Recovery Tests for Analysis of Radionuclides in Spent Resin Using Elution and Microwave Digestion Method

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1. Introduction

Ion-exchange resins are widely used in the nuclear power plant for purification of various aqueous streams. As spent resins are the relatively high activity, an evaluation of radionuclide inventories should be carried out to permanently dispose these resins based on the Korean Nuclear Safety and Security Commission (NSSC) Notice No. 2015-4, in which the acceptance criteria for LILWs are specified. Elution and microwave digestion method was tested for recovery tests of radionuclides in spent ion-exchange resins.

Elution method using a column is a process that the radioactive nuclide is eluted from the resins using the eluent. However, the former is complicated for the control of flow rate and produces secondary waste such as column material. The spent resins are difficult to achieve standard requirement after elution, so it is used to treat some low level radioactive spent resin [1]. Elution method needs concentration process because of much of eluent used.

Closed vessel microwave digestion system offers several distinct advantages such as lower acid consumption, avoiding the loss of volatile elements, rapid reaction time. However, closed microwave digestion system has only one disadvantage, namely, explosion and cracking of digestion tubes due to simultaneous build-up of pressure along with temperature increase.

In this study, we compared recovery tests in fresh ion-exchange resin between elution method and closed vessel microwave digestion method.

2. Experimental

2.1 Preparation

For evaluation of recovery tests, the fresh IRN-150 resin was used. IRN-150 (Amberite^{TM}) is 1:1 mixtures of IRN-77 and IRN-78. The IRN-77 is a strongly acidic cation resin and IRN-78 is a strongly basic anion resin. Approximately 1 g of the sample was dried in an oven at 60℃ for 24 hr in a beaker. After cool, 20 mL distilled de-mineralized water (DDW) was added to the beaker and 0.5 mL of each of Ni, Nb, Re, and Sr (10 mg/L) were added. 12 hr waited for the adsorption of metallic ions, and the ion-exchange resins was separated by a filter paper with 25 μm pore size. The concentration of each element in solution was determined using ICP-AES to know the amount of each element being adsorbed in IRN-150.

2.2 Elution method

A column (D 0.7 cm) was plugged at the bottom with a polyethylene bed support. The column was packed with 1 g of ion-exchange resin with soft tapping to ensure dense packing, and a bed support was packed with a bed support on top of the resin bed for prevention of disturbance during addition of the eluent. The resin bed height was adjusted to 5 cm. Ten mL of DDW, 50 mL of 4 M HCl, 50 mL of 10 M HNO_3, 50 mL of 5 M HNO_3 including 0.2 M HF, and 10 mL of DDW was transferred to the column at a flow rate of 1 mL/min, respectively. The concentration of each element in the elution was determined using ICP-AES.

2.3 Closed vessel microwave digestion system

For recovery test, about 1 g of ion-exchange resin was used to desorb the metal ions in the resin adsorbed using a microwave digestion system (MDS) including mixed acid (10 mL of HNO_3, 4 mL of HCl, 0.25 mL of HF). After operation of MDS, the solution was transferred to a volumetric flask. The concentration of each element in the solution was determined using ICP-AES. Fig. 1 shows elution method and closed microwave digestion system in the glove box.

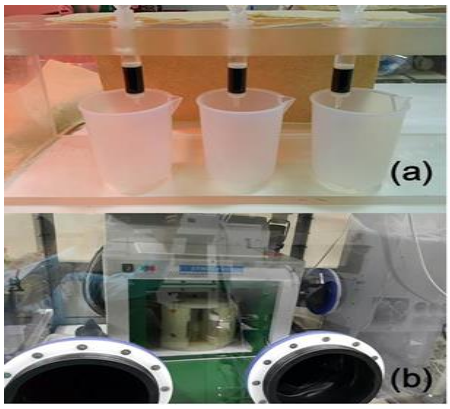

Fig. 1. Elution method (a) and closed vessel microwave digestion system (b).

3. Results & discussion

Fig. 2 shows the result of recovery between elution method and microwave digestion method. The mean recovery of 4 elements for the microwave digestion system was 98.1±2.8%. The recovery of Re and Nb for microwave digestion method was 97.1±3.1% and 94.8±6.3%, respectively.

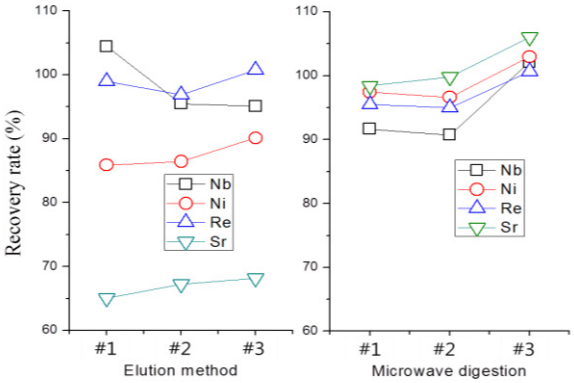

Fig. 2. Comparison of recovery between elution method and microwave digestion method.

However, the average recovery of 4 elements for elution method was 87.9±15.0%. The recovery of Re and Nb for elution method was 98.9±2.0% and 98.3±5.3%, respectively. Especially, although the cause of low recovery for Sr is unknown, the recovery of Sr using elution method was very low as 66.8±1.6%. The recovery of Re and Nb for microwave digestion method was similar to that of the elution method. A standard deviation of both methods was less than 5% except Nb.

4. Conclusion

Nb, Ni, Re, and Sr in the resin adsorbed using the microwave digestion method were quantitatively recovered. The results of microwave digestion method were superior to those of the elution method. Further study will be performed to investigate the cause of low recovery of Sr using elution method.

REFERENCES

[1] Jianlong Wang, Zhong Wan “Treatment and disposal of spent radioactive ion-exchange resins in the nuclear industry”, Progress in Nuclear Energy, 78, 47-55, (2015).