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論 文

Ion Exchange Phenomena of Cs^{+1}, Sr^{+2}, and Th^{+4} on Ion Exchange Resin in Loading and Elution Process

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=Abstract=

The ion exchange behaviour of the Cs^{+1}, Sr^{+2}, and Th^{+4} in the system of Cs^{+1}, Sr^{+2}, Th^{+4}, and $7Cl^{-}-H^{+}$ from Dowex HCR-W2, was examined in the loading and elution processes. Th^{+4} was slowly adsorbed through the entire contact time between resin and solution and Cs^{+1} and Sr^{+2} were adsorbed fast for the first few minutes of contact time. Because of the strong affinity of Th^{+4}, the longer contact time was allowed, the less amount of Cs^{+1} and Sr^{+2} was adsorbed on the resin. The peak concentration of the resin phase Cs^{+1} in the solution concentration of Cs^{+1}:Sr^{+2}:Th^{+4} in the ratio of 2 : 2 : 1 in normality with total normality of 0.1N was produced at about 4 minutes of contact time and the peak time for Sr^{+2} was 20 minutes.

The loaded ions were eluted using hydrochloric acid. The loaded Cs^{+1} was eluted at the low eluent concentration of less than 0.1N with less than 5% contamination of Sr^{+2}. The loaded Th^{+4} was eluted at the high eluent concentration of greater than 1N. The best eluent concentration for eluting Th^{+4} was 4N.

1. INTRODUCTION

At the tail-end process of high level nuclear liquid waste discharged from spent nuclear fuel reprocessing operations, the ion exchange process is currently used to recover useful radioactive nuclides and special alloy elements [1, 2]. For the removal of the contaminated radioactive isotopes in the reactor coolant and for the purification of the spent fuel storage pool water, the ion exchange process is also employed.

To effectively recover and purify or remove the ions from the waste stream, the process device must be effectively designed.

To achieve this goal, several design parameters must be known[3]. In particular, the behaviour of ions to be analyzed on the ion exchange resin of choice are most important among other parameters because they are effective on the design parameters[4]. In a case of the system of mixing high and low valence ions or slow and fast diffusing ions as encountered in the process of nuclear waste, the ion exchange mechanism is so complicate that the behaviour of the ions in such a system should be extensively examined to obtain more correct design parameters.

In this paper, to examine the behaviour of high and low valence ions or slow and fast diffusing ions in their mixture on the resin in the loading and elution processes, a simulated LWR nuclear waste which contains Cs^{+1}, Sr^{+2}, and Th^{+4}(replaced actinide and lanthanide elements) in chloride solution was analyzed. The ion exchange resin used was the cation exchanger Dowex HCR-W2 in hydrogen form.

2. GENERAL CONCEPTS OF ION EXCHANGE MECHANISM OF Cs^{+1}, Sr^{+2}, AND Th^{+4} ON H^+ FORM RESIN

The exchange capability of an ion in solution (solution phase ion) is dependent on the affinity of the ion for the resin. When the affinity of the solution phase ion is greater than that of the counter ion which is initially present in the ion exchange resin (resin phase ion), the exchange occurs. While the ion initially in solution diffuses into the resin, the exchanged ion migrates out of the resin into solution. In this study, hydrogen ion was chosen as the counter ion initially present on the resin.

With respect to the affinities of the ions for the resin, the affinity sequence of the ions analyzed in this study is in the following order[2].

$$H^{+1} < Cs^{+1} < Sr^{+2} < Th^{+4}$$

Following the affinity sequence of the ions, the mechanisms of ion exchange between the resin and solution phase can be expressed by several ion exchange equilibrium equations. For this system, the equilibrium equations are:

$$\text{a) } HR + \left[\begin{matrix} Cs^{+1} \\ Sr^{+2} \\ Th^{+4} \end{matrix}\right]_s \rightleftharpoons \left[\begin{matrix} Cs^{+1} \\ Sr^{+2} \\ Th^{+4} \end{matrix}\right] R + \left[\begin{matrix} H^{+} \end{matrix}\right]_s$$

$$\text{b) } CsR + \left[\begin{matrix} Sr^{+} \\ \\ Th^{+4} \end{matrix}\right]_s \rightleftharpoons \left[\begin{matrix} Sr^{+2} \\ \\ Th^{+4} \end{matrix}\right] R + \left[\begin{matrix} Cs^{+1} \end{matrix}\right]_s \quad (1)$$

$$\text{c) } SrR + [Th^{+4}]_s \rightleftharpoons [Th^{+5}]R + [Sr^{+2}]_s$$

where R = resin matrix subscript
s = solution phase.

Initially, when the resin comes into contact with the solution, the ion exchange reaction occurs with the ion exchange reaction (Eqn. (1)) moving to the right. As the resin sorbs the Cs^{+1}, Sr^{+2}, and the resin phase ions increase a reversible ion exchange occurs and finally, the equilibrium between the two phases is reached. Since Th^{+4} has the strongest affinity, the resin phase Cs^{+1}, Sr^{+2} may be replaced by Th^{+4} as noted in Eqn. (1).

3. MATERIAL

a) Preparation of Resin

The ion exchange resin used was Dowex HCR-W2 which was obtained from the Dow Chemical Company, St. Louis, Missouri, U.S.A., Dowex HCR-W2 is the new trade name for Dowex 50W-X8 with resin particles in the range of 20 to 50 mesh. The properties of Dowex HCR-W2 are summerized in Table 1.

Dowex HCR-W2, as received, was in the wet hydrogen form. To insure that the resin was completely converted to the H^+ form, the resin was preconditioned in a glass chromatographic column by washing with 5% hydrochloric acid[5]. After hydrochloric acid preconditioning, the resin was again washed with demineralized water.

b) Preparation of Solution

All chemicals used in this study were analyzed by the companies supplying them. They were all of more than 99% purity and used without any further analysis. The chemicals used were $SrCl_2 \cdot 6H_2O$, $CsCl$, $ThCl_4 \cdot 8H_2O$, HCl, $NaOH$, and Thorin.

Thorin was used for the detection of

Table 1. Ion exchange properties of dowex HCR-W2*

Type	Strong acid cation exchanger	
Active group	Nuclearsulfanic acid	
Standard crosslinkage	8% DVB	
Ionic form	H*	
Physical form	Hard spherical bead	
Standard mesh size	20~50 mesh	
Shipping density	50 lb/cu-ft	
Moisture content	50~55(52.5**)%	
Total exchange capacity dry resin	4.8(5.1**)meg/g dry resin	
wet resin	1.8(1.86**)meg/g wet resin	
Specific gravity	1.21(1.3**)g/ml	
Size distribution of wet swollen resin particies	mesh	%
	+16	0.6
	+20	37.0
	+30	46.0
	+35	13.0
	+40	2.3
	+50	0.8

* Cited from Dow Chemical Co. Report, Report No. T.D. Inde ×04

** Measured values

thorium. For the preparation of the solution, demineralized water was used. The resistance of the demineralized water was 10 to 15 megaohm/cm.

The solutions used were prepared by mixing the proper amount of chemicals with the correct amount of deionized water to obtain the desired solution normality and amount. To determine the Sr^{+2} and Cs^{+1} concentrations in the experimental solutions, the radioactive isotopes, Sr-85 and Cs-137 which were purchased from Nuclear England Nucleus, U.S.A., were used to "tag" the experimental solutions. The concentration of the radioactive material added to the solutions was about 0.03 μCi/ml for each isotope.

4. EXPERIMENTAL

To examine the adsorption phenomena, 11.75 grams of the centrifuged wet resin in the H form (total ion exchange capacity= 21.15 meq) prepared following the resin preparation procedure was contacted with 640 ml of a solution containing the ions of Cs^{+1}: Sr^{+2}:Th^{+4} in the ratio of 2 : 2 : 1 in a polyethylene bottle. The bottle was tightly covered and left unattended. Before taking a sample, the bottle was first well shaken and then, the samles were taken and analyzed.

The equilibrium concentrations of three ions due to varying total amount of Th^{+4} in the solution was examined. This was conducted by contacting the several bottles containing the same amount of fresh resin with the different amount of fresh solution gradually varying from 80 to 960 ml and in other series, 110 to 750 ml.

For the examination of elution phenomena, hydrochloric acid was used to elute Cs^{+1}, Sr^{+2}, and Th^{+4} from the loaded resin by the gradients elution technique [4, 6, 7, 8], The 5 ml of the loaded resin was prepared by equilibrating the fresh resin in the H^{+1} form with 120 ml of a solution with the Cs^{+1}:Sr^{+2}:Th^{+4} ions in the ratio of 1 : 1 : 3. After washing the loaded resin with demineralized water in a column, the loaded resin was placed in a burette and 40 ml of 0.001 N hydrochloric acid was introduced at the top of the burette with a flow rate of about 0.3 ml/min. After one aliquot eluent was passed, the effluent solution was analyzed. The hydrochloric acid concentration of next eluent were introduced and the effluent was analyzed. This process was repeated until 6N eluent was introduced. (The eluent of low concentration was first introduced to reduce the contamination of the solution due to the residual of the previuos contact.). This procedure was for the non-equilibrium elution process. The batch elution process was also evaluated by placing the same amount of the loaded resin in a flask and contacting it with 40ml of eluent. As in the column process, the elution process began with the lowest concentration of hydrochloric acid and then a higher concentration was subsequentially introduced. After two hour contact period, the solution was analyzed and drained from the resin. The resin was contacted again with the next higher concentration of eluent.

5. RESULTS AND DISSCUSION

Figure 1 shows the variation in the solution phase ion concentrations against contact time for an experiment involving a solution containing Cs^{+1}:Sr^{+2}:Th^{+4} ions in the ratio of 2 : 2 : 1 until the eqilibrium was reached. For the first few minutes of contact, all three ions, Cs^{+1}, Sr^{+2}, and Th^{+4}, were simultaneously adsorbed on the resin, but after about 4 minutes of contact, the adsorbed Cs^{+1} was found to be replaced by Sr^{+2} and Th^{+4}, by judging of incresing of the solution phase Cs^{+1} concentration. As the contact time elapsed, the solution phase Cs^{+1} concentration continuously increased due to the replacement of the previously adsorbed Cs^{+1} by the continuous adsorption of Sr^{+2} and Th^{+4}. After about 25 minutes of contact, the Sr^{+2} concentration in solution started to slightly increase due to being replaced by Th^{+4}, while Th^{+4} was found to be continuously adsorbed until the equilibrium was reached.

Comparing the ion concentrations at equilibrium and initial contact time, a small amount of the Cs^{+1} in the solution was adsorbed but most of the Th^{+4} was adsorbed. However, around 4 minute of contact time, the solution phase Cs^{+1} concentration was at minimum. Namely, the maximum amount of the solution phase Cs^{+1} was adsorbed. Therefore, to adsorb a large of Cs^{+1} in the mixture of Cs^{+1}, Sr^{+2}, and Th^{+4}, a short contact time is recommended[11].

As can be seen from Fig. 1, the adsorptions of Cs^{+1} and Sr^{+2} strongly depend on the adsorbed high valence ion Th^{+4}. This phenomenon can be well explained by judging Fig. 2 on which the data obtained from the equi-

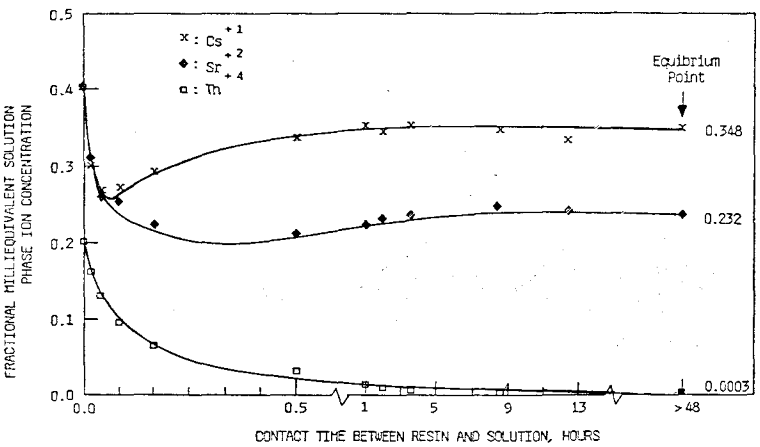

Fig. 1. Variations of solution phase ion concentrations as a function of contact time Cs^{+1}:Sr^{+2}:Th^{+4}=2 : 2 : 1, resin amount=5.62 grams of dry resin, solution=640ml, total solution normality=0.2N

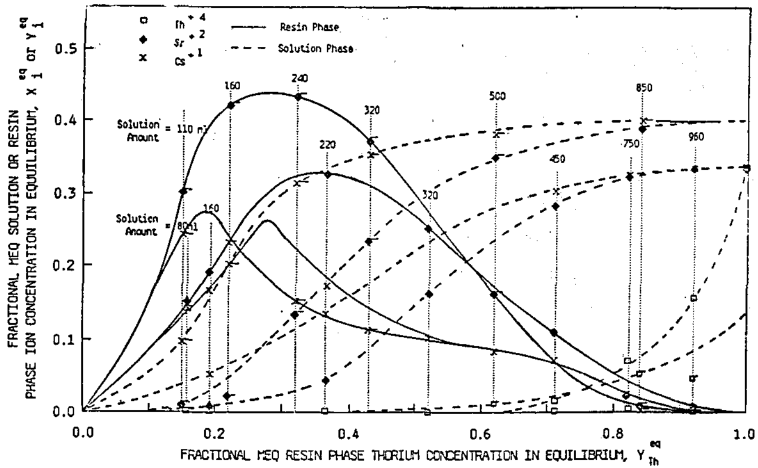

Fig. 2. Equilibrium curves, X_i^{eq}(i=Cs,Sr,Th) aud Y_i^{eq}(i=Cs,Sr) as a function of Y_{Th}^{e}. Untagged symbol: Cs:Sr:Th=1 : 1 : 1, tagged symbol: Cs:Sr:Th=2 : 2 : 1

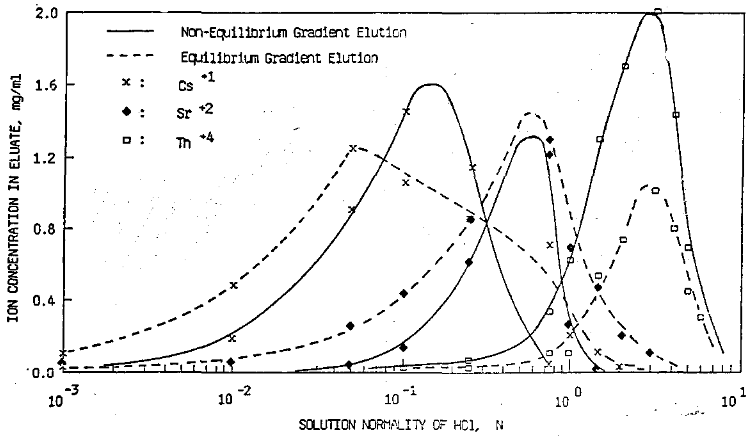

Fig. 3. Ion concentration in eluate as a function of normality of hydrochloric acid flow rate=40 ml/2hours. Resin was equilibriated in Cs:Sr:Th=1 : 1 : 3. Data were obtained passing 40 ml of eluent of the given solution normality through 5 ml of resin bed. Room temperature(78°F) was used. Equilibrium data ware obtained from batch process.

librium experiments were plotted with resin phase Cs^{+1} and Sr^{+2} and solution phase Cs^{+1}, Sr^{+2}, and Th^{+4} as a function of resin phase Th^{+4}. Fig. 2 shows the effects of amounts of Th^{+4} adsorbed on the resin to the resin phase CS^{+1} and Sr^{+1}, and Sr^{+2}. The vertical lines indicates the data obtained from one solution, i.e., at same equilibrium condition. As the volume of the solution increased, the concentration of the resin phase Th^{+4} increased, while the other resin phase ions increased and then decreased. These features are related to the affinities of the ions for the resin as described earlier. In the solution containing the small amount of Th^{+4}(small volume of solution), compared to the total capacity of the resin used, there was not enough Th^{+4} in the solution to replace the resin phase Cs^{+1} and Sr^{+2} so that high amounts of Cs^{+1} and Sr^{+2} remained in the resin. However, the large volume of solution (greater than~150 ml for Cs:Sr:Th=2 : 2 : 1, greater than~190 ml for Cs^{+1}: Sr^{+2}: Th^{+4}=1 : 1 : 1 from Fig. 2) contained enough amount of solution phase Th^{+4} to replace the resin phase Cs^{+1} and Sr^{+2} which replaced H^{+1} earlier and therefore, as the amount of Th^{+4} in the resin increased, the equilibrium amounts of the resin phase Cs^{+1} and Sr^{+2} increased and then decreased (Cs^{+1} more than Sr^{+2} because of the higher affinity of Sr^{+2}) by continueous adsorption of Th^{+4}. Consequently, the peaks were formed at the lower concentration of the resin phase Th^{+4} in two cases as shown in Fig. 2. The peaks of the resin phase Cs^{+1} and Sr^{+2} in case of Cs^{+1}:Sr^{+2}:Th^{+4}=2 : 2 : 1 were formed at lower concentration of the resin phase Th^{+4} than in the case of Cs^{+1}:Sr^{+2}:Th^{+4}=1 : 1 : 1. This feature are related to the affinities of the ions on the resin and also due to the law of mass action of different concentrations of the ions. Considering these ion exchange behaviours between the ions analyzed, if the acceptable short contact time between the resin and the solution is to be allowed, a relatively large amount of Cs^{+1} and Sr^{+2} could be sorbed on the resin.

The results of the elution processes are plotted as shown in Fig. 3. The figure shows two results of the non-equilibrium and equilibrium processes. In both cases, a large amount of Cs^{+1} was eluted compared with Sr^{+2} at the low concentration of eluent less than 0.1 N without eluting any Th^{+4}. But at high concentration of eluent greater than 1N, a large amount of Th^{+4} was eluted with a small amount of Sr^{+2} interfered and none of Cs^{+1}. Figure 3 shows the narrow gaps between the adjacent peaks in the adjacent peaks in the range of 0.1 to 1 N of the eluent concentration. This indicates it would be difficult to obtain nearly pure solution of one ion by the continuous gradient elution process[7]. At particular concentration of the eluent (stepwise

Table 2. Percent purity of ion in eluate predicted from Fig. 3

Solution Normality of Eluent(N)	% Purity					
	Column Process			Bathh Process		
	Cs^{+1}	Sr^{+2}	Th^{+4}	Cs^{+1}	Sr^{+2}	Th^{+4}
0.001	100	—	—	99	—	—
0.01	98	—	—	90	—	—
0.05	95	100	—	82	—	—
0.10	90	90	—	72	100	—
0.25	64	94	—	50	99	—
0.75	2	88	—	33	85	—
1.00	—	66	—	17	75	—
2.00	—	2	—	2	45	—
3.00	—	—	100	—	21	—
5.00	—	—	—	—	7	—
6.00	—	—	—	—	—	100
7.00	—	—	—	—	—	—

Notes: 1. %Purity was calculated using Eq(2).
2. An asssumption that more weakly held ions were completely eluted was used for calculating purities for Sr^{+2} and Th^{+4}.

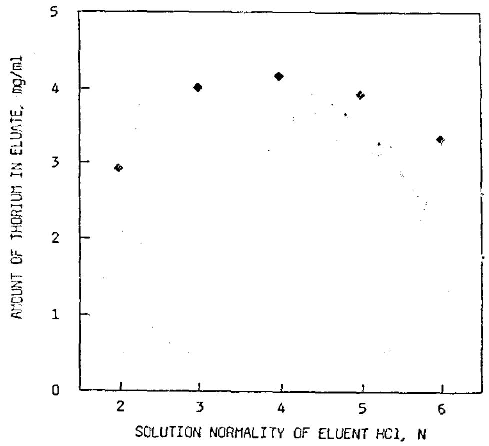

Fig. 4. Elution of thorium from Dowex HCR-H2 depending on the concentration of hydrochloric acid. Resin was initially loaded with thorium in one bottle. A small amount of the loaded resin(4 ml) was placed in 5 columns and then 40 ml of each of 5 different concentrated eluents was passed tnrough each columns.

elution process[7], however, it is possible to obtain the high purity of a particular ion.

Comparing the results of two eluton processes shown in Fig. 3, it can be seen that non-equilibrium process is better process to obtain high pure elements. In the non-equilibrium process, once the resin phase ions were eluted, they were removed out of the system, but in the equilibrium process, the eluted ions remained in the system so that they could elute other ions by mass action until the equilibrium was reachead. Eventually, the more amount of pure elements were obtained in the non-equilibrium process.

The purities of the eluted ions depending on the eluent concentrations obtained from Fig. 3 were shown in Table 2. The % purity of a particular ion was calculated as follows:

$$\% \text{ purity} = \frac{[\text{separated ion}]}{[\text{separated ion}] + [\text{contaminated ion}]} \times 100 \qquad (2)$$

where bracket denotes the solution concentration of ion in effluent.

The analysis of the Table 2 showes that the purities of the Cs^{+1} and Sr^{+2} increase with decreasing concentration of the eluent.

Since Th^{+4} has a very strong affinity for the resin, a strong eluent is needed. According to the literature[9, 10], 4 N hydrochloric acid starts to elute Th^{+4} from Dowex 50W-X8(for particle sizes of 100 to 200 mesh) and Th^{+4} elutes more rapidly, as the hydrochloric acid concentration increases. The literatures concluded that the largest amount of Th^{+4} elutes at the 6 N of hydrochloric acid. However, since a different resin particle size was used in this study, it was necessary to examine the elution strength for eluting Th^{+4}. Five different concentrations of hydrochloric acid(2 to 6N) were used with five different columns containing the same amount of loaded resin and Th^{+4} has been eluted for 2 hours with 30 ml of each eluent. The analysis of the eluent produced the results shown in Fig. 4. It can be seen from this Fig. 4 that 4 N hydrochloric acid produced the highest strength for eluting Th^{+4}. A better understanding of the effect of 4 N hydrochloric acid for eluting Th^{+4} needed another experiment. The loaded resin was placed in a burette and 4 N HCl eluent was continuously introduced at the top of the burette with a flow rate of 0.2 ml/min. Samples of the effluent were periodically taken and analyzed. The same experiment procedure using 6 N HCl eluent was conducted. The results are shown in Fig. 5. The Fig. shows that 70% of the total

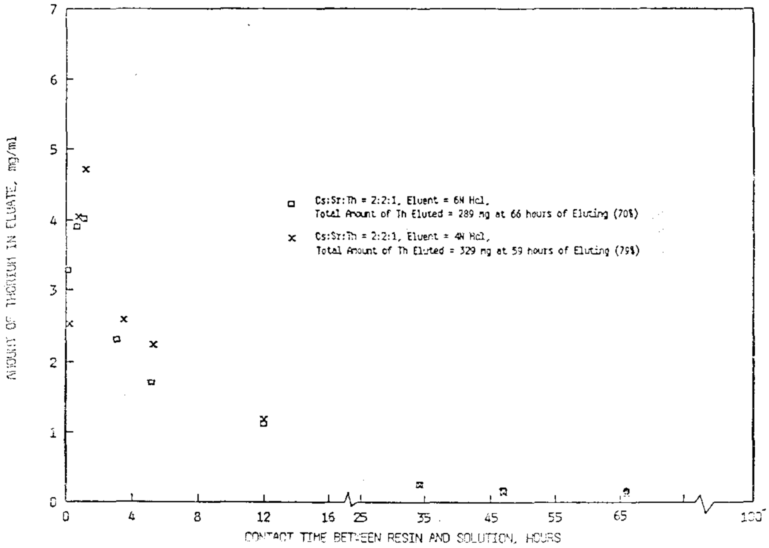

Fig. 5. Comparison of amount of thorium eluted by 6 N and 4 N HCl

amount of the Th^{+4} on the resin was removed in 66 hours using 6 N HCl, while 79% was removed in 59 hours using 4 N HCl. Thus, the 4 N HCl produced greater removal of Th^{+4} in the shorter time. From these experimental results, it can be seen that the 4 N HCl is a sufficient eluent concentration and produces the highest speed for eluting Th^{+4} from Dowex HCR-W2. Fig. 5 shows another important result, i.e., the elution process for Th^{+4} from the resin using hydrochloric acid as an eluent is very slow and, to elute about all of the Th^{+4} from the resin, a long contact time(about 100 hours in this experiment, extrapolately obtained from Fig. 5) should be allowed.

6. CONCLUSION

The ion exchange behaviour of the Cs^{+1}, Sr^{+2}, and Th^{+4} on the ion exchange resin, Dowex HCR-W2, was examined in the loading and elution processes. The examinations showed that Th^{+4} adsorbed slowly on the resin, while the Cs^{+1} and Sr^{+2} adsorbed relatively fast and that the loaded Cs^{+1} and Sr^{+2} were released from the resin after a period of contact time between the resin and solution to produce peaks in their resin phase concentrations. As it has the weakest affinity on the resin, the loaded Cs^{+1} was replaced by both Sr^{+2} and Th^{+2} and therefore the peak of the resin phase Cs^{+1} concentration was produced at earlier contact time than when the resin phase Sr^{+2} peak was producted. Consquently, to recover or to remove more large amount of Cs^{+1} or Sr^{+2} in one batch process, an acceptable short contact time is better than long contact time. The best contact time determined was 4 minutes for Cs^{+1} and 20 minutes for Sr^{+2}.

The elution examinations using non-equilibrium and equilibrium processes with hydrochloric acid in the range of 0.001 to 6 N showed that Cs^{+1} was eluted at the low concentration of eluent less than 0.1 N with slight contamination of only Sr^{+2} and that at high concentration greater than 1 N, Th^{+4} was eluted with only Sr^{+2} slightly contaminated. Such results were about same in both non-equilibrium and equiliqrium processes, but the amounts of the eluted ions were slightly greater in the non-equilibrium proces than in the equilibrium process. Since the elution curves showed the narrow gaps between the adjacent peaks, to obtain high pure elements, a stepwise elution process is recommended wth less than 0.01 N eluent for Cs^{+1} and 0.25 N for Sr^{+2}. The elution concentration of 4 N to elute Th^{+4} was determined for the best eultion process and 10 hours of contact time for elution Th^{+4} is recommended to elute about two thirds of the ta t loaded Th^{+4}.

From these results, some predictions for ion exchange chemical process of nuclear waste can be made; Recovery or removal of low valence fission product ions needs an acceptable short contact time between resin and waste solution, and long contact time is necessry for actinide and lanthanide elements. Batch process is best for recovering or removing actinides and lanthanides.

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Cs^{+1}, Sr^{+2}와 Th^{+4}가 동시에 이온교환수지에 흡착 및 탈착시의 이온교환현상

박종목

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=요 약=

이온교환시스템, Cs^{+1}, Sr^{+2}, Th^{+4}, and $7Cl^{-}-H^{+}$ Dowex HCR-W2,에서 Cs^{+1}, Sr^{+2},이온들이 이온수지 Dowex HCR-W2에 흡착분리될 때의 이온교환 거동에 대하여 연구했다. Th^{+4}이온은 느린 속도의 흡착을 보여 주는 반면에 Cs^{+1}과 Sr^{+2}이온은 대체로 빠른 속도로 흡착되었다. Th^{+4}이온의 강한 흡착력 때문에 수지와 용액의 접촉시간이 길면 이미 흡착된 Cs^{+1}과 Sr^{+2}이온은 다시 수지로 부터 분리되어 용액쪽으로 나오기 때문에 소량의 Cs^{+1}과 Sr^{+2}이온이 용액으로부터 회수 또는 제거되었다. 용액 전체 노르말이 0.1 N 이고 Cs^{+1}: Sr^{+2}: Th^{+4}의 각각의 노르말비가 2 : 2 : 1의 용액인 경우 Cs^{+1}이 최대로 흡착되는 접촉시간은 4분정도이며, Sr^{+2}이온의 흡착정도는 20분정도에서 최대가 되었다.

흡착된 이온을 수지로부터 분리하는데 HCl 용액을 사용했으며 Cs^{+1}이온은 0.1 N 이하의 농도에서 분리가 잘 되었고(Sr^{+2}의 오염이 5%이하였다.) Th^{+4}이온은 1 N 이상의 농도에서 분리가 되는데, 가장 효과적으로 분리되는 HCl 의 농도는 4 N 이었다.