

Solvent extraction of U(VI) by N,N-dimethyl-N', N'-dioctylsuccinylamide and N,N-dimethyl-N', N'-didecylsuccinylamide in cyclohexane

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Abstract The extraction of uranyl nitrate by novel extractants of *N*,*N*-dimethyl-*N'*,*N'*-dioctylsuccinylamide (DMDOSA) and *N*,*N*-dimethyl-*N'*,*N'*-didecylsuccinylamide (DMDDSA) from aqueous nitric/nitrate solutions was investigated. It was found both the concentration of HNO₃ and extractants had an effect on the U(VI) extraction distribution. The extraction mechanism was established, and stoichiometry of the main extracted species was confirmed to be UO₂(NO₃)₂·2DMDOSA and UO₂(NO₃)₂· 2DMDDSA, respectively. Both of the extraction reactions are exothermic. FTIR spectral study of the U(VI) extracted species was also made.

Keywords U(VI) \cdot *N*, *N*-dimethyl-*N'*,*N'*-dioctylsuccinylamide \cdot *N*,*N*-dimethyl-*N'*,*N'*-didecylsuccinylamide \cdot Extraction \cdot Distribution ratio

1 Introduction

Solvent extraction method has been an important technique in concentrating and purifying U(VI). It is widely accepted that the choice of extractants plays a great role in concentration behavior. For example, alkyl amides, which

Guo-Xin Sun sun-guo-xin@hotmail.com are potential extractants of actinides, are considered as an alternate to replace TBP, an extractant used widely in nuclear fuel reprocessing [1-4]. Diamides extractants have attracted widespread attention recently to study the possible use in nuclear fuel reprocessing. Tetraalkyl malonamide [5, 6], tetrabutyl succinamide [7, 8] and tetrabutyl 3-oxo-diglycolamide [9-11] showed good extraction properties for U(VI), Th(IV) and fission products from nitric acid solutions. Mowafy and Mohamed [12] studied the extraction of U(VI) and Th(IV) from nitric acid solutions with N.N.N'.N'-tetrabutylsuccinamide (TBSA) in toluene. Wu et al. [9] found that excess of TBSA could react with uranyl to form $[UO_2(TBSA)_3]^{2+}$, in which two TBSA ligands were in tridentate mode and the third was in monodentate mode in ionic liquid [Bmim][NTf2]. N,N,N',N'-tetrahexylsuccinylamide (THSA) was used for extraction of U(VI) and Th(IV) ions from nitric acid media into n-dodecane [13]. Wang et al. studied solvent extraction of uranyl (II) ion with N, N', N'-tetrabutylsuccinylamide from nitric acid solution [14]. It is well known that extraction ability varies widely in terms of the nature of the alkyl groups on the diamide framework [15–18].

Among the structure of alkyl amide-based extractants, when one alkyl group adjacent to nitrogen atom is a small group (typically methyl) and the other is a bigger one, the steric hindrance around the carbonyl oxygen atoms is kept to minimum and the lipophilic character of the ligands is attained in the presence of a longer chain alkyl. In our previous work, the extraction of U(VI) was achieved by DMDOSA in toluene [19]. In this paper, to further investigate the relationship of diamide structure–extraction function, the extraction behaviors of U(IV) are studied by choosing different alkyl substituents in cyclohexane, and the extraction mechanism is proposed.

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2 Experimental

2.1 Reagents

The DMDOSA (extractant *N*,*N*-dimethyl-*N'*,*N'*-dioctylsuccinylamide) and DMDDSA (*N*,*N*-dimethyl-*N'*,*N'*-didecylsuccinylamide) were obtained by reaction of *N*-methyl octylamine and *N*-methyl decylamine with succinyl chloride [19], respectively. Both DMDOSA and DMDDSA were purified by distillation under vacuum. Structures of the extractants are shown in Fig. 1. The final products were characterized by elemental analysis, IR and ¹HNMR measurements, and the purity was over 98 %. The corresponding data are given as follows:

DMDOSA: (yield 77.4 %), pale yellow oil, 98 % purity, b.p. 240–242 °C/2–4 mmHg; $v_{C=0}$ (film)/cm⁻¹ 1648.2; δ_{H} (300 MHz, DMSO), 0.857 (6H, t, C–CH₃), 1.239–1.260 (20H, m, –CH₂–), 1.346–1.546 (4H, m, –CH₂–C–N–), 2.764–2.940 (6H, t, N–CH₃), 3.200–3.292 (4H, m, N–CH₂–), 2.481 (4H, t, OC–CH₂–CH₂–CO). Element analysis for C₂₂H₄₄N₂O₂ (Calcd.): C: 71.46 % (71.69 %), H: 11.94 % (12.03 %), N: 7.51 % (7.60 %).

DMDDSA: (yield 58.4 %), white solid, 98 % purity, b.p. 246–248 °C/2–4 mmHg; $v_{C=0}$ (film)/cm⁻¹ 1648.3; δ_{H} (300 MHz, DMSO), 0.860 (6H, t, C–CH₃), 1.239–1.260(24H, m, –CH₂–), 1.346–1.546 (4H, m, –CH₂–C–N–), 2.764, 2.939 (6H, t, N–CH₃), 3.200–3.292 (4H, m, N–CH₂–), 2.481 (4H, m, OC–CH₂–CH₂–CO). Element analysis for C₂₆H₅₂N₂O₂ (Calcd.): C 73.53 % (73.29 %), H 12.34 % (12.23 %), N 6.60 % (6.74 %).

Other agents employed in this work were of A.R. grades.

2.2 Extraction procedure

Equal volumes of aqueous phase and organic phase were vigorously mixed for 30 min to reach equilibrium at a constant temperature. The two phases were centrifuged and assayed by taking 100–500 μ L aqueous phase. The U(VI) concentration in a sample was determined by Arsenazo-III visible spectrophotometric analysis, and the U(VI) concentration in organic phase was calculated by subtracting the aqueous concentrations from the total initial aqueous

concentration of U(VI). The distribution ratio (D) was calculated by dividing the U(VI) concentration in the organic phase over the U(VI) concentration in aqueous phase. The *D* values reported were averaged from at least duplicate experiments, with errors of less than 5 %.

Back extraction was conducted as follows: 2 mL of above organic phase after extraction and 2 mL pure water or 0.10 mol dm⁻³ Na₂CO₃ solution were vigorously mixed for 30 min. The two phases were centrifuged and assayed by taking 100 μ L aqueous phase. The concentrations of U(VI) in the sample were determined by Arsenazo-III visible spectrophotometric analysis.

2.3 FTIR characterization

The extractant solutions were shaken with a concentrated solution of $UO_2(NO_3)_2$ and centrifuged to collect the organic phase. The organic solvents were removed by evaporation, and infrared spectra of the residuals were recorded in the wavenumber range of 4000–400 cm⁻¹ on a Perkin-Elmer Spectrum One FTIR spectrometer, using KBr sample windows, in 60 scan times per sample, at a resolution of 2 cm⁻¹.

3 Results and discussion

3.1 Effect of nitric acid concentration

The extractions of U(VI) with 0.20 mol dm⁻³ DMDOSA and DMDDSA (in cyclohexane) from nitric acid solutions of 1.00–5.00 mol dm⁻³ HNO₃ at 298 K were investigated. The results are shown in Fig. 2. The distribution ratio of U(VI) increases with the HNO₃ concentration. This is similar to the results in HNO₃ medium systems [19] and can mainly be attributed to common ion effect of nitrate ion. Comparing the data of extraction of U(VI) with DMDOSA in toluene [19] and in cyclohexane, one knows that it is beneficial to use cyclohexane as diluents at high nitric acid concentrations of over 4.0 mol dm⁻³. Another advantage of using cyclohexane as garaffin is environment friendly.



Fig. 1 Structures of extractants. a N,N-dimethyl-N',N'-dioctylsuccinylamide (DMDOSA), b N,N-dimethyl-N',N'-didecylsuccinylamide (DMDDSA)



Fig. 2 Effect of HNO3 concentration on U(VI) extraction

3.2 Effect of extractant concentration

The extraction equilibrium of UO_2^{2+} from HNO₃ solution by DMDDSA can be described as follows:

$$UO_2^{2+} + 2NO_3^- + nDMDDSA_{(O)}$$

= UO₂(NO₃)₂·nDMDDSA_{(O)} (1)

The conditional extraction constant is

$$K_{\rm ex} = \frac{C_{\rm UO_2(NO_3)_2 \cdot n \rm DMDDSA_{(o)}}}{C_{\rm UO_2^{++}} C_{\rm NO_3^{--}}^2 C_{\rm DMDDSA_{(o)}}^n}$$
(2)

$$D_{\rm U} = \frac{C_{\rm UO_2(NO_3)_2 \cdot n \rm DMDDSA_{(o)}}}{C_{\rm UO_2^{2+}}}$$
(3)

$$K_{\rm ex} = \frac{D_{\rm U}}{C_{\rm NO7}^2 C_{\rm DMDDSA(q)}^n} \tag{4}$$

where $C_{\rm UO_2^{2+}}$ is the total concentration of $\rm UO_2^{2+}$ in the aqueous phase and $C_{\rm UO_2(NO_3)_2:n\rm DMDDSA_{(o)}}$ is the concentration of the extracted species in the organic phase. Equation (4) can be rearranged into Eq. (5) after taking the logarithm:

$$\log D_{\rm U} = \log K_{\rm ex} + n \log C_{\rm DMDDSA_{(0)}} + 2 \log C_{\rm NO_3^-}$$
(5)

where $C_{\text{NO}_3^-}$ is kept constant, DMDDSA is in a large excess to UO_2^{2+} ions under the studied conditions, and then the concentration of DMDDSA can be regarded as constant. Thus, the stoichiometric coefficients *n* can be determined by slope analysis of the relationship between logarithm distribution ratio and logarithm equilibrium concentration. Figure 3 shows the log *D* as a function of logarithm DMDDSA and DMDOSA concentration.

The distribution ratio increases linearly with the DMDDSA or DMDOSA concentration, with the slopes of 2.17 for log *D* versus log $C_{\text{DMDDSA}(0)}$ and 2.25 for log *D* versus. log $C_{\text{DMDDSA}(0)}$ These show that two molecules of



Fig. 3 Effect of extractant concentration on U(VI) extraction

amide are associated with one UO_2^{2+} ions in the extractable complex. However, the average number of TOSA [20] and TBSA [14] coordinated to U(VI) ion was estimated to be one. This is probably connected with nitric acid concentration or the diluents that have a major impact on the extraction behavior due to their polarity [21]. We propose that the difference of the coordinated extractant numbers is possibly due to the size of the substitute. The methyl group of DMDOSA or DMDDSA is smaller compared with octyl group of TOSA or butyl group of TBSA. The methyl group has less steric hindrance resulting in more extractant molecules into the coordination sphere. The extraction mechanism can be described as:

$$UO_2^{2^+} + 2NO_3^- + 2DMDDSA_{(o)}$$

= UO₂(NO₃)₂ · 2DMDDSA_{(o)} (6)

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$$UO_2^{2+} + 2NO_3^- + 2DMDOSA_{(o)}$$

= UO₂(NO₃)₂ · 2DMDOSA_{(o)} (7)

The stoichiometry of extracted species is the same as that obtained in toluene system [19], which shows that diluent does not affect the composition of extracted species.

FTIR spectra of the organic phase with the extracted species were recorded (Fig. 4). Analysis of the spectra of extracted species was carried out in the C=O stretching region ($1550-1760 \text{ cm}^{-1}$) using a curve fitting procedure. The broad band of C=O consists of two sub-bands, and the peaks are centered at 1586 and 1648 cm⁻¹. Compared with the FTIR spectra of the free extractants, the carbonyl group (C=O) stretching vibration of the extracted species shifts from about 1648 to 1586 cm⁻¹, which implies that the amide ligand directly coordinates to the uranyl ion through its carbonyl group. The appearance of bands at about 1030, 1281 and 1522 cm⁻¹ can be indexed to the coordinated nitrate ions [22, 23]. An additional band described as the U–O stretching vibration of the uranyl ion is observed at 934 cm⁻¹.



Fig. 4 IR spectra of the extractant loaded uranyl nitrate

3.3 Temperature effect on extraction

Temperature effect on extraction equilibrium of U(VI) was studied at 15–55 °C (Fig. 5). The distribution ratio decreases with increasing temperature. From the Van't Hoff equations [24], the thermodynamic parameters can be calculated by:

$$\log D = \Delta H / (2.303 \text{RT}) + C \tag{8}$$

where *R* is the gas constant and *C* is a conditional constant. In this case, the slope in Fig. 5 is $\Delta H/(2.303R)$. From Eq. (8) and Fig. 5, we obtained $\Delta H = -26.33 \pm 1.56 \text{ kJ mol}^{-1}$ for DMDOSA and $\Delta H = -23.60 \pm 0.95 \text{ kJ mol}^{-1}$ for DMDDSA. In toluene, we got $-23.9 \pm 1.7 \text{ kJ mol}^{-1}$ for DMDOSA [19]. The results indicate that cyclohexane is a better diluent. The change in enthalpy demonstrates that the extraction reaction is an exothermic process, and it is disadvantageous for the extraction reaction at high temperature.



Fig. 5 Effect of experimental temperature on the extraction of U(VI)

Table 1 $\log K_{ex}$ and carbonyl oxygen charge distribution of DMDOSA and DMDDSA

Extractant	DMDOSA	DMDDSA
log K _{ex} Charge distribution	3.00 ± 0.04 -0.663, -0.663	3.01 ± 0.03 -0.663, -0.660

3.4 Effect of extractant structure

Comparing to the extraction of U(VI) with N,N, N',N'tetraalkylsuccinylamide [14, 25], the adoption of unsymmetrical substituted alkyls, especially with methyl and straight long-chain alkyl, does improve the extractability of succinylamide toward U(VI). The two straight long-chain alkyls adjacent to nitrogen atom in succinylamide extractants lead to a big steric hindrance effect, which is possibly responsible for the difference of the formed extracted species. One alkyl group of DMDOSA or DMDDSA adjacent to nitrogen atom is a small group-methyl, and the other is a bigger one-octyl or decyl; the stereochemical hindrance around the carbonyl oxygen atoms is kept to a minimum, and both oxygen atoms can interact with uranyl ion [26]. On the other hand, the hydrophobic character of the ligands is attained in the presence of a longer alkyl—octyl or decyl. The intercept of $\log D$ versus $\log D_{\text{amide}}$ (Fig. 3) was $\log K_{\text{ex}}$, as given in Table 1.

Generally, the basicity, i.e., charge distribution of coordination group is related to the coordination ability. In view of atoms bonding with U(VI) being oxygen atoms, the charge distributions of carbonyl oxygen of DMDOSA and DMDDSA were calculated by employing density functional theory (DFT) with the B3LYP/6-31+G(d) level of the Gaussian 03 program [27]. The results are given in Table 1.

It is obvious that log K_{ex} of DMDOSA is nearly equal to log K_{ex} of DMDDSA, which is possibly that the steric hindrance effect of octyl is not much different from that of decyl. Moreover, the charge distribution of carbonyl oxygen of DMDOSA is nearly equal to that of DMDDSA, and hence the similar extraction result is obtained. Table 1 also shows that the distal carbon atoms do not influence the charge distribution of carbonyl oxygen effectively.

3.5 Back extraction

The results of stripping U(VI) loaded in the organic phases show that the back extraction of U(VI) extracted by DMDOSA or DMDDSA is efficient. The stripping efficiency is over 96 % with pure water for one step, and nearly 100 % with 0.10 mol dm⁻³ Na₂CO₃ solution.

4 Conclusion

Aliphatic diluent can be used in the extraction of U(VI) with DMDOSA or DMDDSA from nitric/nitrate solutions, which shows actual application prospect. The distribution ratio of UO_2^{2+} increases with concentrations of nitric acid and extractant. The stoichiometry of the extracted species in cyclohexane is $UO_2(NO_3)_2 \cdot 2DMDOSA$ and $UO_2(NO_3)_2 \cdot 2DMDOSA$ 2DMDDSA. The ΔH for the extraction are $-26.33 \pm$ 1.56 kJ mol⁻¹ by DMDOSA and -23.60 ± 0.95 kJ mol⁻¹ by DMDDSA. The adoption of unsymmetrical substituted alkyl groups in DMDOSA and DMDDSA with methyl group does improve the extraction power toward U(VI) compared with a symmetrical extractant (TBSA), in which substituents possess similar numbers of carbon atoms. The conditional extraction constant of DMDOSA and DMDDSA is consistent with the calculated charge distribution of carbonyl oxygen.

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