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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

RESEARCH OF PROCESSES FOR CLEANING OF SOIL CONTAMINATED WITH RADIONUCLIDES

Speciality: 2305.01 – Nuclear chemistry

Field of science: Chemistry

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The work was performed at the Institute of Radiation Problems of the Ministry of Science and Education of the Republic of Azerbaijan.

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Dissertation council FD 1.22 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at the Institute of Radiation Problems of the Ministry of Science and Education of the Republic of Azerbaijan.



GENERAL CHARACTERISTICS OF THE WORK

Relevance of the topic and its degree of development. There is a need for immediate deactivation measures in large areas contaminated with radioactive waste around NPPs where largescale nuclear accidents have occurred in order to make it safe for displaced residents and, in particular, for the personnel of the NPPs located. Numerous scientific research works are continued in the countries with nuclear potential, in order to ensure the effectiveness of the mentioned measures, and the important results are often used in production and deactivation processes.¹

The following options have been used until now: surrounding the soil contaminated with radionuclides with warning signs and a separation fence in order to prevent the entry of outsiders in accordance with the quarantine rules; implementing deactivation measures in case of surface contamination; in case of great necessity to complete transfer of the top layer of the local soil areas extremely contaminated with chemical waste and replacement with other clean soil layer. Decontamination of surfaces is mainly carried out for the purpose of cleaning of equipment, especially military equipment, roads, buildings, weapons and military clothing, and serves to eliminate cases of surface contamination. These processes are accompanied by the partial cleaning of surfaces, as well as the dispersion and absorption of radionuclide extracts into the soil.

Waiting for the long-term natural purification process by tence off the soil will lead to centuries for the complete deactivation of the soil. Periodically mowing and burning the vegetation of the contaminated area and burying the resulting mineral remains in the existing isotope landfill ("isotope cemetery") in order to partially speed up this process are such process that can hardly be realized due to a multi-year series of measures. Therefore, there is a need to develop a more effective (relatively short-term and few-step) method for

¹ Degtyarev, V.V. Sorbent i sorbtsionno-desorbtsionnyy sposob vydeleniya urana i soyedineniy aktinoidov s yego ispolzovaniyem. Patent RU 2256497 C1.– 20.07.2005, p.1-6 / Apkaneyev A.V.

cleaning of soil contaminated with radionuclides. The development of a cleaning method characterized by a high degree of cleaning (high deactivation coefficient) that requires less work time, energy, financial resource compared to existing deactivation methods, for contaminated with nuclear waste of soil and materials is one of the topical areas of radiochemical research.

Object and subject of the research. The object of the research work is soil contaminated with nuclear fuel waste, radioactive material waste separated as a result of deactivation processes and sent for keeping in nuclear material waste storage facilities. Current methods of cleaning soil contaminated with nuclear fuel and radioactive waste are characterized by: low efficiency and multi-stage, as well as sending of the small clay fraction of soil rich in radionuclides together with the separated radionuclides to radioactive waste storage facilities or nuclear cemeteries, i.e. nuclear waste storage facilities quickly fill up. As the subject of the dissertation, it is planned to develop a more effective method of cleaning soil samples contaminated with radioactive substances than existing deactivation methods using existing research methods in the fields of radiation chemistry, radiochemistry and analytical chemistry.

Goal of the dissertation work is to research the processes of effective cleaning of soil contaminated with nuclear fuel and radioactive waste from radioactive substances and nuclear fuel waste in a short time, which is one of the urgent problems of radiochemistry and nuclear chemistry, and to develop a method (deactivation process) of effective separation of radionuclides from soil.

For this purpose, it is planned to use reagents (HNO₃, HCl, their mixture, NaOH) that ensure the effective dissolution of heavy metals and radionuclides and to reduce the consumption of reagents due to washing treated soil with water.

In order to achieve the goal of the dissertation work, it is planned to solve the following issues using radiometric measurement, analytical and physico-chemical research methods:

- monitoring of the general radioactive background in the country and relevant types of radioactive radiation with meters and radiometers equipped with alpha, beta, gamma and neutron detectors; - study of radionuclide contamination of soil samples taken from areas with a relatively high background of radioactive radiation;

- comparative analysis of activities of radionuclides in adsorbents and solution residue after passing nuclear material (uranyl nitrate) solutions through different adsorbents, evaluation of the degree of purification of solution from radionuclides;

- determination of activities of uranium isotopes in soil samples contaminated with uranyl nitrate;

- determining the degree of purification of soil samples by gamma spectroscopic analysis of soil contaminated with nuclear material waste and their extracts by extracting soil samples contaminated with uranyl nitrate in different sequences and proportions with weak acid, alkaline solutions and distilled water;

The candidate defends the following provisions:

- determination of the activities of certified standard samples of individual isotopes of radioactive elements during the experimental period and their energy spectral intensities necessary for comparative analysis;

- determination of the activities of individual radioactive isotopes detected in soil samples contaminated with radionuclides and adsorption of uranium isotopes from uranyl nitrate solutions with various adsorbents by comparing the results of gamma spectroscopic analysis of soil with the results of gamma spectroscopic analysis of standard individual isotope samples;

- determination of purification rates of soil samples contaminated with uranyl nitrate by extracting with weak acid and alkaline solutions and then washing with distilled water.

Scientific novelty of the research:

- determination of the activities of radioactive elements in soil samples contaminated with radionuclides, nuclear material and rich in natural radionuclides by comparing them with the activities of certified individual isotope samples during the experimental period and determination of the degree of adsorption of uranium isotopes from aqueous solutions obtained from soil samples contaminated with nuclear material waste with various adsorbents;

- determination of the degree of purification of soil samples from

radionuclides by successive extraction of soil samples contaminated with uranyl nitrate attributed to nuclear materials with strong acid, alkaline solutions and distilled water and by repeated analysis of residual soil samples;

- feasibility study and determination of optimal parameters of the cleaning process of soil samples contaminated with uranyl nitrate by sequential extraction with strong acid, alkaline solutions and distilled water.

Theoretical and practical significance of the research. The obtained results serve to enrich the important information base in some fields such as chemistry, ecology, radiochemistry, nuclear chemistry, radiation chemistry, radioecology, radiation materials science. The results of the scientific study and research methods can be used in the following cases and areas:

- in order to clean up soil contaminated with radionuclides in the areas of accidental events in nuclear technology and energy facilities explained in the introduction and literature review part of the dissertation, and to make these areas safe and suitable for living beings;

- in order to bring areas contaminated by rivers flowing from lands rich in natural radionuclides or contaminated by nuclear material waste and contaminated as a result of accumulation of radioactive isotopes near the mouth of river into suitable condition for agriculture, if necessary;

- it can be used for the detection of radioactive substances in areas with high radioactive radiation background, determination of their types, their separation from soil, obtaining solid extracts of radionuclides and carrying out complex isotope enrichment process.

Approbation of the research. The results of the dissertation work were reported and discussed at the following international and republican conferences and published in the materials of those conferences:

- International Scientific and Technological Conference. Natural disasters and human life safety. Scientific and Practical Conference under the Ministry of Education of the Republic of Azerbaijan and AzACU. December 4-6, 2017, Baku;

- International Scientific and Practical Conference. Radiation and chemical safety problems. Scientific and Practical Conference under the Ministry of Education of the Republic of Azerbaijan and AzACU. November 5-6, 2019, Baku;

- Scientific Achievements of Modern Society. XII International Scientific and Practical Conference. July 22-24, 2020, Liverpool, UK;

- International Conference on "Scientific research of the SCO countries: synergy and integration". October 28, Beijing, China, 2020;

- Modern science: problems and innovations. Abstracts of the 9th International Scientific and Practical Conference. November 17, 2020, Stockholm, Sweden;

- International Scientific and Practical Conference. Emergency risks in the de-occupied territories. Scientific and Practical Conference under the Ministry of Education of the Republic of Azerbaijan and AzACU. May 21, 2021;

- I International Scientific Conference on "Multidisciplinary Academic Explorations". Proceedings of Conference. Amsterdam, Netherlands. January 10-12, 2022.

Publications: The results of the research were reflected in 22 scientific works (15 articles and 7 conference materials) published in foreign and republican scientific journals and conference materials.

Name of the institution where the dissertation work was carried out:

The dissertation work was carried out at the Institute of Radiation Problems of ANAS in 2015-2022.

The dissertation work consists of 4 chapters.

Structure and scope of the work: The dissertation consists of 65 figures, 17 tables, an annotated introduction, four chapters, conclusions and a list of references on 171 pages. 204 scientific literature in the list of references were used in the dissertation.

MAIN CONTENT OF THE RESEARCH:

In the introduction, the relevance of the dissertation work, the formulation of the goal and the main tasks, the innovations of the scientific results, the experimental and applied importance of the work, the main provisions defended, the approbation of the work and the brief content of the chapters are explained.

In the first chapter, the analysis on the contamination of environmental objects as a result of environmental disasters and nuclear accidents that occurred on Earth, and on the created ecological situation was carried out. A comparative analysis of the applied cleaning methods was carried out in order to investigate the cases of contamination of large land areas with radionuclides and nuclear fuel waste after major nuclear accidents and to ensure the radiation safety of residents living in those areas and NPP employees.² It has been proposed to replace these processes, which are mostly multi-stage and less profitable, with a more effective deactivation method. For this purpose, the directions of conducting systematic research for the development of a new deactivation method more effective than the existing cleaning methods of soil contaminated with nuclear material waste have been justified. There is a need to develop the scientific basis of a new, effective, fast deactivation (cleaning) method that ensures cleaning of contaminated soil to a safe level, since the landseparated part of the existing deactivation methods is in the form of a mixture of radionuclide fraction and fine sand fraction, that is, they lead to quick filling of waste storage facilities, they are either less effective (with a lower deactivation coefficient) or they consist of multi-stage processes, respectively, less profitable, they require more financial resources and time. Development of methods for cleaning contaminated soil from radioactive components in order to ensure radiation safety is one of the urgent problems of radiochemical research. In the current work, it is planned to study the possibilities of

² Kotov, Y.B. Mikrovolnovoye izlucheniye pri krupnykh avariyakh na AES / Y.B.Kotov, T.A.Semenova // Atomnaya energiya, - 2015, i.118, v.4, - p. 219-224.

cleaning soil contaminated with uranyl nitrate and uranium isotopes along with natural radionuclides.

For this purpose, it is planned to use aqueous solutions contaminated with uranium isotopes, adsorbents used in water treatment technology (activated gravelly sand mass, coal, anthracite, cationite) as a model system. In order to study the possibility of relatively short-term and more effective treatment of soil contaminated with radionuclides, i.e. for cleaning soil samples contaminated with radionuclides, it is planned to use reagents that ensure effective dissolution of heavy metals and radionuclides (HNO₃, HCl, their mixture, NaOH), and then washing with distilled water, thus to reduce reactive consumption due to washing of treated soil with water.

In the second chapter, the methodology of the conducted experiments is described. The following are explained: During radio monitoring in the country, radiometric measurements were made with a dosimeter "Thermo Eberline R020 SI" (Thermo Electron Co.), with a gamma ray counter "PRM-470CG" (Tesla Systems Ltd.), with a radiometer-dosimeter "InSpector 1000" (Canberra Co., USA), with a alpha-spectrometer (Canberra Co., USA), with radiometer-counters for alpha, beta, gamma, neutron radiation "Radiagem 2000" (Canberra Co., USA), with a search gamma-neutron radiometer-dosimeter "ИСП-РМ1401К-01 IP65" (Polimaster-Minsk, Belarus), with a radiation 'dosimeter-isotope ionizing determiner' gamma "IdentiFINDER" (Thermo Scientific Co., AFR) (Fig. 1); The presence of alpha, beta, gamma, neutron radiation types was evaluated; The intensity of the available radiation types was determined; Analysis was carried out in a Gamma Spectrometer with a high-purity HPGe detector to determine the type and activity of radioisotopes based on calibration with certified standard solutions of radionuclides and standard point sources; analysis methodology.











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Figure 1. General view of counter-dosimeter-radiometers (Thermo Eberline R020 SI, PRM-470CG, InSpector-1000, IdentiFINDER, GR-135 Plus) and XRF-x-ray spectrometer (A), HPGe-gamma spectrometer (B), Radiagem 2000 alpha, beta, gamma, neutron radiation radiometer-counter (C), *U*CII-PM1401K-01 IP65 search radiometer-dosimeter (D), monitoring "RESA" radiometer (E), dried uranyl nitrate salt extracted from contaminated soil /nuclear material/ (F) and its spectrum taken on HPGe-Gamma spectrometer (J).

According to the comparative analysis of the spectra of the point radionuclide sources whose activities have been determined, with the gamma spectrum of the mineral of unknown composition of the same size, the same weight, which radionuclides are contained in that mineral and in what quantity were determined by comparing with the radionuclide navigator program and radiochemistry catalogs.³

During the conducted radiometric measurements and spectrometric analysis (in accordance with the law of radioactive decay with probabilistic nature and statistical characteristics), despite the fact that during the half-life the atoms of the radioactive

³ Yefimov, A.I. "Svoystva neorganicheskikh soyedineniy". A.I.Yefimov, L.P.Belorukova, I.V.Vasilkova, V.P.Chechev. // Spravochnik, - 1983, - p. 21-55.

substance decay on average by half, the difference between the calculated value and the device reading in any arbitrarily small time interval is up to 5% (+ 5%), i.e. fluctuations characteristic of natural radioactive radiation were observed.

During the radio monitoring carried out in the regions of the country, the samples taken from the upper layer of the soil, dug up to a depth of 15 cm from the surface, were analyzed using analytical chemical and physico-chemical research methods in stationary laboratory conditions. For this purpose, extraction with aqueous solutions of reagents "chemically pure for analysis", filtration, separation in centrifugation, passing through activated and ionexchange adsorbents, precipitation with a reagent, separation of mineral from solution by boiling and evaporation, quality reactions, determination of concentrations of cations and anions with standard tests on "HANNA instrument" HI 96707, HI 96708, HI 96728 spectrometers, determination of soil minerals, including heavy metals, with X-ray fluorescence spectrometers "Expert-3L" (Ukraine) and "XRF" (USA) and atomic absorption spectrometer "AA-6800" (Shimadzu, Japan), radiometric measurements for the identification of radionuclides in soil, alpha spectrometric ("Canberra", USA) and gamma spectrometric (gamma spectrometer with HPGe detector, "Canberra", USA) analysis were carried out. Model soil samples contaminated with nuclear material waste used for analysis were prepared by adding uranyl nitrate solution with predetermined activity to 200 gram portions of soil taken from the regions of the country. Tests and reagents (HANNA Instruments, HI 93728-01, HI 93707-01, HI 93708-01 /Romania-USA/, Merck kGaA /Germany/, VWR PROLABO /France/, Lachema /Czech Republic/, АО "База №1 Химреактивов" /RF/) that meet the requirements of ISO 9001 and 9002 standards were used for complex analysis of nuclear material-contaminated soil samples prepared as model samples.

Based on calibration with standard solutions of radionuclides, alpha and gamma spectrometric analyses were performed to determine the type of uranium radioisotopes and their activity during analysis. In the third chapter, the results of radiometric measurements, existing types and intensities of radiation, activity values of determined radionuclides, the regularities of adsorption of uranyl nitrate (nuclear material) solution with different amounts of adsorbents from standard solutions of different concentrations with analyses carried out in the Gamma Spectrometer with HPGe detector were interpreted. Determination of radioisotopes in soil samples contaminated with radionuclides and nuclear material and rich in natural radionuclides (by comparison with certified individual isotope samples), adsorption rates of radioisotopes separated from soil by extraction with chemical reagents from aqueous solutions with various adsorbents were shown. The types and activities of the existing radionuclides in the soil samples taken from the territory of the country were determined.

The permissible value of the average annual dose for the population in the territory of the Republic of Azerbaijan is 1 mZv, which is proportional to the absorbed dose power of 0.115 (about 0.12) μ Sv/h for people who are in constant contact with the radiation source (living in this area). The results of radiometric measurements carried out in the territories of the country show that the intensity of total radioactive radiation, that is, the power of the absorbed dose (0.03-0.12 μ Sv/hour) does not exceed the Allowable Limit/AL = 0.12 μ Sv/hour) in most areas of the country. The level of alpha radiation for soil in the regions of the country is 0-0.03 Bq_{eq}/cm². In addition, it was found that there are local areas in the country where the absorbed dose power of total radioactive radiation (0.15-3.75 μ Sv/h) is many times higher than AL, and the level of alpha radiation is 0.03-0.35 Bq_{eq}/cm².

The deactivation coefficients found after washing the soil samples containing different concentrations of natural radioactive elements with solutions of different amounts of nitric acid or sodium alkali, as well as a mixture of nitric and hydrochloric acids in distilled water, i.e. reduction of the activity of isotopes (2-20 times) in soil samples, show the possibility of deactivation of contaminated soil with chemical reagents. Spectroscopic analysis of minerals (dry residue) in the form of white salt obtained by complete evaporation of solutions in small volumes allows to determine natural radionuclides (Na²², K⁴⁰, etc.) in water and soil. Effective adsorbents need to be found for the purification of large quantities of waste water from radionuclides. While using the same type of adsorbent, uranyl nitrate decreases from the solution in inverse proportion to the increase in the amount of adsorbent. The regularities of adsorption of radioisotopes from aqueous solutions of nuclear material waste in granular activated coal, anthracite, gravelly sand, DOWEX HCR S/S cationite, expanded clay mass have been studied. The results of the studies are shown in the Tables 1-5.

Table 1.

Adsorption of 238 U and 235 U radioisotopes with specific activities of 90 and 60 Bq, respectively, on activated carbon mass from uranyl nitrate solution and the deactivation coefficient of the process (DC = Ainitial solution / Aresidual solution).

	Adsorption of uranium isotopes from an aqueous									
	solution of uranyl nitrate to the activated carbon mass									
Amount	during 1 hour and the change in the deactivation									
of	coefficie	ent of the corr	responding proces	S						
adsorben	$A_{adsorbent}(^{238}U)$			$DC(^{235}U)$						
t, g	or	$DC(^{238}U),$	$A_{adsorbent}(^{235}U),$,						
	$/A_{initial \ solution} -$	$(A_{i.s.}/A_{r.s.})$	$(A_{i.s.}$ - $A_{r.s.})$	(A _{i.s.} /						
	Aresidual solution/ Ar.s.)									
3	5	1.06	2	1.03						
10	10	1.13	7	1.13						
20	15	1.20	12	1.25						
50	25	25 1.38 22 1.58								
100	45	2.00	38	2.73						

Trace amounts of U²³⁴, U²³⁶, U²³⁷ isotopes in uranyl nitrate solution are completely absorbed by small amounts of activated

carbon mass, 50% of U²³⁸ isotope in uranyl nitrate solution in 100 g of activated carbon mass ($A_{ads.} \times 100\%$ / $A_{i.s.} = 45$ Bq $\times 100\%$ / 90 Bq = 50%), and 63% of the U²³⁵ isotope ($A_{ads.} \times 100\%$ / $A_{i.s.} = 38$ Bq $\times 100\%$ / 60 Bq= 63%) adsorb.

Table 2.

Adsorption of 238 U and 235 U radioisotopes with specific activities of 90 and 60 Bq, respectively, from uranyl nitrate solution on granular anthracite mass and the deactivation coefficient of the process (DC = Ainitial solution / Aresidual solution).

Amount of	The change in the deactivation coefficient								
adsorbent, g	corresponding to the adsorption of uranium								
	isotopes from a	isotopes from an aqueous solution of uranyl nitrate							
	to the granu	to the granular anthracite mass within 1 hour							
	$A(^{238}U)$ $DC(^{238}U)$ $A(^{235}U)$ $DC(^{235}U)$								
3	2	1.02	2	1.03					
10	5	1.06	5	1.09					
20	8	1.10	8	1.15					
50	18	1.25	13	1.27					
100	30	1.5	19	1.46					

Table 3.

Adsorption of 238 U and 235 U radioisotopes with specific activities of 90 and 60 Bq, respectively, from uranyl nitrate solution on gravelly sand mass and the deactivation coefficient of the process (DC = Ainitial solution / Aresidual solution).

Amount	The change in the deactivation coefficient corresponding to the									
of	adsorption of uranium isotopes from an aqueous solution of uranyl									
adsorbe	nitrate to the gravelly sand mass within 1 hour									
nt, g	$A(^{238}U)$	$A(^{238}U)$ $DC(^{238}U)$ $A(^{235}U)$ $DC(^{235}U)$								
3	5	1.06	3	1.05						
10	7	1.08	7	1.13						
20	13	1.20	12	1.25						
50	25	1.38	20	1.50						
100	37	1.70	24	1.70						

Trace amounts of U^{234} , U^{236} , U^{237} isotopes in uranyl nitrate solution are completely absorbed by small amounts of anthracite mass, 33% of U^{238} and U^{235} isotopes in uranyl nitrate solution are adsorbed in 100 g of anthracite mass.

Trace amounts of U^{234} , U^{236} , U^{237} isotopes in uranyl nitrate solution are completely absorbed by small amounts of gravelly sand mass, 41% of U^{238} and U^{235} isotopes in uranyl nitrate solution are adsorbed in 100 g of gravelly sand mass.

Table 4.

Adsorption of 238 U and 235 U radioisotopes with specific activities of 90 and 60 Bq, respectively, from uranyl nitrate solution on DOWEX HCR S/S cationite and the deactivation coefficient of the process (DC = Ainitial solution / Aresidual solution).

	The change in the deactivation coefficient									
	corresponding to the adsorption of uranium									
Amount of	isotopes from an aqueous solution of uranyl nitrate									
adsorbent, g	to the granular DOWEX HCR S/S cationic mass									
	within 1 hour									
	$A(^{238}U)$ $DC(^{238}U)$ $A(^{235}U)$ $DC(^{235}U)$									
3	5	1.06	2	1.03						
10	15	1.20	7	1.13						
20	25	1.38	15	1.33						
50	48 2.14 30 2.00									
100	64	3.46	44	3.75						

Trace amounts of U^{234} , U^{236} , U^{237} isotopes in uranyl nitrate solution are completely absorbed by small amounts of DOWEX HCR S/S cationite mass, 72% of U^{238} isotope and 75% of U^{235} isotope in uranyl nitrate solution are adsorbed in 100 g of DOWEX HCR S/S cationite mass.

Among the large-scale production areas operating in the country are brick, glass, glass containers, cement, bentonite, aluminum production, metallurgy, chemistry, petrochemical, etc. industries have a special weight. Among the products and wastes of these production areas, the substance with the highest adsorption property is "high-temperature baked porous clay residue scraps" – expanded clay mass, accumulated in the surrounding areas as a waste of brick production plants operating in most regions of the country. Porous expanded clay mass processed at high temperatures, typical for the brick "baking" industry with a large reserve of raw materials, was used as an adsorbent in our experiments.

Table 5.

Adsorption of 238 U and 235 U radioisotopes with specific activities of 90 and 60 Bq, respectively, from uranyl nitrate solution on expanded clay mass and the deactivation coefficient of the process (DC = Ainitial solution / Aresidual solution).

Amount of	The change in the deactivation coefficient										
adsorbent, g	corresponding to the adsorption of uranium										
	isotopes from	isotopes from an aqueous solution of uranyl nitrate									
	to the e	to the expanded clay mass within 1 hour									
	$A(^{238}U)$	A $\binom{238}{238}$ DC $\binom{238}{235}$ DC $\binom{235}{235}$ DC $\binom{235}{235}$									
	11(0)	20(0)	11(0)	20(0)							
3	4	1.05	2	1.03							
10	8	1.10	6	1.10							
20	12 1.15 10 1.20										
50	23	23 1.34 20 1.50									
100	41	1.84	30	2.00							

Trace amounts of U^{234} , U^{236} , U^{237} isotopes in the uranyl nitrate solution are completely absorbed by small amounts of expanded clay, 47% of the U^{238} isotope and 50% of the U^{235} isotope in the uranyl nitrate solution are adsorbed in 100 g of expanded clay. Among the mentioned adsorbents, the samples of granular DOWEX HCR S/S cationite (72-75%), activated carbon (50-65%) and expanded clay (47-50%) have the highest

adsorption properties while gravelly sand (41%) and granular anthracite (33%) have the weakest adsorption properties.

Although the deactivation coefficient (1.05-3.75) of the cleaning processes carried out with the application of the studied adsorbents are smaller than the deactivation coefficients of the existing cleaning methods (DC > 4) shown in the scientific literature, it can be used as a cleaning method for the adaptation of the physical purification stage of oil to the safety standards of waste water by using expanded clay which has large natural raw material reserves in our country.

Table 6.

Adsorption of ²³⁸U isotopes from uranyl nitrate solutions on studied 100 g adsorbents at different temperatures.

Type of	Partial	Value of							
adsorbent	on t	activation							
	pro	proportion to the temperature increase,							
	-	-	Bq/100 g	g.		desorption of			
						²³⁸ U isotope			
	298		323 K	3	43 K	from			
	Κ					adsorbents,			
						E _a (kcal/mol).			
	A298	D ₃₂	ln(D ₃₂₃ /A	D343	ln(D ₃₄₃ /				
		3	298)		A298)				
DOWEX	64	18	-1.269	32	-0.693	26.5			
HCR S/S									
cationite									
Activated coal	45	13	-1.242	22	-0.716	24.2			
Expanded	41	12 -1.229		20	-0.718	23.5			
clay									
Gravelly sand	37	11	-1.213	18	-0.721	22.6			
Anthracite	30	9	-1.204	14	-0.762	20.3			

In the "value of activation energy" column of the Table 6, the values of the activation energy of the desorption process of 238 U isotopes from the adsorbents used in the research are shown, calculated on the basis of the graphical dependences in the Figure 2.



Figure 2. Proportional desorption of 238 U isotopes adsorbed to adsorbents (100 g) from uranyl nitrate solutions at 298K temperature.

$$-E_{a}/R = [\ln(D_{343}/A_{298}) - \ln(D_{343}/A_{298})]/[(1000/T1) - (1000/T2)] (1)$$

$$-E_{a}/R = [\ln(18/64) - \ln(32/64)]/[(1000/323) - (1000/343)] =$$

$$= -1.269 - (-0.693)/(3.096 - 2.915) = -0.576/0.181 = -3.18;$$

$$-E_{a}/R = -3.18; E_{a} = 3.18 \times 8.3144 = 26.5 \text{ kC/mol}.$$

After performing calculations with the same algorithm for other adsorbents, it is shown that the value of (E_a/R) and the value of activation energy (E_a) for all adsorbents used in the experiments varies in the range of 2.44-3.19 and 20.3-26.5 kcal/mol respectively (Table 6).

These values confirm that the adsorption process is a weak exothermic process, while the desorption process is a weak endothermic process. The values of the activation energy determined for the desorption process show that adsorption has a physical nature (physical sorption) in all 5 types of adsorbents used in the experiments, and only weak Van der Waals forces exist between the absorbed uranium isotopes and the adsorbent.

The fourth chapter is devoted to the development of effective deactivation processes for soil samples contaminated with nuclear material waste. Model samples of soil contaminated with nuclear material waste (uranyl nitrate) were developed and separation of radionuclides and uranium isotopes from soil with reagents was studied in order to develop effective deactivation processes of those soil samples. The effectiveness of the proposed method of cleaning (deactivation) of soil samples contaminated with nuclear material waste (corresponding values of the deactivation coefficient) was evaluated and compared with the effectiveness of similar processes (prototypes) described in the scientific literature for the treatment of soil contaminated with nuclear waste.⁴

During the experiments, "chemically pure for analysis" reagents (Merck kGaA /Germany/, VWR PROLABO /France/, Lachema /Czech Republic/, AO "База №1 Химреактивов" /RF/) were used. For the analysis, the following solutions were prepared in bidistilled water obtained from the GFL-2304 bidistiller: in density 1.4 g/cm³ at 20°C, 0.2 M, 0.5 M, 1.0 M and 2.0 M solutions of 67% nitric acid and crystalline granular sodium hydroxide, and in density 1.19 g/cm³ at 20°C, 0.4 M, 1.0 M, 2.0 M and 4.0 M solutions of 38% hydrochloric acid. Which isotopes are present in the uranyl nitrate salt and in what concentration were determined by gamma spectrometry with HPGe detector.

The soil samples taken during the radio monitoring carried out in the regions of the country were divided into equal parts weighing 200 grams. These soil samples were mixed with uranyl nitrate solution in order to obtain model samples contaminated with nuclear material waste. The activity of ²³⁸U, ²³⁵U, ^{234,236}U isotopes in the uranyl nitrate salt solution soaked in soil samples

⁴ Dmitriyev, S.A. Sposob reagentnoy ochistki gruntov ot radionuklidov tseziya / Patent RU 2361301 C1. – 10.07.2009, Bul. №19 / Prozorov L., Kuptsov V.M.

weighing 200 g was determined to be in the ratio of 1200:400:20. Nitric acid, hydrochloric acid, sodium hydroxide solutions and distilled water were used to separate radioisotopes from soil samples.

Since radioactive substances, including the elements that form the basis of nuclear materials, have metallic properties, it is expected that they react with solutions of nitric and hydrochloric acids of various concentrations, dissolve in these acid solutions or their mixtures, like other non-radioactive, stable metallic elements. For this reason, in our experiments, extraction for radionuclides was carried out from 0.2 M, 0.5 M, 1.0 M and 2.0 M concentrated solutions of nitric acid, then re-extraction was carried out with aqueous solutions of 0.2 M, 0.5 M, 1.0 M and 2.0 M concentrated sodium hydroxide in the last stage of the experiments in order to restore the neutral reaction property of the extracted soil sample residues. For more effective extraction of radionuclides, we also used aqueous solutions of nitric and hydrochloric acids in a ratio of 1:2. Taking into account that the radioactive substance dissolved in the reaction with chemical reagent solutions did not completely pass into the solution during filtration after extraction, and a part of it remained in the residual soil, after extraction with the reagent, washing the soil residue with 1 liter of distilled water, gamma-spectroscopic analyses of the soil residue and the obtained extract were performed.

The regularities of the 2-stage deactivation processes of the soil samples contaminated with uranyl nitrate and with the activity of ²³⁸U, ²³⁵U, ^{234,236}U isotopes in the ratio 1200:400:20 with aqueous solutions of nitric acid are shown in the Figure 3/1st and 2nd stage/.



I stage

II stage

Figure 3. Reduction of 238 U isotope content in soil after extraction (1 h) of uranyl nitrate-contaminated soil samples (200 g) with nitric acid solution (1 h) followed by washing with 1 l of distilled water (1 h).

It is possible to reduce the amount of uranium isotopes in those soil up to 20 times by the 2-stage deactivation processes of soil samples (200 g) with the 1200 Bq activity of 238 U isotopes in their contents with aqueous solutions of nitric acid.

As a model sample, the regularities of the 2-stage deactivation processes of the soil samples (200 g.) contaminated with uranyl nitrate and containing the activity (activity concentration) of ²³⁸U, ²³⁵U, ^{234,236}U isotopes in the ratio of 1200:400:20 with aqueous solutions of sodium alkali have been presented in the following pictures (Fig. 4/1st and 2nd stage/).



I stage

II stage

Figure 4. Reduction of 238 U isotope content in soil after extraction (1 h) of uranyl nitrate-contaminated soil samples (200 g) with sodium alkali solution (1 h) followed by washing with 1 l of distilled water (1 h).

It is possible to reduce the amount of uranium isotopes in those soil up to 15 times with the 2-stage deactivation processes of soil samples (200 g) contaminated with uranyl nitrate and containing 1200 Bq of activity (activity concentration) of ²³⁸U isotopes with aqueous solutions of sodium alkali.

The regularities of the 2-stage deactivation processes of soil samples (200 g.) contaminated with uranyl nitrate and containing ²³⁸U, ²³⁵U, ^{234,236}U isotope activity (activity concentration) in the ratio of 1200:400:20 with a mixture of nitrate and hydrochloric acid solutions have been presented in the following pictures (Fig. 7/1st and 2nd stage/).



I stage

II stage

Figure 5. Reduction of 238 U isotope content in soil as a result of extraction (1 hour) of uranyl nitrate-contaminated soil samples (200 g) with a mixture of nitrate and hydrochloric acid solutions (1 hour) followed by washing with 1 l of distilled water (1 hour).

It is possible to reduce the amount of uranium isotopes in those soil up to 2000 times with the 2-stage deactivation processes of soil samples (200 g) contaminated with uranyl nitrate and containing 1200 Bq of activity (activity concentration) of 238 U isotopes with a mixture of nitrate and hydrochloric acid solutions (1 hour) and then with 1 l of distilled water (1 hour).

From the obtained regularities, it can be seen that when a reagent (HNO₃, HCl or NaOH) or a mixture (HNO₃-HCl) is used for the extraction of contaminated soil, the amount of radionuclides decreases in the soil, inversely proportional to the increase in the amount or concentration of the reagent, as well as the increase in the amount of water which the soil is subsequently washed with. A 2-step deactivation process of soil contaminated with uranyl nitrate wastes with a mixture of nitrate and hydrochloric acid solutions followed by distilled water (or an aqueous solution of sodium

alkali) was determined to be a more effective cleaning method, compared to deactivation with aqueous solutions of nitric acid, hydrochloric acid, or sodium alkali alone, as well as compared to all existing prototypes. With this method, it is possible to completely deactivate soil contaminated with nuclear material waste.

As a model sample, comparative analyses of the efficiency indicators of 2-stage deactivation processes of soil samples (200 g.) contaminated with uranyl nitrate and containing ²³⁸U, ²³⁵U, ^{234,236}U isotope activity in the ratio of 1200:400:20 with a mixture of nitrate and hydrochloric acid solutions and then with distilled water have been presented in the Table 7.

Table 7.

Results of deactivation of 200-gram soil samples contaminated with uranyl nitrate (specific activity of ²³⁸U, ²³⁵U and ^{234,236}U radioisotopes with 1200, 400 and 20 Bq, respectively) with a mixture solution of nitric and hydrochloric acids.

n, mol		I stage			II stage			Other II stage				
(n	Soil	sample	is extra	icted	Soil	Soil residue is extracted with		Soil residue is extracted with				
HNO ₃	wit	th nHNC)3+2nH	ICl	nHNO ₃ + +2nHCl solution for			nHNO3++2nHCl solution for				
+2n	solutio	on for 1	hour ar	nd soil	1 hour and final soil residue is			1 hour and final soil residue is				
HCl	residue is washed with 1		washed with 1 liter of distilled			extracted with nNaOH (M)						
in 1	liter o	f distille	d wate	r for 1	water for 1 hour.			solution for 1 hour.				
liter		hou	ır.									
of water)	A_1 238U,	A ₁ ²³⁵ U,	A ₁ 234,2	DC_1	A ₂ ²³⁸ U,	A_2 235U,	A ₂ 234,236	DC_2	A2' 238	$A_2'^{235}$ U,	A2' 234,236	DC2'
,	Bq	Bq	³⁶ U		Bq	Bq	U,		U,	Bq	U,Bq	
			,Bq				Bq		Bq			
0.2	650			1.85	21			57	17			70
		210		1.9		7		57		5		80
			12	1.7			0.3	70			0.2	100
0.5	370			3.3	14			85	11			110
		100		4.0		4		100		3		133
			7	2.9			0.2	100			0.1	200
1.0	130			9.2	6			200	4			300
		30		13		1.4		333		1		400
			3	6.7			0.05	400			0.03	667
2.0	30			40	1.1			1100	1			1200
		10		40		0.3		1330		0.3		1330
			1	20			0.01	2000			0.01	2000

Notes: A_i – specific activity of U isotopes in the residues of the soil sample after the processing stages;

 DC_i – deactivation coefficient of the soil sample after the processing stages (the ratio of the specific activity of the initial soil sample to the specific activity of the soil residue after the processing stage).

As a result of the conducted studies, the 2-stage deactivation process of soil contaminated with nuclear material waste with a mixture of nitrate and hydrochloric acid solutions and then with distilled water (or with an aqueous solution of sodium alkali) was determined to have a greater deactivation coefficient compared to deactivation processes with various reagents (aqueous solutions of nitric acid, hydrochloric acid, sodium alkali with different concentrations) and all other existing prototypes.

Chapter 1 of the dissertation describes a 3-stage deactivation method with the largest deactivation coefficient (DC=830) and high efficiency among the cleaning methods of soil samples contaminated with ¹³⁷Cs isotopes published in the scientific literature. Aqueous solutions of sulfuric and phosphoric acids were used for relaying the process. In order to reduce radiation activity (299 kBq/kg) from soil samples contaminated with ¹³⁷Cs isotopes by 830 times (DC=830), in the 1st stage, 60 grams of soil mass was extracted with 2M H₂SO₄ and 0.5M H₃PO₄ aqueous solutions heated to 90°C for 7 hours, after the 1st stage, radiation activity from contaminated soil decreased 16.3 times (DC₁=16.3). In the 2nd stage, as a result of separating the soil into sandy and fine dispersed (clay) fractions by water-gravity separation, the second (fine dispersed clay) fraction, which is 3.8% of the total soil mass and is rich in isotopes, was sent directly to the nuclear waste cemetery. As a result, the value of the deactivation coefficient of the 2nd stage increased to 25.7. In the 3rd stage, after the extraction of the residual sandy fraction of the soil in a 10-section percolation unit at a temperature of 90°C with 2M H₂SO₄ solution for 24 hours, it was determined that the radiation activity from the residual soil decreased by 830 times.

The method of deactivation of soil samples contaminated with nuclear waste (uranyl nitrate) with chemical reagents (nitric and hydrochloric acids and sodium alkali solutions) is characterized by a higher deactivation coefficient (DCi=1200-2000). In the 1st stage of the process, 200 grams of soil mass was extracted with a mixture of 2M HNO₃ and 4M HC1 aqueous solutions at room temperature for 1 hour (the solution was filtered), then washed and filtered with 1 liter of distilled water for 1 hour, and the residual soil was analyzed. In 2nd step, the residual soil was extracted (the solution was filtered through a filter) with the same amount of mixed solution (a mixture of 2M HNO₃ and 4M HC1 aqueous solutions) at room temperature for 1 hour, then the residual soil was washed with 2M NaOH solution for 1 hour. 238U, 235U, 234,236U isotopes contained in the uranyl nitrate salt (in the residue of the separated alkaline solution) dissolved in aqueous solution residue of 2M HNO₃ and 4M HC1 acids collected after soil extraction in both stages were filtered and collected in the mixture residue (in the water residue separated after washing the residual soil with 1 liter of distilled water in the 1st stage and in the alkaline solution residue filtered and separated after washing the residual soil with 2M NaOH solution in the 2nd stage), are converted into minerals and sent to the radioactive waste repository by complete evaporation of the liquid phase or by adding 10 ml of potassium ferrocyanide solution with a concentration of 10^{-3} M to the solutions.

For comparative analysis, information on the manufacturers and wholesale prices of the reactants used for the deactivation process developed by us with the most effective deactivation method was collected and used in the calculations for the feasibility studies of the processes.^{5,66}

⁵ Jafarli H. A. Management analysis. Textbook. / H.A.Jafarli - Baku: "University of Economics" Publishing House, - 2018. - p. 188-219.

⁶ Zhou, Y. A basic study of optimal investment of power sources considering environmental measures: Economic evaluation of CCS through a real options approach. / Y. Zhou, M. Kato // Electrical Engineering in Japan. - 2010, v. 174, Issue 3, - p.9-17, February 2011, First published: 29 November 2010, https://doi.org/10.1002/eej.21065.

$$S = \tau + E + R + \Delta s \tag{1}$$

Here, S – total amount of the financial resources (currency) spent on all deactivation works and necessary to ensure radiation safety (in RF Rubles or AZN);

 τ – currency equivalent of the working hours spent on deactivation (in RF Rubles or AZN);

E – currency equivalent of the energy spent on deactivation, earth excavation and transportation works (in RF Rubles or AZN);

R – value of the reagents used for deactivation in currency equivalent (in RF Rubles or AZN);

 Δs – the value of the cost of storage of the radioactive substance released from the soil as a result of deactivation or the soil fraction enriched with radionuclides in nuclear waste storage facilities ("cemetery") in currency equivalent (in RF Rubles or AZN).

If using the most effective deactivation method available, 2,000 tons = (20×100) tons or 1 ha = 10,000 m² of soil contaminated with ¹³⁷Cs isotopes are deactivated up to 830 times at a depth of 15 cm, and the separated ¹³⁷Cs isotopes, as well as the 3.8% soil fraction rich in radionuclides are stored in the nuclear waste storage for 1 year: then S = τ + E + R + Δ s = 51000 + 6600000 + 7666700 + 1300 = 14 319 000 AZN or 954 600 000 RF Ruble currency equivalent financial expenditure is required.

If using the developed new deactivation method, the contaminated top layer of 2000 tons = (20×100) tons or 1 ha = 10000 m² soil area contaminated with nuclear material waste are deactivated completely (up to 1200 times) at a depth of 15 cm and the fraction of separated uranium isotopes is stored in nuclear waste storage for 1 year: then S = τ + E + R + Δ s = 400000 + 266700 + 164000000 + 8670 = 164 675 370 RF Rubles or 2 470 131 AZN currency equivalent financial expenditure is required.

The currency equivalent of 164675370 RF Rubles (2470131 AZN) financial expenditure is required for the complete (up to 1200 times) deactivation of top layer of 2000 tons or 1 h=10000 m² soil area contaminated with radioactive material waste at a depth of 15 cm with the new deactivation method described in the dissertation. According to the scientific literature, for the deactivation of the soil

of the same area up to 830 times with the most effective deactivation method available, financial expenditure is required in the currency equivalent of 954600000 RF Rubles (14319000 AZN).

The developed new deactivation method is more effective and profitable than the existing most effective deactivation method (954 600 000 RF Rubles / 164 675 370 RF Rubles) = (14 319 000 AZN / 2 470 131 AZN) = 5.8 times more effective and profitable.

Only 2.47 million AZN, 247 million AZN and 2 470 million AZN of national currency are required for the complete deactivation (1200-2000 times) of land areas contaminated with nuclear material waste, in 1 ha, 100 ha and 1000 ha, respectively.

Taking into account the contamination of the top 5 cm thick layer of the soil in the surrounding areas during accidents in nuclear reactors and as a result of rains⁷, the above-mentioned process will require less financial expenditure.

Since the financial resources spent on the first batch of the contaminated soil deactivated with a mixture of nitrate and hydrochloric acids according to the production volume are at least 100 times more than the financial resources spent on the next soil batches to be cleaned, that is, the extraction of the next soil mass to be cleaned with the residue of the acid mixture left after the initial extraction (provided that only 1% of the initial clean solution is added), taking into account the last 2 arguments mentioned (the top layer of the soil is cleaned not 15 cm, but 5 cm thick, and the residual acid mixture solution separated from the previously cleaned soil mass is added to the next cleaned soil batches), - Deactivation of 100-300 times larger land area is possible with the financial expenditure of AZN 2.47 billion. This shows that even 20% of the country's territory is polluted, with 14.3 billion AZN funds, it is possible to fully restore those lands.

⁷ Bondarkov, D.M. Izucheniye Povedeniya Radionuklidov na Silnozagryaznennykh Poligonakh 5-Kilometrovoy Zony CHAEC / D.M.Bondarkov, I.N.Vishnevskiy, V.A.Zheltonozhskiy [et al.] // Yaderna Fizika Ta Yenergetika, – 2016, i. 17, № 4, – p.383.

MAIN CONCLUSIONS:

1. The results of radiometric measurements carried out in the territories of the country show that the intensity of total radioactive radiation (0.03-0.12 μ Sv/h) does not exceed the allowed limit AL = 0.12 μ Sv/h in most areas of the country. The level of alpha radiation for the soil of the regions of the country is 0-0.01 Bq_{eq}/cm². However, local areas have also founds where the intensity of total radioactive radiation (0.15-3.75 μ Sv/h) is many times higher than AL, and the level of alpha radiation is 0.03-0.35 Bq_{eq}/cm² were also found.

2. The determined values of deactivation coefficients found after washing the soil samples containing different concentrations of natural radioactive elements with solutions of different amounts of nitric acid or sodium alkali, as well as a mixture of nitric and hydrochloric acids in distilled water, i.e. reduction of the activities of isotopes in the soil samples by 2-20 times shows the possibility of deactivation with chemical reagents.

3. The study of the regularities of adsorption of isotopes from aqueous extracts of nuclear material waste on adsorbents shows that granular DOWEX HCR S/S cationite (72-75%), activated carbon (50-65%) and expanded clay (47-50%) samples have the high adsorption properties, while gravelly sand (41%)) and granular anthracite (33%) samples have relatively weak adsorption properties. Despite the fact that the deactivation coefficient of the cleaning processes carried out by applying the studied adsorbents is smaller than the deactivation coefficients of the existing cleaning methods shown in the scientific literature, expanded clay, which has large natural raw material resources in our country, can be used to adapt the wastewater of physical purification stage of oil to the safety standards.

4. It was determined that the values of activation energy of the process of thermal desorption of uranium isotopes from the studied adsorbents varies in the range of 20.3-26.5 kcal/mol. The values of the activation energy determined for the desorption process show that the adsorption process on the adsorbents used in the experiments has a physical nature (physical sorption), and only weak Van der Waals forces exist between the absorbed uranium isotopes and the adsorbent.

5.It has been determined that the concentration of uranyl nitrate in soil samples decreases in inverse proportion to the increase in the amount or concentration of the reagent or the amount of water that is used to wash the soil when using individual reagents (HNO3, HCl or NaOH) or their mixtures. I have been determinated also the contaminated with nuclear material waste soils 2-stage deactivation process with a mixture of nitrate and hydrochloric acid solutions and then with distilled water (or with an aqueous solution of sodium alkali) to has a higher deactivation coefficient value (DC=1200-2000) than this value for deactivation processes with various reagents (aqueous solutions of nitric acid, hydrochloric acid, sodium alkali with different concentrations) and other prototypes using solutions of sulfuric and phosphoric acids for deactivation.

6. It was determined on the basis of the feasibility studies, that the 2-stage deactivation process with a mixture of nitrate (2 M) and chloride (4 M) acid solutions and then with an aqueous solution of sodium alkali (2 M) requires only 2.47 billion AZN for complete (1200-2000 times) deactivation of 1000 ha of soil contaminated with nuclear material waste. The developed new deactivation method is 5.8 times more effective and profitable than the existing most effective deactivation method (prototype). Taking into account that after the accidents at nuclear reactors and the subsequent rains, the upper 5 cm thick layer of the soil on the surrounding areas is contaminated with nuclear wastes and due to the return of the treated acid mixture solution to the next batches of soil is required up to soo times less reagent consumption than for imitial batch of cleaned soil, the developed deactivation process will require less financial expenditure.

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