Sensitive detection of uranium in water samples using differential pulse adsorptive stripping voltammetry on glassy carbon electrode

Zhiping Zhou[1] · Yueming Zhou[1] · Xizhen Liang[1] · Fang Xie[1] · Shujuan Liu[1] · Jianguo Ma[1]

Abstract

Direct and rapid determination of trace amounts of uranium in natural water remains a challenge. Herein, determination of uranium by differential pulse adsorptive stripping voltammetry (DPAdSV) in presence of cupferron and diphenylguanidine on glassy carbon electrode was investigated. Under optimized conditions, the DPAdSV peak current was proportional to the concentration of uranium in the range of 3–80 µg L^{-1} with the detection limit of 1.0 µg L^{-1} and a linear correlation coefficient of 0.999. The DPAdSV method based on GCE was successfully applied to direct determination of trace uranium in natural water samples.

Keywords Uranium · Differential pulse adsorptive stripping voltammetry · Cupferron and diphenylguanidine · Glassy carbon electrode

Introduction

As one of the primary clean energy sources, nuclear power can provide reliable and safe electricity power without emitting greenhouse gases. Uranium is called the new energy elite because it is one of the most important raw materials in nuclear energy industry [1]. However, its radioactive waste can endanger human health, like causing kidney damage and acute arterial disease. These potential hazards have aroused public concern, especially after the Fukushima Daiichi nuclear accident in 2011. The World Health Organization (WHO) has regulated the maximum concentration level of uranium in drinking water no more than 30 µg L^{-1} [2–5]. Thus, it is important to monitor the concentration of U(VI) in nuclear power plant, uranium mining area and environmental waters [6–9].

Many efforts have been made to detect uranium in environmental water samples using various of methods, such as inductively coupled plasma optical emission spectrometry (ICP-OES) and ICP-MS [10–13], fluorescence spectroscopy [5, 7, 8, 14–16], UV–vis spectrophotometry [17–19], neutron activation analysis [20, 21] and radiometric techniques [22, 23] (alpha- and gamma-spectrometry) combined with some separation techniques to further improve sensitivity. However, these methods are usually based on complex operation, high cost of apparatus and maintenance, which are not available for monitoring trace uranium in small laboratories and on-site environment [10]. Electrochemical analysis is relatively simple, convenient and low cost in operation [4, 6, 24]. Adsorptive stripping voltammetry (AdSV) based on complexing reagent of uranium [4, 25–30] on mercury electrodes provides low limits of detection and reliable analytical procedures [6, 31–34]. A parallel chemical re-oxidation of U(III) to U(V) was due to the complexation and oxidation of cupferron, and hence to an enhanced signal on mercury electrode [30]. The commonly used mercury electrode shows excellent electrochemical performances due to a fresh electrode surface and forming amalgams; however, its toxicity and the complex of operation device cannot be ignored [3, 27, 31].

The mercury-free electrodes hold promise to monitor uranium in environmental water samples [28, 35–38]. Zhang et al. [38] have made progress in the determination

✉ Yueming Zhou
  ymzh@iccas.ac.cn; zyming11@gmail.com

✉ Jianguo Ma
  mjgyh8125@163.com

1 State Key Laboratory of Nuclear Resources
   and Environment, Jiangxi Province Key Laboratory
   of Polymer Micro/Nano Manufacturing and Devices, College
   of Chemistry, Biology and Materials Science, East China
   University of Technology, Nanchang 330013, China

of uranium by tin–bismuth alloy electrode and proposed a more detailed mechanism of U(VI)-cupferron. Among these mercury-free electrodes, carbon material electrodes, like glassy carbon electrode (GCE), carbon paste electrode and graphite electrode, have received extensive attention in radioanalytical chemistry due to their non-toxicity, wide potential window and low cost [35, 39–43]. The GCE was employed as a common electrode material for trace uranium analysis combined with a complexing agent due to good electrical conductivity and electrocatalytic activity in diverse redox reactions. This suggests that GCE as a work electrode combined with cupferron is promising to determine trace uranium by DPAdSV [44, 45].

In this work, the GCE was used for direct determination of uranium by DPAdSV in presence of cupferron and exhibited decent sensitivity and reproducibility. Cupferron as a complexing agent and diphenylguanidine as a synergistic reagent were studied. Under the controllable conditions, the determination of uranium reached a lower detection limit of part-per-billion level. This method was applied for uranium detection in real water samples.

Experimental

Reagents and solutions

All chemicals, namely, cupferron, ethanol and HNO_3 (Sinopharm Chemical Reagent Co., Ltd), diphenylguanidine (Shanghai Aladdin Biochemical Technology Co., Ltd) were used as received. Uranyl nitrate stock solution was prepared by dissolving U_3O_8 in diluted nitric acid. 0.1 M diphenylguanidine was prepared by diluting appropriate amount of reagent in ethanol. 0.1 M acetate buffer solution was obtained under different conditions by mixing the stock solution of acetic acid and sodium acetate. A fresh solution of 0.2 M cupferron was prepared in double-distilled water (DDW).

Apparatus

The cyclic voltammetry (CV) and DPAdSV were performed using PARSTAT 2273 electrochemical workstation (AMET Co., Ltd., Princeton, USA) and CHI 660D electrochemical workstation (Shanghai Chenhua Instrument Co., Ltd., China). A conventional three-electrode cell containing GCE (Φ 1, 3 mm) as the working electrode, a saturated calomel electrode (SCE) as the reference electrode and a platinum foil as the counter electrode was used. The pH measurements were performed with a PHS-3C pH meter (INESA Scientific Instrument Co., Ltd., China).

Electrode preparation

GCE was carefully polished to a mirror-like surface with 0.3 μm and then 0.05 μm gamma alumina slurry, rinsed with DDW in each polishing step, next ultrasonicated in DDW for 2 min to remove some adsorbed alumina particles or dirt from the electrode surface [46, 47].

Analytical procedure

The detection of UO_2^{2+} was performed in an electrochemical cell containing 0.1 M acetate buffer solution and a certain concentration of cupferron and diphenylguanidine. The deposition of UO_2^{2+} was carried out at -0.95 V (vs SCE) under stirring with a deposition time of 120–180 s, and then the stripping voltammogram was recorded in quiescent solution from -0.8 to -1.3 V (vs SCE) by DPAdSV. Each measurement was repeated thrice. All electrochemical experiments were carried out at room temperature.

Results and discussion

Voltammetry determination in uranium–cupferron system

An excellent complexing agent cupferron was used to determine metal ions by voltammetry [6, 48]. The uranium–cupferron complex formed with diphenylguanidine synergistic effect was strongly adsorbed on the surface of GCE, leading to higher peak current of uranium [30, 38]. Figure 1a shows the cyclic voltammogram of 0.5 mg L^{-1} U(VI) in 0.1 M pH 4.5 acetate buffer solution. The cathodic peak current of uranium (solid curve) was obtained obviously while not observed at concentration of 10 μg L^{-1} U(VI). Figure 1b shows the DPAdSV voltammogram of 10 μg L^{-1} U(VI) in 0.1 M pH 4.5 acetate buffer solutions. Compared to the uranium stripping peak current, it can be clearly seen that a sharper increase of the uranium stripping current was exhibited with the addition of diphenylguanidine in the cupferron and uranyl solution. The diphenylguanidine radical cations may induce adsorption of uranium–cupferron complexes during preconcentration, resulting in higher response currents [38].

Effect of pH

The pH values play an important role on the complexation of uranyl ions in solution [49]. Herein, acetate buffer solution chosen as the supporting electrolytes is ascribed to the stable complexes of acetate and uranyl ion [50]. As shown

in Fig. 2a, uranyl species with positive or neutral charge are present in the solution of the pH range of 3–4, whereas at pH > 4.5 neutral and anionic species prevail. In addition, the effect of pH suggests that the coordination interaction of the ligand cupferron with U(VI) competes with the acetate in solution. As shown in Fig. 2b, the uranium stripping peak current was strongly affected by pH values. The uranium stripping response decreased at pH < 4.5, which was a consequence of the reduction of cupferron to phenylhydrazine on the surface of electrode [6, 30]. The uranium peak decreased at pH > 4.5 because the hydrolysis of UO_2^{2+} ions resulted in great decrease of electroactive complex on the surface of electrode [38].

Effect of the cupferron and diphenylguanidine concentration

The impact of the concentration of cupferron and diphenylguanidine on the uranium stripping peak current was further investigated. As shown in Fig. 3a, it was clear that net
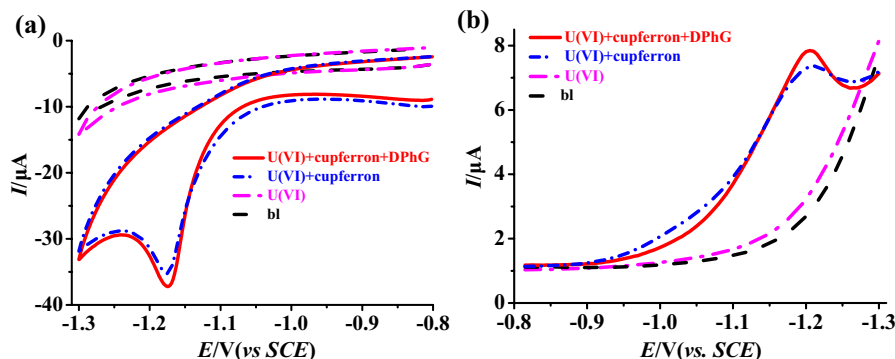

Fig. 1 **a** Cycle voltammetry in 0.1 M acetate buffer of pH 4.5 for 0.5 mg·L^{-1} U(VI) with 160 μM cupferron and 40 μM diphenylguanidine (solid), 0.5 mg·L^{-1} U(VI) with 160 μM cupferron (short dash dot), 0.5 mg·L^{-1} U(VI) (dash dot) and reagent blank (dash). Scan rate, 100 mV s^{-1}. **b** DPAdSV in 10 μg L^{-1} U(VI) in the same solution composition as **a**. Scan rate, 50 mV s^{-1}; pulse height, 25 mV; step height, 5 mV; step time, 0.1 s, deposition time 120 s, deposition potential −0.95 V

Fig. 2 Effect of different pH on **a** U(VI) speciation [49], as predicted by MINEQL + for conditions of 10 μg L^{-1} U(VI) in 0.1 M acetate buffer, and **b** the peak current of 10 μg L^{-1} U(VI), with 160 μM cupferron and 40 μM diphenylguanidine in 0.1 M acetate buffer using deposition of 120 s and deposition potential of −0.95 V
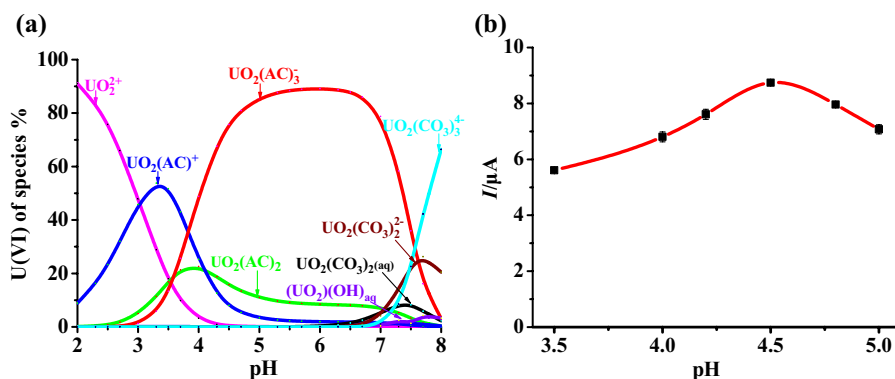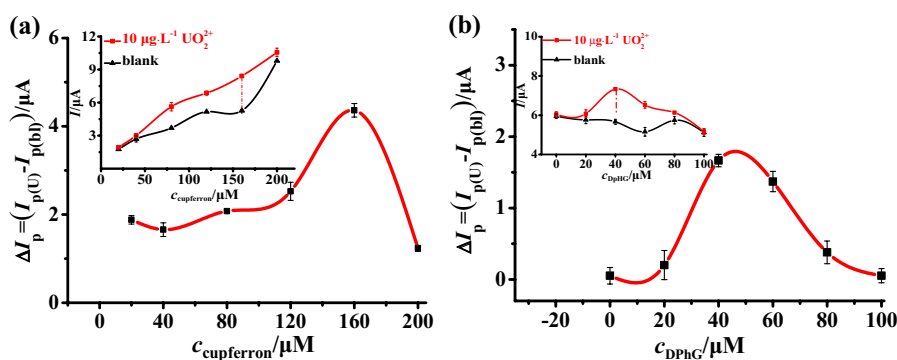

Fig. 3 DPAdSV in 0.1 M acetate buffers of pH 4.5 for the effect of **a** cupferron and **b** diphenylguanidine concentration on net current of cupferron and U(VI)-cupferron ($\Delta I_p = I_{p(U)} - I_{p(bl)}$). The inset shows current curve of 10 μg·L^{-1} U(VI) and blank solution

current of cupferron and U(VI)-cupferron ($\Delta I_p = I_{p(U)} - I_{p(bl)}$) reached a maximum with the concentration of 160 µM cupferron and 40 µM diphenylguanidine. The ΔI_p increased at the concentration of cupferron lower than 160 µM, which was the effect of complex between cupferron and UO_2^{2+} in solution. The ΔI_p rapidly decreased at the concentration of cupferron higher than 160 µM, which was attributed to the increased ligand concentrations to reduce the stability of U(VI)-cupferron complex [38]. The effect of concentration of diphenylguanidine was exhibited in Fig. 3b. The ΔIp increased rapidly with the diphenylguanidine concentration before the concentration of diphenylguanidine is up to 40 µM. In the weak acid buffering solution, the protonation of diphenylguanidine might induce uranium–cupferron adsorption on the surface of electrode, resulting in higher peak current of uranium [51]. However, the net current dropped sharply after the diphenylguanidine concentration was higher than 40 µM. It was probably because of diphenylguanidine affecting the coordination between cupferron and U(VI). Therefore, 160 µM cupferron and 40 µM diphenylguanidine were selected for the following experiments.

Effect of deposition potential and deposition time

The different deposition potential causes the change of interface potential, surface charge and potential distribution of the electrode/solution electric double layer of a solid electrode surface [52]. In order to further study the effect of the deposition potential on the peak current of uranium, it was investigated between -0.75 and -1.10 V during a constant deposition time of 120 s. The voltammetry scan was initiated after a pause of 10 s without stirring to allow the electrode to equilibrate. As shown in Fig. 4a, the uranium stripping peak current increased at potential range of -0.75 to -0.95 V because of high-efficiency deposition of U(VI)-cupferron complex on the surface of electrode [31]. The uranium peak decreased at potential range of -0.95 to -1.1 V as a result of the U(VI)-cupferron adsorption saturation of the electrode surface [29, 38]. The effect of deposition time on the uranium stripping peak current was investigated

between 0 and 300 s at deposition potential of -0.95 V. As shown in Fig. 4b, it was clear that the response current rapidly increased before deposition time up to 120 s and then almost leveled off, which indicated that electroactive substance adsorptive equilibrium was achieved at electrode surface [28, 29]. Therefore, deposition potential of -0.95 V and deposition time of 120 s were chosen for the following investigation.

Real sample analysis

The main deficiency of stripping voltammetry methods based on the adsorption accumulation of U(VI) complexes is that they are susceptible to disturbances caused by organic compounds present in natural environmental samples, like surfactants [4, 29]. Some common surfactants are known to interfere with analysis quality of environmental water samples [32]. As shown in Fig. 5a, 2 mg L^{-1} Triton X-100 decreased the uranium signal for about 10%; however, caused peak sharp decrease at higher concentrations. Although the uranium signal decreased about 20% at the concentration of 4 mg L^{-1} SDS, uranium peak trended to level off with the concentration increasing. An interesting phenomenon observed was that the uranium peak decreased in the concentration range of 0 to 4 mg L^{-1} CTAB and then increased from 4 to 8 mg L^{-1} CTAB. It seems that the competitive adsorption of CTAB and uranium–cupferron was generated on the surface of electrode resulting in the change of the ratio of redox uranium species. Since environmental water samples usually contain 0.01–2 mg L^{-1} surfactant like Triton X-100 [53], there is almost no effect on the determination of uranium. The effect of foreign ions on the uranium stripping peak current was studied in Fig. 5b. It clearly shows that the uranium peak decreased more than 20% with the addition of Co^{2+}, Cu^{2+} and Pb^{2+}, but other ions have less effect on uranium response current. Considering the low concentration level of Co^{2+}, Cu^{2+} and Pb^{2+} in the environment water, the uranium may be detected directly in real environmental water samples.

Fig. 4 DPAdSV in 0.1 M acetate buffers of pH 4.5 for **a** Effect of deposition potential on the peak current of 10 µg L^{-1} U(VI), **b** Effect of deposition time on the peak current of 10 µg L^{-1} U(VI)

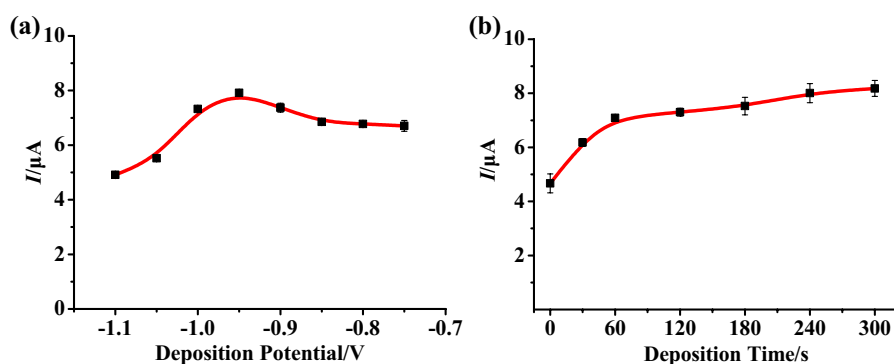**Fig. 5 a** Effect of surface-active substances (Triton X-100, SDS, CTAB) on 10 µg L^{-1} U(VI) determination, **b** Effect of various interference ions at a concentration of 1000 µg L^{-1} on the determination of 10 µg L^{-1} U(VI)
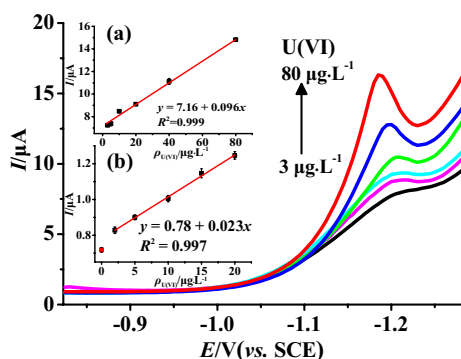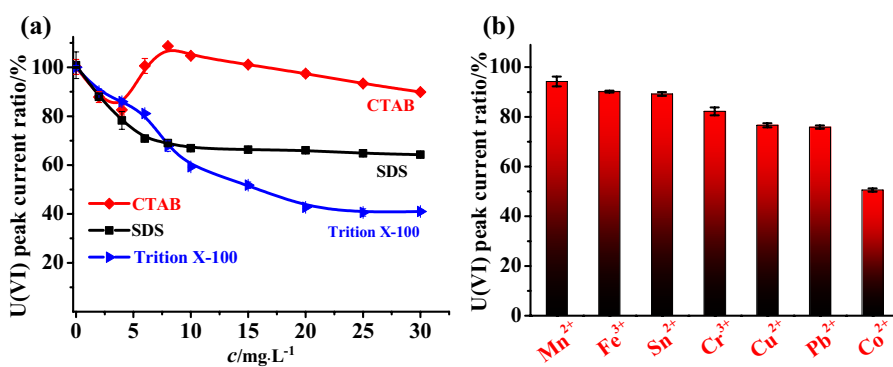

Fig. 6 The DPAdSV for different concentration of U(VI) from down to up, 3.0, 5.0, 10.0, 20.0, 40.0 and 80.0 µg·L^{-1}. The inset shows plot of the peak current of the U(VI) concentration on **a** Φ 3 mm and **b** Φ 1 mm GCE

Figure 6 shows that the peak current rises linearly with the increase of U(VI) concentration based on DPAdSV with the following calibration equation: (a) $y = 7.16 + 0.096 x$ ($R^2 = 0.999$) and (b) $y = 0.78 + 0.023 x$ ($R^2 = 0.997$). The detection limit, calculated $3s_{bl}$/slope, was 1.0 µg L^{-1}. The precise uranium stripping peak currents were obtained with a relative standard deviation (*RSD*) of 5.03% (n = 6). The stripping response of the GCE was examined by carrying out a series of repetitive experiments for a fixed concentration of U(VI) of 10 µg L^{-1}. Repeatability and reproducibility of peak currents were obtained with RSD of 4.4% (n = 30) and 6.1% (n = 21), respectively.

The electrodes proposed in this work were used for direct determination of U(VI) in environmental water samples. The spiked method was used to detect low concentration of U(VI) in natural water to evaluate the performance of the proposed GCE in real sample analysis. The water used in this work was sampled from tap water and East China University of Technology Twin Lakes. The water samples were simply filtered with a long-neck funnel to remove insoluble substances, then the sample acidification before sealed for refrigeration. 5.00 mL water sample was fractioned in 25 ml volumetric flask containing 0.1 M acetate buffer pH 4.5, 160 µM cupferron, and 40 µM diphenylguanidine, and then detected using DPAdSV. Table 1 shows good recovery rates of spiked amount of uranyl ions in the tap water and lake water sample. The comparison test results obtained from the DPAdSV are in accordance well with those detected by ICP-MS, which confirmed the accuracy and precision of the proposed DPAdSV. In addition, Table 2 provides a comparison of the results of the present work versus that of other voltammetric procedures of U(VI) determination in natural waters. The GCE seems to be a promising chemical sensor for the determination of uranium in environmental water.

Conclusion

In summary, a convenient and sensitive differential pulse adsorptive stripping voltammetry method for the determination of trace uranium in presence of cupferrron on a GCE

Table 1 Determination of U(VI) in real water samples

Sample	Added (µg L^{-1})	Found ± SD (µg L^{-1})[a]	*RSD* (%)	Recovery (%)	Detected by ICP-MS (µg L^{-1})
Tap water	10.0	10.11 ± 0.2	3.0	101.1	9.95
	20.0	19.48 ± 0.2	2.7	97.4	19.65
Lake water	10.0	8.97 ± 0.5	7.1	89.7	9.71
	20.0	18.21 ± 0.6	6.6	90.6	19.16

[a]Average value of three experiments

Table 2 Comparison of different voltammetric procedures of uranium determination in water samples

Number	Electrode	Method	Linear range ($\mu g\ L^{-1}$)	Detection limit ($\mu g\ L^{-1}$)	Water samples	References
1	Lead film electrode	SWAdSV[d]	0.119–4.76	0.048	River water	[25]
2	Bismuth film electrode	SWAdSV[d]	10.0–350	0.3	Seawater	[27]
3	Tin–bismuth alloy electrode	DPAdSV[a]	0.119–7.14	0.057	Tap water etc.	[38]
4	RuNPs/GCE	DPV[c]	45.2–352	1.95	Lake water etc.	[2]
5	Au microelectrode	DPASV[b]	No reported	0.24	Tap water	[36]
6	NPAA/GCE	DPAdSV[a]	0.5–30.0	0.15	Tap water	[39]
7	CNT/GCE	SWASV[e]	1.19–28.57	0.23	Tap water	[45]
8	GCE	DPAdSV[a]	3.0–80.0	1.0	Tap water and lake water	This work

[a]Differential pulse adsorptive stripping voltammetry

[b]Differential pulse anodic stripping voltammetry

[c]Differential pulse voltammetry

[d]Square wave adsorptive stripping voltammetry

[e]Square wave anodic stripping voltammetry

was developed. Effects of several parameters, like pH values, concentration of complexing agent, deposition potential and deposition time were investigated. The uranium was detected quickly and efficiently with a detection limit of 1.0 $\mu g\ L^{-1}$, which was well below the concentration of 30 $\mu g\ L^{-1}$ by WHO guideline in drinking water. In addition, the GCE based on DPAdSV was evaluated for its analytical performance using tap water, lake water and interfering substances, such as surfactants and some metal ions. Our results showed a very simple and economic approach for the determination of uranium in environmental water samples. Therefore, the GCE holds promise to be used in the field of electrochemical sensing in the environment water samples.

Compliance with ethical standards

Conflict of interest There are no conflicts to declare.

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