Contents lists available at ScienceDirect

Electrochimica Acta

journal homepage: www.elsevier.com/locate/electacta

Studies on the feasibility of electrochemical recovery of palladium from high-level liquid waste

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ARTICLE INFO

Article history: Received 15 April 2008 Received in revised form 21 August 2008 Accepted 21 August 2008 Available online 28 August 2008

Keywords: Palladium Fission product High-level liquid waste Electrodeposition Voltammetry

ABSTRACT

The electrochemical behavior of palladium (II) in nitric acid medium has been studied at platinum and stainless steel electrodes by cyclic voltammetry. The cyclic voltammogram consisted of a surge in cathodic current occurring at platinum electrode at a potential of -0.1 V (vs. Pd), which culminates in a peak at -0.3 V was due to the reduction of Pd(II) to Pd. This was accompanied by a broad scant anodic peak (I_p^a) at 0.25 V during scan reversal. Reduction of Pd(II) was irreversible and the diffusion coefficient was found to be 2.35 × 10⁻⁸ cm²/s at 298 K. At stainless steel electrode, a surge in the cathodic current occurring at -0.4 V (vs. Pd) was due to palladium deposition, which was immediately followed by a steep increase in cathodic current at -0.66 V due to H⁺ reduction. Electrolysis of palladium nitrate from 1 M to 4 M nitric acid medium at stainless steel electrode resulted in complete recovery of palladium with reasonably high Faradaic efficiency depending upon nitric acid concentration. However, the recovery and Faradaic efficiency were significantly lowered (to 40%) in the case of electrolysis from simulated high-level liquid waste due to other interfering competitive reactions.

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1. Introduction

PUREX process is being adopted for the separation of uranium and plutonium from the spent nuclear fuel dissolver solution [1]. The nitric acid raffinate obtained after extraction is known as "highlevel liquid waste" (HLLW), which contains significant quantities of valuable fission platinoids [2]. Most of the isotopes of platinum group metals (Ru, Rh and Pd) formed from fission reaction are nonradioactive or weakly radioactive. For example fission palladium is composed of few stable isotopes (83%) and a radioactive ¹⁰⁷Pd(17%), which is a soft β^- emitter with a half-life of 6.5 × 10⁶ years. The intrinsic radioactivity of ¹⁰⁷Pd is very weak and can be tolerated for several industrial applications. Therefore, there is a growing interest in the recovery of palladium from HLLW [3].

Several separation methods such as solvent extraction [4], ion exchange [5], non-aqueous methods [6], etc. have been reported for the recovery of palladium. Excellent reviews in this regard by Kolarik and Renard [3,7,8], and Pokhitonov and Romanovskii [9], detail the methods reported to date for the separation of fission platinoids. The electrochemical method for the separation and recovery of palladium is one of the easy and promising techniques due to its simplicity, accessibility of reduction potential of palladium in nitric acid medium. Besides, this method does not

demand the addition of external reagents in HLLW. Koizumi et al. [10] reported the electrolytic extraction of fission platinoids from nitric acid medium. A recovery of 90%, 23% and 10% was reported respectively for the deposition of Pd, Rh and Ru, and the deposition rates were reported to decrease with increase of nitric acid concentration. Ozawa et al. developed a new strategy for the back end of nuclear fuel cycle, known as Adv.-ORIENT, to enhance the separation of potentially useful fission products such as palladium and transmutation of minor actinides [11,12]. Kirshin et al. [13] studied the electrolytic recovery of palladium from nitric acid solutions and reported the efficiency of the process in the presence of HNO₃, NaNO₃, uranium and other admixtures. However, detailed electrochemical behavior of palladium (II) in nitric acid medium, speciation and recovery from simulated waste of fast reactor fuels has not been reported so far. Therefore the objective of the present paper is to report the results of the electrochemical behavior of Pd²⁺ in nitric acid medium, investigated by cyclic voltammetry. Electrodeposition of palladium in the presence of various interfering elements present in high-level liquid waste, effect of nitric acid concentration, and recovery from simulated high-level waste, etc. are also reported.

2. Experimental

All the chemicals used in the study were of analytical AR grade. Palladium (II) nitrate in nitric acid solution (10%, w/v) was procured from Arora Matthey, Kolkata, India.





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^{0013-4686/\$ -} see front matter © 2008 Elsevier Ltd. All rights reserved. doi:10.1016/j.electacta.2008.08.034

2.1. Voltammetry of palladium (II) nitrate in nitric acid

A solution of palladium nitrate (20 mM) in nitric acid medium was prepared by dissolving a required quantity of palladium nitrate stock in appropriate concentration of nitric acid (25 mL). Electrochemical studies and electrolysis were conducted at 298 K. In voltammetric measurements, platinum wire was used as the working (cylindrical, SA=0.11 cm²) and counter electrodes, and palladium wire was used as quasi-reference electrode. All the voltammetric data were obtained after IR compensation. Electrodeposition was conducted on a stainless steel (6 cm²) plate with platinum plate as counter electrode and palladium wire as quasi-reference electrode. The choice of stainless steel as working electrode during electrolysis was due to its inexpensiveness and its easy availability for scaling-up operations. After the deposition, the plate was washed extensively with acetone and deionized water before subjecting it for morphological examination.

2.2. Instrumentation

Cyclic voltammograms of the solutions were recorded using Autolab (PGSTAT- 030) equipped with an IF 030 interface. UV–Vis absorption spectrum was recorded using Shimadzu UV–Vis spectrometer model 2500. A Philips field effect scanning electron microscope (SEM), model XL 30, with energy-disperse spectrometer (EDS) working at 30 kV was used to examine the surface morphology and elemental composition of the deposit.

3. Results and discussions

Fig. 1 shows the cyclic voltammogram of palladium (II) in nitric acid medium recorded at platinum electrode at the potential sweeping rate of 100 mV/s. A surge in the cathodic peak current occurring at a potential of -0.1 V (vs. Pd), which culminates in a peak at -0.3 V, is due to the reduction of palladium. A broad oxidation wave is observed at 0.25 V during scan reversal, indicating that the reduction of palladium (II) at platinum electrode is irreversible. Increasing the nitric acid concentration decreases the cathodic peak current (I_p^c) and broadens the cathodic wave. This effect could be attributed to the conversion of free palladium (II) ion in to various nitro-complexes of palladium. The absorption spectra of Pd(II), shown in Fig. 2, also exhibits a bathochromic shift with increase in the concentration of nitric acid due to nitrate



Fig. 1. Comparison of cyclic voltammograms of Pd(II) in different concentrations of HNO₃ recorded at platinum electrode. Quasi-reference: palladium wire, T = 298 K, scan rate = 0.1 V s⁻¹.



Fig. 2. Comparison of UV-vis absorption spectrum of Pd(II) in various concentrations of nitric acid. [Pd] = 1×10^{-4} M.

complexation. Purans et al. [14] studied the structure and speciation of Pd²⁺ in nitric and perchloric acid medium. The stability constants for the complexation of palladium (II) ion with nitrate ions in nitric acid medium, derived from solvent extraction data, were reported by Tarapcik [15]. Based on those stability constants $(\beta_1 = 3.28, \beta_2 = 2.13, \beta_3 = 0.223, \beta_4 = 0.004)$ speciation of palladium in nitric acid medium was determined and is depicted in Fig. 3. It is seen that in 0.1 M nitric acid medium, majority of palladium exists as free ion $[Pd(H_2O)_4]^{2+}$ and its concentration decreases with increase in the concentration of HNO₃. The concentration of [Pd(NO₃)(H₂O)₃]⁺ is maximum at 1 M HNO₃, and the concentration of neutral species, $[Pd(NO_3)_2(H_2O)_2]$, increases with increase in concentration of nitric acid. Accordingly, the λ_{max} observed in the visible absorption spectrum of palladium nitrate solution, shown in Fig. 2. is also shifted bathochromically with increase in the concentration of nitric acid. Thus, the charge of the electroactive species decreases with increase in the concentration of nitric acid that seems to reduce the diffusion of palladium species and lowers the cathodic peak current $(I_{\rm p}^{\rm c})$.



Fig. 3. Speciation of various Pd(II) nitrate complexes in various concentrations of nitric acid.



Fig. 4. Comparison of cathodic sweep voltammograms of Pd(II) present in various concentrations of nitric acid recorded at platinum rotating disc electrode (r = 500 rpm). Counter electrode: platinum wire. Reference electrode: palladium wire. T = 298 K, scan rate: 0.01 V s^{-1} .

The electrochemical behavior of palladium (II) was also investigated at rotating disc electrode. Fig. 4 shows the cathodic sweep voltammogram of palladium (II) present in nitric acid medium recorded at platinum rotating disc electrode (radius = 1.5 mm) at the scan rate of 10 mV/s. It is observed that the cathodic current increases with increase of applied potential due to the reduction of palladium (II) ion, which is subsequently followed by increase of cathodic current arising from the reduction of H⁺ ion (H₂ evolution). A limiting current for the reduction of palladium (II) is obtained only in the case of reduction from 0.1 M nitric acid medium, where distinct curves for the reduction of palladium (II) as well as for H⁺ are observed. However, at higher nitric acid concentrations the onset of reduction of palladium is shifted to more negative potentials and merges with H⁺ reduction. In addition, since the concentration of H⁺ is very high (4 M) as compared to palladium (II) ion (in 20 mM), the reduction of palladium (II) at RDE is not apparent in the voltammogram shown in Fig. 4. A similar behavior of broadening of cathodic wave and shifting of cathodic peak potential (E_p^c) to more negative values is also observed during the cyclic voltammetric investigations (Fig. 1). Therefore the diffusion coefficient of palladium (II) could be determined only for the reduction from 0.1 M nitric acid medium using Levich equation, shown in Eq. (1) [16,17], that relates the limiting current (i_1) and angular velocity ($\omega = 2\pi r/60$, r is revolutions per minute).

$$i_1 = 0.62n \text{FCAD}^{2/3} \eta^{-1/6} \omega^{1/2} \tag{1}$$

$$\frac{1}{i} = \frac{1}{i_{\rm K}} + \frac{1}{0.62n{\rm FCAD}^{2/3}\eta^{-1/6}\omega^{1/2}}$$
(2)

where η is the kinematic viscosity (10^{-2} cm²/s). Fig. 5 shows the cathodic sweep voltammogram of palladium (II) in 0.1 M nitric acid at various rotation rates (300-1500 rpm) recorded at platinum rotating disc electrode. It is observed that the limiting current increases with increase of rotation speed. The limiting current is measured at a potential of -0.40 V and plotted against $\omega^{1/2}$. The linear regression of the data showed that the intercept is marginally deviating from the origin, as shown in Fig. 5. Therefore, Koutecky-Levich equation (Eq. (2)) is used for determining the diffusion coefficient [16,17], where *i* is the magnitude of current at a potential of -0.4 V in the present case and $i_{\rm K}$ is the current in the absence of any mass transfer effects. A plot of inverse of current (1/*i*,) against $\omega^{-1/2}$ (Koutecky–Levich plot) is also shown in Fig. 5. From the slope of the straight line the diffusion coefficient of palladium (II) in 0.1 M nitric acid medium was found to be $2.35 \times 10^{-8} \text{ cm}^2/\text{s}.$

4. Electrolysis experiments

4.1. Selection of applied potential

Electrodeposition of palladium from nitric acid medium was studied at constant applied potential. The experiments involved



Fig. 5. Cathodic sweep voltammogram of Pd(II) present in 0.1 M nitric acid recorded at platinum rotating disc electrode at various rotations. *Insert*: Levich (i_1 vs. $\omega^{1/2}$) and Koutecky–Levich (1/i vs. $\omega^{-1/2}$) plots. The limiting current measured at -0.4 V.

Table 1

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Applied potential (vs. Pd)	Charge passed in coulombs	Time, h	Recovery obtained	Expected of charge pas	deposit for the ssed (grams)	Faradaic efficiency (%)	
				grams	%		
-0.4	0.252	8.5	0	0	0	0	
-0.45	2	8.5	<0.005	<5	0.0028	36.3	
-0.5	191	9.3	0.055	100	0.1053	52.2	
-0.6	154	4.6	0.055	100	0.0848	64.8	

electrolysis of a 20 mM palladium nitrate solution in nitric acid medium with stainless steel (6 cm^2) as cathode and platinum plate as anode. The potential was varied from -0.4 V (onset of palladium (II) reduction at stainless steel electrode) to -0.6 V (vs. Pd) to choose the optimum potential required for electrolysis. The result of electrolysis of palladium nitrate solution from 4 M nitric acid medium is tabulated in Table 1. It is observed that the recovery of palladium deposit increases with increase of applied negative potential and at potentials more negative than -0.5 V (vs. Pd), the recovery is quantitative. However, increasing the applied negative potential may also favor other undesirable redox reaction and considering this and duration of electrolysis the potential of -0.5 V is chosen as the optimum potential for electrolysis using stainless steel as cathode.

4.2. Variation of [HNO₃]

The influence of nitric acid concentration on the electrodeposition of palladium was studied at a constant applied potential of -0.5 V. The concentration of nitric acid was varied from 1.0 M to 4.0 M. Fig. 6 shows the number of coulombs passed as a function of time at various nitric acid concentrations. It is observed that there is a gradual increase in the coulombic charge density (number of coulombs passed per unit area) in the initial stages of electrolysis and after three hours there is an abrupt increase in the passage of coulombic charge. Significant deposition of palladium was visually observed on cathode during this abrupt increase in passage of coulombic charge density. This is accompanied by a rapid change in the color of the solution from dark orange to colorless. The concentration of palladium left in the solution after six hours of electrolysis was found to be below the detection limit



Fig. 6. Charge–time curves for the electrodeposition of palladium on a stainless steel plate from different nitric acid concentrations. $A = 5.92 \text{ cm}^2$. Time = 8 h. Potential = -0.5 V (vs. Pd). Counter electrode: platinum.

(<0.5 ppm) of a spectrophotometric method using Arsenazo III as coloring agent. In all these experiments, significant evolution of gases, hydrogen and NO_x, was observed at the working electrode only when the experimental solution was becoming colorless. The recovery of palladium was quantitative at all nitric acid concentrations investigated and the Faradaic efficiency (calculated after eight hours of electrolysis) was -75% at 1.0 M nitric acid and was lowered to -55% at 4.0 M nitric acid. This could be due to the electrochemical reduction [18] of nitrate or H⁺ ion (discussed below). The deposit obtained from the electrolysis was subjected to surface morphological examination by scanning electron microscopy (SEM). The deposit, shown in Fig. 7, is uniform all over the surface of stainless steel electrode with a dendrite growth observed in all cases. The dendrites seem to increase with increase in the concentration of nitric acid.

The interference of various cations and anions during the deposition of palladium was investigated by cyclic voltammetry using stainless steel as working electrode, platinum as counter electrode and palladium as quasi-reference (same electrode system used for electrolysis). The cyclic voltammogram of 0.1 M nitric acid solution recorded at stainless steel electrode is shown in Fig. 8A and the nitric acid is stable from -0.9 V to 0.17 V (vs. Pd). The reduction of palladium (II) from nitric acid medium is compared with the reduction of various ions such as Ag(I), UO_2^{2+} , NO_3^{-} , Fe(II), etc., that are likely to be present in high level liquid waste and also lower the Faradaic efficiency of palladium deposition. A surge in the cathodic current occuring at -0.4V (vs. Pd), is due to palladium deposition (Fig. 8B), which is subsequently accompanied by a steep increase in cathodic current at -0.66 V, is due to H⁺ reduction. Such behavior is not observed during the deposition of silver (one of the fission products) on cathode (Fig. 8C). This clearly indicates that the reduction of palladium (II) from nitric acid medium results in a deposition of metallic palladium on stainless steel electrode and due to the characteristic property of palladium metal, the deposit seems to catalyze the underpotential reduction of H⁺ ion [18] at the electrode. It is also observed from Fig. 8 (voltammograms C to F), that the onset of reduction for a solution of silver nitrate, uranyl nitrate, ferric nitrate and sodium nitrate in nitric acid medium at stainless steel electrode occurs at the potentials, of -0.3 V, -0.71 V, -0.81 V, -0.65 V respectively. These potentials are very close to the onset of reduction (-0.4 V) of palladium (II). However, when palladium is co-existing in the solution along with these ions the reduction reactions are shifted to lower negative potentials (i.e. underpotential reduction) as shown in Fig. 8 (voltammograms G to J). This indicates that the initial deposition of palladium on stainless steel electrode modifies the electrode surface to metallic palladium, favors underpotential reduction and also substantially increases the cathodic current in all cases. These factors tend to lower the Faradaic efficiency of palladium deposition.

It was also found that lowering the applied negative potential (towards nobler) lowered the recovery palladium (as tabulated in Table 1) and the presence of rare-earth elements, strontium and cesium (equal to the amounts present in HLLW shown in Table 2) did not affect the recovery of palladium to any significant extent.

Fig. 7. SEM images of palladium obtained by electrolysis from different nitric acids and simulated high-level waste. (A) 1 M HNO₃, (B) 2 M HNO₃, (C) 3 M HNO₃ and (D) fast reactor high-level waste. Magnification 2000× for A, B and C images and 1000× for image D.

However, when the mole ratio of uranium to palladium exceeds four and presence of sodium nitrate (>0.5 M) in 0.1 M HNO₃ solution lowers the recovery (40% after 8 h at -0.5 V) and Faradaic efficiency significantly. This could be due to other undesirable electrochemical reactions as discussed above. A similar behavior was also reported by others [10,13]. Krishin et al. [13] suggested that the presence of U(VI) in HLLW promotes the reduction of nitric acid and lowers the current efficiency.



Fig. 8. Cyclic voltammograms of various interfering ions present in 0.1 M HNO₃ medium recorded in the absence of palladium (C–F) and in the presence of palladium (G–J), at stainless steel working electrode. Counter electrode: Pt wire. Reference electrode: Pd wire. T = 298 K. (A) 0.1 M HNO₃, (B) 50 mM Pd, (C) 2 mM AgNO₃, (D) 0.3 M NaNO₃ (high concentration of NaNO₃ required to obtain the reduction wave), (E) 6 mM Fe(NO₃)₃, (F) 30 mM UO₂(NO₃)₂, G = 9 mM AgNO₃, H = 0.3 M NaNO₃, I = 6 mM Fe(NO₃)₃, J = 30 mM UO₂(NO₃)₂. The voltammograms G–J were obtained in the presence of 50 mM PdNO₃.

Table 2

Elemental compositions of simulated high-level liquid waste (HLLW) showing pressurized heavy water reactor and fast breeder reactor spent fuel compositions [19]

Element	Elements present (g/L)							
	PHWR (6500 MWd/tonne)	FBR (80,000 MWd/tonne)						
Sodium	3.01	3.00						
Iron	0.50	0.50						
Nickel	0.10	0.10						
Selenium	0.01	0.01						
Strontium	0.19	0.14						
Zirconium	0.77	0.89						
Cesium	0.55	1.12						
Barium	0.31	0.41						
Lanthanum*	0.26	0.48						
Cerium	0.54	0.69						
Praseodymium	0.24	0.34						
Neodymium	0.86	1.131						
Samarium	0.17	0.05						
Europium	0.02	0.31						
Gadolinium	0.02	0.07						
Silver	0.02	0.13						
Cadmium	0.02	0.04						
Yttrium	0.10	0.08						
Promethium	0.03	0.05						
Terbium	0.001	0.01						
Dysprosium	0.001	0.005						
Uranium	18.33	2.64						
Technetium	0.18	0.26						
Molybdenum	0.73	1.09						
Ruthenium	0.46	0.81						
Rhodium	0.13	0.26						
Palladium	0.27	0.60						
Chromium	0.10	0.10						
Rubidium	0.08	0.06						
Tin	0.02	0.02						
Antimony	0.01	0.01						
Tellurium	0.10	0.16						
Acidity (M)	4.0	4.0						

*La: added for Y, Pm, Te, Dy; Tc, Mo: not added; Co: added as Ni; Ru, Rh, Rb: added as Pd; Sn, Sb, Te: not added due to poor solubility. Excess palladium taken to study the feasibility of extraction.

4.3. Recovery of palladium from simulated high level waste

Electrolysis of simulated waste solution was carried out to assess the feasibility of separating palladium from high-level liquid waste solution. The composition of simulated HLLW [19] is shown in Table 2. Electrolysis was carried out at -0.5 V (vs. Pd) using stainless steel as cathode and platinum as anode for eight hours. The results obtained from the electrolysis of palladium nitrate present in simulated high-level waste of PHWR and FBR are identical. In these experiments, gradual deposition of palladium on cathode was observed during initial stages of electrolysis. However, copious evolution of gases accompanied by rapid increase in coloumbic charge density was observed after 4–5 h of electrolysis. Subsequently there was no significant deposition of palladium even after eight hours. Therefore, the electrolysis was discontinued after five hours and the recovery was determined to be -40% (in some experiments it was 25-30%) and Faradaic efficiency was 30%. The lower recovery and Faradaic efficiency could be due to the presence of several interfering ions like uranium (VI), nitrate, iron, silver, etc. in HLLW whose redox reactions to lower valent state also occur near palladium deposition potential in nitric acid medium (Fig. 8). In contrast, electrodeposition of palladium was quantitative when electrolysis was carried out in nitric acid medium alone as described earlier. In those experiments, significant gas evolution was observed only after 80% of palladium was deposited. However, in the present case, the presence of elements such as iron, silver, nitrate in simulated HLLW limits the deposition of palladium and facilitates the competitive redox reactions and gas evolution (H_2, NO_x) , that seems to be responsible for lower recovery and Faradaic efficiency. The SEM image of the palladium deposit obtained from the HLLW of fast reactor is shown in Fig. 7, which also indicates the formation of dendrites during deposition.

5. Conclusions

The neutral palladium (II) nitrate specie existing in 3-4 M nitric acid medium undergoes an irreversible single step two-electron transfer to metallic palladium at platinum electrode. While the palladium nitrate present in nitric acid medium could be quantitatively recovered by electrolysis at -0.5 V (vs. Pd), several complications aggravated in the presence of interfering elements of HLLW such as silver, nitrate, iron, etc. during electrolysis. Due to these, the recovery and Faradaic efficiency was below 40%. The study, therefore, suggests the requirement of other separation methods either independent or coupled with electrochemical method for the quantitative recovery of palladium from high-level liquid waste.

Acknowledgements

The authors are thankful to the reviewers for their constructive suggestion. The authors also thank Ms. R. Sudha, Materials Chemistry Division, IGCAR and Dr. R. Rangarajan, Water and Steam Chemistry Division, BARC-F, Kalpakkam for providing SEM images and RDE respectively.

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