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$ZnWO_4$ Nanorod Modified Electrode for Uric Acid Electrocatalytic Sensing and Application

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In this paper $ZnWO_4$ nanorod was modified on carbon ionic liquid electrode (CILE) and used for voltammetric sensing of uric acid (UA). $ZnWO_4$ nanorod on CILE surface exhibited excellent electrocatalytic activity to UA redox reaction. On cyclic voltammogram the oxidation peak current of UA was enhanced and the oxidation peak potential was decreased, showing a typical electrocatalytic process. Electrochemical behaviors of UA were carefully studied on $ZnWO_4$/CILE with a quasi-reversible diffusional-controlled electrode process. Electrochemical parameters including, the number of electron transfer (n) and the diffusional coefficient (D) were calculated with the results as 2.26 and 3.26×10^{-5} cm^2/s. At the optimal conditions the oxidation peak current of UA was proportional to its concentration from 0.2 to 800.0 μmol/L by differential pulse voltammetry with the detection limit of 0.0637 μmol/L (3σ). The urine from healthy human was chosen as the real sample for the detection of UA content with satisfactory results.

Keywords: Uric acid; $ZnWO_4$ nanorod; Electrocatalysis; Electrochemistry; Carbon ionic liquid electrode.

1. INTRODUCTION

As the product of purine metabolism in living system, uric acid (UA) is often present in blood and urine samples [1]. Being an important analytical target in biological diagnosis the concentration of UA is often related with serious diseases such as kidney damage, gout, renal insufficiency and so on, which requires precise method for UA analysis at lower concentration [2,3]. Various detection methods have been reported for UA detection including spectrophotometry, liquid chromatography, flow-injection analysis and electrochemical method [4-7]. Due to direct electrooxidation of UA on the electrode, various electrochemical methods have been proposed [8-11]. Electroanalysis has exhibited the advantages including higher sensitivity, wider linear range and lower detection limit with cheap instrument and simple procedure. However the UA oxidation on traditional electrodes often gives a high oxidation potential and low sensitivity with the interference of ascorbic acid present in biological fluid. Therefore various modified electrodes have been reported to decrease the oxidation potential with increased sensitivity. Among them nanocomposites have been selected for the decoration and the specific properties of nanomaterials endowed the electrode interface with excellent electrochemical performances [12-14].

Nanosized $ZnWO_4$ is a metal tungstate that has the characteristics including intriguing luminescence and structure, which has been applied to the construction of lase hosts or optical fibers [15,16]. Zhao et al fabricated $ZnWO_4$ nanoparticle modified electrochemical platform for photoelectrocatalysis of azo dyes [17]. Ruan et al checked direct electron transfer of hemoglobin on $ZnWO_4$ nanorod modified electrode [18]. Carbon ionic liquid electrode (CILE) is defined as the usage of ionic liquid as the modifier in carbon paste electrode, which can be acted as the substrate working electrode with excellent performances including large electrochemical windows, stable voltammetric responses and antifouling ability [19]. Nanomaterial decorated CILE has been reported for preparation of electrochemical biosensors with improved performances [20-22].

In this paper electrochemical behavior of UA was studied on $ZnWO_4$ modified CILE. The existence of nanosized $ZnWO_4$ on the electrode was benefit for the redox reaction of UA with the enhancement of the oxidation peak current, providing a sensitive method for UA analysis with urine sample detected. The results indicated that $ZnWO_4$ nanorods had potential applications in electroanalysis for the electroactive molecules detection.

2. EXPERIMENTAL

2.1 Chemicals

UA was got from Fluka (USA) with a 1.0×10^{-4} mol/L stocking solution prepared every day. $ZnWO_4$ nanorods were synthesized according to the reference [23]. Ionic liquid 1-hexylpyridinium hexafluorophosphate ($HPPF_6$) was purchased from Lanzhou Yulu Fine Chem. Co. (China) and acted as the modifier for CILE construction. Chitosan (CTS) was purchased from Dalian Xindie Ltd. Co. (China) and used to immobilize nanomaterial on the electrode surface. 0.1 mol/L phosphate buffer solutions (PBS) were acted as the supporting electrolytes. All the other chemicals were analytical grade and doubly distilled water were used throughout.

2.2 Instrumentation

Electrochemical experiments including electrochemical impedance spectroscopy (EIS), cyclic voltammetry (CV) and differential pulse voltammetry (DPV) were performed with a CHI 750B electrochemical analyzer (Shanghai CH Instrument, China) and a three-electrode cell. CTS/$ZnWO_4$/CILE was homemade to act as working electrode with a saturated calomel (SCE) reference electrode and a platinum wire counter electrode. Scanning electron microscope (SEM) image was recorded using a JSM-7500F SEM (Japan Electron Company, Japan).

2.3 Fabrication of CTS/$ZnWO_4$/CILE

Based on the reference [24], CILE was home-made with $HPPF_6$ and pretreated to get a mirror-like surface. Then 6 µL 0.5 mg/mL $ZnWO_4$ nanorod and 1.0 mg/mL CTS mixture was cast on the CILE surface. After dried at room temperature, CTS/$ZnWO_4$/CILE was got as the working electrode for the following investigation.

3. RESULTS AND DISCUSSION

3.1 Characteristics of the Modified Electrode

SEM image of $ZnWO_4$ nanorod on CILE surface was shown in Fig. 1A, which gave a rod-like appearance that similar to reference [23]. Therefore the interface roughness was increased due to the presence of disordered nanorod. Cyclic voltammetry was applied to probe the interfacial properties of different working electrodes with the curves shown in Fig.1B. A standard redox peak of ferricyanide was found on CILE (curve a), CTS/CILE (curve b) and CTS/$ZnWO_4$/CILE (curve c) with the responses increased gradually. The results were ascribed to the positive charged CTS and large surface of $ZnWO_4$ nanorod that could absorb more $[Fe(CN)_6]^{3-/4-}$ on the electrode, then the peak currents increased gradually.

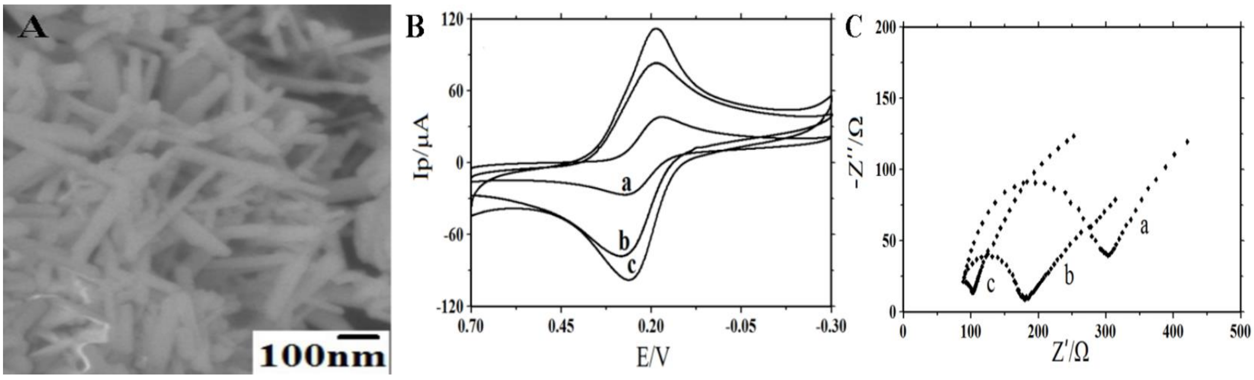

Figure 1. (A) SEM image of $ZnWO_4$ nanorod on CILE; (B) Cyclic voltammograms of CILE (curve a), CTS/CILE (curve b), CTS/$ZnWO_4$/CILE (curve c) in 1.0 mmol/L $K_3[Fe(CN)_6]$ and 0.5 mol/L KCl with the scan rate as 100 mV/s; (C) EIS of CILE (curve a), CTS/CILE (curve b) and CTS/$ZnWO_4$/CILE (curve c) in a 10.0 mmol/L $[Fe(CN)_6]^{3-/4-}$ solution containing 0.1 mol/L KCl with the frequency from 10^5 Hz to 0.1 Hz.

EIS results of different electrodes was further recorded and shown in Fig. 1C with the appearance of a linear part and a semicycle part. The electron transfer resistance (R_{et}) that can reflect the interfacial information after the modification is got from the semicircle part. The R_{et} value of CILE (curve a) was 207.8 Ω, and that of CTS/CILE (curve b) and CTS/$ZnWO_4$/CILE (curve c) decreased to 89.4 Ω and 11.1 Ω. Therefore the presence of CTS and $ZnWO_4$ nanorod on the electrode surface lowered the interfacial resistance and increased the redox peak currents, which could be ascribed to the increased conductivity and large surface area with positive charged interface.

3.2 Electrochemistry of UA

Fig. 2 showed cyclic voltammetric curves of 1.0×10^{-4} mol/L UA on CILE (curve a) and CTS/$ZnWO_4$/CILE (curve b) in pH 5.5 PBS. On CILE the oxidation peak located at 0.434 V (*vs*. SCE) without the reduction peak and the peak current was 13.02 µA. On CTS/$ZnWO_4$/CILE the oxidation peak could be observed at 0.425 V with current value as 28.73 µA. The increase of the oxidation peak current (2.2 times) proved that the presence of $ZnWO_4$ nanorod was benefit for the UA electrooxidation. $ZnWO_4$ nanorod exhibited certain conductivity, large surface area and electrocatalytic ability. More UA molecules could be accumulated on the electrode surface and involved in electrode reaction. Therefore the oxidation reaction of UA was accelerated with the peak current increased. Also only a large oxidation peak with a very small reduction peak was observed, showing a nearly irreversible electrochemical process.

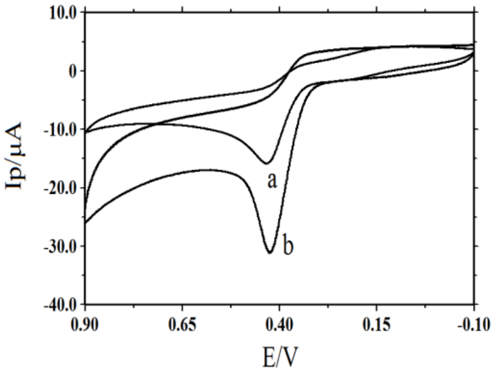

Figure 2. Cyclic voltammograms of 1.0×10^{-4} mol/L UA on (a) CILE and (b) CTS/$ZnWO_4$/CILE in pH 5.5 PBS with the scan rate as 100 mV/s.

3.3 Electrochemical Investigation

The effect of potential scan rate on the electrochemical reaction of UA on CTS/$ZnWO_4$/CILE was recorded by cyclic voltammetry with voltammograms shown in Fig. 3A. The stepwise increase of scan rate induced the increase of the oxidation peak currents and the oxidation potential was positively shifted. As shown in Fig.3B, the oxidation peak current had a good linear relationship with square root of scan rate from 10 to 450 mV/s with the equation as I_{pa} (µA) = 11.89+9.02 $\upsilon^{1/2}$ (V/s) (γ = 0.998), indicating a diffusional controlled electrode process [25]. From Fig.3A the increase of scan rate also led to the positive move of the peak potential, and the linear equation was E_{pa}(V) = 0.465 + 0.0113 ln υ (V/s) (γ=0.998) with the plot in Fig.3C. From the following equation [25]: $E_p=E^{\theta}+(RT/\alpha nF)$ [0.780 + $\ln(D_R^{1/2}/k^{\theta})$ + $\ln(\alpha nFv/RT)^{1/2}$], the value of αn_{α} was got as 1.13. Assuming charge transfer coefficient (α) as 0.5 for an irreversible process, the electron transfer number (n_{α}) was got as 2.26, close to the theoretical value (2) for UA reaction.

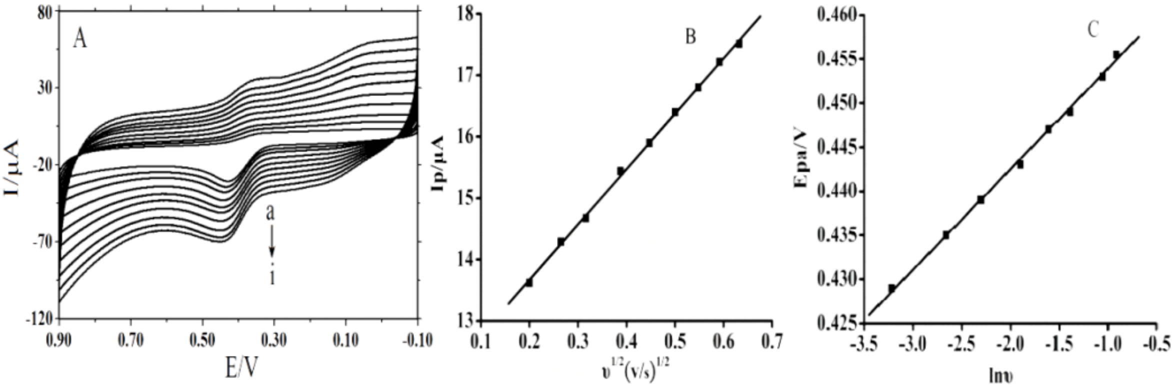

Figure 3. (A) Cyclic voltammograms of 1.0×10^{-4} mol/L UA on CTS/$ZnWO_4$/CILE with different scan rate (υ) in pH 5.5 PBS (from a to i: 40, 70, 100, 150, 200, 250, 300, 350, 400 mV/s); (B) Linear relationship of the oxidation peak current (I_{pa}) versus $\upsilon^{1/2}$; (C) Linear relationship between the oxidation peak potential (E_{pa}) and lnυ.

3.4 Effect of Solution pH

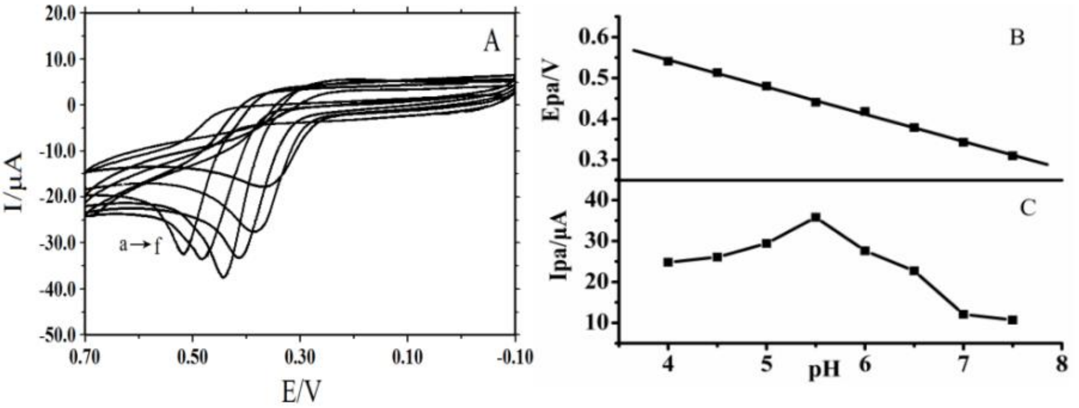

Figure 4. (A) Cyclic voltammograms of 1.0×10^{-4} mol/L UA on CTS/$ZnWO_4$/CILE with different pH PBS (from a to f were 4.5, 5.0, 5.5, 6.0, 6.5, 7.0), scan rate 100 mV/s; (B) Relationship between the oxidation potential (E_{pa}) and pH; (C) Relationship between the oxidation current (I_{pa}) and pH.

The influence of solution pH on cyclic voltammograms of UA was analyzed with the curves overlapped in Fig.4A. In the pH range from 4.0 to 7.5, the increase of solution pH led to the negative move of the oxidation potential, proving that H^+ were took part in the reaction [26]. As shown in Fig.4B and 4C, the relationship of pH with the oxidation current and the oxidation potential were plotted. The equation of E_{pa} and pH was got as E_{pa}(V)=0.811 - 0.0665 pH (γ=0.999). The slope value (0.0665) was close to the Nernstian value (0.059), indicating equal amount of protons and electrons in the reaction [27]. Therefore the value of X and n were equal to 2. The oxidation current increased from 4.0 to 5.5 and then decreased gradually with the maximum data located at pH 5.5, which was chosen in the experiment. Also the result indicated that the electrooxidation of UA involved in the protons and the electrochemical process was expressed with the following equation (Fig.5).

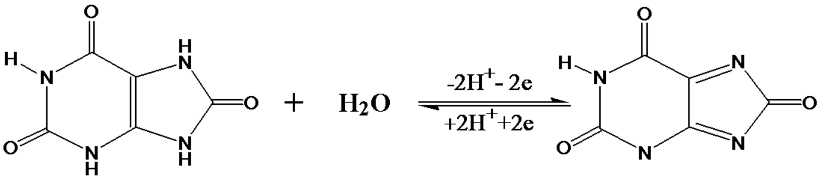

Figure 5. The electrode redox process of UA.

3.5 Chronocoulometry

Chronocoulometric measurements of UA on CTS/$ZnWO_4$/CILE was carried out with the curves shown in Fig. 6A, which was performed in pH 5.5 buffer (curve a) and pH 5.5 buffer with 1.0×10^{-4} mol/L UA (curve b). The relationship of Q with $t^{1/2}$ was plotted and shown in Fig. 5B with a linear relationship got as $Q(\mu C) = 14.88\ t^{1/2} + 1.88$ (γ=0.997). Based on the Anson's equation: $Q=2nFAD^{1/2}ct^{1/2}/\pi^{1/2}+Q_{dl}+nF\Gamma$, the diffusional coefficient (D) was calculated from the slope value as 3.26×10^{-5} cm^2/s.

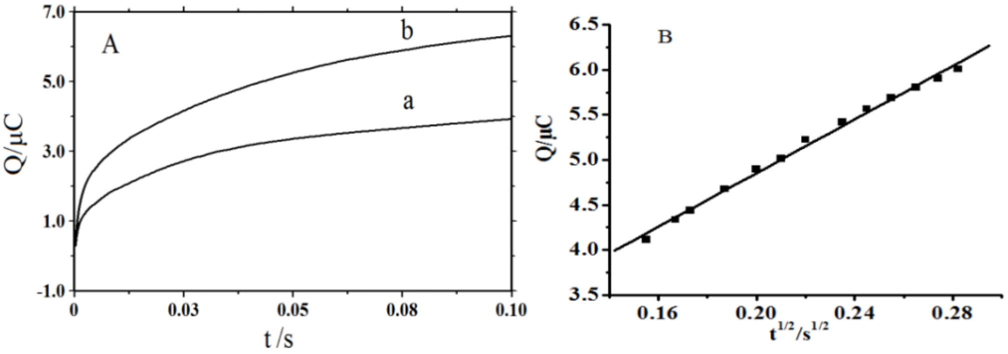

Figure 6. (A) Chronocoulometric curve of CTS/$ZnWO_4$/CILE in (a) pH 5.5 PBS and (b) pH 5.5 PBS containing 1.0×10^{-4} mol/L UA; (B) Relationship of Q against $t^{1/2}$.

3.6 Calibration Plot and Detection Limit

At the optimal conditions the oxidation current of UA on CTS/$ZnWO_4$/CILE was applied to the UA detection with DPV, which had a relatively better sensitivity and peak-shape curve than CV. As shown in Fig.7, the oxidation peak was increased with UA concentration and the peak current was proportional to concentration from 0.2 to 800.0 μmol/L. The linear regression equation was I_{pa} (μA)=1.158+0.114C (μmol/L) (n=16, γ=0.999) and the detection limit was 0.0637 μmol/L (S/N=3).

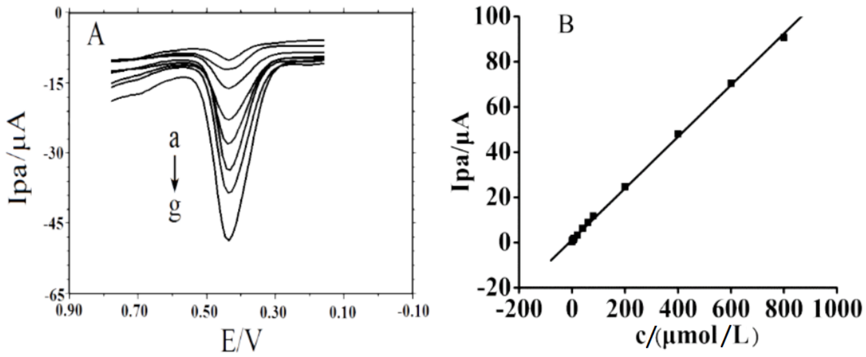

Figure 7. (A) Differential pulse voltammograms of various concentrations of UA on CTS/$ZnWO_4$/CILE (a-g: 6.0, 8.0, 20.0, 40.0, 60.0, 80.0, 200.0 µmol/L). (B) Relationship of the oxidation peak current with the UA concentration.

Table 1 summarized the analytical parameters for UA detection with various kinds of chemically modified electrodes. It can be deduced that the proposed electrode exhibited wider linear range and lower detection limit for UA analysis with the usage of one kind of nanomaterial.

Table 1. Comparison of the analytical performances of different modified electrodes

Electrode	Linear range (µmol/L)	Detection limit (µmol/L)	Ref
PTH/GCE	5.0-30.0	1.3	[27]
CTS/GR/GCE	2.0-45.0	2.0	[28]
Pt/GR/GCE	0.5-180.0	0.22	[29]
TCPP-RGO/GCE	20-5000	1	[30]
GR/CFE	0.194-49.68	0.132	[31]
ERGO/GCE	0.5-60.0	0.02	[32]
CILE	2-2200	1.0	[33]
Pt-MWCNT/GCE	0.46-50.0	0.35	[34]
Fe_3O_4-SnO_2-GR/CPE	0.015-2.4	0.000005	[35]
CdSe QDs-GR/GCE	3.0-600.0	1.0	[36]
NiHCF/PDAN/GCE	600.0-1000.0	0.037	[37]
Pd/RGO/GCE	6.0-469.5	1.6	[38]
CTAB-GO/MWNT/GCE	3.0-60.0	1.0	[39]
GNP/LC/GCE	8.0-5500.0	0.2	[40]
Pd/CNF-CPE	2.0-200.0	0.7	[41]
PImox-GO/GCE	3.6-249.0	0.59	[42]
Poly(eriochrome black T)/GCE	10.0-130.0	1.0	[43]

Poly-sulfonazo III /GCE	0.2-100.0	0.11	[44]
CTS/$ZnWO_4$/CILE	0.2-800.0	0.0637	This work

Note: PTH, poly(thionine); GCE, glassy carbon electrode; GR, graphene; GO, graphene oxide; TCPP, Porphyrin 5, 10, 15, 20-Tetra (4-pyridyl)-21H, 23H-porphine; CFE, carbon fiber electrode; ERGO, electrochemically reduced graphene oxide; Pt-MWCNT, Pt nanoparticles decorated multiwall carbon nanotube; CPE, carbon paste electrode; QDs, quantum dots; NiHCF, nickel hexacyanoferrate; PDAN, poly 1,5-diaminonaphthalene; RGO, reduced graphene oxide; CTAB, hexadecyltrimethylammonium bromide; GNP, gold nanoparticles; LC, L-cysteine; Pd/CNFs, palladium nanoparticle-loaded carbon nanofibers; PImox, polyimidazole;

CTS/$ZnWO_4$/CILE was applied to 9 parallel detections of 100.0 μmol/L UA with the relative standard deviation (RSD) calculated as 4.1%, showing good reproducibility. 5 modified electrodes were made by same procedure and applied to the analysis of 100.0 μmol/L UA with the RSD value of 3.9%. The CTS/$ZnWO_4$/CILE was stored in the lab for 3 weeks without the changes of the oxidation responses, indicating the good stability.

3.7 Interference

The interference of commonly coexisting substances on the detection of 1.0×10^{-4} mol/L UA was checked. Experimental results indicated that 100 times of K^+, Na^+, Cl^+, citric acid, glucose, NH_4^+, 50 times of sucrose, L-cysteine, L-phenylalanine, L-tyrosine, 30 times of glycine, and Ca^{2+}, 25 times of L-lysine and 10 times of H_2O_2, Fe^{3+}, Cu^{2+} did not interfere the analysis with the deviation less than ±5%, therefore CTS/$ZnWO_4$/CILE had good selectivity for the UA detection.

3.8 Sample Detection

The urine sample from the healthy volunteer was diluted for the UA detection with the results listed in table 2. The standard addition procedure was chosen for the calculation of recovery. The result was in the range of normal concentration with the recovery from 98.9% to 102.3%, which indicated that this modified electrode was able to detect the real samples with satisfactory results.

Table 2. Detection of UA concentration urine samples (n=3)

Sample	Found (μmol/L)	Added (μmol/L)	Total (μmol/L)	Recovery (%)
1	36.02	40.0	76.93	102.3
2	30.37	30.0	60.06	98.9

4. CONCLUSION

A new modified electrode was prepared with $ZnWO_4$ nanorod and further applied to differential pulse voltammetric analysis of UA. The presence of $ZnWO_4$ nanorod could accelerate the oxidation of UA, which underwent an almost irreversible reaction with an obvious oxidation peak on cyclic voltammogram. Electrochemical parameters of UA were calculated and a new differential pulse voltammetric procedure for UA analysis was established. The oxidation current was in linear with the UA concentration from 0.2 to 800.0 μmol/L and the modified electrode was successful applied to urine sample detection. Therefore this paper extended the application of $ZnWO_4$ nanorod in the field of electrochemical sensor.

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