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Recovery of Uranium from Ammonium Uranyl Carbonate (AUC) Effluents by Combined Ion Exchange and Membrane Separation

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ABSTRACT

Ammonium uranyl carbonate (AUC) production processes have effluents with a high uranium content (5 g/L), which is conventionally recovered by evaporation. In this study, an alternative process, recovery of uranium by strong anion exchange (AE) membranes is investigated. Solutions were modeled by MINTEQA2 for determining the species present. Laboratory scale AUC precipitations with three different C/U mole ratios were carried out using uranyl nitrate (UNH) feed solution from the pilot plant for the purpose of determining the remaining concentration of uranium in the effluents. Strongly basic AE membrane separations of

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these effluents were carried out, but separation was not effective because of the limited surface area of the membranes. Synthetic solutions of three different C/U mole ratios were prepared and passed through a column of strongly basic AE resin. Membrane separations were then carried out on the resulting solutions. Membrane separations proved to be effective only in dilute solutions of uranium. There was a complementarity between the AE resins and membranes for U(VI) recovery from carbonate solutions in that different uranyl carbonate anionic species were extracted to differing extents with the two procedures.

Key Words: Uranium recovery; AUC effluent; Carbonate solution; Membrane separation; Ammonium uranyl carbonate; MINTEQA2 modeling; Uranyl carbonate anionic species.

INTRODUCTION

Some examples of the basic techniques for the recovery of uranium from various solutions and effluents include biological treatment,[1] solvent extraction,[2–4] ion exchange,[5] and precipitation.[6] However, the search for full exploitation of natural resources combined with strict environmental regulations often necessitates some novel and emerging techniques be used for U recovery at specific applications.[7]

In countries utilizing nuclear power plants for the generation of electricity, one of the problems faced is achieving a reliable technique for the treatment of streams containing low concentrations of uranium. The nuclear fuel cycle involves several steps such as purification of yellow cake or uranium concentrate by solvent extraction, precipitation as either ammonium diuranate or ammonium uranyl carbonate (AUC) by addition of ammonia or ammonium carbonate, respectively, conversion to uranium dioxide (UO_2), then to uranium tetrafluoride (UF_4), and finally to uranium hexafluoride (UF_6). After enrichment of the latter, it is converted to AUC, and then to UO_2 for manufacturing reactor fuel.[8]

Thus, the established route for uranium dioxide nuclear fuel is ammonium diuranate precipitation, and it is preferred in many fabrication plants. The alternate route for nuclear fuel production is the AUC process, which is preferred in some installations because of the capability of producing better flowing powder and direct pressability (without the addition of binder). The drawback of this method of production is that the remaining uranium concentration in the effluent is high for direct discharge of the solution. Uranium forms strong complexes with carbonate ions [e.g., the log (stability constant) values for the dicarbonato- and tricarbonato-uranate (VI) species are at the order of 14 and 18][9] and, therefore, effective removal may prove to be difficult. As conventional methods of removal, evaporation, and ion exchange separations have been tried. The unstabilization of the carbonato-uranate species, held in the resin by decreasing the pH to achieve partial conversion to diuranate anion,[10] bears the risk of sorption of low-solubility products onto the resin causing clogging (e.g., in U recovery from carbonate solution using strongly basic anion exchanger).[10] The purpose of this study is to investigate the utilizability of membrane separations for carbonate solutions of uranium.[11]

MODELING

The theoretical modeling of the solutions was carried out by using the chemical equilibria code MINTEQA2 (Version 4) obtained from the Center for Exposure Assessment Modeling at the US Environmental Protection Agency (EPA) in Athens, Georgia. The MINTEQA2 code calculations include aqueous speciation, solubility, and saturation state (i.e., saturation index), adsorption, oxidation–reduction, gas phase equilibria, and precipitation/dissolution of solid phases.[12] The AUC effluents and the synthetic solutions prepared in increasing uranium concentrations for three C/U molar ratios (7.5, 9, and 12, respectively) were modeled at the pH 4–12 range with 0.5 pH increments. The region of interest in evaluating the theoretical results was chosen according to AUC production parameters, and, therefore, only the charged species in the pH range of 8.5–10 were evaluated. The species in the region of interest are $UO_2(CO_3)_3^{4-}$, $UO_2(CO_3)_2^{2-}$, and $UO_2(CO_3)_{aq}$. As seen in Fig. 1, with increasing uranium concentration at equilibrium pH, and at all three C/U mole ratios, the dominant species is $UO_2(CO_3)_3^{4-}$. The results of the modeling graphs were found to be in agreement with the species diagrams in literature.[13]

EXPERIMENTAL

The experimental setup is given in Fig. 2. Uranium-containing carbonate solutions were placed in membrane cells. The retentate used was distilled deionized water. The membranes were preconditioned with ammonium carbonate for 24 hr before use. The experiments were carried out for 72 hr with constant magnetic stirring of the receiver solution. Synthetic uranium solutions of 1000, 500, 100, 50, and 25 mg/L concentrations for each C/U ratio were prepared from the high purity feed uranyl nitrate (UNH) solution of 110 g/L from pilot plant. The actual effluents from the pilot plant tested had a 5 g/L of U concentration. The membranes used were anion exchange

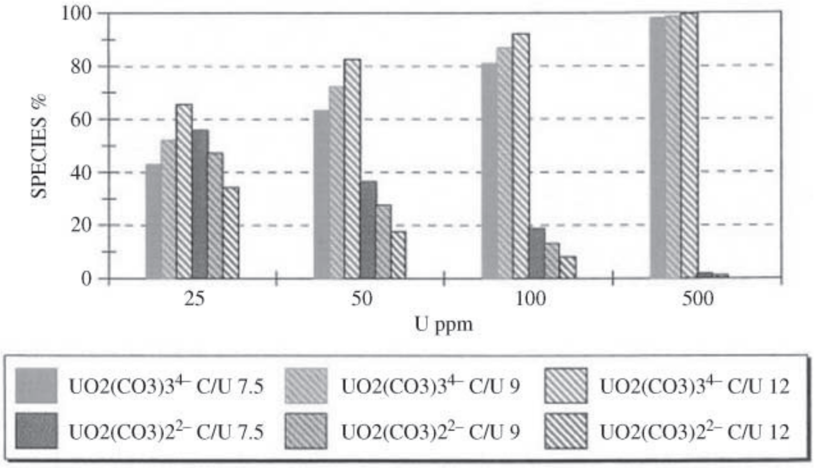

Figure 1. Species distribution diagram of di- and tricarbonato-uranate(VI) species as a fraction of total uranium concentration (drawn for the C/U ratios of 7.5, 9, and 12).

(AE) membranes of quaternary ammonium type and 47 mm in diameter from Pall-Gelman Sciences (the declared AE capacity was 0.8 meq/g dry weight). The membranes, permeate, and retentate solutions were analyzed for uranium after 72 hr by inductively coupled plasma atomic emission spectrometry (ICP-AES), and alternately by spectrophotometric 2-pyridylazo resorcinol sodium salt (PAR) method for very low levels of uranium. Uranium analysis was carried out on the retentate and the permeate, and the adsorbed (surface precipitated) uranium on the membrane was dissolved with 7.5 M HNO_3 and then analyzed. Some uranium was retained on the surface of the membrane as mentioned in other studies by AE membranes.[14] The membranes could be reused provided that there was no mechanical damage and tear. Because of the limited surface of the membranes, dilution of actual effluent solutions from the pilot plant was necessary. The uranyl carbonate solutions containing 25, 50, 100, and 500 ppm U had a volume of 100 mL. Membrane separation by itself on actual and synthetic solutions proved to be ineffective. Therefore, ion exchange separations prior to membrane separations were introduced as a step to increase effectiveness of the method. Strongly basic AE resin of quaternary ammonium type (Dowex 1X8, 200–400 mesh, functional group: trimethyl ammonium, shipping density: 0.7 kg/L, volume change from Cl^- to OH^- forms: +20%, declared total exchange capacity: 1.3 eq/L), was used in 1.3 cm diameter and 300 mm height glass columns throughout the experiments. The resin bed height was 200 mm. The columns were conditioned with 2 M $(NH_4)_2CO_3$ prior to use. In the case of carbonate

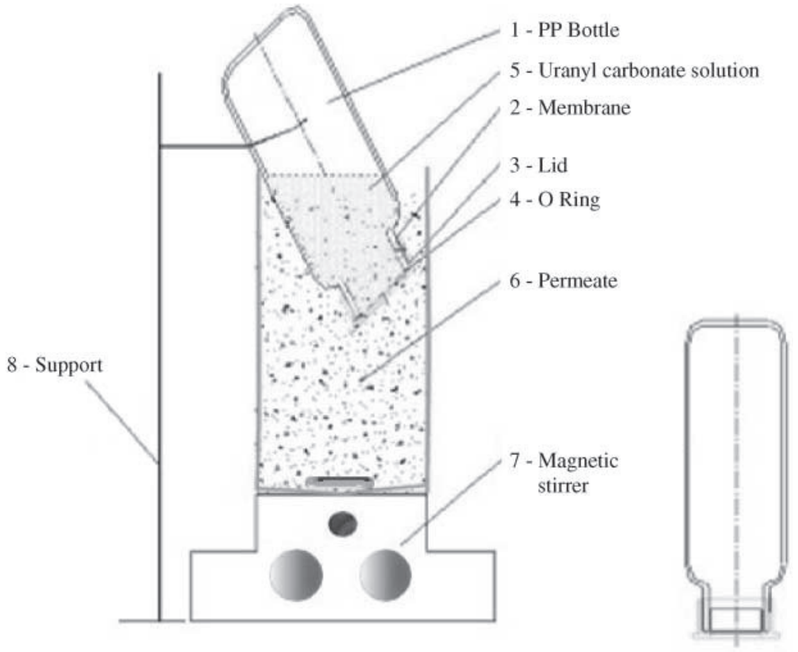

Figure 2. The experimental setup for the AE membrane separation of U from carbonate solution.

leaching, only NH_4NO_3 or NaCl could be used as eluants. Acid elution could not be used, because the retained carbonato-uranate species would cause the evolution of free carbon dioxide. The quantity of eluant (1 M NaCl) was about 15 bed volumes for uranyl carbonate leaching. Preliminary experiments (not shown) indicated that the Langmuir binding constant of $UO_2(CO_3)_3^{4-}$ for the resin was higher than that of divalent common anions, and was about two orders of magnitude higher than that of univalent common anions like Cl^- and NO_3^-. Thus, a high selectivity of U retention with AE resins is possible from AUC effluents and similar synthetic solutions. An ARL 3520 ICP-AES and Schimadzu UV-visible photometer were used for atomic and molecular spectrometric analysis.

RESULTS AND DISCUSSION

The membrane experiments carried out with only deionized water in the permeate resulted in very little uranium transfer to the permeate; uranium basically stayed in the retentate, and was adsorbed on the membranes to be redissolved for analysis. The adsorption isotherms of quaternary ammonium membranes for uranium retention of AUC effluents are given in Fig. 3. Adsorption by the membrane material is common in membrane separations reported in the literature, e.g., uranium adsorption onto the amidoxime adsorbents in seawater proceeded via the diffusion of uranyltricarbonate ions from the bulk solution to the external surface of the adsorbent (membrane), interior diffusion through the pores, and intrinsic complexation of $UO_2(CO_3)_3^{4-}$ ions with the amidoxime groups of the membrane.[15] Because the area of the membranes used in this work were limited and could not be increased, membrane separation by itself was not very efficient for the recovery of relatively high concentrations of uranium. Ion exchange separation prior to membrane separation was used for developing a more efficient process. The AE of carbonato-uranate(VI) solutions also presented some problems.

The theoretical modeling studies showed that in AUC production conditions (pH >8.5 and C/U ratio >7.5), the $UO_2(CO_3)_3^{4-}$ and $UO_2(CO_3)_2^{2-}$ species are predominantly present, depending on the uranium and carbonate concentrations. The relative concentration of $UO_2(CO_3)_3^{4-}$ increases with increasing uranium concentration (Fig. 1). Figures 4–6 show that for a fixed total U concentration of 25 ppm in aqueous solution, the predominant species in the region of interest are $UO_2(CO_3)_2^{2-}$ and $UO_2(CO_3)_3^{4-}$. Especially at pH ≥ 9.1, the predominance of the tricarbonate species shifts to lower pH as the C/U ratio is increased from 7.5 to 12.0 (Figs. 4–6). Ion exchange resin capacities increase with increasing ammonium carbonate concentration, and the highest capacity is reached at C/U 12 mole ratio (Fig. 6). The breakthrough capacity (i.e., calculated from the breakthrough volume of 225 mL of 5 g/L U influent solution when U started to appear in the effluent) was 80 mg U/g resin, and this maximal capacity was reached for a C/U ratio of 12 (i.e., the breakthrough capacities for C/U: 9 and 7.5 were 71 and 54 mg/g, respectively). Naturally these values should increase and approach theoretical capacities when the dynamic column operation was carried out at very low flow rates for equilibration, and when the dynamic capacity was found by integrating the upper portion of the breakthrough curve (drawn with C/C_0 vs. effluent volume) up to the point when $C/C_0 = 1$, i.e., influent and effluent concentrations were equal. This result is to be expected in the case of ion exchange resins, because retention depends on size and charge of the ion, meaning that the $UO_2(CO_3)_3^{4-}$ species should have higher retention. This is in accordance with the literature findings stating that the tricarbonato-uranate(VI) species show the highest tendency and selectivity for the strongly basic AE resins[10] where the highest "reversible sorption capacity" was obtained at pH 9.1 for the $UO_2(CO_3)_3^{4-}$ species, whereas higher but "irreversible sorption capacities"

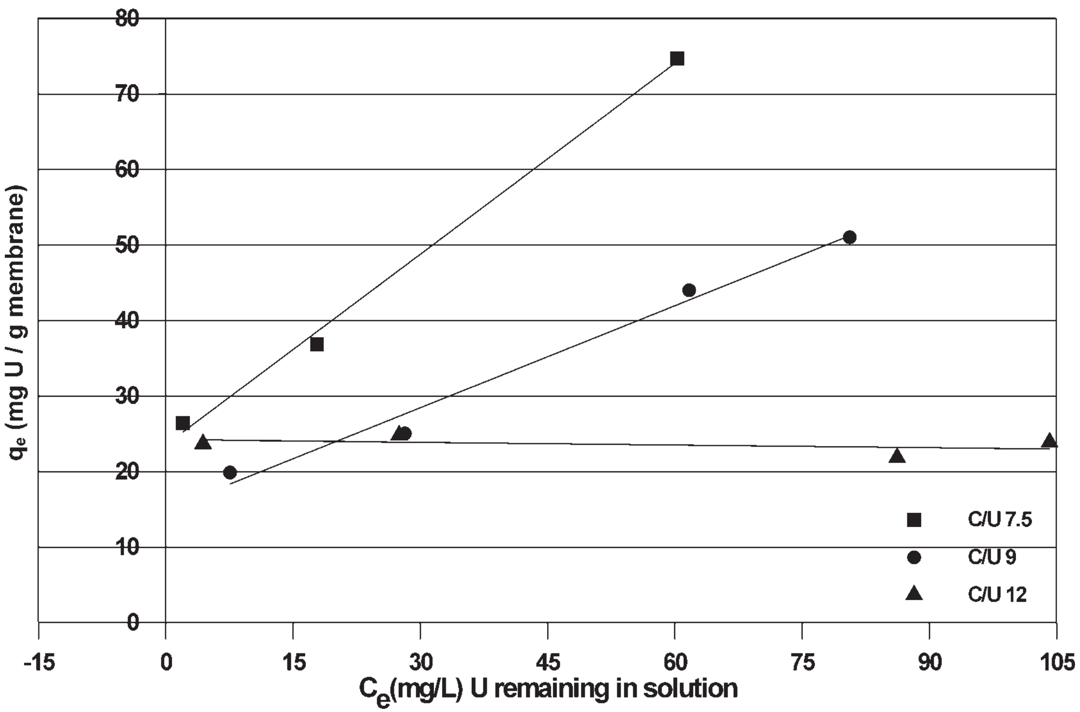

Figure 3. The adsorption isotherms of quaternary ammonium membranes for U retention of AUC effluents for varying C/U ratios [C_e, equilibrium U concentration remaining in solution (mg/L); q or (x/M), amount of U retained per mass of membrane (mg/g)].

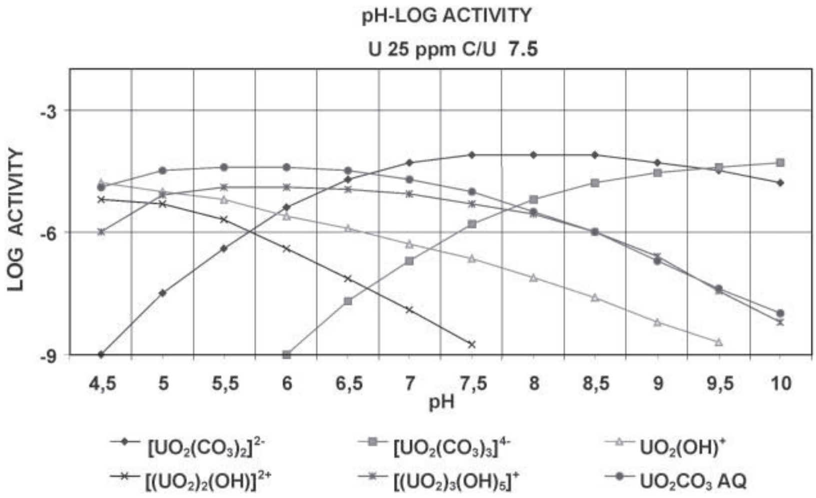

Figure 4. Log activity vs. pH diagram of uranyl carbonate solution of 25 ppm U at C/U 7.5 ratio.

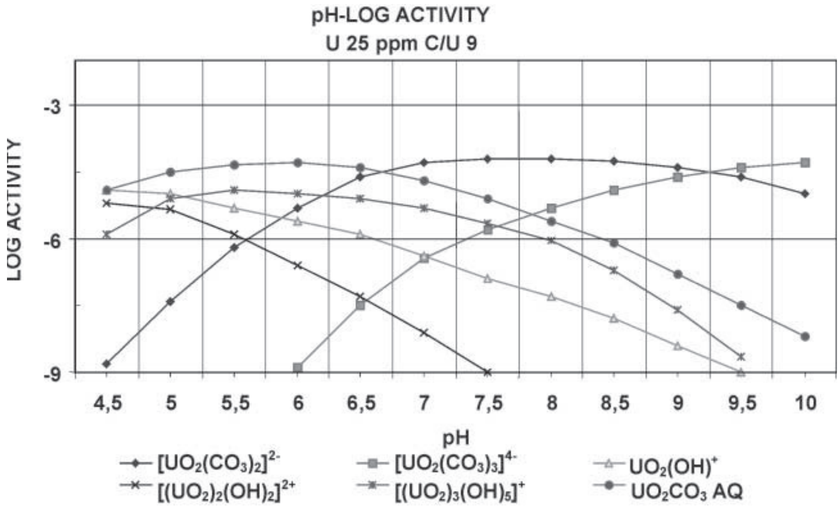

Figure 5. Log activity vs. pH diagram of uranyl carbonate solution of 25 ppm U at C/U 9 ratio.

were recorded, especially at pH <8 due to partial conversion and precipitation of diuranates in the resin phase. On the other hand, for the AE membrane separation, the order of affinity was reversed, and maximum U retention was reached for C/U 7.5 mole ratio (Fig. 3) with the irreversible sorption capacity decreasing as the C/U ratio was increased (i.e., the maximal capacities of U retention within the studied concentration range were 75, 51, and 25 mg U/g membrane for the C/U ratios of 7.5, 9, and 12, respectively). Considering that about 85%–90% of the total surface area of membranes was used, and that very high initial concentrations of U were not tested for membrane separation for the sake of process integrity, the reported capacities could be increased further. This can be explained by the preferential sorption on the membrane of the dicarbonato-species, $UO_2(CO_3)_2^{2-}$, which has a lower hydration energy than the tricarbonato-complex. Thus, the species of lower charge can be more easily excluded from the aqueous phase (similar to the exclusion of an aqueous phase species into an organic solvent by solvent extraction) and subsequently adsorbed on the quaternary amine-type anion exchanger membrane. Again this complies with the literature report mentioning that the high charge is an obstacle in the electrodialytic mobility of an ion such as tricarbonato-uranate(VI), e.g., the selectivity of MoO_4^{2-} over $UO_2(CO_3)_3^{4-}$ is high owing to its lower charge, and that the maximum electrodialytic U recovery in the form of tricarbonato-uranate(VI) species may reach 15% using an AE membrane.[16]

The predominant reactions in the resin and membrane phases may be postulated as:

$$4\,\overline{R_4N^+Cl^-} + UO_2(CO_3)_3^{4-} \rightleftharpoons \overline{(R_4N)_4UO_2(CO_3)_3} + 4Cl^-$$

(resin ion-exchange)

$$2\,\overline{R'_4N^+Cl^-} + UO_2(CO_3)_2^{2-} \rightleftharpoons \overline{(R'_4N)_2UO_2(CO_3)_2} + 2Cl^-$$

(membrane adsorption)

When liquid emulsion-type membrane processes in the literature are considered, it becomes apparent that the conventional liquid–liquid extractants [used for the recovery of U(VI) from aqueous solution] have recently been adapted as membrane materials. For example, high molecular weight amines like trioctylamine (TOA) and aliquat-336 that may effectively extract hexavalent uranium from sulfuric acid solution[17] have been used as membrane materials where Na_2CO_3 was used as stripping agent in the internal phase.[18,19] Another selective extractant for U(VI) that has a high distribution coefficient is tri-*n*-octylphosphine oxide (TOPO),[20] which also has been used recently as liquid membrane material with Na_2CO_3 as the stripping agent.[21,22] The situation in this study that is distinctly different from the

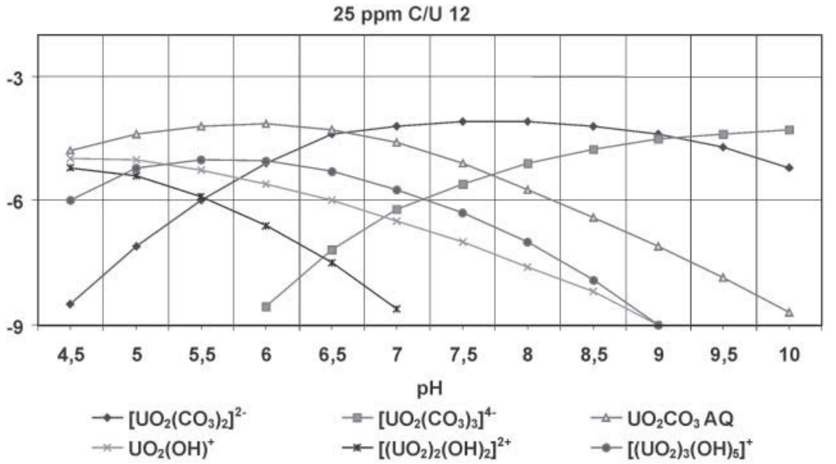

Figure 6. Log activity vs. pH diagram of uranyl carbonate solution of 25 ppm U at C/U 12 ratio.

previous membrane processes is that the solution from which U(VI) is to be recovered is an AUC process effluent of the uranium industry that is highly carbonated, and such carbonate solutions constitute the internal phase (stripping agent) of other membrane processes. Thus, there is no stronger internal solution for this study, i.e., the overall stability constant in logarithmic scale, $\log \beta_3$, for $UO_2(II)\text{-}CO_3^{2-}$ is 21.5.[10] The uranium in the AUC effluent, whose major proportion is recovered previously by an anion exchanger resin, is finally adsorbed on a quaternary ammonium-type AE membrane. Had a sulfuric acid internal phase been used, CO_2 would have evolved as a result of H_2SO_4 and HSO_4^- neutralization of the uranyl-carbonate species, and the evolved CO_2 would have an adverse effect upon the stability of the membrane leading to its breakage and consequent leakage of the permeated uranium.[19] Thus, the uranium values in the AUC process effluent are won with an environmentally clean process, i.e., the AUC process effluent containing 5 g/L of U predominantly as the tricarbonate-anion may first be fed to a quaternary ammonium type AE resin up to breakthrough capacity, and the effluents containing less than 500 ppm U could be sorbed on an AE membrane having similar functional groups until surface coverage. Regeneration of the resin and membrane may be performed with NH_4NO_3/NaCl and HNO_3 solutions, respectively. Both sorbents should be thoroughly washed with water before reuse. Uranium recovery in each phase (ion exchange and membrane steps) of the treatment is theoretically interpreted with respect to U species distribution in the aqueous phase. In general, reversible sorption (i.e., elutable with relatively concentrated NH_4NO_3 or NaCl solutions) of uranyl carbonate species on a strongly basic AE resin has been associated with the preferential binding of $UO_2(CO_3)_3^{4-}$ species by the R_4N^+ functional groups of the sorbent, whereas irreversible sorption has been attributed to the retention of lower carbonates and possibly $U_2O_7^{2-}$ anions formed in the sorbent phase by partial conversion at lower pH.[10]

CONCLUSION

An integrated laboratory-scale separation process has been developed for separating and recovering uranium from AUC effluents of the fuel production departments of nuclear power plants by the use of an anion-exchanger resin in the first phase to reduce the U concentration of the effluent to an acceptable level, followed by the incorporation of a quaternary amine-type anion exchanger membrane for recovering the remaining uranium from the relatively dilute solution. The mechanism of uranium recovery for the whole process involves ion exchange and adsorption retention of the tricarbonato- and dicarbonato-uranate(VI) species that predominantly exist in AUC effluents. A high ratio of C/U is necessary for reversible AE resin removal of U from ammonium carbonate solution, but a lower C/U ratio is preferable in AE membrane removal, once the U removal mechanism in the latter is irreversible sorption and the retained U is recovered with acid dissolution. Thus, there is a complementarity between the AE resins and membranes for U recovery (though the AE membranes—due to their limited surface area—have much lower operation capacities for possible industrial use). By the proposed scheme of separation and recovery, the uranium values of the AUC effluents can be won, and these effluents may be decontaminated to fairly acceptable levels.

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