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PURIFICATION OF URANIUM(VI) FROM IMPURITIES OF FISSION PRODUCT SURROGATES BY SOLVENT EXTRACTION IN THE CARBEX PROCESS

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Abstract. *The article presents the results on the extraction and purification of uranium(VI) from impurities of surrogates of some fission products (Cr(III), Mn(IV), Cu(II), Sr(II), Cs, Sb(V), Mo(VI), Ba(II), La(III), Ce(IV), Sm(III), Eu(III), Re(V), Al(III), Y(III), Nd(III)) and Na from aqueous solutions of sodium carbonate and ammonium carbonate by solvent extraction using methyltrioctylammonium carbonate. The values of uranium purification factors were 10^3–10^6. Based on the carried out studies, a scheme of solvent extraction reprocessing of multicomponent carbonate solutions containing fission product surrogates was developed and optimized. This scheme showed the high efficiency of the solvent extraction technique for fractionating the fission products in the CARBEX process.*

Key words: *Uranium, fission products surrogates, carbonate solutions, solvent extraction, scrubbing, re-extraction, stripping, methyltrioctylammonium carbonate.*

1. INTRODUCTION

Solvent extraction (SE) is one of the most promising methods for the separation and purification of less common elements and non-ferrous metals, including radioactive ones, from impurities from acidic and carbonate-alkaline media.

The PUREX process, a well-known technology for reprocessing of spent nuclear fuel (SNF), is based on the application of the SE technique for separation and purification of uranium and plutonium from nitric acid media using tri-*n*-butyl phosphate (TBP) as a solvent in a hydrocarbon diluent [1]. Recently, alternative hydrometallurgical non-acidic approaches to SNF reprocessing have been developed [2]–[4]. All of them are based on the use of carbonate/bicarbonate or carbonate/alkaline media. One of such methods is the CARBEX (CARBonate EXtraction) process, the concept of which was published in 2008 [5]. The most promising solvents for reprocessing of uranium from impurities from carbonate and alkaline solutions are quaternary ammonium compounds (QAC). The authors of [6]–[8] found that the QAC can extract uranium(VI) from alkaline solutions after leaching of mineral raw materials. For quantitative extraction of uranium from carbonate and alkaline solutions, it is preferable to use QAC, which is confirmed in previously published works [8]–[24].

In the CARBEX process, at the stage of SE separation of uranium(VI) from carbonate solutions and its purification from all impurities of fission products (FPs), it is proposed to use methyltrialkylammonium (MTAA) carbonate or methyltrioctylammonium (MTOA) carbonate in a hydrocarbon diluent. Currently, to develop an effective option for the SE partitioning of carbonate solutions formed in the CARBEX process at the SNF oxidative dissolution stage and to develop an operational scheme of SE processes, it is necessary to carry out additional lab tests with modeling of multi-stage countercurrent SE processes, as well as to carry out a full cycle of SE reprocessing of multicomponent U(VI)-containing carbonate solutions in the loading/scrubbing/stripping mode.

The goal of this research was to optimize the process of SE separation and purification of uranium from multicomponent carbonate solutions after oxidative leaching of a spent uranium oxide fuel surrogate using a laboratory multi-stage countercurrent extraction cascade.

2. MATERIALS AND METHODS

Solid salts Na_2CO_3, $(NH_4)_2CO_3$, NH_4HCO_3, and 35% aqueous solution of H_2O_2 of the chemically pure grade were used. MTOA carbonate was obtained according to the original procedure.

As the initial (feed) solution, a U(VI)-containing carbonate solution was used in the work, Table 1, which was obtained by dissolution of the SNF surrogate. The composition of the initial surrogate powder was chosen according to the mass content of U and FPs in the SNF of the water-water energetic reactor VVER-1000, with the burnup rate of 40 MWd kgU^{-1}, after 5 years of cooling period. To prepare the surrogate powder with accurate composition, powders of metals' oxides and carbonates have been weighted with an accuracy of ±0.0001 g, placed in a porcelain mortar, and carefully grinded for 30 min. The grinded powder mixture was

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placed in a sealed metal container and shaken for 30 min. Obtained homogeneous SIMFUEL powder was heat-treated in the air at 500°C for four hrs and cooled to room temperature. After alkaline pretreatment with 1.0 mol/L NaOH (to remove the bulk of molybdenum and cesium), the suspension was filtered [25]. The insoluble residue was used for the dissolution stage with 1.0 mol/L Na_2CO_3 - 0.1 mol/L H_2O_2 solution at 60°C in the mode of 10 min agitation – 10 min ultrasonication. The resulting carbonate solution containing 98.0 gU(VI)/L and impurities (see Table 1) was used for investigation.

Table 1. Chemical composition of the initial multicomponent U(VI)-containing carbonate solution used for SE tests, in mg/L

Cr	Mn	Cu	Sr	Te	Al
2.5	0.03	1.16	0.01	0.049	0.06
Ba	La	Ce	Sm	Eu	Re
4.45	3.52	57.3	1.17	0.05	0.001
Sb	Nd	Sn	Y	Cs	Mo
0.0355	0.511	0.115	0.572	2.93	4.42

Solvent extraction, scrubbing, and re-extraction (stripping) procedures were carried out at room temperature (20±2°C), organic-to-aqueous phases (O/A) volume ratio equals 1/1 and with intensive mixing for 15 min. The mixing phase time equal to 15 min is determined based on preliminary kinetic experiments. Mixing and separation of the aqueous and organic phases were carried out in glass separation funnels with a volume of 10–50 mL.

As the initial extraction mixture, an organic solution of MTOA carbonate ($[R_4N^+]$ = 1.5 mol/L, $[CO_3^{2-}]$ = 0.7 mol/L) in toluene (chemical purity grade) was used.

The content of U(VI) in the aqueous solutions with a concentration over 1.0 g/L was established by titration, using 8.4 mmol/L solution of ammonium vanadate as the titrant and diphenylamine-4-sulfonic acid sodium salt as the indicator [26]. The content of U(VI) in the solutions with a concentration lower than 1.0 g/L was established by the spectrophotometric method with Arsenazo III, per the absorbance spectra of the green-blue complex compound arsenazo-uranyl (λ_{max} = 651 nm, detection limit ~0.025–0.05 μgU/L) [27].

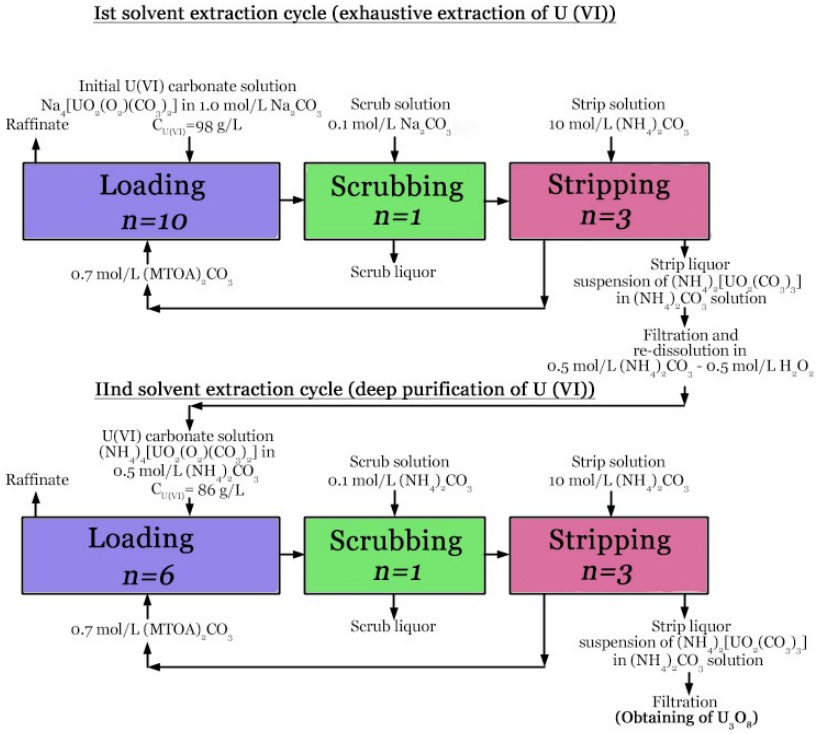

Figure 1. Operational scheme for processing of carbonate U(VI)-containing multicomponent solutions in the loading/scrubbing/stripping cycle. O/A = 1/1, 20±2°C, mixing phase time – 15 min, *n* – number of steps.

The concentration of metals in multicomponent solutions and solid samples (U_3O_8) was analyzed by the ICP-MS method on the iCARTM Q device (Thermo Fisher Scientific, USA) and Agilent 7500ce (Agilent Technologies, USA).

The degree of U(VI) extraction (Ex) was calculated by the following equation:

$$Ex(\%)=(M_U/M_{U,i})\cdot 100 \quad (1)$$

where $M_{U,i}$ is the quantity of the U(VI) in the initial carbonate solution and M_U is the quantity of U(VI) contained in the organic phase after extraction (at equilibrium conditions).

The values of the purification factor (K_{PUR}) of U from impurities were calculated according to the equation:

$$K_{PUR}=[(W_U)/(W_M)]/[(W_{U,i})/(W_{M,i})] \quad (2)$$

where $W_{U,i}$ and $W_{M,i}$ are the masses of U and impurities in the initial (feed) carbonate solution while W_U and W_M are the masses in the U_3O_8 powder sample obtained from crystalline ammonium uranyl carbonate (AUC) $(NH_4)_4[(UO_2)(CO_3)_3]$ after loading/scrubbing/stripping cycle in according with Figure 1.

3. Results and discussion

Based on previous studies [6] on the SE of carbonate $[UO_2(CO_3)_x]^{2-2x}$, where x = 2 or 3 and mixed U(VI) peroxo-carbonate species, mainly corresponding to the general formula $[UO_2(O_2)_x(CO_3)_{3-x}]^{4-}$, where x is 1 or 2 from carbonate solutions, an operational scheme of a laboratory multi-stage countercurrent extraction cascade based on 25 mL glass separation funnels was developed (see Figure 1). This cascade was used for laboratory testing of U(VI) recovery and purification from impurities in loading/scrubbing/stripping mode.

During the development of the SE processing scheme of the CARBEX process, it was found that it was necessary to carry out two stages of SE. The first stage is the SE separation of U(VI) and Pu(IV/VI) from carbonate solutions after SNF oxidative dissolution stage, as a result of which few amounts of some FPs (Mo, Cs, rare-earths elements (REEs), and some others) are coextracted with uranium and plutonium. For deep purification of uranium and plutonium from all impurities and compliance with all requirements for modern SNF reprocessing methods, during the SE processing of carbonate solutions in the CARBEX process, an additional (refining) extraction cycle is required.

Table 2. Uranium(VI) concentration values in aqueous and organic phases on the loading, scrubbing, and stripping steps at stationary mode (first (exhaustive) SE cycle). The designation A is the aqueous phase; O is the organic phase. The digit after the phase designation is the step number. A(O)1–10 is aqueous(organic) phases in the extraction part of the cascade; A(O)11 – equilibrium aqueous(organic) phase after scrubbing; A(O)12–14 – equilibrium aqueous(organic) phases during stripping.

Aqueous phases designation	[U(VI)], g/L	Organic phases designation	[U(VI)], g/L
A1	0.011	O1	0.007
A2	0.022	O2	0.017
A3	0.033	O3	0.028
A4	0.04	O4	0.032
A5	0.46	O5	0.73
A6	1.14	O6	1.1
A7	1.6	O7	1.5
A8	2.5	O8	5.0
A9	7.0	O9	40.0
A10	47.0	O10	91.0
A11	1.0	O11	90.0
A12	0.022	O13	0.2
A13	0.016	O13	0.007
A14	0.004	O14	0.004

After starting the laboratory multi-stage countercurrent extraction cascade and achieving stationary (operation) mode, two full loading/scrubbing/stripping cycles with multicomponent U(VI)-containing carbonate solution were carried out. Table 2 shows U(VI) concentration values in the aqueous and organic phases at operating mode.

According to Figure 1, the first (exhaustive) extraction cascade included ten SE steps, one scrubbing step, and three stripping steps and was intended for quantitative extraction of U(VI) from the initial carbonate solution. At the same time, the initial multicomponent carbonate solution (containing 98 gU(VI)/L and 1.0 mol/L Na_2CO_3) was fed to the 10th extraction step, and unloaded aqueous phase (raffinate) was removed from the 1st extraction step. The concentration of U(VI) in the raffinate after the extraction cycle was 0.011 g/L (see Figure 1 (phase A1)), which corresponded to more than 99.9% of uranium extraction into the organic phase. The U(VI)-loaded organic phase (with [U(VI)] = 91.0 g/L) from the 10th extraction step was supplied to the scrubbing (one step), which was carried out with 0.1 mol/L aqueous Na_2CO_3 solution at O/A = 1/1. The concentration of U(VI) in the carbonate solution after scrubbing (scrub liquor) was 1.0 g/L, which corresponded to ~1.0% of U(VI) losses.

The organic phase after scrubbing was supplied to the 1st step of stripping. Aqueous 10 mol/L $(NH_4)_2CO_3$ solution was used as a strip solution. This made it possible not only to quantitatively extraction of U(VI) from the loaded organic phase at the stripping stage but also to obtain crystalline AUC in the strip liquor. In this mode, the process is called precipitative stripping (re-extraction) [7]. After the first stripping step, the U(VI) concentration in the aqueous phase was 0.02–0.03 g/L. The degree of extraction of U(VI) into the crystalline AUC from the organic phase was ~99.8%.

The second SE cycle (deep purification of U(VI), see Figure 1) was carried out from carbonate solutions obtained by re-dissolution of the crystalline AUC (obtained on the 1st SE cycle) in aqueous solutions of 1.0 mol/L of $(NH_4)_2CO_3$ - 0.5 mol/L H_2O_2 (option 1) and 1.0 mol/L $(NH_4)_2CO_3$ (option 2) at room temperature. In the first option, a carbonate solution was obtained which contained 86 gU(VI)/L (see Figure 1). In the case of using hydrogen peroxide when the dissolution of crystalline AUC, mixed U(VI) peroxo-carbonate complexes are formed, the solubility of which is higher in aqueous solutions of $(NH_4)_2CO_3$ and Na_2CO_3 compared to carbonate complexes of U(VI). In the second option, the carbonate solution contained no more than 15–20 gU(VI)/L.

Aqueous carbonate solution containing 86 gU(VI)/L was used as an initial solution at the second(refining) extraction cascade for deep purification of U(VI) from impurities. In this case, the second SE cascade included six extraction steps, one scrubbing step, and three stripping steps (see Figure 1).

The initial carbonate solution was fed on the 6th extraction step and the raffinate was removed from the first step in which the concentration of U(VI) was 0.016 g/L which corresponded to the degree of U(VI) extraction equal 99.9%. The U(VI)-loaded organic phase, as in the first (exhaustive) cascade was fed for

scrubbing. In the second SE cycle for scrubbing, an aqueous 0.1 mol/L $(NH_4)_2CO_3$ solution was used to exclude sodium contamination of the final uranium product (AUC). The losses of U(VI) for the one scrubbing step was 0.2–0.25% (at a concentration of U(VI) in the scrub liquor equal 0.2–0.25 g/L). After the scrubbing stage, the organic phase was feed to the first stripping step. The degree of U(VI) extraction for three consecutive stripping steps was more than 99.9%. The degree of U(VI) recovery into AUC crystals was 99.7%.

Table 3 presents data on the values of the purification factors of U(VI) from some impurities achieved in experimental laboratory tests (in two options) with the use of a simulated multi-stage countercurrent solvent extraction process. Carrying out modeling of multistage solvent extraction process on a laboratory cascade in various options and in extraction/scrubbing/stripping mode, allowed to reach values of uranium purification factors from impurities of some FPs surrogates (Mo, Cs, Sr, Sn, Ba, Al, Cr, Cu, Sb, Re, Te, Mn, Zr, and some REEs) and also from sodium at value level 10^3-10^6.

Table 3. The values of the purification factors of U(VI) from some impurities of FPs surrogates

$K_{PUR} \cdot 10^6$										
Option 1										
Elements	Na	Al	Cr	Mn	Cu	Sr	Y	Zr	Mo	Sn
Exhaustive cascade	–	0.01	0.1	0.1	0.04	0.1	0.01	0.1	0.03	0.001
Refining cascade	>0.1	>0.01	>0.1	>2.0	>0.04	>0.4	>0.01	>0.1	>0.1	>0.01
Elements	Sb	Te	Cs	Ba	La	Ce	Nd	Sm	Eu	Re
Exhaustive cascade	0.1	0.001	0.001	0.02	0.08	0.01	0.0	0.08	0.01	0.01
Refining cascade	>0.8	>0.001	>0.01	0.4	0.3	0.8	0.2	0.4	0.1	>0.03
Option 2										
Elements	Na	Al	Cr	Mn	Cu	Sr	Y	Zr	Mo	Sn
Exhaustive cascade	–	0.01	0.07	1.9	0.04	0.04	0.01	0.1	0.07	0.01
Refining cascade	>0.01	>0.01	>0.07	>2.0	>0.04	>0.4	>0.01	>0.1	>0.1	>0.01
Elements	Sb	Te	Cs	Ba	La	Ce	Nd	Sm	Eu	Re
Exhaustive cascade	0.07	0.007	0.003	0.1	0.06	0.5	0.1	0.4	0.1	0.01
Refining cascade	>0.7	>0.007	>0.02	0.8	0.6	5.2	1.2	4.4	1.3	>0.03

4. Conclusion

According to the data of two laboratory tests, the values of the purification factors of U(VI) from Mo, Cs, Sr, Sn, Ba, Al, Cr, Cu, Sb, Re, Te, Mn, Zr, and some REEs as well as from sodium were 10^3–10^6. The obtained results are close to those of the industrial PUREX technology. Thus, the developed and optimized scheme of solvent extraction processing of multicomponent carbonate solutions after oxidative leaching of the VVER–1000 SNF surrogate on a laboratory scale showed high efficiency of the solvent extraction technique using methyltrioctylammonium carbonate for fractionation of carbonate solutions in the CARBEX process.

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