Preliminary Test of ^{55}Fe and ^{63}Ni Analysis Method in Concrete Sample

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1. Introduction

The radiochemical analysis is an essential necessity for the management and disposal of radioactive wastes. Both ^{55}Fe and ^{63}Ni are neutron activation products. ^{55}Fe ($t_{1/2}$=2.7y) decays via electron capture to stable ^{55}Mn with the emission of Auger electrons and low energy X-rays (5.89 keV, 16.9%). ^{63}Ni is a pure beta emitting radionuclide with maximum beta energy of 66.95 keV and half life of 100.1 y. Due to low beta energy emitted from ^{55}Fe and ^{63}Ni, both have to be separated completely from other radionuclides before the counting. In this study, a sample procedure based on anion exchange is investigated for the simultaneous determination of ^{55}Fe and ^{63}Ni. The recovery rate of stable isotope Fe and Ni were also confirmed for the standard solution and concrete samples.

2. Experimental

2.1 Equipment and chemicals

Inductively coupled plasma optical emission spectrometry (ICP-OES, Teledyne leeman laboratory Inc, Prodigy) is used for the determination of stable Fe and Ni. The automatic fusion system (K2 prime, Katanax) is used to melt the concrete samples. Fe and Ni standard solution was purchased from the AccuSandard, Inc., (Accutrace reference standard, MES-16-1). This standard solution contains stable isotopes such as Ca, Co, Cu, Pb, etc. In order to primarily separate Fe and Ni, Bio-Rad anion exchange resin was purchased from Bio-Rad laboratory(USA).

2.2 Preparation of samples

The stable isotope Fe and Ni carrier are 25 mg and 2 mg, respectively, and 1mL of cobalt standard (1,000ug/mL) is added to the sample. The prepared sample is adjusted to pH 8~9 using 8M NaOH. The precipitate is separated by centrifugation, and then dissolved with concentrated nitric acid, transferred to a teflon beaker and evaporated to dryness. This sample is dissolved with 10 mL of 9M HCl solution to prepare a sample for separation.

2.3 Separation of Fe and Ni from interfering radionuclides by anion exchange chromatography

The solution is then loaded to a Bio-Rad AG 1x4 anion exchange column, which has been conditioned with 20mL of 9M HCl solution. 9M HCl is added to elute Ni^{2+} and wash the column, 6M HCl is added to elute Co^{2+} and Cu^{2+}, finally DIW(or 0.05M HCl) is added to elute Fe^{3+}. The effluent, washes and eluate are collected in polyethylene vials of 2mL each. These vials are then measured by ICP-OES.

2.4 Applied to concrete samples

Concrete samples are completely melted by automatic fusion system and then separated according to 2.3.

2.5 Determination of Fe and Ni by ICP-OES

2mL of each eluate in accordance with 2.3 was diluted and the concentration was measured with ICP-OES. Then the recovery rate was calculated.

3. Results and discussion

In a concentrated HCl solution, many metals form anion complexes with Cl^-, and will therefore be absorbed on an anion exchange resin. Depending on the stabilities of the metal chloride anion complexes in different concentration of HCl, Fe, Ni and other metals can be separated by anion exchange chromatography. Fig. 1 shows the eluting curves of Fe, Ni and other metals on an anion exchange column. Ni^{2+} does not absorb on the column even in 9M HCl, so Ni can be separated from Co^{2+} and Fe^{3+} very well. Fe^{3+} retained on the column can be isolated by elution of Cu^{2+}, Co^{2+}, etc., with 6M HCl, and then eluting Fe with DIW.

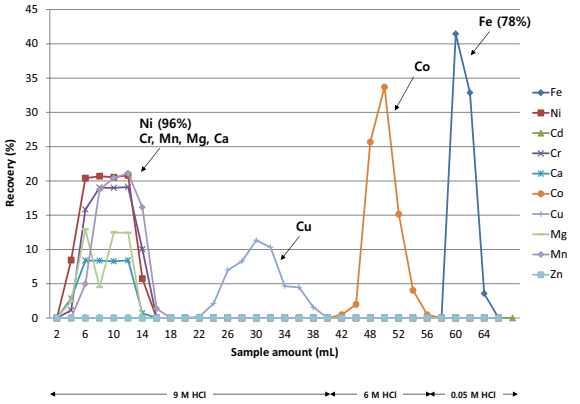

Fig. 1. Eluting curves on anion exchange column.

4. Conclusion

The Fe and Ni were separated each other by an anion exchange chromatography and the recovery rate of Fe and Ni are higher than 90%, 80%, respectively.

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