

Electrochemical Behavior of Ce(IV)/Ce(III) Couple in N,N-Di(2-ethylhexyl)-n-butanamide (DEHBA), N,N-Di(2-ethylhexyl)-iso-butanamide (DEHiBA), and N,N-Di(2-ethylhexyl)-3,3-dimethyl Butanamide (DEHDMBA)

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Electrochemical behavior of Ce(IV)/Ce(III) couple in N,N-di(2ethylhexyl)-n-butanamide (DEHBA), N,N-di(2-ethylhexyl)-isobutanamide (DEHiBA), and N,N-di(2-ethylhexyl)-3,3-dimethyl butanamide (DEHDMBA)

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Abstract

In this work we report on the electrochemical behavior of Ce(IV)/Ce(III) redox couple in pure N,N-dialkyl amides (N,N-DA), namely N,N-di(2-ethylhexyl)-n-butanamide (DEHBA), N,N-di(2-ethylhexyl)-iso-butanamide (DEHiBA), and N,N-di(2-ethylhexyl)-3,3-dimethyl butanamide (DEHDMBA) equilibrated with nitric aqueous solutions as an entry to the direct electrochemical characterization of plutonium in these extractants. Ce(IV)/Ce(III) redox process was used as a model. Its potential (E $_{1/2} \cong 1.02$ V/SCE) is not affected by the temperature and the nature of the N,N-DA and this clearly indicates that the functionalities of the these extractants produce the same relative effect on both +IV and +III oxidation states of the cerium cation. Linear variations of the current intensity of the reduction peak of Ce(IV) with the concentration of Ce(IV)/N,N-DAs/HNO₃(5M) solutions were obtained from cyclic voltammograms recorded at 25°C and 40°C. Due to the poor definition of the voltammograms in DEHiBA and DEHDMBA, such characterization allows only the evaluation of the performances of the chemical extraction of Ce(IV)/Ce(III) in N,N-DAs was not previously studied and our findings will for sure open the door for further investigations in this field.

Keywords: cerium, dialkylamide, PUREX, cyclic voltammetry

1. Introduction

The fission of the impair isotopes of uranium and plutonium is an important source of energy from nuclear reactors. In particular, the fission of uranium produces plutonium, which is also fissile, and thus a new source of energy. In France, it has been decided to recycle recoverable materials such as uranium and plutonium. To do this, the PUREX process (for Plutonium Uranium Reduction EXtraction) is used industrially to separate and then purify both elements from the fission products. After separation, these elements are converted into the form of dioxide $((U,Pu)O_2)$ that can again be used as nuclear fuel. This process consists in extracting U(VI) and Pu(IV) from the original aqueous nitric solution to an organic phase of n-tributyl phosphate TBP and an alkane diluent (1). But TBP has particular limitations such as the formation of degradation products in the presence of nitric acid and strong ionizing radiations. New families of alternative extractants, such as N,N-dialkylamides (called N,N-DAs), are currently being investigated (2-20). N,N- DAs, diluted in an alkane solution (0.5<[N,N-DA]<1.5 mol.L-1), are indeed very promising and one of their characteristics is the strong influence of their alkyl groups on their extraction properties. Indeed, their extraction strength and selectivity towards U(VI) and Pu(IV) can be tuned by adjusting the length and branching of the monoamide alkyl chains (2,7,10,21-23). However, few fundamental studies have been reported, and the influence of the structure of N,N-dialkylamides on the extraction properties is not yet fully explained. The structures of Pu(IV) and U(VI) complexes with N,Ndialkyl amides ligands were recently characterized by Guillaumont and coworkers (24,25) using single-crystal X-ray diffraction (XRD), extended X-ray absorption fine structure (EXAFS), and theoretical calculations. These studies show that these species are tetra-nitrato complexes solvated by two extractants. In these organic phases ([N,N-DA] = 1 mol.L⁻¹), the structure of the Pu(IV) complex is $Pu^{IV}(NO_3)_4(N,N-DA)_2$.

The monitoring of the performance of each step of the nuclear fuel reprocessing is crucial and it is done through the characterization of the speciation of the extracted complexes and their relative concentration. In this context, electrochemistry has several advantages over other analytical techniques, especially for the analysis of the organic phase. Indeed, it allows a simple, fast, economic and non-destructive determination of the samples and can be carried out on volumes of the order of micro/milliliter. This the also allows lowering significantly the exposure of people and equipment to radiations. In this work we report our investigations of the electrochemical behavior of couple Ce(IV)/Ce(III) in pure N.N-DAs. namelv N.N-di(2-ethvlhexvl)-nbutanamide (DEHBA), N,N-di(2-ethylhexyl)-isobutanamide (DEHiBA), and N.N-di(2-ethylhexyl)-3,3dimethyl butanamide (DEHDMBA; see scheme 1) equilibrated with nitric aqueous solutions as an entry to the direct electrochemical characterization of plutonium in these extractants. Indeed, it is important to note that the high radioactivity of all the plutonium isotopes brings important constraints to the handling of this element, and thus to obtain experimental results. Moreover, obtaining experimental data requires specific installations with particular safety measures, and in particular glove boxes for handling radioactive elements. In order to ovoid these constraints, the methodology usually developed consists in a first step to mimic the plutonium element by a nonradioactive one that presents similar behavior and Ce(IV)/Ce(III) couple is usually used to do so (26). Such characterization would allow the evaluation of the performances of the chemical extraction of Ce(IV) by the undiluted N.N-DAs. To our knowledge, the electrochemical behavior of Ce(IV)/Ce(III) in these extractants was not previously reported and our findings will for sure open the door for further investigations in



this field.

Scheme 1. studied N,N-dialkyl amides

2. Experimental

2.1. Reagents and solutions

All chemicals and solvents were analytical grade and used without further purification. All aqueous solutions were made with Milli-Q purified water (Millipore). The N,N-dialkyl amides DEHDMBA, DEHBA and DEHiBA were synthesized by Pharmasynthese (France). The source of cerium was Ce(III) nitrate or Ce(IV) ammonium nitrate (Alfa Aesar) with suprapure grade. 69% nitric acid was purchased from VWR.

2.2. Instrumentation

Electrochemical measurements were carried out using a "VoltaLab PST50" potentiostat (Radiometer Analytical). Cyclic Voltammetry was performed under dynamic mode compensation of the ohmic drop. All electrochemical experiments were driven using the "Voltamaster 4" Software. The electrochemical cell was a thermostated conventional three electrodes-one compartment glass cell filled with 10 mL of N,N-DA, previously equilibrated as depicted below. Vitreous carbon electrodes (CVJ; purchased from radiometer, $A = 0.07 \text{ cm}^2$) were used as working electrodes. Prior to each run, they were polished on "coarse polishing pad" and "Emery polishing paper (UF800)" (ALS Co, LTD) and rinsed with MilliO water and ethanol. Platinum gauze was used as counter electrode. A saturated calomel electrode (SCE) was used as reference electrode. The ohmic drop correction in dynamic mode allowed evaluating the resistance of the different electrolytic solutions. The resistances are of the order of magnitude of 10 K Ω , 20 K Ω and 40 K Ω , respectively for DEHBA, DEHiBA and DEHDMBA equilibrated with HNO₃ 5M at 25°C. As expected, they decrease to 5 K Ω , 15 K Ω and 20 K Ω at 40°C, respectively. These high values confirm that the studied N,N-DAs are very little dissociating.

2.3. Procedure for the preparation of N,N-DAs solutions equilibrated nitric aqueous solutions

First, undiluted N,N-DA was contacted 3 times with the same volume of nitric acid aqueous solution (5M) to obtain equilibrated nitric acid-amide. In brief, equal volumes (25 mL) of pure N.N-DA and aqueous nitric acid solution were contacted in a 100 mL separating funnel and vigorously mixed for 20 min, and after decanting, both phases are separated. The organic phase recovered was re-contacted with 25 mL of fresh nitric acid solution (5M). This operation was repeated three times. The compositions of the organic solutions after equilibrium with HNO₃ 5M are shown in Table 1. Then, a millimolar solution of Ce(IV) or Ce(III) was prepared by dissolving [(NH₄)₂Ce^{IV}(NO₃)₆] or Ce^{III}(NO₃)₃.6H₂O salt in the equilibrated nitric acid-N,N-DA. The concentration range studied is from 2 to 15 mM. The solutions obtained are denoted as Ce(X)/DEHBA/HNO₃(5M), Ce(X)/DEHiBA/HNO₃(5M) and $Ce(X)/DEHDMBA/HNO_3(5M)$ with X = IV or III.

Table 1. Chemical composition of the N,N-DA organic phases in equilibrium with the aqueous nitric acid phase $(5 \text{ mol. } L^{-1})$.

	[N,N- DA] (M)	[HNO ₃] _{org.} (M)	[H ₂ O] _{org} (M)
DEHBA/HNO ₃ (5M)	2.78	2.53 ± 0.06	$\begin{array}{ccc} 0.34 & \pm \\ 0.01 & \end{array}$
DEHiBA/HNO ₃ (5M)	2.76	2.23 ± 0.04	$\begin{array}{ccc} 0.43 & \pm \\ 0.01 & \end{array}$
DEHDMBA/HBO ₃ (5M)	2.53	2.01 ± 0.05	$\begin{array}{ccc} 0.31 & \pm \\ 0.01 & \end{array}$

3. Results and discussion

3.1. Cyclic voltammetry of Ce(IV) in N,N-DAs equilibrated with aqueous nitric acid

The electrochemical behavior of Ce(III) and Ce(IV) cations in undiluted N,N-DAs solutions equilibrated with nitric acid are identical. Therefore we will only detail the results obtained with Ce(IV). Figure 1 shows the cyclic voltammograms of Ce(IV)/DEHBA/HNO₃(5M) solution at different potential scan rates at 25°C and 40°C at a vitreous carbon electrode. At both temperatures, the voltammograms exhibit a well-defined reversible couple of peaks that is related to the redox process:

$$Ce^{IV}(NO_3)_4(DEHBA)_n + e^- \Leftrightarrow Ce^{III}(NO_3)_3.(DEHBA)_n + NO_3^-$$

with n = 2 or 3.





Fig. 1. Cyclic voltammograms of Ce(IV)/DEHBA/HNO₃(5 M) at different potential scan rates at (a) 25°C ([Ce(IV)] = 15.3 mM) and (b) 40°C ([Ce(IV)] = 15.2 mM) at a vitreous carbon electrode with ohmic drop correction (dynamic mode) of $(7 \pm 1) \text{ K}\Omega$ and $(4 \pm 1) \text{ K}\Omega$, respectively.

This reversible couple Ce(IV)/Ce(III) is observed at E $_{1/2} \cong 1.02$ V/SCE (E $_{1/2} = (E_{pa} + E_{pc})/2$, where E_{pa} and E_{pc} are the anodic and cathodic peak potentials, respectively). The evolution of the variation of the anodic and cathodic peak currents with the square root of the potential scan rate allows confirming that the electrochemical process is under diffusion control (Fig. 2). This result shows clearly that the electrode is not prone to passivation and/or adsorption processes in this medium. The Randle-Sevick equation (27) allows attaining the diffusion coefficients of Ce(IV) at both temperatures. The obtained values are $(3.3 \pm 0.3) \times 10^{-12} \text{ m}^2 \text{ s}^{-1}$ and $(5.6 \pm 0.5) \times 10^{-12} \text{ m}^2 \text{ s}^{-1}$ at 25°C and 40°C, respectively. These remarkably low values can be explained by the relatively high dynamic viscosity of DEHBA ((59.8 \pm 0.1) mPa.s and (26.8 \pm 0.1) mPa.s at 25°C and 40°C, respectively). The increase in the diffusion coefficient with temperature in the DEHBA follows the stokes-Einstein law as the ratio of the diffusion coefficients at both temperatures is close to that of the product of viscosities and temperatures. The hydrodynamic radius of Ce(IV) in DEHBA calculated from Stokes-Einstein Law (27) is (13 ± 3) Å.

Figure 3 shows the linear variation of the current intensity of the reduction peak of Ce(IV) with the concentration of Ce(IV)/DEHBA/HNO₃(5M) solutions obtained from cyclic voltammograms recorded at 25°C and 40°C. As expected, the slope of the linear variation obtained at 40°C ((1.19 ± 0.12) μ A.mM⁻¹) is higher than that the one at 25°C ((0.94 ± 0.1) μ A.mM⁻¹). These data will be of interest when it comes to quantify the extraction process of Ce(IV) by this N,N-DA.



Fig. 2. Variation of anodic (Ipa) and cathodic peak currents (|Ipc|) depending on the square root of the scanning rate at (a) 25°C ([Ce(IV)] = 15.3 mM) and (b) 40°C ([Ce(IV)] = 15.2 mM).

and

Ce(IV)/DEHiBA/HNO₃(5M)

Ce(IV)/DEHDMBA/HNO₃(5M) show an electrochemical behavior similar that of Ce(IV)/DEHBA/HNO₃(5M) at 25°C and 40°C at a vitreous carbon electrode, except that the low conductivity of DEHDMBA/HNO₃(5M) at 25°C does not allow obtaining well defined and reproducible voltammograms. Also, linear variations of the current intensity of the reduction peak of Ce(IV) with the concentration of Ce(IV)/DEHiBA/HNO₃(5M) and Ce(IV)/DEHDMBA/HNO₃(5M) solutions were obtained from cyclic voltammograms recorded at 40°C.



Fig. 3: Variation of the current intensity of the reduction peak of Ce(IV) depending on the cerium concentration of Ce(IV)/DEHBA/HNO₃ (5M) solutions obtained from cyclic voltammograms recorded at 25°C (\star) and 40°C (\Box) at a scan rate of 75mV.s⁻¹. The dotted lines represent the 10% uncertainty.

Table 2 recapitulates the obtained data. Three main concluding remarks can be emphasized:

(i) the Ce(IV)/Ce(III) redox process exhibit a welldefined reversible couple. The potential is not affected by the temperature and the nature of the N,N-DA and this, clearly indicates that the functionalities of the three N,N-DAs extractants produce the same relative effect for both +IV and +III oxidation states of the cerium cation,

(*ii*) the hydrodynamic radii are similar for the three extractants, from 13 Å to 28 Å (Table 2). For the pure DEHBA extractant, the hydrodynamic radius of Ce(IV) is about 13 Å. This value is larger (factor 2.5) than the one estimated to be 7.75 Å by NMR (28, 29) and theoretical calculations (25) for Pu(NO₃)₄(DEHBA)₂ in dilute medium ([DEHBA] < 1 mol.L⁻¹). This difference can be explained by the existence of a hydrogen bonding network between the solvates Ce^{IV}(NO₃)₄(DEHBA)_n and the DEHBA molecules in the pure solvent, whereas in dilute medium (< 1 mol.L⁻¹) the solvates are isolated.

(*iii*) in spite of the high viscosity and low conductivity of these media, and the low diffusion coefficients of Ce(IV) and Ce(III), it was possible to establish calibration curves for potential quantification of these cations after their extraction.

Table 2. Electrochemical characterization of Ce(IV) in	
DEHBA, DEHiBA and DEHDMBA equilibrated with nitr	ic

	E _{1/2} (V/SCE)		Coefficient diffusion $D(x10^{-12}m^2s^{-1})$		Hydrodynamic radius of Ce(IV) (Å)	Linear s calibratio (µA.mM	lope for n ¹)*
	25°C	40°C	25°C	40°C		25°C	40°C
Ce(IV)/DEHBA/HNO ₃ (5M)	1.02	1.03	3.3	5.6	13 ± 3	0.94	1.19
	±0.04	± 0.04	±0.3	±0.5		±0.1	±0.12
Ce(IV)/DEHiBA/HNO ₃ (5M)	1.1	1.07	2.8	$5.1 \pm$	19 ± 3	$0.86\pm$	$1.13 \pm$
	± 0.06	± 0.05	±0.2	3		0.15	0.15
Ce(IV)/DEHDMBA/HNO ₃ (5M)	-	1.03	-	$3.8 \pm$	28 ± 3	-	$0.975~\pm$
		± 0.04		1			0.2

acid (5M)

* Electrode area : 0.07 cm^2

3.2. Quantification of Ce(IV) upon its extraction by DEHBA equilibrated with aqueous nitric acid

In order to evaluate the input and performances of the use of electrochemistry to quantify the extracted metal cations from highly concentrated nitric acid solution using the examined N,N-DAs, Ce(IV) was extracted from an aqueous nitric acid solution (5M) to DEHBA, DEHiBA and DEHDMBA previously equilibrated with HNO₃ (5M). For a HNO₃ (5M) aqueous solution containing initially 15.2 mM of Ce(IV), the cyclic voltammograms of the extracted Ce(IV) at 40°C are shown in Fig. 4. The use of the calibration curves shown in Fig. 3 allows determining the concentration of Ce(IV) in DEHBA at 25°C and 40°C. The obtained value is 12.8 \pm 1.3 mM which is compared to that obtained by ICP-OES of Ce(IV) in nitric acid aqueous solution before and after its extraction. These values are reported in Table 3 and they clearly indicate that cyclic voltammetry can be used to determine successfully yield of extraction of Ce(IV) with DEHBA.





Figure 4. Cyclic voltammograms of Ce(IV) extracted in (a) DEHBA/HNO₃(5M), (b) DEHiBA/HNO₃(5M) and (c) DEHDMBA/HNO₃(5M) at 40°C with several scanning rates and with ohmic drop correction (dynamic mode) of 5 K Ω , 15 K Ω and 20 K Ω , respectively. (see Fig S1 (supplementary information section) for the characterization of the

residual current in DEHBA/HNO₃(5M) and in absence of Ce(IV)).

Table 3. Analysis data of Ce(IV) upon its extraction from nitric acid solution with DEHBA

Analyzed solution	HNO ₃ (5M) before extraction	HNO ₃ (5M) after extraction with DEHBA	DEHBA/HNO ₃ (5M) extract	DEHBA/HNO ₃ (5M) extract
Quantification method	ICP/OES	ICP/OES	Cyclic voltammetry	Calculated
Ce(IV) concentration	(15.2±0.3) mM	$(3.0 \pm 0.3) \text{ mM}$	$(12.8 \pm 1.3) \text{ mM}$	$(12.2 \pm 0.3) \text{ mM}$

The high viscosity of the two other extractants and the illdefined cyclic voltammograms do not allow unfortunately to obtain very reproducible data and thus the validation of the potential of this approach is not as promising as the one implementing DEHBA.

3. Conclusion

In this work we report on the electrochemical behavior of Ce(IV)/Ce(III) redox couple in pure DEHBA), DEHiBA) and DEHDMBA equilibrated with nitric aqueous solutions as an entry to the direct electrochemical characterization of plutonium in these extractants. The redox process exhibits a reversible couple which potential (E $_{\rm 1/2}\cong$ 1.02 V/SCE) is not affected by the temperature and the nature of the N,N-DA. This clearly indicates that the functionalities of the extractants produce the same relative effect for both +IV and +III oxidation states of the cerium cation. Calibration curves for possible quantification of these Ce(IV) after its extraction by these solvants were also obtained but due to the high viscosity and low conductivity of DEHiBA and DEHDMBA, such allows of characterization the evaluation the performances of the chemical extraction of Ce(IV) from aqueous nitric acid solution only by the undiluted DEHBA. Our findings will for sure open the door for further investigations in this field.

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4. References

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