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pH Effect on the Separation of Uranium Fluoride Effluents by the Reverse Osmosis Process

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Abstract

Ammonium fluoride solutions and uranium fluoride effluents (UFE) with solute concentrations from 0.101 to 7.920 kg/m^3, at pH 2.80 to 9.60, have been treated with a continuous feedback reverse osmosis (RO) process. The solute rejections of NH_4^+, F^-, and U^{6+} depend heavily on the feed pH value. For ammonium fluoride solutions, the rejection ratio of NH_4^+ decreases sharply from ca. 90 to 44.2% with the feed pH increased from 3.30 to 9.60, while that of F^- increases abruptly from 44.8 to 99.9% at the same pH change. For UFE solutions, the rejection ratio of U^{6+} remains greater than 90% at pH 2.80–7.13, while that of F^- decreases steadily from 96.4 to 18.8% with decreasing feed pH. Accordingly, the fluoride ions can be separated from UFE solutions under acidic conditions. The changes of solute rejection with feed pH can be explained by the different solubilities of the solutes in the membrane at different pH values. The UFE solutions with α and β activities at 20.4–53.7 and 8.99–21.3 ($\times 10^5$ Baq/m^3) can be reduced to a level lower than 2.41 and 3.37 ($\times 10^5$ Baq/m^3), respectively, by the current RO process.

INTRODUCTION

Modest energy consumption is one of the main advantages of the membrane process over many other conventional separation processes. With the improvements in membrane quality and module design, the application areas for the membrane process have expanded greatly in the last decade. Reverse osmosis (RO), for instance, has developed as one of the most important membrane processes in industrial applications (*1*).

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Seawater desalination has been one of the best known RO applications (*2*, *3*). Applications have also extended to free radical separation (*4*), rare earth separation (*5*), whey and milk concentration (*1*), wastewater treatment (*6*, *7*), etc. A very interesting and practical application of the RO process is in the metal finishing industry. Many plating chemicals, such as those for the nickel, copper, and zinc plating industry, are well recovered with the RO process (*8*). In addition to the practical investigations, a variety of theoretical analyses of the RO processes have also been reported (*9–13*).

The uranium conversion process effluent (UCPE) can be classified into two different types at the Institute of Nuclear Energy Research (INER), Taiwan (*14*).

1. Uranium nitrate effluent (UNE), such as yellow cake or ammonium uranyl tricarbonate (AUT), which comes from the extraction and conversion process of natural uranium. The basic precipitation reaction is

$$2UO_2(NO_3)_2(aq) + 6NH_4OH(aq) \rightarrow (NH_4)_2U_2O_7(s) + 4NH_4NO_3(aq) + 3H_2O(l)$$

2. Uranium fluoride effluent (UFE), which comes from the ammonium diuranate (ADU) or ammonium uranyl carbonate (AUC) reconversion process of UF_6. The basic precipitation reaction is

$$2UO_2F_2(s) + 4HF(aq) + 10NH_4OH(aq) \rightarrow (NH_4)_2U_2O_7(s) + 8NH_4F(aq) + 7H_2O(l)$$

The composition and concentrations of UCPE are very complicated and depend on the process conditions. The major components in UCPE are uranium compounds, nitrate, ammonia, ammonium ions, and fluoride ions. The UCPE is radioactive, corrosive, and toxic. If it is released without effective treatment, not only will the environment be polluted, but also valuable uranium will be wasted.

Several studies concerning the use of RO membrane in the treatment of UNE have been done by INER (*15*, *16*). The uranium rejections of Filmtec's FT-30 membrane module are higher than 98%, and the radioactivity of the permeate stream can be reduced to lower limit detection (LLD). However, RO treatment of UFE has not been successful due to the presence of a large amount of fluoride ions.

Based on the results, it is clear that the primary separation of fluoride ions from UFE is a prerequisite for effective treatment of UFE solutions. This research focuses on the separation characteristics of UFE and NH_4F solutions, using a new uranium recovery membrane (URM) process designed by INER. It is expected to separate fluoride ions from the uranium-containing UFE solutions so that the concentrated uranium can be recovered and the corrosive fluorides can be treated separately.

EXPERIMENTAL

The separation experiments were carried out with a URM test unit (Fig. 1) equipped with three kinds of spiral-wound RO modules and a ultrafiltration (UF) module. The RO module used in this study was composed of Filmtec's FT-30 element SW30-2521 with a pore diameter of 325 Å and a membrane area of 2.56 m^2. The thin film composite membrane was composited of a crosslinked aromatic polyamide deposited on a porous polysulfone.

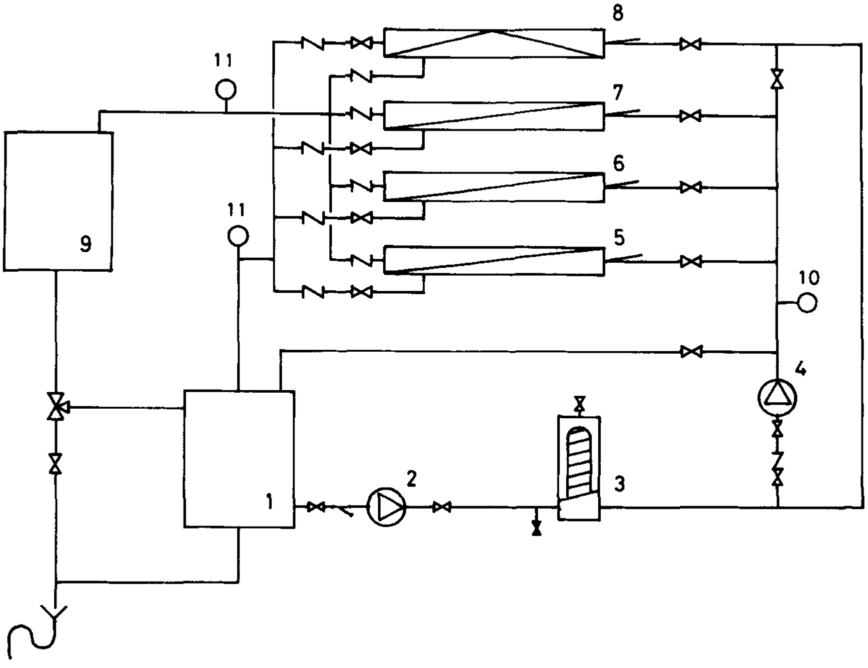

FIG. 1. Schematic flow diagram of URM-INER test unit. (1) Feed tank, (2) supply pump, (3) microfilter, (4) high pressure pump, (5)–(7) RO module, (8) UF module, (9) permeate tank, (10) pressure indicator, (11) flow rate indicator.

The experiments were first performed on aqueous solutions of ammonium fluoride to evaluate the pH effect on the rejection of fluoride ions. Then experiments were performed on various uranium fluoride effluent (UFE) solutions obtained from ammonium diuranate (ADU) or ammonium uranyl carbonate (AUC) reconversion processes.

The effluent solutions (UFE) contained fine precipitates of submicron size. In addition to aqueous ammonium fluoride solutions and uranium-containing effluents, water streams of different grades were also investigated on the RO system prior to the ammonium fluoride solution and uranium effluent investigation, to assure that the equipment functioned satisfactorily.

All the investigations were carried out at operating pressures consecutively increased from 1.38 to 5.87 MPa at different feed pH values (2.80 to 9.60). The total operation period was 50 min. The concentrate stream and the pass flow through the safety valve when the operation pressure was high (>4.83 MPa) were recycled to the feed tank continuously. Samples of the corresponding concentrate stream and permeate stream were collected simultaneously. Composition analysis was performed for the concentrate stream collected 2 min after the desired pressure was reached, as well as for the permeate samples. The fluoride ion was analyzed with an ion selective electrode (Orion Research, Model 710A/Digital Ion Analyzer); the solution pH was analyzed by the acid–base titration method. The concentration of ammonium ions (NH_4^+-NH_3) was determined by measuring the absorptivity (450 μm) of the complexes formed by reacting with Nessler solution ($2KI{\cdot}HgI_2$) after titration with NaOH(aq) to basic conditions. The uranium ion concentration was analyzed by polarographic measurement (EG & G Princeton Applied Research, Model 384B Polarographic Analyzer), while the alpha and beta activities were obtained with a Beckman Wide B-II.

The pH of the feed solutions was regulated by adding nitric acid or ammonia water. The flow rates and pressures were read from the flowmeters and the pressure gauge, respectively, for both the permeate and the concentrate stream.

RESULTS AND DISCUSSION

The results of a series of preliminary experiments on water purification with the RO system used in this study indicate that the system performance was very satisfactory. It is able to purify water with a conductivity of 2.01 and 0.017 Ω/m to as low as less than 0.0003 Ω/m. The experimental results for aqueous ammonium fluoride and UFE solutions are discussed below.

Separation Characteristics of Ammonium Fluoride

The main solutes in the UFE solutions are uranium compounds together with a large amount of ammonium fluorides. Treatment of the uranium-containing UFE can be accomplished if primary separation of the fluoride ions from UFE solutions is possible. In order to test the permselectivity of fluoride ions, the separation characteristics of ammonium fluoride were first studied by the current RO membrane process.

The solute rejection (ratio) is defined as

$$\% \text{ Rejection} = (C_c - C_p)/C_c \times 100\%$$

where C_c and C_p are the solute concentrations in the concentrate and permeate streams, respectively. The permeate flow rate F_p of the RO separation system can be expressed as

$$F_p = \Lambda(\Delta P - \Delta\pi)$$

where Λ is the permeability of water in the membrane, ΔP is the static pressure of the liquid (MPa), and $\Delta\pi$ is the osmotic pressure (MPa).

The separation results of ammonium fluoride solutions at low and high concentrations (F^-, 0.604 kg/m^3; NH_4^+, 1.116 kg/m^3; and F^-, 6.840 kg/m^3; NH_4^+, 7.920 kg/m^3) are shown in Tables 1 and 2 and Figs. 2 and 3. The permeate flow rates increase linearly as the pressure increases from 1.38 to 5.52 MPa, indicating that the permeability of Filmtec's FT-30 is independent of the operating pressure. At both concentrations, the F^- rejections are higher than 95.6% and increase in proportion to the operating pressure. The NH_4^+ rejections show a similar trend, although the rejection ratios are much lower than those of F^-. The increase in rejection

TABLE 1
Separation Results of Aqueous Ammonium Fluoride by RO Process[a]

		Consecutive operating pressure (MPa)							
		1.38		2.76		4.14		5.52	
	Feed	P	C	P	C	P	C	P	C
pH	9.20	10.9	9.2	10.9	9.2	10.9	9.2	10.9	9.2
F^- (kg/m^3)	0.604	0.004	0.745	0.003	0.771	0.002	0.804	0.002	0.836
NH_4^+ (kg/m^3)	1.116	0.360	1.098	0.342	1.134	0.340	1.134	0.342	1.188
Permeate flow (m^3/h)		0.022		0.035		0.047		0.060	
Concentrate flow (m^3/h)			0.614		0.601		0.590		0.576
F^- rejection (%)		99.4		99.6		99.6		99.7	
NH_4^+ rejection (%)		67.8		69.4		69.5		69.4	

[a] P = permeate; C = concentrate.

TABLE 2

Separation Results of Aqueous Ammonium Fluoride by RO Process[a]

		Consecutive operating pressure (MPa)							
		2.07		2.76		4.14		5.52	
	Feed	P	C	P	C	P	C	P	C
pH	8.80	10.0	8.80	10.0	8.80	10.0	8.80	10.0	8.80
F^- (kg/m^3)	6.840	0.304	7.581	0.226	7.904	0.125	7.904	0.104	7.999
NH_4^+ (kg/m^3)	7.920	1.134	8.514	1.044	8.406	0.954	8.622	0.900	8.784
Permeate flow (m^3/h)		0.008		0.015		0.028		0.041	
Concentrate flow (m^3/h)			0.629		0.621		0.608		0.595
F^- rejection (%)		95.6		96.7		98.2		98.5	
NH_4^+ rejection (%)		85.7		86.8		88.0		88.6	

[a] P = permeate; C = concentrate.

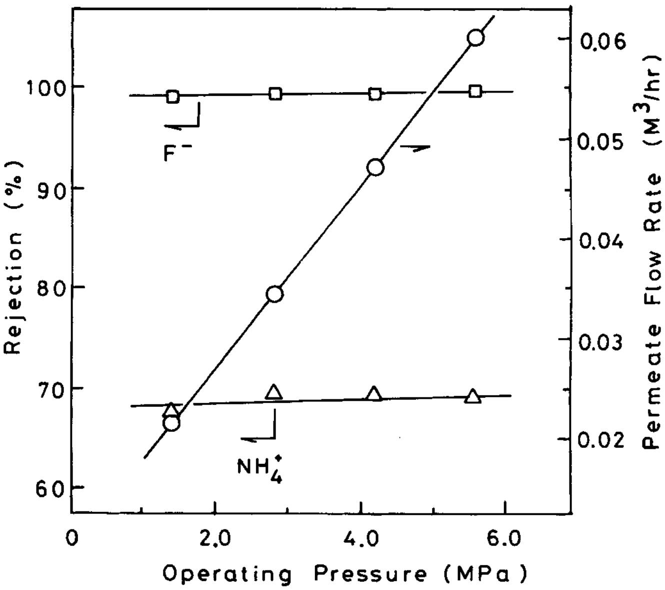

FIG. 2. Separation results of $NH_4F(aq)$ by the RO process (rejection and flow rate vs operating pressure at pH 9.2). Feed: $[F^-]$ = 0.604 kg/m^3, $[NH_4^+]$ = 1.116 kg/m^3.

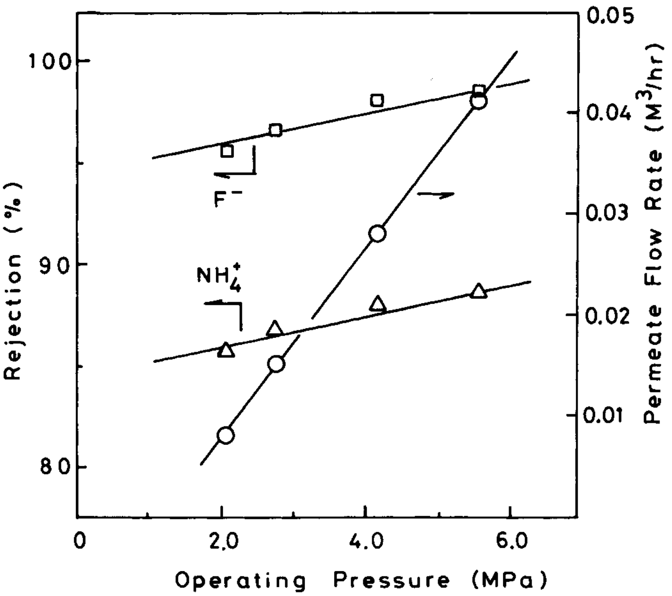

FIG. 3. Separation results of NH_4F(aq) by the RO process (rejection and flow rate vs operating pressure at pH 8.8). Feed: $[F^-] = 6.840$ kg/m³, $[NH_4^+] = 7.920$ kg/m³.

ratio with increasing operating pressure is expected because the effective pressure difference across the membrane is increased.

Furthermore, the differences of the rejection ratio of F^- between feed pH 9.20 and 8.80 are less than 4% (Tables 1 and 2), while those of NH_4^+ are greater than 17.4%. This result suggests that feed pH may play an important role in the rejection of NH_4^+ and F^-. Therefore, RO experiments were performed on ammonium fluoride solutions at different feed pH values to evaluate its influence.

The results of separation of ammonium fluoride solutions at different feed pH values are shown in Table 3. As expected, the rejection ratios of ammonium and fluoride show an opposite dependence on feed pH. The rejection ratios of both species are higher than 96% between pH 6.60 and 7.20, but the rejection ratios of ammonium and fluoride decrease abruptly at high and low feed pH values, respectively. For example, NH_4^+ rejection is only 44.2% at pH 9.60, while that of fluoride ions is 44.8% at pH 3.30. This result can be attributed to the selective permeation of NH_4^+-NH_3 or F^- through the membrane at different feed pH values. Under alkaline conditions, ammonia molecules, which are the predominant species, can easily be dissolved in the RO membrane matrix due to strong hydrogen-

TABLE 3
Effects of Feed pH on the Separation of Aqueous Ammonium Fluoride by RO Process[a,b,c]

	Feed pH											
	3.30		3.60		6.60		7.20		8.70		9.60	
	P	C	P	C	P	C	P	C	P	C	P	C
F^- (kg/m^3)	0.239 (0.433)	0.534	0.115 (0.434)	—	0.007 (0.433)	0.593	0.003 (0.456)	—	0.003 (0.513)	—	<0.001 (0.450)	—
NH_4^+ (kg/m^3)	0.054 (0.504)	0.558	0.036 (0.450)	—	0.018 (0.504)	0.594	0.018 (0.900)	—	0.198 (1.602)	—	0.864 (1.548)	—
Permeate flow (m^3/h)	0.061		0.060		0.059		0.060		0.060		0.061	
Concentrate flow (m^3/h)		0.575		0.576		0.577		0.576		0.576		0.575
F^- rejection (%)	44.8		73.5		98.4		99.4		99.5		99.9	
NH_4^+ rejection (%)	89.3		92.0		96.4		98.0		87.6		44.2	

[a] Feed flow rate, 0.636 m^3/h; temperature, 22°C; operating pressure, 5.52 MPa.

[b] P = permeate; C = concentrate.

[c] The values in parentheses are those of the corresponding feeds.

bonding formation with the membrane materials (*17*). The dissolution of ammonia in the membrane matrix results in decreased rejection ratios of the ammonium ions. On the contrary, fluoride ions show significant permeability under acidic conditions. The strong hydrogen-bonding of fluoride ions in acidic solution results in their enhanced solubility in the membrane, and hence the permeability of the fluoride ions is increased. The permeate flow rates remain at ca. 0.060 m^3/h at pH feed values of 3.30 to 9.60. This implies that variation of the feed pH does not affect the performance of the RO membrane and that Filmtec's FT-30 is stable in this pH range.

pH Effect on the Separation of UFE Solutions

The results mentioned above about the separation of ammonium fluoride solutions under acidic conditions seem to be applicable to UFE solutions whose main solutes are ammonium fluoride and uranium. The separation of the corrosive fluoride ions from uranium and NH_4^+ in UFE should be readily attained by pH control of the feeds.

Investigations were performed on various UFE solutions with different feed pH values under operating pressures of 3.80, 4.83, and 5.87 MPa. The fine particles present in the feed UFE were removed by the microfilter of the URM test unit; no particulate was observed either in the permeate stream or in the concentrate stream in any case.

The results of the separation of UFE solutions at feed pH values of 4.75 and 7.13 are described in Tables 4 and 5 and Figs. 4 and 5. The permeate flow rates increase linearly with increasing operating pressure at both feed pH values, indicating that Filmtec's FT-30 functions satisfactorily under these conditions. At a feed pH of 7.13, the rejection ratios of both F^- and U^{6+} are higher than 96%, while that of NH_4^+ decreases from 97.9 to 90.7% with increasing pressure from 3.80 to 5.87 MPa (Table 4 and Fig. 4). The decreasing NH_4^+ and F^- rejections are contrary to those observed in ammonium fluoride solutions (Figs. 2 and 3). This abnormal behavior in UFE solutions is not understandable currently. However, the α and β activities of the feed can be reduced from 48.8 and 14.2 ($\times 10^5$ Baq/m^3) to a level lower than 1.07 and 0.48 ($\times 10^5$ Baq/m^3), respectively.

The rejection ratios of U^{6+}, NH_4^+, and F^- at a feed pH value of 4.75 increase slightly with increasing operating pressure (Fig. 5), similar to those observed in ammonium fluoride (NH_4F) solutions. However, the differences of rejection ratios between U^{6+} and F^- are greater than 20%, indicating that fluoride ions can be separated from UFE solution under acidic conditions. In addition, the α and β activities of the feed can be reduced from 43.3 and 14.6 ($\times 10^5$ Baq/m^3) to a level lower than 3.03 and 2.85 ($\times 10^5$ Baq/m^3), respectively.

TABLE 4
Separation Results of UFE Solutions by RO Process[a]

		Consecutive operating pressure (MPa)					
		3.80		4.83		5.87	
	Feed	P	C	P	C	P	C
pH	7.13	6.70	7.45	6.90	7.50	7.05	7.50
U^{6+} (kg/m^3)	0.116	0.003	0.107	0.002	0.116	0.003	0.181
F^- (kg/m^3)	4.978	0.055	5.187	0.180	6.156	0.243	7.562
NH_4^+ (kg/m^3)	1.038	0.308	0.417	0.028	0.484	0.035	0.573
Permeate flow (m^3/h)		0.048		0.070		0.092	
Concentrate flow (m^3/h)			0.170		0.140		0.110
U^{6+} rejection (%)		97.4		98.1		97.4	
F^- rejection (%)		98.9		96.5		96.0	
NH_4^+ rejection (%)		97.9		92.5		90.7	
Activity,	α = 48.8	0.74	42.9	0.48	68.5	1.07	78.4
(Baq/m^3), $\times 10^5$	β = 14.2	<LLD[b]	13.5	<LLD	19.7	0.48	23.2

[a] P = permeate; C = concentrate.

[b] Lower limit of detection: $\alpha = 0.25 \times 10^5$ Baq/m^3; $\beta = 0.33 \times 10^5$ Baq/m^3.

TABLE 5
Separation Results of UFE Solutions by RO Process[a]

		Consecutive operating pressure (MPa)					
		3.80		4.83		5.87	
	Feed	P	C	P	C	P	C
pH	4.75	3.83	5.10	3.70	5.05	3.75	5.15
U^{6+} (kg/m^3)	0.101	0.010	0.164	0.004	0.179	0.006	0.208
F^- (kg/m^3)	4.408	1.535	6.099	1.499	6.764	1.488	7.961
NH_4^+ (N)	0.36	0.03	0.50	<0.03	0.57	0.03	0.70
Permeate flow (m^3/h)		0.070		0.086		0.100	
Concentrate flow (m^3/h)			0.147		0.117		0.090
U^{6+} rejection (%)		90.1		97.6		96.7	
F^- rejection (%)		65.2		75.4		78.0	
NH_4^+ rejection (%)		91.8		92.7		90.7	
Activity,	α = 43.3	3.03	37.4	2.41	58.5	2.33	68.1
(Baq/m^3), $\times 10^5$	β = 14.6	1.70	14.1	1.78	18.5	2.85	24.5

[a] P = permeate; C = concentrate.

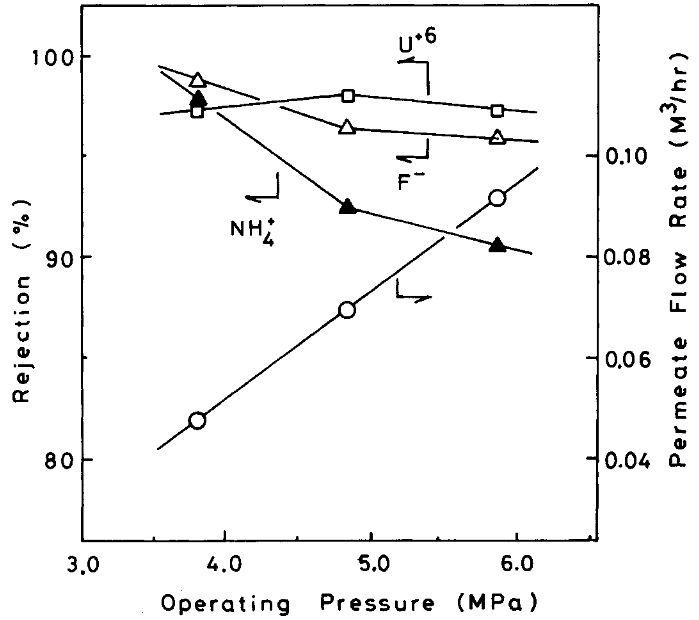

FIG. 4. Separation results of UFE by the RO process (rejection and flow rate vs operating pressure at pH 7.13). Feed: $[U^{6+}]$ = 0.116 kg/m³, $[F^-]$ = 4.978 kg/m³, $[NH_4^+]$ = 1.038 kg/m³.

The results of the separation of U^{6+} and F^- from UFE solutions at feed pH values of 2.80 to 7.13 are presented in Table 6 (and Fig. 6). The fluoride rejection, similar to that obtained in ammonium fluoride solutions, decreases significantly with decreasing feed pH. The rejection ratio is greater than 96% at pH 7.13, and then decreases quickly to 18.8% at pH 2.80. However, the uranium rejection does not change significantly in the same pH range and remains greater than 96.7%. The ammonium ion rejection remains at a level higher than 89.4%. In addition, the fluoride rejection increases slightly with increasing operating pressure, while U^{6+} rejection does not change greatly with pressure and remains higher than 90% (Fig. 6). In these pH ranges, the α and β activities of the permeates are lower than 2.41 and 3.37 ($\times 10^5$ Baq/m³), respectively.

As expected, the fluoride rejections in the treatment of UFE solutions are dependent on pH control, and they can be applied to the primary separation of corrosive fluoride ions from UFE by the RO membrane process at low feed pH. In such a case, regulation of the feed pH in UFE solutions is necessary. In consideration of the operating pH range of Filmtec's FT-30 module (pH 2.0–12), the best pH range would be between 2

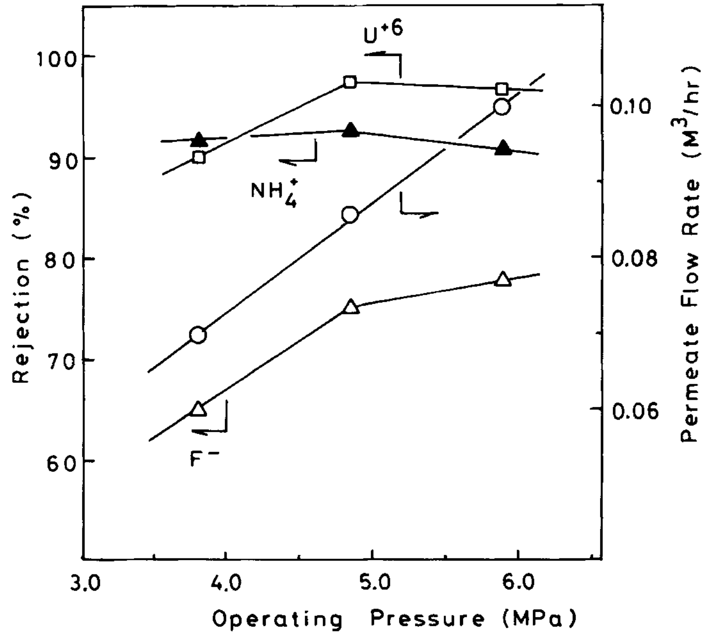

FIG. 5. Separation results of UFE by the RO process (rejection and flow rate vs operating pressure at pH 4.75). Feed: [U^{6+}] = 0.101 kg/m^3, [F^-] = 4.408 kg/m^3, [NH_4^+] = 0.36 *N*.

and 4, in which the differences of the rejection ratios between U^{6+} and F^- are greater than 67%.

Through a combination of the influence of pH control and pressure control on fluoride rejection, it is realized that higher permeability of fluoride ions is at a lower feed pH and a lower operating pressure range. However, the optimal feed pH and pressure in designing RO membrane separation processes depends on membrane stability and the required flow rate of the permeate stream.

CONCLUSION

It is possible to satisfactorily separate fluoride ions and U^{6+} from UFE solutions under acidic conditions with a continuous feedback RO process. For ammonium fluoride solutions, the NH_4^+ rejection decreases sharply from ca. 90 to 44.2% with the feed pH value increased from 3.30 to 9.60, while that of F^- increases abruptly from 44.8 to 99.9% with the same pH change. For UFE solutions, U^{6+} rejection remains greater than 90% at feed pH values of 2.80 to 7.13, while that of F^- decreases steadily from

TABLE 6
Effects of Feed pH on the Separation of UFE Solutions by RO Processes[a,b,c]

	Feed pH									
	2.80		3.95		4.75		6.80		7.13	
	P	C	P	C	P	C	P	C	P	C
F^- (kg/m^3)	2.869 (3.534)	3.325	3.116 (4.446)	5.263	1.499 (4.408)	6.764	0.403 (4.712)	7.467	0.180 (4.978)	6.156
U^{6+} (kg/m^3)	<0.001 (0.083)	0.153	0.004 (0.093)	0.180	0.004 (0.101)	0.179	0.003 (0.106)	0.178	0.002 (0.116)	0.116
NH_4^+ (kg/m^3)	0.036 (5.742)	8.154	0.540 (6.318)	10.566	0.486 (6.408)	10.188	0.684 (6.480)	9.882	0.504 (6.768)	8.712
Permeate flow (m^3/h)	0.070		0.086		0.086		0.090		0.070	
Concentrate flow (m^3/h)		1.40		0.110		0.117		0.120		0.140
U^{6+} rejection (%)	100		97.4		97.6		97.4		98.1	
F^- rejection (%)	18.8		29.9		66.0		91.4		96.4	
NH_4^+ rejection (%)	99.4		91.5		92.4		89.4		92.6	
Activity, (Baq/m^3), × 10^5 α =	<LLD[d] (40.0)	21.2	2.30 (20.4)	32.4	2.41 (43.3)	58.5	2.29 (53.7)	83.6	0.48 (48.8)	68.5
β =	<LLD (21.3)	33.3	3.37 (8.99)	16.30	1.78 (14.6)	18.50	1.07 (17.8)	25.1	<LLD (14.2)	19.7

[a] Feed flow rate, 0.210 m^3/h; temperature, 22°C; operating pressure, 4.83 MPa.

[b] P = permeate; C = concentrate.

[c] The values in parentheses are those of the corresponding feeds.

[d] Lower limit of detection: $\alpha = 0.25 \times 10^5$ Baq/m^3; $\beta = 0.33 \times 10^5$ Baq/m^3.

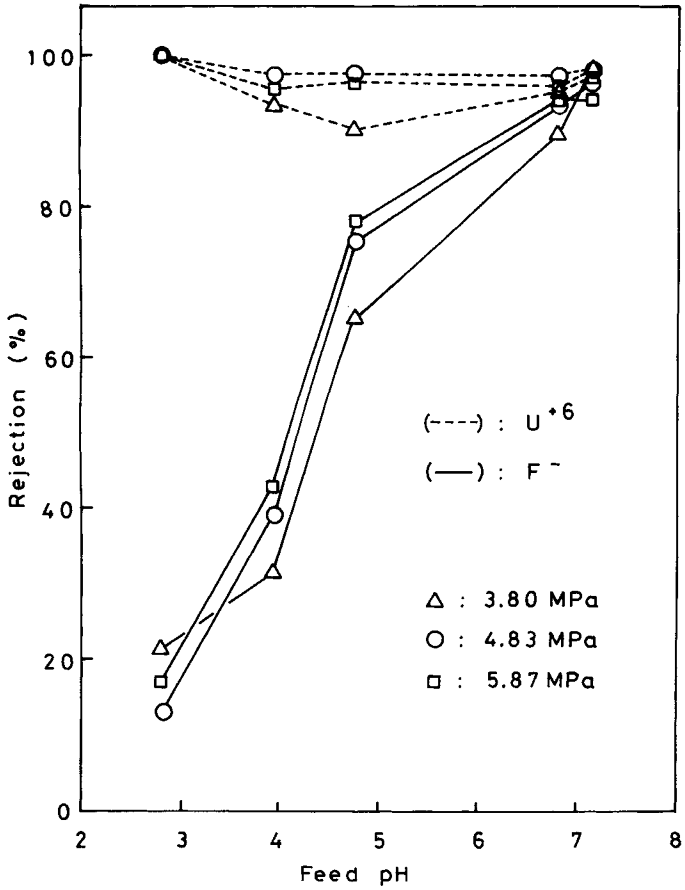

FIG. 6. Effects of feed pH on the separation of UFE by the RO process (rejection vs feed pH).

96.4 to 18.8% with decreasing feed pH. The change of solute rejections can be explained by the different solubilities of the solutes in the membrane at different feed pH values. This difference in solute rejections for U^{6+} and F^- between feed pH values of 2.80 and 7.13 can be applied to the primary separation of corrosive fluoride ions from UFE solutions. In addition, the α and β activities of the feed can be reduced from 20.4–53.7 and 8.99–21.3 ($\times 10^5$ baq/m^3) to a level lower than 2.41 and 3.37 ($\times 10^5$ Baq/m^3), respectively.

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