2 - 36 Simultaneous Determination of U and Nd by Binary Ration and X-ray Fluorescence Spectrometry

Fan Fangli and Qin Zhi

X-ray fluorescence spectrometry is a powerful tool for the determination of elements in a wide variety of sample matrices[1]. It is a relatively fast method of analyzing the elemental composition of a sample. Specimens placed in the instrument can be in the form of either solids or liquids, and the specimen is neither consumed nor destroyed during the analysis[2]. In the present work, a rapid method for determination of U and Nd in simulative spent fuel by binary ration and X-ray fluorescence spectrometry was developed. The determination of U and Ce in simulative MOX fuel samples by binary intensity ratio and X-ray fluorescence spectrometry has been studied[3].

The method of binary intensity ratio determine the amount of each element by measuring the net intensity of a line of each constituent and making a log-log plot of net intensity ratio *vs.* concentration ratio of each constituent. The relation of the net intensity and the concentration of element is as follows:

$$\log[I(A)/I(B)] = \log K + n\log[C(A)/C(B)] \ ,$$

where I and C are the intensity of a line and the mass concentration of each element, respectively.

Firstly, the calibration curve was established according to the above relation. The standard samples were prepared from U_3O_8 and Nd_2O_3 powders by weighing in suitable proportions and mixing in a Mixer Mill. The U_3O_8-Nd_2O_3 mixtures may be regarded as binary for this study because of the virtually negligible effect of oxygen. The measurement condition by X-ray fluorescence spectrometry in our experiment shows in Table 1. Fig. 1 shows the calibration curve with binary ration for U_3O_8-Nd_2O_3 mixtures, and R^2 is greater than 0.999. Then the amount of each element in the test samples of U_3O_8-Nd_2O_3 mixtures can be deduced by the calibration curve.

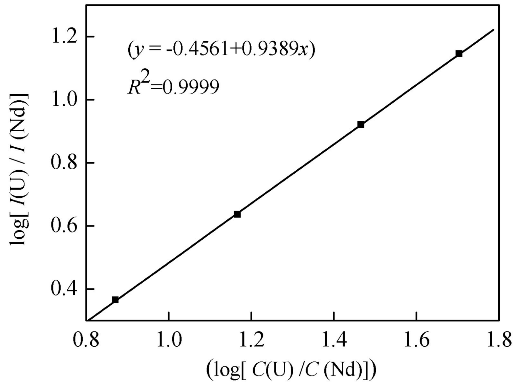

Fig. 1 The calibration curve with binary ration for U_3O_8-Nd_2O_3mixtures.

Table 1 Measurement condition for analytical elements.

Element	Analytical line	Excite condition		Detector	Measuring time / s
		U / kV	I / μA		
U	U La	30	15	CdTe	120
Nd	Nd La	30	15	CdTe	120

References

[1] E. P. Bertin, Anal Chem, 36(1964)826

[2] E. B. Buchanan, Jr. Foo-Chong Tsai, Analytical chemistry, 46(1974)1701.

[3] You Song, Weiming Zheng, GuiJiao Liu, Journal of nuclear and radiochemistry, 27(2005)7.

2 - 37 Desorption of Uranium from Amidoximed Silica

Yin Xiaojie and Bai Jing

Previous work suggested that amidoximed silica can be used as a highly efficient adsorbent for uranium removal from salt lake water. In this work, desorption of uranium from amidxoximed silica was investigated.

Amidoximed silica, which were loaded with uranium(VI) from salt lake water were used for desorption studies. 0.1 mol / L of HNO_3 Na_2CO_3, $NaHCO_3$ and NaOH solution were used as desorbing solutions, respectively. The uranium desorbed into the solution was determined after shaking for certain time. Desorption efficiency was calculated directly from the difference between the desorbed and absorbed uranium. The desorption ratio (%) was calculated by the following equation:

$$\text{Desorption}(\%) = \frac{\text{Amount of } U(\text{VI}) \text{ desorbed}}{\text{Amount of } U(\text{VI}) \text{ adsorbed}} \times 100\%$$

As it is shown in Table 1, HNO_3 was the best desorption reagent and the influence of HNO_3 concentration for desorption was also investigated. The results suggested that HNO_3 concentration had no obvious influence on uranium desorption when it is greater than 0.1 mol/L. 0.1 mol/L HNO_3 was the best desorption solution for uranium desorption with desorption ratio of 62%. More efficient desorption reagent is desired in the future work.

Table 1 Desorption of uranium with different desorbing agents.

Agent	Concentration / (mol/L)	Desorption / %
HNO_3	0.05	54.07
HNO_3	0.10	62.10
HNO_3	0.25	62.86
HNO_3	0.50	61.78
Na_2HCO_3	0.10	38.78
Na_2CO_3	0.10	4.89
NaOH	0.10	6.06

2 - 38 Separation of Rare Elements from Spent Nuclear Fuel

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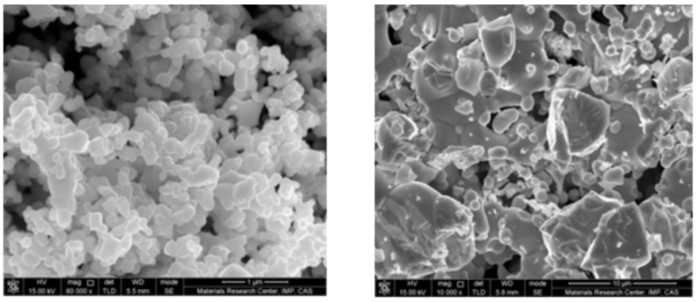
Rare-earth (RE) elements (La, Ce, Pr, Nd, Pm, Sm, Eu, Gd and others) are produced in nuclear fuel by uranium fission and by the decay of other fission products. Rare-earth (RE) elements in spent nuclear fuel are strong neutron absorbers, which affect the fission efficiency of fissile materials and decrease the thermal conductivity of the UO2 matrix. Thus, to enhance the thermal conductivity and nuclear energy production of reused fuel, the separation of RE elements from spent nuclear fuel is very necessary.

In the present work, the separation of rare-earth elements from simulated spent fuel by high temperature oxidation was investigated. Samples of U_3O_8 doped with varying quantities of RE (where RE was Nd or Sm) were prepared by the co-precipitation method. Ammonium hydroxide (25%) was used to co-precipitate neodymium and uranium from a solution of their nitrates. The resulting Nd-doped ammonium diuranate (ADU) was collected by centrifuging, and then was washed several times with dilute ammonium hydroxide before being air-dried at 100 °C. The ADU powders were then heated in air at 900 °C for 4 h to covert then to U_3O_8. The resulting $(U_{1-x}Nd_x)_3O_8$ powder as the starting material were carried out the heat treatment experiment in the temperature range between 1 000 °C and 1 700 °C for 6 h in air. Finally, the thermally treated powders were cooled to room temperature in air.



（a） 900 ºC 4 h (b) 1300 ºC 6 h

Fig. 1 (color online) SEM of the powders produced by the heat treatment of the $(U_{1-x}Nd_x)_3O_8$.

The samples after heat treatment were analyzed by SEM and XRD. Fig. 1 shows the SEM micrographs of the powders produced by the heat treatment of the $(U_{1-x}Nd_x)_3O_8$. From Fig. 1(a), one can see that the uniform small particles appeared in the starting material $(U_{1-x}Nd_x)_3O_8$. But after heat at 1 300 °C, the phase changed into two different particles. The small particles named as "rich-rare phase", $(U_{1-y}Nd_y)O_{2+v}$ particles were observed and most