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Osamu Tomioka[a]; Youichi Enokida[a]; Ichiro Yamamoto[a]
[a] Department of Nuclear Engineering, Nagoya University, Nagoya, Japan

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SELECTIVE RECOVERY OF NEODYMIUM FROM OXIDES BY DIRECT EXTRACTION METHOD WITH SUPERCRITICAL CO_2 CONTAINING TBP–HNO_3 COMPLEX

**Osamu Tomioka, Youichi Enokida,* and
Ichiro Yamamoto**

Department of Nuclear Engineering, Nagoya University,
Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

ABSTRACT

A method for the direct extraction of a metal utilizing supercritical CO_2 containing tri-*n*-butylphosphate (TBP)–HNO_3 (TBP), which is the TBP solution of TBP–HNO_3 complex, has been developed for the recovery of Nd from its oxide. This method was applied using a flow reactor to several oxides (Nd_2O_3, ZrO_2, MoO_3, and RuO_2), and binary mixtures of the oxides (Nd_2O_3–ZrO_2, Nd_2O_3–MoO_3, and Nd_2O_3–RuO_2). Neodymium (Nd) was extracted almost quantitatively from 0.01 mol Nd_2O_3 powder with supercritical CO_2 containing TBP–HNO_3 (TBP) at 313K and 12 MPa, while Zr, Mo, and Ru were hardly extracted from their oxides and remained in the reactor. Nd was extracted quantitatively and selectively also from the binary mixtures of the oxides. From these results, it is found that the supercritical CO_2 extraction process using TBP–HNO_3 (TBP) as reactant is feasible for the selective recovery of lanthanides directly from the various oxide mixtures.

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*Corresponding author. Fax: +81 (52) 789-3785; E-mail: yenokida@nucl.nagoya-u.ac.jp

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Key Words: Supercritical carbon dioxide; Extraction; Lanthanide; Oxide; HNO_3–tri-*n*-butylphosphate complex

INTRODUCTION

Conventional solvent-extraction processes generate a large amount of organic solvent wastes. Supercritical CO_2 is one of the most promising substitutes as a diluent for the minimization of the amount of the solvent wastes, since CO_2 can be removed readily from the extractant and the extracted substances by gasification under atmospheric pressure and ambient temperature, and can be recycled in the process (1–4). CO_2 has other attractive properties such as being nontoxic and stable, chemically and radiochemically. Additionally, controlling the pressure and/or temperature can enhance the extraction efficiency. These advantages are important for the chemical separation process in the field of nuclear technology dealing with various radioactive elements.

There are a number of research papers and several review articles published regarding supercritical fluid extraction (SFE) of metal ions and metal oxides. The charge neutralization of the ion is required to enhance significantly the solubility of the metal in a supercritical fluid. For this purpose, typical chelating agents such as dithiocarbamates (1–4), β-diketones (5,6), and crown ethers are used (7). The SFE methods for heavy metals from acidic aqueous solution have been developed (8–14). A series of studies (12–14) show that the SFE with tri-*n*-butylphosphate (TBP) can be applied to the separation of uranium and plutonium for the reprocessing of nuclear spent fuels. The results on the extraction of U(VI) and other metal ions show that the SFE is feasible for the separation and recovery of uranium from fission products. The SFE technology provides a new method of removing uranium and other metals from solid materials for environmental monitoring and remediation purposes (1–3,5,8,10,13,15–19).

In previous studies (20,21), we found that some lanthanide metals were extracted directly from their oxides (Nd_2O_3 and Gd_2O_3) with supercritical CO_2 containing TBP–HNO_3 (TBP), i.e., the TBP solution with an appropriate portion of TBP–HNO_3 complex. Using the supercritical CO_2 containing only TBP as the extractant, however, the lanthanides were found to be hardly extracted. In the present study, we applied this direct extraction method to several oxides (Nd_2O_3, ZrO_2, MoO_3, and RuO_2) and mixtures of the oxides (Nd_2O_3–ZrO_2, Nd_2O_3–MoO_3, and Nd_2O_3–RuO_2). Zr, Mo, and Ru were selected as typical fission products in the nuclear spent fuel. The purpose of this study is to show a feasibility of the direct extraction method for the selective recovery of Nd from such metals as Zr, Mo, and Ru with supercritical CO_2 containing TBP–HNO_3 (TBP).

EXPERIMENTAL

Chemicals

All the chemicals used were of reagent grade. Nd_2O_3 (Katayama Chemical Industries, Nagoya, Japan), ZrO_2 (Rare Metallic, RM, Tokyo, Japan), MoO_3 (RM), and RuO_2 (RM) were used without further purification. We prepared seven oxide samples. The single oxide samples were Nd_2O_3 (0.01 mol), ZrO_2 (0.01 mol), MoO_3 (0.01 mol), and RuO_2 (0.001 mol) and binary mixtures of the oxides were Nd_2O_3 (0.01 mol)–ZrO_2 (0.01 mol), Nd_2O_3 (0.01 mol)–MoO_3 (0.01 mol), and Nd_2O_3 (0.01 mol)–RuO_2 (0.001 mol). A 100 cm^3 of anhydrate TBP (Katayama) was contacted with 100 cm^3 of 15.5 mol/dm^3 (M) HNO_3 (Katayama) by shaking vigorously to prepare TBP–HNO_3 (TBP) solution. The concentration of HNO_3 in prepared TBP–HNO_3 (TBP) solution was determined to be 4.8 *M* by titrimetry. Here, for the determination of the HNO_3 concentration in TBP–HNO_3 (TBP), HNO_3 in the TBP solution was back-extracted into the aqueous solution, which was subjected to the titration with NaOH aqueous solution. An amount of 4.8 *M* TBP–HNO_3 (TBP) was diluted twice with anhydrate TBP to obtain 2.4 *M* TBP–HNO_3 (TBP) solution. The concentration, e.g., 2.4 *M* of HNO_3 in the TBP–HNO_3 (TBP) solution denotes that 2.4 mol TBP–HNO_3 complex is contained in 1 dm^3 of TBP–HNO_3 (TBP) solution, and therefore the total TBP contained in 2.4 *M* TBP–HNO_3 (TBP) is 3.1 mol. When the concentration of HNO_3 in the TBP–HNO_3 (TBP) solution is less than 3 *M*, HNO_3 forms (1:1) TBP–HNO_3 complex (22). The TBP–HNO_3 (TBP) solution was mixed with supercritical CO_2 and used for the extraction of Nd in oxide samples as Nd(III)-TBP nitrate complex.

Supercritical Fluid Extraction Apparatus

The apparatus used in the SFE experiment, which consisted mainly of a flow reactor (50 cm^3), a constant-temperature water bath, plunger pumps, a restrictor, and a collection vessel, was almost identical to that reported previously (20,21). A schematic diagram of the apparatus is shown in Fig. 1. The equilibrium vessel used for preparing the homogenous mixture of TBP–HNO_3 (TBP) and CO_2 in the apparatus reported previously, was removed and the TBP–HNO_3 (TBP) was injected directly to the supercritical CO_2 through a mixing joint (Kyowa Seimitsu, Tokyo, Japan, KSY-16) by a plunger pump (GL Science, Tokyo, Japan, GLS PUS-1.5).

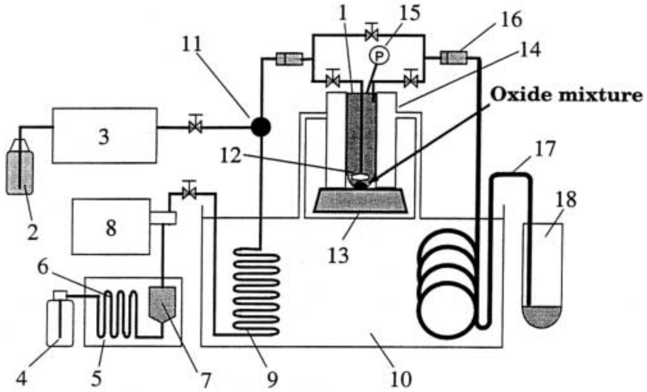

Figure 1. Schematic diagram of the experimental set-up for SFE system: (1) flow reactor, (2) TBP$-$HNO$_3$ (TBP) solution container, (3) plunger pump, (4) liquid CO$_2$ cylinder, (5) pre-cooling bath, (6) pre-cooling coil, (7) strainer, (8) plunger pump, (9) pre-heating coil, (10) constant-temperature water bath, (11) mixing joint, (12) stirring bar, (13) magnetic stirrer, (14) water-jacket type thermostat, (15) pressure gauge, (16) filter, (17) restrictor, (18) collection vessel.

Procedure

The oxide sample was taken in the flow reactor. Neat CO$_2$ from CO$_2$ cylinder was filled in the SFE system and then the CO$_2$ was pressurized up to 12 MPa with a plunger pump (Gilson Medical Electronics, Villiers-le-Bel, France). At the beginning of the extraction, at an extraction time $t_{ex} = 0$, the TBP$-$HNO$_3$ (TBP) solution was supplied to the CO$_2$ flow by the plunger pump. The flow rate of the injected 2.4 M TBP$-$HNO$_3$ (TBP) was maintained at 0.5 cm^3 min^{-1}. Through the mixing joint, the TBP$-$HNO$_3$ (TBP) was mixed with CO$_2$. The mixture medium was heated at 313K by passing through a pre-heating coil. The mixture medium was then introduced into the flow reactor. During the extraction procedure, pressure and temperature inside the flow reactor were kept at 12 MPa and at 313K, respectively, which are higher than the critical pressure ($P_C = 7.4$ MPa) and critical temperature ($T_C = 304.4$K) of CO$_2$ (3). It is usually difficult to keep both the pressure and the flow rate of the supercritical CO$_2$ medium constant in the SFE process using a continuous flow of CO$_2$ medium. In the present work, the pressure was adjusted by controlling the flow rate of the CO$_2$ using plunger pump and the flow rate of the TBP$-$HNO$_3$ (TBP) solution was kept at 0.5 cm^3 min^{-1}. The flow rate of the extraction medium, i.e., the mixture of the TBP$-$HNO$_3$ (TBP) and CO$_2$, varied and was in the range 1.8–3.6 cm^3 min^{-1}, i.e., the flow rate of CO_2 was in the range 1.3–3.1 $cm^3\,min^{-1}$ as the flow rate of the liquid CO_2 at the plunger pump outlet.

The TBP–HNO_3 (TBP) in CO_2 reacted with the Nd_2O_3 or the metal oxides in the flow reactor and Nd(III)-TBP complex was formed and dissolved in the supercritical CO_2. The effluent from the flow reactor was introduced into the collection vessel through the restrictor (GL Sciences, stainless steel tubing of 100 $\mu m\varnothing$ in internal diameter and 1.5 m in length). Because CO_2 gasified at atmospheric pressure and the solutes contained in the CO_2 medium such as Nd(III)-TBP complex, TBP–HNO_3 complex, and free TBP were recovered in the collection vessel. The restrictor was submerged in a constant-temperature water bath for depressing a plugging of the restrictor. Nd(III) recovered in the collection vessel as the Nd(III)-TBP complex was back-extracted into 0.01 M HNO_3 solution. The concentrations of the metal ions in the stripped samples were analyzed by using an ICP–MS (Finnigan MAT, Bremen, Germany, ELEMENT) to determine the recovery efficiencies of Nd. Here, the recovery efficiency (%) is defined as the ratio of the cumulative amount of the metal collected in the collection vessel during the experiment to that initially loaded. Burford et al. defined and discussed the extraction efficiency (percent removed from the sample matrix) and the collection efficiency (percent recovered in the collection solvent) in the SFE experiment (23). The recovery efficiency measured in the current study corresponds to the collection efficiency defined in Ref. (23), except for the fraction of the metal in the flushed solvent being not counted.

RESULTS AND DISCUSSION

The extraction behavior of Nd in the sample of Nd_2O_3 powder (0.01 mol, 3.365 g) was investigated using the mixture medium of 2.4 M TBP–HNO_3 (TBP) (0.5 $cm^3\,min^{-1}$) and CO_2 (2.2–2.5 $cm^3\,min^{-1}$) at 313K and 12 MPa. The recovery efficiency increases with increase in the extraction time t_{ex} as shown in Fig. 2.

The TBP–HNO_3 (TBP) solution was added to CO_2 flow at the extraction time, $t_{ex} = 0$. When t_{ex} was less than 30 min, the concentration of TBP–HNO_3 complex in the flow reactor was not high enough, and therefore, the recovery efficiency was negligible. At t_{ex} of 30–150 min, Nd was recovered efficiently because the concentration of TBP–HNO_3 complex in the flow reactor was high.

As Nd_2O_3 was dissolved completely at $t_{ex} = 150$ and the residual medium containing Nd in the flow reactor was flown out and the recovery efficiency of Nd gradually increased with an increase of t_{ex} in the range of 180–300 min. After the experiment, Nd_2O_3 remnant of an appreciable quantity was not observed in the flow reactor.

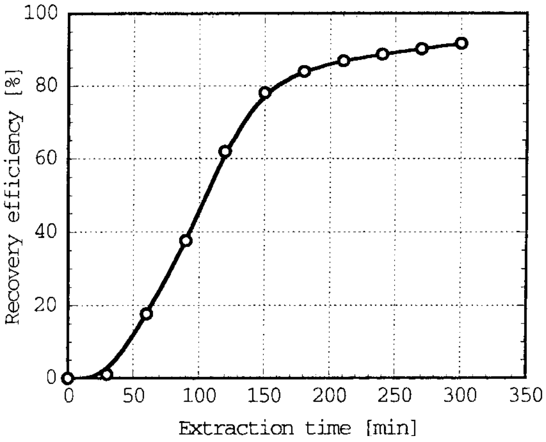

Figure 2. Recovery efficiency of Nd against the extraction time. Extraction: 12 MPa, 313K.

Nd was reported to form nitrate–TBP complex and to be extracted in the conventional solvent extraction (24). We have no experimental evidence so far that Nd forms the same chemical complex in the supercritical CO_2, but the most probable form seems $Nd(NO_3)_3(TBP)_2$. At t_{ex} in the range 60–150 min, the extraction reaction, which is expressed by the following equation, may proceed steadily.

$$Nd_2O_{3\,(s)} + 6TBP{-}HNO_{3\,(CO_2)} \rightarrow 2Nd(NO_3)_3(TBP)_{2(CO_2)} + 2TBP_{(CO_2)} + 3H_2O \quad (1)$$

During these 60 min, i.e., $60 \leq t_{ex} \leq 120$ min, HNO_3 and TBP supplied into the flow reactor were calculated to be 0.072 and 0.094 mol, respectively, and the extracted Nd was 0.0088 mol. Almost one-third of TBP–HNO_3 complex supplied into the flow reactor was consumed by the extraction reaction of Nd as Eq. (1). For the enhancement of the reaction efficiency of Nd_2O_3 with TBP–HNO_3 complex, it is required to optimize the flow rate of the medium as well as the extraction temperature and pressure.

The recovery efficiency of Nd from the Nd_2O_3 powder sample (0.01 mol, 3.365 g) by the 2-hr extraction, experiment #1, is shown in Table 1 together with the flow rate of the CO_2. The recovery efficiency of Nd is 66%. Identical extraction experiments #2–4 were performed for the oxides, i.e., ZrO_2 (0.01 mol, 1.232 g), MoO_3 (0.01 mol, 1.439 g), and RuO_2 (0.001 mol, 0.133 g) and the results of the recovery of the metal are summarized in Table 1. Zr, Mo, and Ru were hardly recovered from their oxides. In this connection, ZrO_2, MoO_3, and RuO_2 were insoluble even in the 2.4 *M* TBP–HNO_3 solution (nondiluted by CO_2)

Table 1. Recovery Efficiency of Nd, Zr, Mo, and Ru from Their Oxides and from Binary Mixtures of Nd_2O_3 with ZrO_2, MoO_3, or RuO_2

Experiment Number	Sample	Recovery Efficiency		CO_2 Flow Rate ($cm^3\ min^{-1}$)
		Element	Percent	
1	Nd_2O_3	Nd	66.30	0.6–2.9 (2.5)[a]
2	ZrO_2	Zr	0.01	1.6–2.9 (1.9)[a]
3	MoO_3	Mo	0.02	3.0 (3.0)[a]
4	RuO_2	Ru	0.00	1.0–2.8 (1.3)[a]
5	Nd_2O_3–ZrO_2	Nd	50.87	3.0–3.5 (3.1)[a]
		Zr	0.02	
6	Nd_2O_3–MoO_3	Nd	18.69	1.0–2.0 (1.8)[a]
		Mo	0.01	
7	Nd_2O_3–RuO_2	Nd	50.26	2.3–3.5 (2.5)[a]
		Ru	0.00	

Extraction: 12 MPa, 313K, $t_{ex} = 2$ hr.

[a] The mean flow rate of CO_2 as liquid CO_2 at the plunger pump outlet (12 MPa, 268K).

at 313–343K, i.e., Zr, Mo, and Ru were hardly extracted so that they did not form stable nitrate–TBP complexes. After each experiment, it was found that ZrO_2, MoO_3, and RuO_2 remained as taken in the flow reactor.

The results of the recovery efficiency for the binary mixtures of oxides, i.e., Nd_2O_3 (0.01 mol)–ZrO_2 (0.01 mol), Nd_2O_3 (0.01 mol)–MoO_3 (0.01 mol), and Nd_2O_3 (0.01 mol)–RuO_2 (0.001 mol), experiments #5–7, are summarized in Table 1. Zr, Mo, and Ru were hardly recovered from their oxide mixtures identical to the results from their single oxides. The recovery efficiency of Nd from the binary oxide mixtures could not be evaluated directly from these results, since the flow rate of the extraction medium varied greatly from experiment to experiment. For the evaluation of recovery efficiency of Nd, the recovery efficiencies of Nd were plotted against the total volume of the solutes, which are the mixture of Nd(III)-TBP complex, TBP–HNO_3 complex, and free TBP eluted from the reactor and collected in the collection vessel during the 2-hr extraction procedure. Figure 3 involves the plots of the recovery efficiencies of Nd from the binary oxide mixtures.

It is found that the recovery efficiencies of Nd from different oxide mixture samples to the cumulative solute samples are practically the same, independent of the composition of the oxide mixture. It should be noted that every plot shows a clear linearity between the recovery efficiency of Nd and the total volume of the solutes collected regardless of the flow rate of the extraction medium in the range 1.8–3.6 $cm^3\ min^{-1}$.

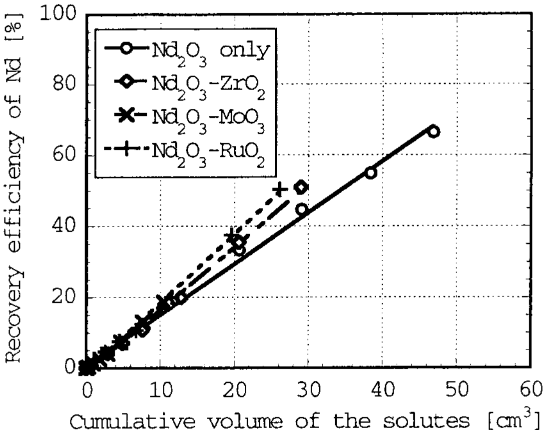

Figure 3. Recovery efficiency of Nd against the cumulative volume of the solutes. Extraction: 12 MPa, 313K.

This indicates that the extraction reaction of Nd from oxide mixture proceeds steadily, independent of the flow rate of the medium and the kind of oxide sample.

CONCLUSION

Neodymium is extracted directly from Nd_2O_3 powder and the mixtures of Nd_2O_3–ZrO_2, Nd_2O_3–MoO_3, and Nd_2O_3–RuO_2 with the extraction medium of TBP–HNO_3 (TBP) solution diluted by supercritical CO_2. Quantitative extraction of Nd from Nd_2O_3 powder was achieved by the extraction at 12 MPa and 313K. Since Zr, Mo, and Ru are not extracted from their oxides and the mixture with Nd_2O_3, Nd is selectively recovered from Zr-, Mo-, and Ru-containing oxide samples.

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