20	1.25	10
0	0	1	10	0.5	2
0.5	0	0.5	20	0.5	1.25
0	0.5	0.5	10	1.25	1.25

The initial criteria (parameters) were density, pH, sedimentation rate after contact of the CIS with radioactive water, degree of cesium ion extraction (sorption coefficient), and activity of treated water samples. The rate of sedimentation of the suspension after purification was determined visually in a measuring cylinder.

The degree of cesium ion removal (sorption coefficient) we determined by the formula:

$$\alpha=\frac{(C_{in}-C_{ec})}{C_{in}}\cdot 100\%, \quad (1)$$

where C_{in} is the initial concentration of cesium ions in solution, μg/l; C_{ec} is the equilibrium concentration of cesium ions in solution, μg/l.

The sorption capacity of CIS for cesium ions we determined by the formula:

$$A_s=\frac{(C_{in}-C_{ec})}{m}\cdot V, \quad (2)$$

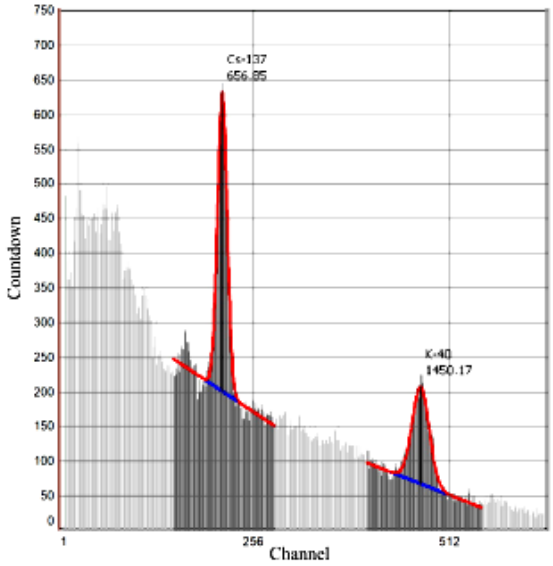
where C_{in} is the initial concentration of cesium ions in solution, μg/l; C_{ec} is the equilibrium concentration of cesium ions in solution, μg/l; V is the volume of solution (sorbate), l; m is the mass of the CIS, g.

RESULTS AND DISCUSSION

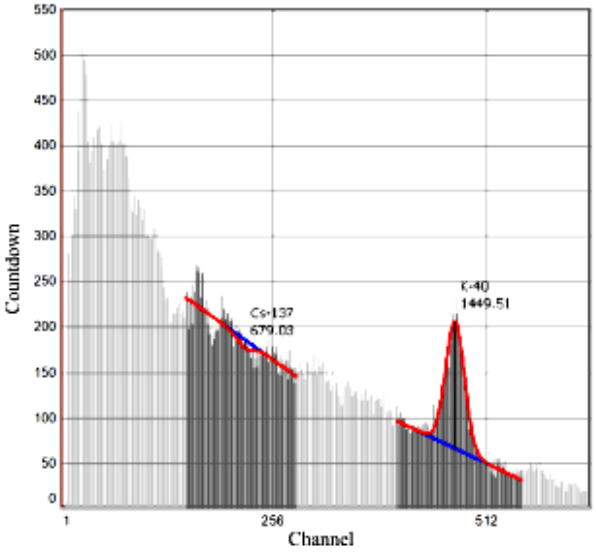
The activity of low-level radioactive water samples after treatment according to the plan points they shown in Fig. 5.

The numerical values of the output parameters they shown in Table 3.

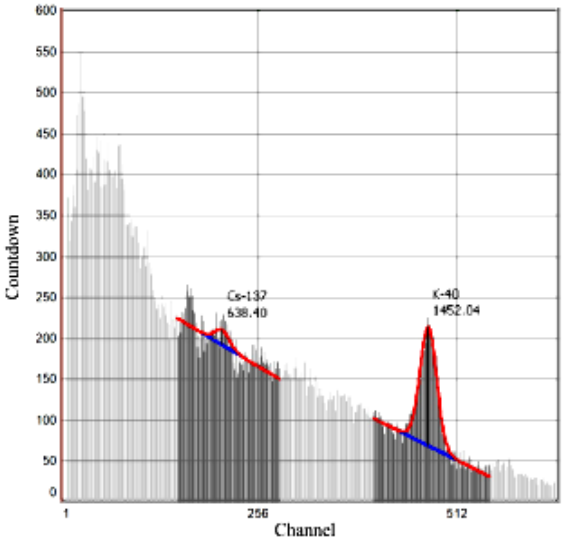
The pH of the source radioactive water is 8.5, and the pH of the treated water samples within the plan points is 9.3–9.4. The pH of the source radioactive water is 8.5, and the pH of the treated water samples within the plan points is 9.3–9.4. The density of the source water is 1 g/cm^3, and the density of the treated water samples within the plan points is 0.744…1.05 g/cm^3.



Initial water



P1



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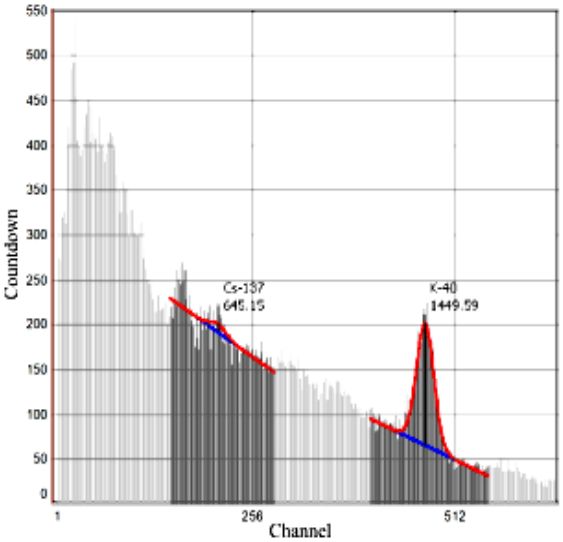

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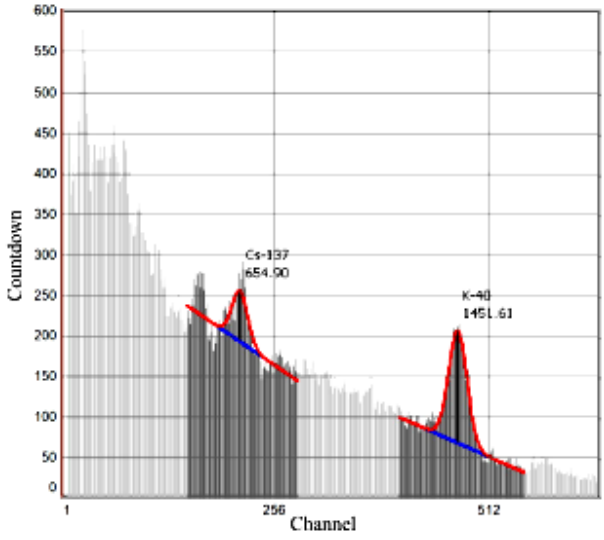

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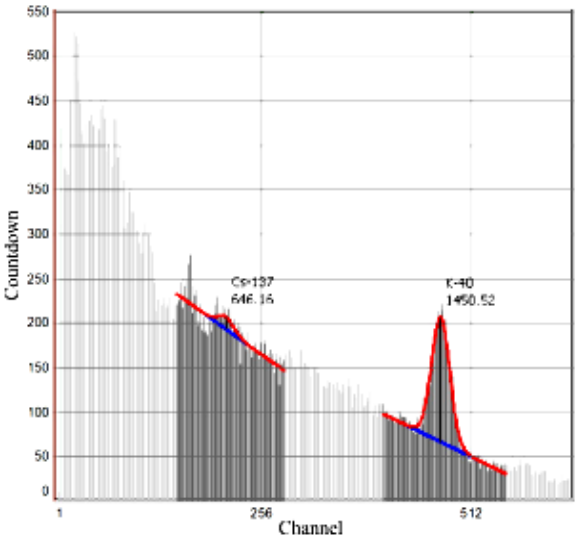
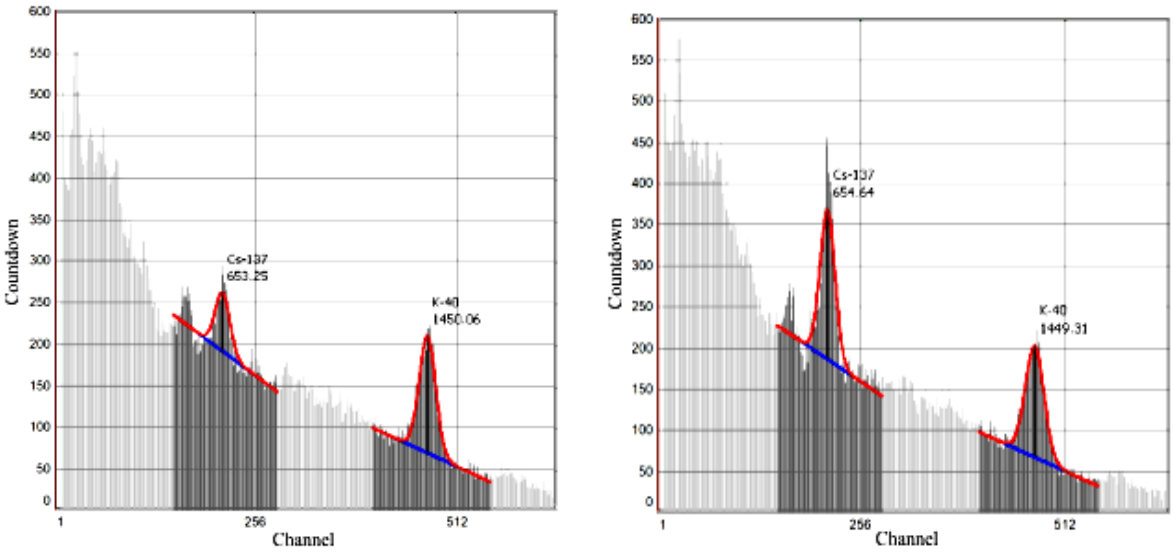


P5

Fig. 5 (Start)



P6 P7

Fig. 5. Activity of samples of low-level radioactive water after treatment according to the plan points

Table 3

Results of experiment

No point plan	V, ml/min	Aw, Bq (^{137}Cs)	α, %	Ac, mg/g
1	8.04	-2	100	1.9
2	4.32	5	94	9.5
3	1.88	3	96	6
4	5.72	17	79	7
5	2.69	4	95	4.2
6	4.72	19	76	6.6
7	8.6	50	37	5.6
Initial RW	–	80	–	–

Using the three-factor simplex central plan, mathematical models (3)–(8) and Pareto diagrams - histograms showing the quantitative ratios of the varied factors on the output parameters in descending order of frequency were obtained [16, 17]. Columns, the order of which follows a descending line from top to bottom [18], represent a Pareto diagram. The length of the columns shows the frequency of the influence of the variation factors on the properties of composites (Fig. 6) and the isoparametric surfaces of the response functions with the expected responses (Figs. 7–12) of the influence of the varied factors on the change in the properties of CIS.

Regression equations for the influence of variable factors on changes in the properties of the initial parameters:

$$pH=9.4x1+9.4x2+9.3x3-0.4x1x2+0.2x1x3-0.6x2x3+0.6x1x2x3; \quad (3)$$

$$\rho=0.987x1+1.05x2+0.856x3+0.098x1x2+0.23x1x3-0.716x2x3+3.477x1x2x3; \quad (4)$$

$$V=1.88x1+8.039x2+2.69x3+3.042x1x2+9.74x1x3+13.022x2x3-74.253x1x2x3; \quad (5)$$

$$Aw\ (^{137}Cs)=3x1-2x2+4x3+66x1x2+62x1x3+196x2x3-882x1x2x3; \quad (6)$$

$$\alpha=96x1+100x2+95x3-76x1x2-78x1x3-242x2x3+1107x1x2x3; \quad (7)$$

$$As=6x1+1.9x2+4.2x3+12.2x1x2+6x1x3+10.2x2x3+62.4x1x2x3. \quad (8)$$

The analysis of the regression equations (3) shows that the change in pH values is most influenced by the main variation factors x1, x2 and x3 and the combined effect of the products of factors x1x3 and x1x2x3, which is confirmed by the distribution of their frequencies (see Fig. 6,a).

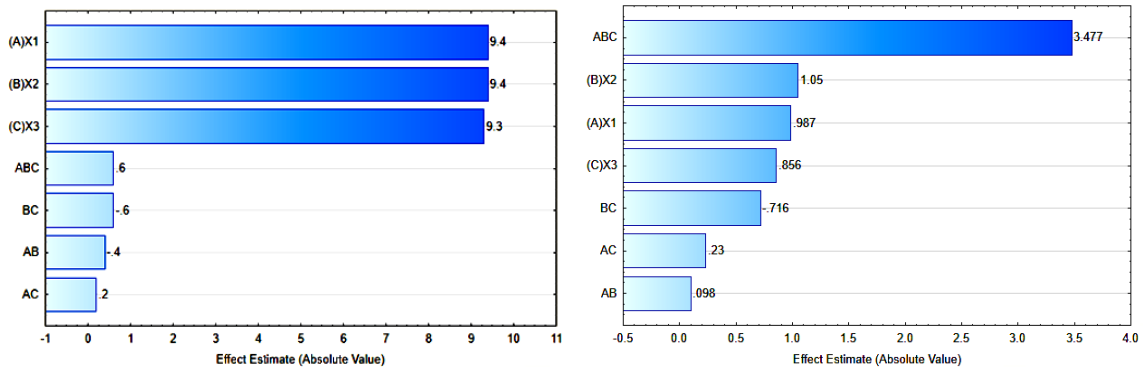


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Fig. 6. Pareto diagrams of the influence of variable factors on the output parameters: a – pH; b – ρ

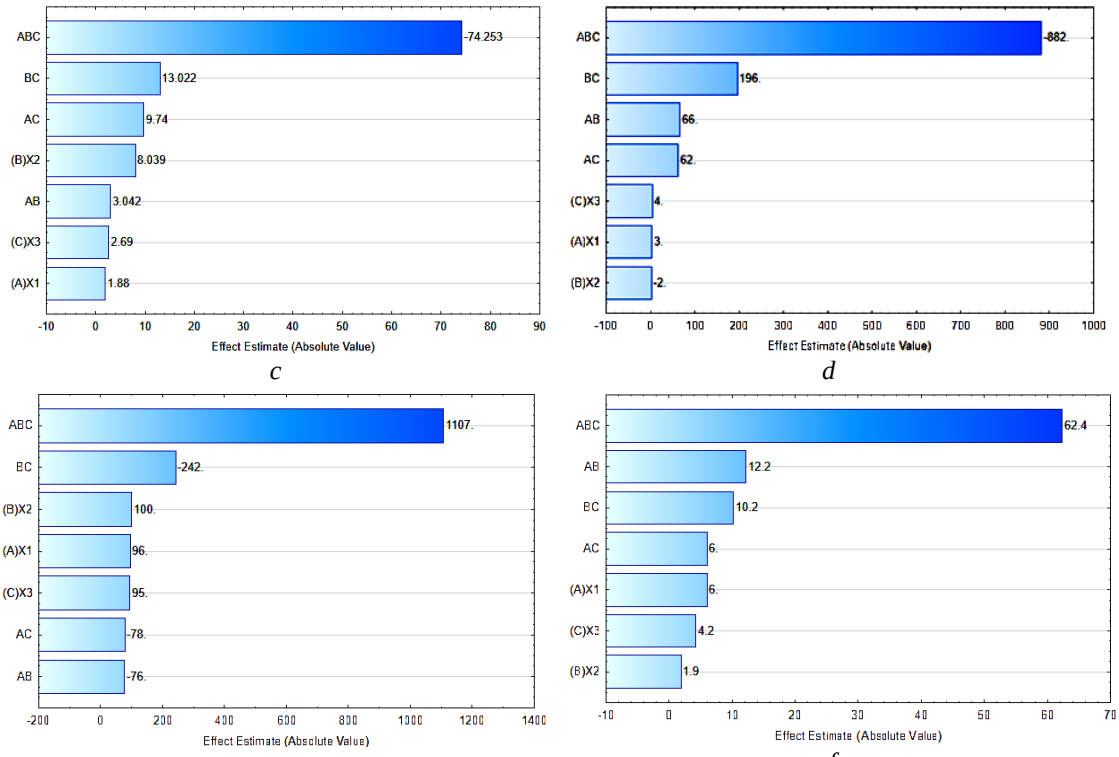

Fig. 6. Pareto diagrams of the influence of variable factors on the output parameters: c –V; d – Aw; e – α; f – As

The change in the values of the CIS solution density (4) is most significantly influenced by the combined effect of the products of factors x1x2x3, then x2, x1, and x3, and less influenced by the combined effect of the products of factors x1x2 and x1x3, which is confirmed by their distribution by frequency of occurrence (see Fig. 6,b).

The change in the deposition rate (5) of CIS is most significantly we influenced by the combined effect of the products of factors x1x2x3, then by x2x3, x1x3, and x2. The less influenced by the combined effect of the products of factors x1x2 and the main variation factors x1 and x3, as evidenced by their distribution by frequency of occurrence (see Fig. 6,c).

The sorption of ^{137}Cs ions (6) is most significantly influenced by the combined effect of the products of factors x2x3, x1x2, and x1x3, and less by the effect of the main variation factors x3 and x1, which is confirmed by their distribution by frequency of occurrence (see Fig. 6,d).

The degree of extraction of cesium ions (7) from the radioactive solution (sorption coefficient) is most significantly influenced by the product of factors x1x2x3, to a lesser extent – by the influence of the main variation factors x2, x1, and x3, which is confirmed by the distribution of their frequencies (see Fig. 6,e).

The sorption capacity of the CIS (8) is most significantly influenced by the products of factors x1x2x3, x1x2, x2x3, and x1x3, with a lesser influence of the main variation factors x1, x2, and x3, as evidenced by the distribution of their frequencies (see Fig. 6,f).

From the above regression equations and the data on the frequencies of occurrence (see Fig. 6), the variation factors and their products in terms of the effect on the degree of cesium ion extraction from solution and the sorption capacity of the CIS we can be ranked in the following sequence, namely: x1x2x3>x1x2>x2x3>x1x3>x3>x2>x1. The influence of the variation factors on other properties of the initial parameters is less relevant.

From Fig. 7,a, it follows that the increase in pH from 9.32 to 9.4 in low-level radioactive solutions is influenced by the introduction of iron nanoparticles in the amount of 11 to 30 ml (factor x1) and copper ferrocyanide in the amount of 0.5 to 0.09 and 1.5 to 2 g (factor x2) into the complex sorbent.

The decrease in the alkalinity of the solution from 9.28 to 9.2 is influenced by the introduction of Portland cement in the amount of 1.9 to 0.8 g (factor x3), which is confirmed by the data of expected reactions shown in Fig. 7,b.

The increase in the density of low-level radioactive solutions from 0.9 to 1.05 g/cm^3 (see Fig. 8,a) is influenced by the combined introduction of nanoparticles of the iron oxide component in the amount of 15 to 30 ml (factor x1), copper ferrocyanide in the amount of 0.5...2 g (factor x2) and Portland cement from 0.5 to 0.7 g (factor x3), which is confirmed by the data of expected reactions given in Fig. 8,b. The isoparametric surface shows a clear area of densities, which they limited by the effect of the variation factors, namely: x1 – the number of nanoparticles of the iron oxide component from 21 to 24 ml; x2 – the amount of copper ferrocyanide from 0.9 to 1.25 g; x3 – the amount of Portland cement from 0.6 to 0.92 g. According to Fig. 8,b, the second component of the CIC has the most significant contribution.

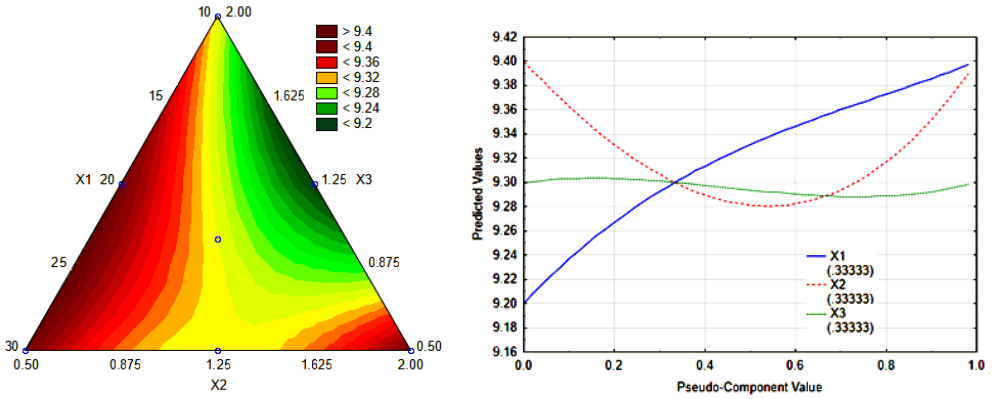

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Fig. 7. Isoparametric response surface (a) and expected responses (b) of the influence of the factors of variation of the CIC on the pH change of aqueous low-level radioactive solutions

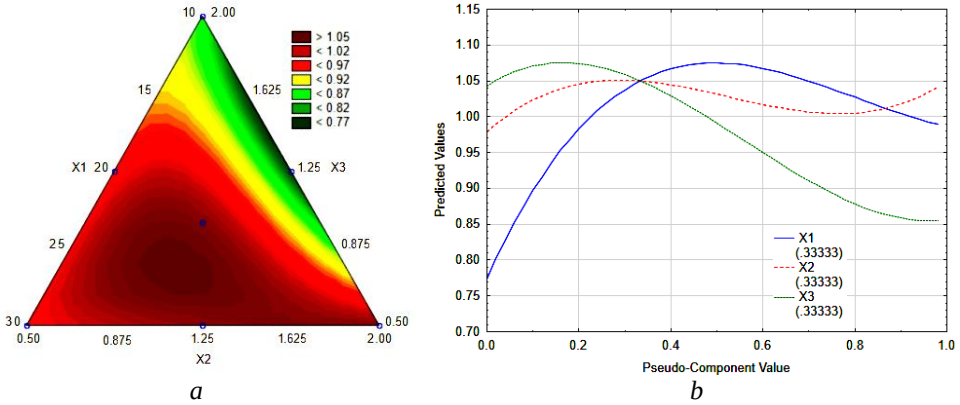

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Fig. 8. Isoparametric response surface (a) and expected responses (b) of the influence of the factors of variation of the CIC on the change in the density of ρ, g/cm^3, of aqueous low-level radioactive solutions

The increase in the rate of deposition of the complex sorbent from 6 to 9 ml/min (see Fig. 9,a) is influenced by the combined introduction of copper ferrocyanide in the amount of 1.52...2 g (factor x2) and Portland cement from 0.5 to 1.625 g (factor x3). This they confirmed by the data of the expected reactions shown in Fig. 9,b.

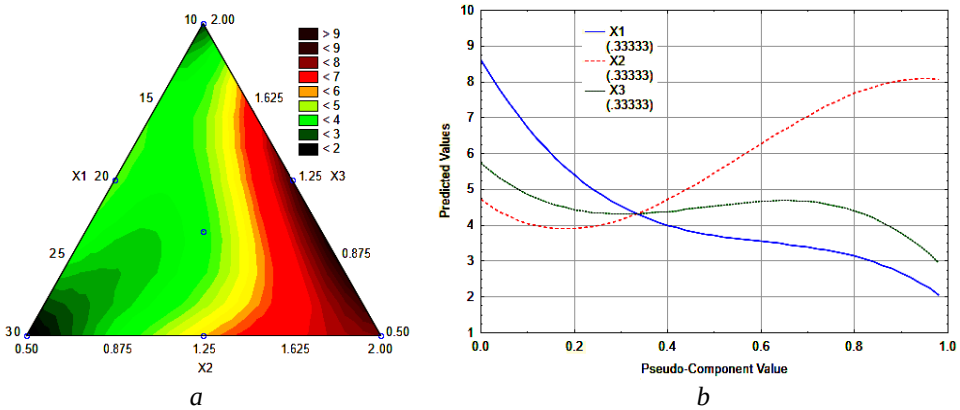

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Fig. 9. Isoparametric response surface (a) and expected responses (b) of the influence of the factors of variation of the CPS on the change in the deposition rate V, ml/min of the substrate

The highest activity of radioactive solutions for cesium ions from 40 to 48 Bq (see Fig. 10,a) is observed when the CIS slide contains nanoparticles of the iron component from 10 to 12.5 ml (factor x1), copper ferrocyanide from 0.9 to 1.9 g (factor x2) and Portland cement from 0.7 to 1.8 g (factor x3). This they confirmed by the data of expected reactions relative to the central point of the plan (see Fig. 10, b).

The most significant contribution has made by factors x3 and x2 – the content of Portland cement and copper ferrocyanide or the third and second components of the CIS and reactions shown in Fig. 10,b. Results of measurements of samples of activity of weakly active radioactive water on the Atoll-3 M device according to the plan points (see Table 3).

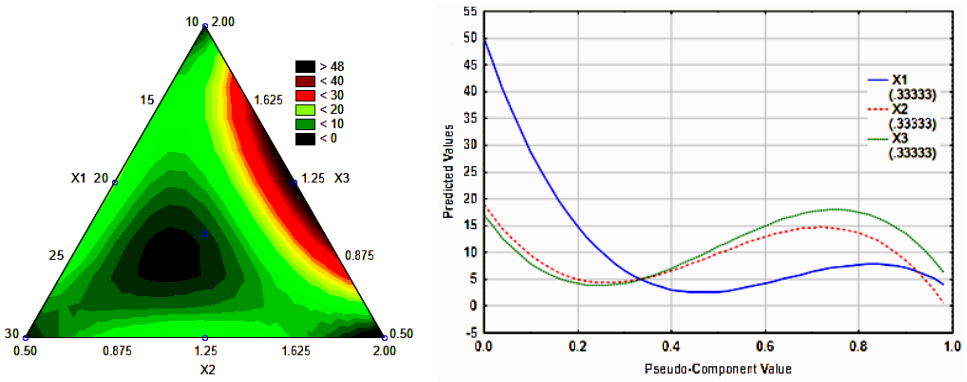

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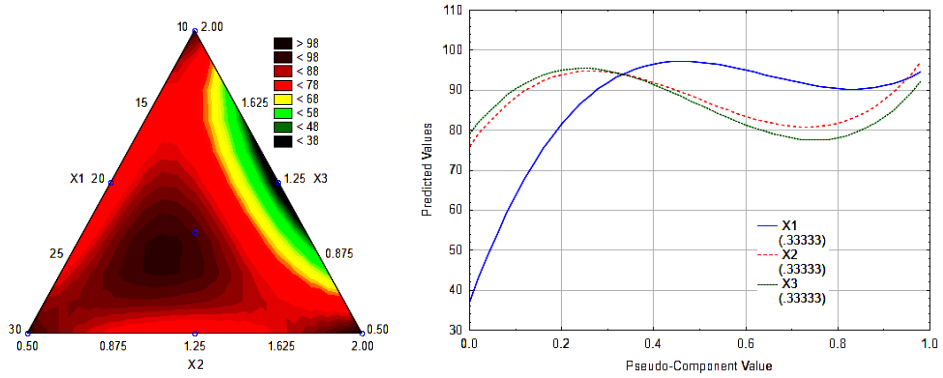
Fig. 10. Isoparametric response surface (a) and expected responses (b) of the influence of factors of variation of the CIC on the change in activity Aw, Bq, by cesium ions of weakly radioactive water

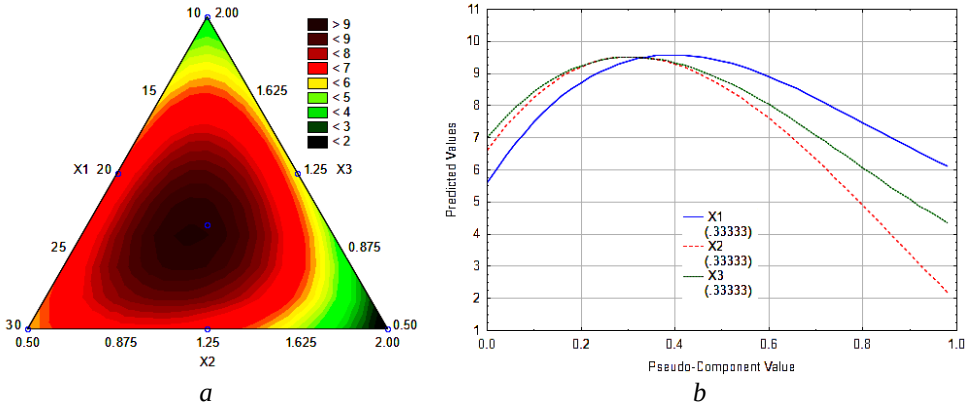
The degree of cesium ion extraction (sorption coefficient) from 78 to 98% (see Fig. 11,a) is affected by the combined introduction of iron nanoparticles in the amount of 15 to 30 ml (factor x1), copper ferrocyanide in the amount of 0.5 to 2 g (factor x2), and Portland cement from 0.5 to 0.7 g (factor x3) into the sorbent. This is confirmed by the data of the expected reactions shown in Fig. 11,b.



a *b*

Fig. 11. Isoparametric response surface (a) and expected responses (b) of the influence of factors of variation of the CIS on the change in the degree of extraction of α, %, cesium ions from weakly radioactive water

The largest sorption capacity – 9.5 mg/g (see Fig. 12,a) has a complex sorbent containing iron nanoparticles from 15 to 25 ml, copper ferrocyanide from 0.9 to 1 g, portland cement from 0.88 to 0, 92 g. This we confirmed by graphs of expected reactions (see Fig. 12,b).



a *b*

Fig. 12. Isoparametric response surface (a) and expected responses (b) of the influence of the factors of variation of the CIC on the change in the adsorption capacity Ac, mg/g, of cesium ions from low-level radioactive water

By the method of superimposing isoparametric diagrams of the studied initial parameters, the region of existence of the component composition of a complex inorganic sorbent intended to remove residual concentrations of cesium ions from low-level radioactive aqueous solutions was established, namely: by x1 – the number of nanoparticles of the iron-iron component from 18 to 19. 5 ml, by x2 – the amount of copper ferrocyanide from 0.9 to 1.05 g, by x3 – the amount of Portland cement from 0.88 to 0.95 g, which ensures the extraction of cesium ions from 98 to 100% and the capacity of the CSC from 9 to 9. 5 mg/g at the

alkalinity of the solution pH within 9.2...9.3, density from 1.02 to 1.05 g/cm^3 and the deposition rate of CIS from 4 to 5 ml/min at a constant processing time (15 min) and mixer speed (120 rpm).

CONCLUSIONS

Because of mathematical modelling, the region of existence of the component composition of a complex inorganic sorbent designed to remove residual concentrations of cesium ions from low-level radioactive aqueous solutions was determined. The area we limited by x1 – the number of nanoparticles of the iron oxide component from 18 to 19.5 ml, x2 – the amount of copper ferrocyanide from 0.9 to 1.05 g, and x3 – the amount of Portland cement from 0.88 to 0.95 g. The determined concentrations of the complex inorganic sorbent provide for the removal of cesium ions from 98 to 100% and the capacity of CIS from 9 to 9.5 mg/g. This is ensured by observing the alkalinity of the solution pH within 9.2...9.3, density from 1.02 to 1.05 g/cm^3 and the deposition rate of CNS from 4 to 5 ml/min at a constant processing time (15 min) and mixer speed (120 rpm).

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ВИДАЛЕННЯ ЗАЛИШКОВИХ КОНЦЕНТРАЦІЙ ІОНІВ ЦЕЗІЮ ІЗ СЛАБОАКТИВНИХ РАДІОАКТИВНИХ РОЗЧИНІВ

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За допомогою математичного моделювання визначено область існування компонентного складу комплексного неорганічного сорбенту, призначеного для видалення залишкових концентрацій іонів цезію з низькорадіоактивних водних розчинів. Область обмежена по x1 – кількістю наночасток залізооксидного компоненту від 18 до 19,5 мл, по x2 – кількістю фероціаніду міді від 0,9 до 1,05 г, по x3 – кількістю портландцементу від 0,88 до 0,95 г. Визначені концентрації комплексного неорганічного сорбенту забезпечують видалення іонів цезію від 98 до 100%; ємність комплексного неорганічного сорбенту становить від 9 до 9,5 мг/г. За даними про частоти появи варіаційних факторів та їх продуктів за впливом на ступінь вилучення іонів цезію з розчину та сорбційну здатність складного неорганічного сорбенту їх розташували в такій послідовності, а саме: x1x2x3>x1x2>x2x3>x1x3>x3>x2>x1. Менш актуальним є вплив варіаційних факторів на інші властивості вихідних параметрів