

PROCESSING METHOD FOR RECYCLED URANIUM-CONTAINING WATER FROM RADIOACTIVELY CONTAMINATED SOILS TREATED BY HYDROSEPARATION

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The paper focuses on a method used to process water from the treatment of uranium-contaminated soil resulting from the operation of JSC AECC sublimation facilities by hydroseparation, in particular, on its development and testing results. During the tests, the feasibility of recycled water treatment was demonstrated both suggesting the use of membrane and ion exchange methods with process parameters of the treatment stages identified. The study shows the possibility of producing uranium-containing materials in the form of ammonium polyuranates as secondary products of recycled water processing. A schematic diagram of recycled water purification following hydroseparation treatment of soil excluding the generation of radioactive waste was developed based on the research.

Keywords: *radioactively contaminated soil, radioactive waste, hydroseparation, reverse osmosis, absorption, prioranet of ammonium, laboratory bench.*

Nuclear decommissioning, as well as remediation of contaminated areas (RCA) are considered as quite topical tasks to accomplish. In 2019, the fuel company JSC TVEL was appointed by the State Atomic Energy Corporation Rosatom as an integrator in the field of nuclear decommissioning, including the operations dealing with reactor installations of nuclear power plants and radioactive waste (RW) management. Testing of the developed decommissioning methods, as well as of those providing the decontamination of radioactively contaminated soil (RCS) with its significant amounts being generated during nuclear decommissioning and RCA cleanup operations has been actively implemented at the sites of enterprises pertaining to the fuel division. Practical experience shows that the level of radioactive contamination varies for the excavated soil which can be partially categorized as radioactive waste and partially as industrial waste with its

content of radionuclides being below the level allowing waste categorization as radioactive. In this regard, the developed RCS decontamination methods are aimed at reducing RW amounts and RCS release from radiation control.

JSC VNIINM has developed an installation designed for RCS decontamination based on the hydro-separation method [1]. This method has been tested on the soils of AECC's sublimate production (SP) contaminated with uranium isotopes and categorized both as industrial waste and very low-level radioactive waste (VLLW) [2, 3].

The decontamination process based on the hydro-separation method, water-gravitational separation of soil fractions by size, implies multiple use of aqueous media being considered as recyclable [3]. During its testing that involved some contaminated soil categorized as VLLW [3], the specific activity of uranium isotopes in the circulating water was

growing gradually. When the mass ratio between the treated soil and the circulating water became equal to 1, the latter one was removed from the hydro-separation cycle. By the content of uranium radionuclides, it corresponded to the category of liquid radioactive waste. Thus, it became quite obvious that minimization of contaminated aqueous media inventory should be achieved based on RCS decontamination method. Therefore, development of a processing method for spent solutions resulted from RCS cleanup seems to be of a great practical importance.

In practice, ion-exchange desalination and sorption methods involving selective extraction of pollutants are used to treat the waste present in the form of aqueous saline solutions and LRW. Generic methods such as membrane desalination, distillation and their combinations are applied in case of highly polluted waters with complex multicomponent salt composition [4, 5].

Water purification and uranium concentration methods both by reverse osmosis and sorption extraction of uranium on a strongly basic anion-exchange resin with subsequent precipitation from the concentrates in the form of ammonium polyuranate (PUA) were proposed to process uranium-containing recycled water from RCS decontamination by hydro-separation.

This article overviews the development and laboratory testing of a processing technology for recycled water resulting in some treated water complying with the standards established for the discharges into centralized water disposal systems and some secondary waste not belonging to the RW category.

Experimental part

The study investigates a batch of recycled water resulted from the treatment of AECC soil belonging to VLLW at a laboratory bench. The spent water held for a long time in storage tanks was an aqueous solution with some sediment at its bottom. Its separation by settling resulted in a solid sediment, which was a finely dispersed fraction of soil with the specific activity of uranium isotopes (234, 235, 238) being equal to 64 Bq/g, and a clarified aqueous solution seen as the main object of further research. The specific activity of uranium isotopes (234, 235, 238) in the clarified solution was equal to 6 Bq/g. The chemical composition of salts in the clarified aqueous solution was as follows (wt. % of the total salt content equal to 1,100 mg/dm³): HCO₃⁻ — 35.7; Ca²⁺ — 15.2; NO₃⁻ — 12.5; SO₄²⁻ — 6.4; Na⁺ — 3.2; Mg²⁺ — 2.4; K⁺ — 0.8; uranium — 23.5; other — 0.3. Table 1 presents the assessment results for the

spent water sample, the discharge standards in accordance with [6–9] and the purification factors. The required and achieved purification factors for various indicators (K_{pur}) were set as a ratio between the content of components in the initial solution, the discharge rates and their content in the treated water, respectively.

The main processing stages were tested using the equipment of a VNIINM's purification bench for high-salt solutions [5] that involved a pre-treatment unit to remove some mechanical impurities, two stages of a reverse osmosis-based purification unit and another one designed based on ion exchange method.

Pre-treatment allowing to remove some mechanical impurities was performed at a cascade involving two sand filters connected in series to the circuit. Quartz sand with a grain size of 0.8–2 mm and 0.2–0.7 mm, respectively, was used as a resin charge. The sand filters were washed before and after spent water batch filtration was done. The filtrate was supplied to the tank of the high-pressure reverse osmosis unit or to the column of the ion exchange unit. The data considering each processing option were compared to identify the most effective indicators.

Water treatment and concentration of impurities by reverse osmosis was done in two stages. At the first stage, a circulation loop was set through the membrane element, on which desalination and concentration of the main contaminating components took place. pH level of the solution was adjusted to 4–5 pH. The concentrate was supplied back to the tank of the first stage unit which accounted for the concentration mode. Dissolved contaminating components got accumulated in the concentrate of the circulation tank. After passing through the membrane, the water (permeate) entered the second-stage tank for subsequent post-treatment. The working pressure above the membrane was maintained at a level of no less than 4.5 MPa providing high permeate performance, the highest degree of purification and maximum concentration.

The second stage of reverse osmosis has been run similarly to the first one. The hydrogen index of the averaged permeate after the first stage was adjusted from 4 to 6–7 pH. The working pressure above the membrane amounted to 1.0 MPa. Permeate from the second stage was transferred to a container filled with clean water.

During the treatment implemented within the units of the first and second stages of reverse osmosis, the concentrate and permeate samples with different concentration degrees were monitored: the pH was measured with pH-420 pH-meter, specific electrical conductivity was monitored using

Table 1. Results of a study focused on chemical and radionuclide composition of the initial recycled water and water resulted from the purification following two stages of reverse osmosis

Indicator, unit	Value of the indicator		Nominal error, %	Discharge standards	Purification factor (K_{pur})	
	Spent water	Treated water			Required	Achieved
Sample pH, pH units	8.0±0.2	6.5±0.2	–	6.5–8.5	–	–
Total iron, mg/dm ³	1.2	–	15	1.5	–	–
Manganese, mg/dm ³	1.1	0.007	30	0.074	15	157
Copper, mg/dm ³	0.6	0.007	32	0.018	34	85.7
Zinc, mg/dm ³	0.04	0.013	30	0.014	3	3.1
Nickel, mg/dm ³	0.05	0.007	32	0.013	4	7.1
Sodium, mg/dm ³	36	2.4	15	–	–	15
Potassium, mg/dm ³	8.4	<1	16	–	–	>8.4
Calcium, mg/dm ³	170	1.3	16	–	–	131
Magnesium, mg/dm ³	27	0.6	15	–	–	45
Sulfates, mg/dm ³	72	1.5	25	69.5	1.1	48
Nitrates, mg/dm ³	140	4.2	15	40.7	3.4	34
Hydrocarbonates, mg/dm ³	400	10	12	–	–	40
Phenol, mg/dm ³	0.02	<0.01	–	0.25	–	2
Dry residue	850	<50	9	3,000 [6]	–	60
Thorium-234, Bq/kg	1,290	<0.021	–	400	3.3	>6·10 ⁴
Uranium-234, Bq/kg	2,650	<0.032	–	28	95	>8·10 ⁴
Uranium-235, Bq/kg	142	<0.023	–	29	4.9	>6·10 ³
Uranium-238, Bq/kg	3,100	<0.036	–	30	103.3	>8·10 ⁴

MARK-603/1 conductometer with the specific activity of radionuclides calculated as well. The pH level was adjusted with sodium hydroxide and nitric acid.

Concentrate from the first stage of reverse osmosis was decarbonized while maintaining constant stirring of the solution for 2–3 hours providing a pH level equal to 3. Concentrate decarbonization stage of the reverse osmosis was deemed necessary since carbonate and bicarbonate ions preventing quantitative release of uranium into the precipitate were found in the system. Preliminary tests have shown that the precipitation mother liquors resulted from the acidification of the concentrate with sulfuric acid to pH=3 provided more complete extraction of uranium as compared to the application of nitric acid.

To treat the clarified spent water from uranium by the ion exchange method, a 50 cm³ column filled with anion-exchange resin of AV-17-8 brand in a Cl-form with a layer thickness of 22.4 cm was used. The feed was supplied at a rate of 5 column volumes per hour assuming a downward flow pattern. The filtrate was collected in a container and the specific activity of uranium was measured. Whenever the specific activity of uranium in the filtrate sample was found to be higher than the one equivalent to

the uranium content of 1 mg/dm³, filtration was stopped. The spent resin was washed with demineralized water and uranium was desorbed with {0.9 M NH₄NO₃ + 0.1 M H₂SO₄} solution according to an ascending flow pattern at a rate of 5 column volumes per hour. Desorbate was collected portion-wise and put into individual containers. According to the specific activity measurements, portions of the desorbate with the highest uranium content were combined and sent to the precipitation stage in the form of ammonium polyuranate. The resin was converted into a usable form by its washing with water and a 5 % NaCl solution.

Direct precipitation method with a 25 % aqueous solution of ammonia was used to extract uranium from concentrates in the form of ammonium polyuranate until a pH level of 9.5 units was reached assuming continuous stirring of the resulting slurry. Part of the solution clarified by settling was supplied into the mother liquor collection container. The pulp was fed to a nutsch filter. A "white tape" paper filter was used as a filtering material, the mother liquor was supplied to a collector tank, the wet precipitate of ammonium polyuranate was unloaded manually and sent to an air-drying unit providing its constant weight.

Results and discussion

The wash waters from the coarse mechanical treatment stage contained some minor amount of suspensions and the filtrate had a light yellow color.

At the first stage of purification and concentration by reverse osmosis, nitric acid flowrate used to adjust the pH level from 8 to 4.5–6.0 amounted to 0.3 g/dm³. The specific electrical conductivity of the permeate was increasing gradually along with the increase in the concentration degree of the solution in the circulation tank. Thus, the first stage of reverse osmosis resulted in a concentration of the dissolved circulating water components by a factor of 8–9. Specific activity of uranium-235 in the concentrate sample was found to be equal to 1.2 Bq/g, which corresponds to a specific activity of 53 Bq/g for uranium isotopes (234, 235, 238) in case of a natural ratio and a uranium content of 2 g/dm³ in the solution. Some brown precipitate was found in the storage tank of the first concentration stage, which is likely to be seen as the main contaminant of membrane elements. The pulp with sediment had a uranium concentration of about 6 g/dm³. Obviously, the sediment in the pulp contained insoluble products of uranium hydrolysis: polynuclear hydroxo complexes U(VI) (U₂O₅²⁺, U₃O₈²⁺, UO₂(OH)⁺, U₃O₈(OH)⁺, U₃O₈(OH)₂, U₃O₈(OH)₃⁻ [10], which are formed at a pH level off more than 3.

The averaged permeate of the first stage, which entered the second stage of purification, had a specific electrical conductivity of 57 μS/cm and a pH of 3.8. The flowrate of sodium hydroxide used to adjust the pH from 3.8 to 6.5 amounted to 0.03 g/dm³.

Table 2 summarizes the operating parameters of the first and second stages in the desalting and concentration modes, as well as data on the selectivity (expressed as an equivalent amount of NaCl retained by the membrane, %) and the concentration factor.

Table 2 presents the specific activity of radionuclides in permeate samples. These data evidence that at the first stage of reverse osmosis, the spent water purification factor from uranium-238 was about 1.8·10⁴ which is explained by the hydrolyzed state of uranyl cations in the solution at a pH level of more than 3.

Table 1 summarizes the results of a study focused on the chemical and radionuclide post-purification composition of water after two stages of reverse osmosis. Two-staged reverse osmosis allowed to purify the spent water to a state when the monitored parameters would meet the requirements providing its discharge into the centralized water disposal system [7].

Sorption concentration of uranium was performed by treating clarified spent water to a level of uranium content in the filtrate of 1 mg/dm³, which

Table 2. Parameters of the purification and concentration stages based on the reverse osmosis method during testing

Parameter	Unit	Parameter value		
		First stage		Second stage under the treatment mode
		At the start of operation (demineralization)	Under operating mode (concentration)	
Temperature of the solution	°C	21–25	25–38	21–25
Working pressure above the membrane	MPa	4.5–5.0		1
pH of the solution	pH	4.3		6.5
pH of the permeate	pH	3.7		5.7–6.5
Specific membrane capacity	dm ³ /(m ² ·h)	38	34–36	37
Specific electrical conductivity of the permeate	μS/cm	3	140	15.6
Equivalent salt content of NaCl in permeate	mg/dm ³	1.4	66.7	6.5
Specific electrical conductivity of the concentrate	μS/cm	860	13,550	1,490
Equivalent NaCl salt content in the concentrate	mg/dm ³	415	6,590	720
Total salt content in the concentrate	g/dm ³	1.1	13.7	–
Concentration factor	–	–	12.5	–
Calculated selectivity	%	99.4	99.0	99.1
Specific activity of uranium-238 in permeate	Bq/kg	–	0.7	<0.2
Specific activity of uranium-235 in permeate	Bq/kg	–	0.16	
Specific activity of uranium-234 in permeate	Bq/kg	–	0.19	

was achieved by filtering through 190 column volumes. The content of uranium-238 in the averaged sample of the filtrate amounted to 0.3 mg/dm^3 , thus, compared with the initial content in the spent water (250 mg/dm^3), the purification factor (K_{pur}) amounted to 830. After being treated from uranium, the filtrate was either discharged into centralized water disposal system, if its indicators did meet the discharge standards given in Table 1, or further treated by reverse osmosis from the remaining contaminants.

Thus, sorption concentration allows up to 99.9% of uranium from the initial content to be extracted from the spent water. The average amount of uranium in the column with the sorbent amounted to some 47 g/dm^3 , which corresponds to uranium sorption-based concentration by a factor of 190.

Uranium desorption by a mixed nitrate-sulfate $\text{NH}_4\text{NO}_3 + \text{H}_2\text{SO}_4$ solution was accompanied by carbonate desorption with carbon dioxide release; therefore, a bottom-up flow pattern was chosen for the desorbing solution in the column. Samples were taken after each portion of the desorbing solution with a volume equal to one column volume was filtered through it with the specific activity of uranium evaluated for each sample. Figure 1 shows the results of uranium desorption from AV-17-8 ion-exchange resin with $\{0.9 \text{ M NH}_4\text{NO}_3 + 0.1 \text{ M H}_2\text{SO}_4\}$ solution. Desorption has provided the extraction of a fraction containing 98% of absorbed uranium with a concentration of 8 g/dm^3 .

Concentrates generated by reverse osmosis and ion exchange methods were precipitated using a 25% aqueous ammonia solution, which allowed to extract ammonium polyuranate with its flowrate maintained at a level of 107 ml/dm^3 to allow the precipitation from reverse osmosis concentrate and 130 ml/dm^3 for the one from the desorbate. The precipitation solutions had a specific activity of uranium isotopes (234, 235, 238) of no more than 0.05 Bq/g and a total salt content of $50\text{--}100 \text{ g/dm}^3$.

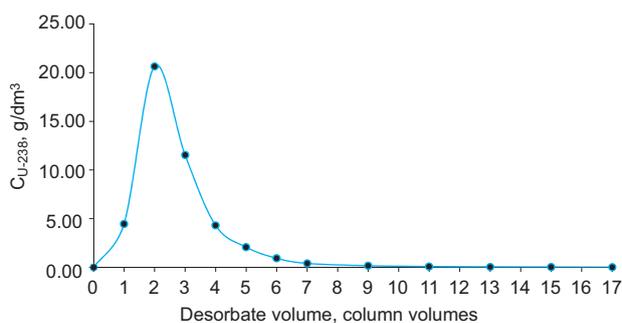


Figure 1. Output curve for uranium desorption from AV-17-8 saturated anionite phase by $\{0.9 \text{ M NH}_4\text{NO}_3 + 0.1 \text{ M H}_2\text{SO}_4\}$ solution

Given such indicators, after being dried to dry salts, the specific activity of secondary solid waste would be lower than the level allowing its categorization as RW.

After being precipitated from the reverse osmosis concentrate, the air-dried precipitate of ammonium polyuranate contained about 17 wt.% of uranium, whereas after being precipitated from the desorbate its content amounted to 63 wt.%. Obviously, membrane concentration of uranium with its subsequent precipitation in the form of ammonium polyuranate appears to be less effective. The indirect data also indicates that the desorbate obtained during the sorption concentration of uranium contains only minor impurities that can be coprecipitated as hydroxides.

The flowchart for the spent water treatment stage

Figure 2 presents the proposed flowchart for the recycled water treatment. The treatment process includes the stages of spent water mechanical filtration at a cascade of bulk sand filters, sorption of uranium from clarified water on anionite in a sulfate form, additional purification of non-radioactive solution in the first stage of reverse osmosis and concentration of chemical pollutants in the second stage, concentration of the solution from the second stage of reverse osmosis by evaporation and thermal drying of non-radioactive salts. Treated water is either supplied to the soil treatment unit to allow its recycling and reagent fabrication or discharged into centralized wastewater systems. Non-radioactive salts following radiometric control are disposed of at an industrial waste landfill.

The treatment process also involves uranium desorption, ammonium polyuranate precipitation, precipitate filtration and drying, as well as subsequent management of non-radioactive precipitation mother liquor. The flowchart suggests the use of AV-17-8 anion exchanger in sulfate form to minimize the amount of corrosive chloride ions in the solution and to provide closed solution circulation in the system. After the anion-exchange resin gets saturated, uranium is desorbed in two portions of a solution having the following composition: $72 \text{ g/dm}^3 \text{ NH}_4\text{NO}_3 + 10 \text{ g/dm}^3 \text{ H}_2\text{SO}_4$. The desorbate is segregated into two streams: the first (commercial fraction) is sent to allow for ammonium polyuranate precipitation, the second one is handed over to a storage tank and can be further used as the first portion of the stripping solution. Then, the sorbent is washed with water, the column is drained, the sorbent is converted into a sulfate form allowing its further recycling.

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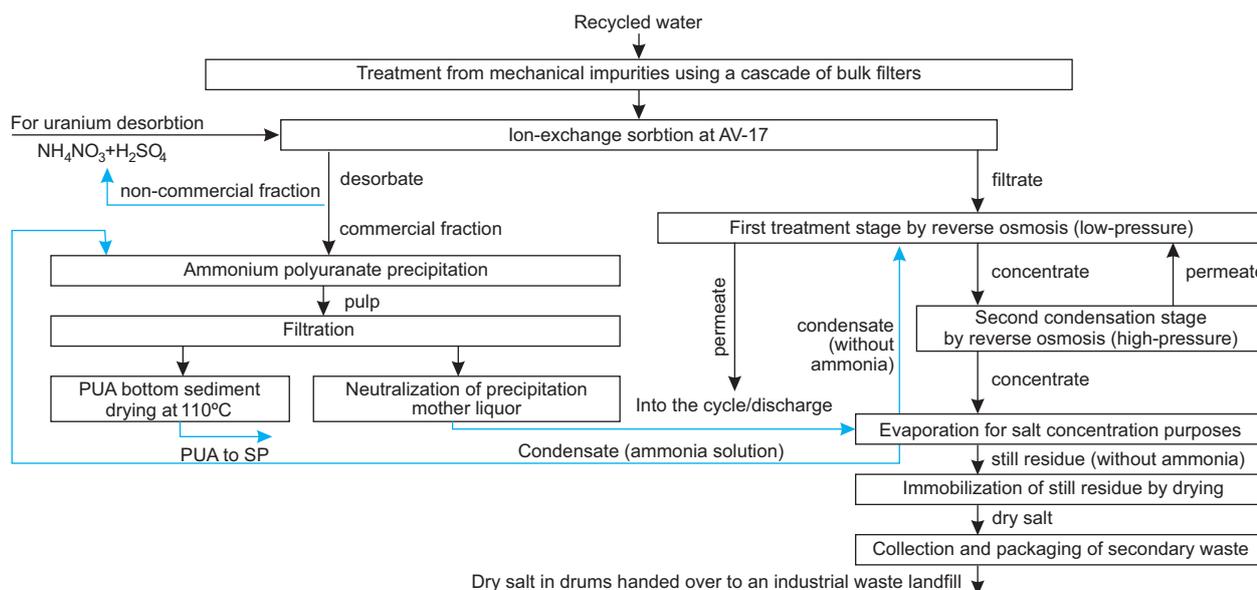


Figure 2. Treatment flowchart for uranium-containing spent water generated from RCS treatment by hydro-separation method

Ammonium polyuranate gets precipitated from the commercial desorbate fraction by a method providing for simultaneous merging of the fraction and the precipitant (25% ammonia solution) assuming a $\text{pH}=9.5 \div 10$. When the ammonium polyuranate precipitate is formed, the pulp is handed over to clarification and filtration. Upon completing the filtration process, the precipitate is handed over to a thermal drying unit. The content of uranium and impurity elements is evaluated to check if the resulting product meets TU 95 2822-2002 requirements to allow its transfer to sublimate production facility. The above flowchart allows optimal use of various reagents to minimize the volume of secondary liquid waste. The precipitation mother liquor is neutralized to neutral $\text{pH} 6-8$ and evaporated to a saturated solution concentration. Ammonium was proposed to be recycled at the stage of precipitation mother liquor evaporation in accordance with [11]. Alkali is dosed into the bottoms of the mother liquor at a rate of 1.2 mol of NaOH per 1.0 mol of NH_4^+ which is followed by evaporation with distillation and subsequent condensation of ammonia solution. After being evaluated, the condensate is sent to ammonium polyuranate precipitation stage as a precipitating agent. The distillation residue, which is a mixture of sodium nitrate and sodium sulfate, is sent to immobilization by thermal drying. Dry salt after being packaged and checked by its radiometric characteristics is disposed of at an industrial waste landfill.

Thus, we have uranium-containing spent water at the inlet of the processing stage and treated water and solid non-radioactive salts at the outlet.

Conclusion

Laboratory tests exploring spent water purification from uranium by reverse osmosis and ion exchange methods have shown that the following goals can be attained: compliance with the discharge standards for the treated water; separation of uranium-containing fraction with a uranium concentration of at least 8 g/dm^3 and subsequent precipitation of uranium into an insoluble compound in the form of ammonium polyuranate with uranium content of at least 63 wt.%. Ammonium polyuranate extraction from uranium-containing fraction obtained by sorption extraction on anion exchanger has proved to be more effective.

The data obtained allowed to develop a basic processing flowchart for uranium-containing spent water generated at a soil treatment plant by hydro-separation method resulting in uranium concentrate and avoiding RW generation (Figure 2).

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