

Poly(sulfosalicylic acid)/multi-walled Carbon Nanotube Modified Electrode for the Electrochemical Detection of Catechol

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A novel carbon paste electrode (CPE) modified by the poly(sulfosalicylic acid) and multi-walled carbon nanotubes (MWCNTs) composite (poly-SA/MWCNTs) has been easily constructed as an electrochemical sensor for the catechol determination. The electrochemical behaviors of catechol are investigated by cyclic voltammetry (CV) and differential pulse voltammetry (DPV) at the modified electrode. It is found that (i) poly-SA/MWCNTs/CPE exhibits a better electrical conductivity than bare CPE and MWCNTs/CPE; (ii) the anodic peak current of catechol at the poly-SA/MWCNTs/CPE electrode is about 8-fold larger than that at the bare CPE electrode; (iii) the detection limit of concentrate of catechol is $0.16 \mu\text{mol L}^{-1}$ ($S/N = 3$) and the linear dynamic range varies from 3 to $240 \mu\text{mol L}^{-1}$. The newly developed sensor exhibits many advantages for catechol detection, such a low-cost, easy preparation, high sensitivity, good anti-interference ability, excellent reproducibility and long-term stability and so on, which will be a potential candidate for the detection of phenolic derivatives.

Keywords: Electrochemical detection, multi-walled carbon nanotube, sulfosalicylic acid, catechol, electrode modification

1. INTRODUCTION

Catechol is an important phenolic compound and widely used in industrial production of pharmaceuticals, dyes and food and so on [1]. However, just like every coin has two sides, it is also a hazardous compound, which can cause depression of the central nervous system of human beings, inhibit the replication of DNA, and bring about the chromosomal aberration [2]. In general, catechol coexists with hydroquinone and resorcinol in the environmental samples due to a similar structure and property, which make them difficult to determine the isomers. Therefore, it is important to develop a sensitive and reliable method for catechol determination.

At present, a number of methods have been established to determine catechol including high-performance liquid chromatography [3], spectrophotometry [4, 5], electrochemiluminescence [6] and electrochemistry [7, 8, 9]. The former two techniques usually need a complicated and time-consuming pretreatment process, an expensive instrument and an overlong analysis time, which make them unsuitable for the routine analysis [10]. In comparison, electrochemical methods with high sensitivity and selectivity have been widely employed for catechol determination owing to their simplicity and low-cost [11, 12, 13, 14].

Since the 1990s, multi-wall carbon nanotubes (MWCNTs) have attracted much attention due to their remarkable physical, mechanical and electrochemical properties. They have been widely applied to construct electrochemical sensors to improve the detection sensitivity [15]. There are massive works on the fabrication of the MWCNTs-modified electrodes, such as the MWCNTs-polymer nanocomposite electrode [16], the MCNT-glassy electrodes [17] and the layer-by-layer assembly of MWCNTs film electrode [18]. All of them have been successfully applied in developing the electroanalytical methods for the biological and pharmaceutical analysis.

The electropolymerization has drawn enormous attention as a modification method because of its simplicity, selectivity and sensitivity towards analyses and strong adherence of the polymer film to the electrode surface [19]. Recently, the electropolymerization modified carbon paste electrodes are reported to offer an excellent effort as a sensor to investigate catechol [20].

In this work, a poly(sulfosalicylic acid)/multi-walled carbon nanotube (poly-SA/MWCNTs) electrode was fabricated by the electropolymerization of sulfosalicylic acid on the MWCNTs/CPE surface and the electrochemical behaviors of catechol were investigated using cyclic voltammetry (CV) and differential pulse voltammetry (DPV). The poly-SA/MWCNTs/CPE sensing platform showed great promising potential in the rapid detection of electroactive substances.

2. EXPERIMENTAL

2.1 Reagents

Catechol and sulfosalicylic acid (SA) were purchased from Aladdin Co. (Shanghai, China) and Shanghai Chemical Reagents Company (China), respectively. Multi-walled carbon nanotube (MWCNTs) with a diameter of 40/60 nm was provided by Shenzhen Nanotech Port Co. Ltd. A phosphate buffer solution (PBS, 0.1 M, pH 7) was prepared with 1 mol L⁻¹ dihydrogen phosphate and 1 mol L⁻¹ potassium phosphate and its pH was adjusted by phosphoric acid or potassium hydroxide. The solutions were prepared with double distilled water and the reagents used were of analytical grade.

2.2 Apparatus

All electrochemical measurements were carried out using a Reference 600 Electrochemical Workstation (Gamey Instruments in Philadelphia, USA) under ambient conditions. A conventional three-electrode system was used with a modified or bare carbon paste electrode (CPE) as the working

electrode, a saturated calomel electrode (SCE) as the reference electrode and a platinum wire as the counter electrode. The surface morphologies of the complex films were characterized by scanning electron microscope (JSM-6700F, Japan).

2.3 Preparation of CPE electrodes

The CPE was prepared by mechanically mixing 5 g of graphite powder and 0.07 g of paraffin until a uniformly wetted paste was obtained. The proportion of these materials was obtained by optimization. The paste was packed into a plastic tube (with a diameter of 3 mm and the depth of 3 cm). A copper wire was inserted into the carbon paste to provide electrical contact. Then the surface of the carbon paste was polished with a smooth paper to obtain a shiny appearance [21].

2.4 Preparation of modified electrodes

2 g of MWCNTs was dispersed in 2 mol L⁻¹ NaOH for 2 h with the aid of condensation reflux device and washed thoroughly with distilled water to neutrality. After totally dried, the pretreated MWCNTs was dispersed in 60 mL mixture of concentrated HNO₃ and H₂SO₄ (1/3, v/v) [22] with the aid of condensation reflux device for 0.5 h, then washed with distilled water to neutrality. Finally, this MWCNTs was dried in oven for 2 h.

The MWCNTs/CPE was prepared by dropping 1 mg mL⁻¹ of the suspension of the pretreated MWCNTs on the surface of the carbon paste electrode. The MWCNTs modified electrode was left to dry naturally at room temperature to form MWCNTs thin-film. Then, the electrode was immersed in PBS (pH 5.5) containing 0.01 mol L⁻¹ sulfosalicylic acid under repeated potential scan from -1 V to 2 V at a scanning rate of 100 mVs⁻¹ for 8 cycles until a reproducible CV characteristic was obtained (see Fig.1). Finally, the poly-SA/MWCNTs/CPE was carefully washed with double distilled water.

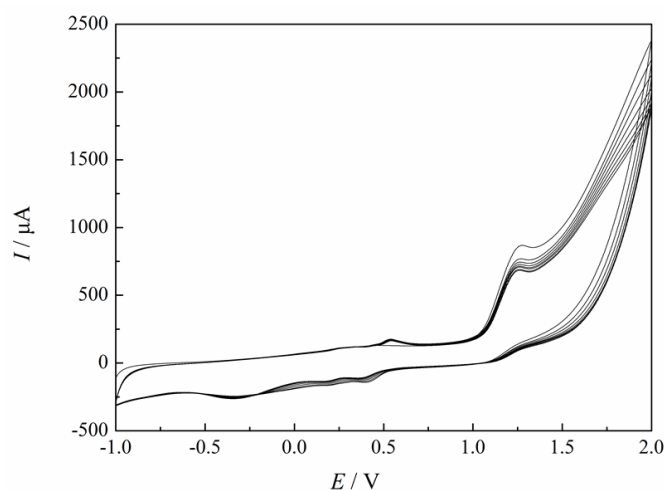


Figure 1. Continuous CV for the electrochemical polymerization of 10 mmol L⁻¹ SA on a MWCNTs/CPE in PBS (pH 7.0) under repeated potential scan from -1.0 V to 2.0 V at the scan rate 100 mV s⁻¹ for 8 cycles.

3. RESULTS AND DISCUSSION

3.1 Characterization

The surface morphology of the modified electrodes was recorded by the scanning electron microscope (SEM). Fig.2 showed the SEM images of CPE, MWCNTs/CPE and poly-SA/MWCNTs/CPE, respectively. As can be seen in Fig.2B, for the three-dimensional network structure of MWCNTs/CPE electrode consists of many small diameter-nanotubes in a densely-packed reign. It is obvious in Fig.2C that the MWCNTs was embedded into a polymer film after the electropolymerization of sulfosalicylic acid, indicating that the SA was successfully deposited on the surface of MWCNTs/CPE [23, 24].

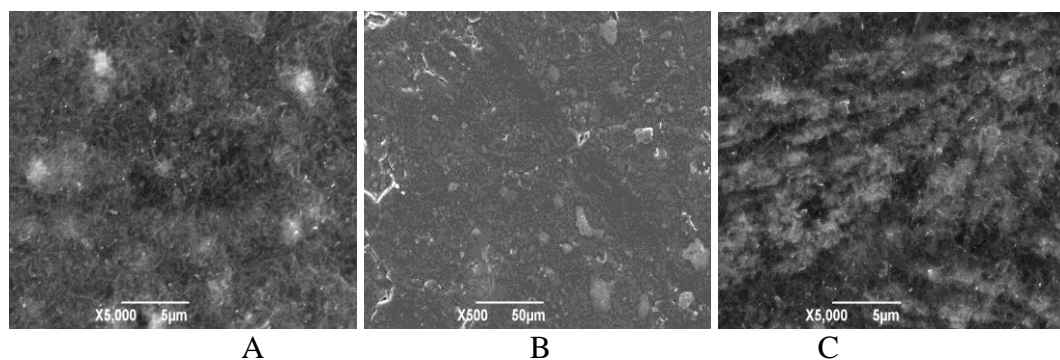


Figure 2. SEM images of CPE (A), MWCNTs/CPE (B) and poly-SA/MWCNTs/CPE (C)

The redox probe $[\text{Fe}(\text{CN})_6]^{3-/4-}$ was utilized to evaluate the electrochemical properties of different electrode. In electrochemical impedance spectroscopy (EIS), the semicircle diameter equals the electron-transfer resistance (R_{et}). The EIS measurements were performed in $5 \text{ mmol L}^{-1} \text{ K}_3[\text{Fe}(\text{CN})_6]/\text{K}_4[\text{Fe}(\text{CN})_6]$ (1:1) solution containing $0.1 \text{ mol L}^{-1} \text{ KCl}$ over the frequency range of 0.1Hz-10 kHz and the results were plotted in Fig.3A. The bare CPE displays the largest semicircle, suggesting the highest R_{et} of ca. 5230Ω . After coating MWCNTs on CPE, the semicircle is sharply decreased with $R_{\text{et}}=132 \Omega$ due to the improvement of electrical conductivity of MWCNTs. The poly-SA/MWCNTs/CPE exhibits the smallest semicircle ($R_{\text{et}}=86 \Omega$), indicating its excellent electrical conductivity.

The CVs of different electrodes were measured in $5.0 \text{ mmol L}^{-1} [\text{Fe}(\text{CN})_6]^{3-/4-}$ containing $0.1 \text{ mol L}^{-1} \text{ KCl}$ in a potential range of -0.4 to 0.8V and the CV curves were shown in Fig. 3B. The CV curves of the three electrodes all show a couple of redox peaks. However, the peak potential separations (ΔE_p) are different for the three electrodes. The (ΔE_p) of the bare CPE is about 871.6 mV. After coating MWCNTs on CPE, the redox peak currents of $[\text{Fe}(\text{CN})_6]^{3-/4-}$ increase significantly and the ΔE_p decreased obviously to 179.8 mV. After SA was electropolymerized on MWCNTs/CPE, the redox peak currents further rise dramatically, but the ΔE_p reduces slightly to 165.9 mV. These suggest the poly-SA/MWCNTs/CPE would possess the fastest electron-transfer rate [25].

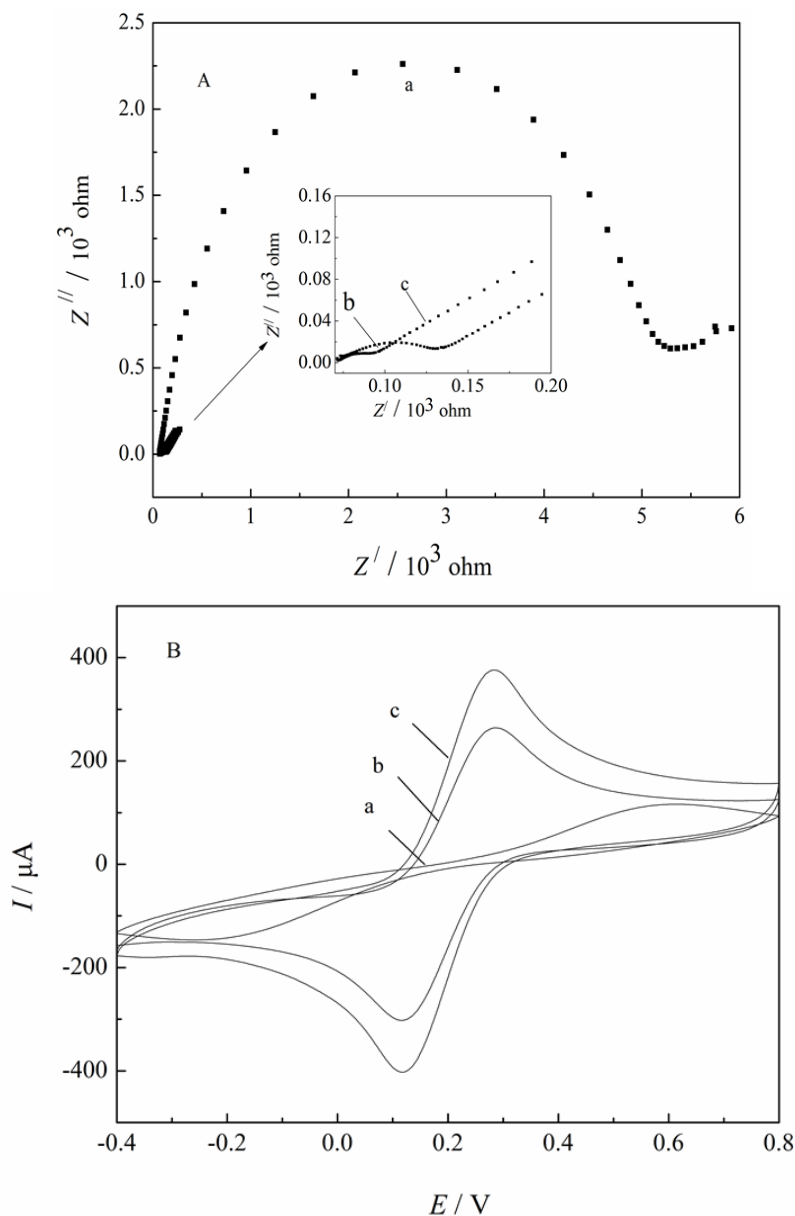


Figure 3. EIS (A) and CV curves (B) in $5.0 \text{ mmol L}^{-1} \text{ K}_3\text{Fe}(\text{CN})_6/\text{K}_4\text{Fe}(\text{CN})_6$ (1:1) with $0.1 \text{ mol L}^{-1} \text{ KCl}$ in potential range of -0.4 to 0.8 V at bare CPE (a) and MWCNTs/CPE (b), and poly-SA/MWCNTs/CPE (c) respectively.

3.2 Electrochemistry of catechol at poly-SA/MWCNTs/CPE

Fig.4 gave the CVs of catechol of 0.1 mmol L^{-1} in PBS (pH 7.0) at CPE, MWCNTs/CPE and poly-SA/MWCNTs/CPE electrodes at the scan rate of 100 mV s^{-1} , respectively. The oxidation and reduction peak potentials of the bare CPE electrode appear at 0.404 and -0.019 V , respectively and the oxidation peak current is observed at $5.296 \mu\text{A}$. For the MWCNTs/CPE electrode, the reduction peak potentials shift positively while the cathodic peak potential shifted negatively, and the oxidation peak current was increased largely to $18.39 \mu\text{A}$. After SA was electropolymerized on the MWCNTs/CPE, the oxidation peak current continually increased up to $40.06 \mu\text{A}$, approximately 8 times larger than that

of bare CPE, which is caused by the effective conductivity and catalytic activity of the immobilized MWCNTs and SA [26].

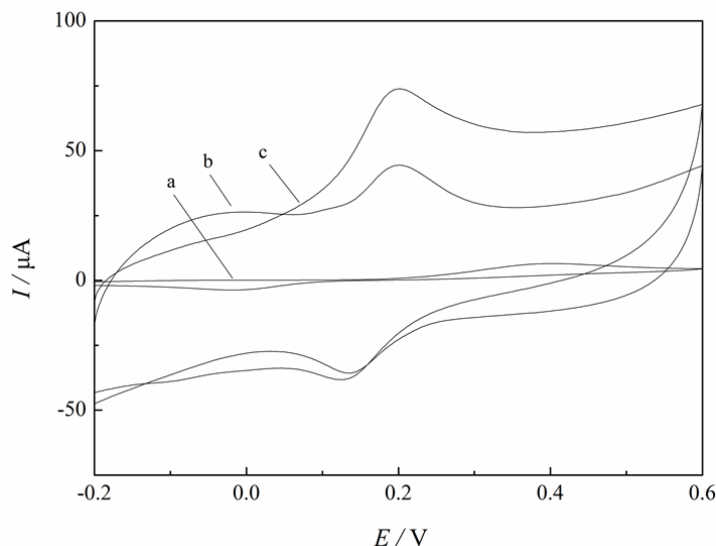


Figure 4. CV of 0.1 mmol L⁻¹ catechol at bare CPE (a), MWCNTs/CPE (b) and poly-SA/MWCNTs/CPE (c) in a PBS (pH 7.0) under potential scan from -0.2 V to 0.6 V at the scan rate of 100 mV s⁻¹.

3.3 Effect of the scan rate on the electrochemical response of catechol at poly-SA/MWCNTs/CPE

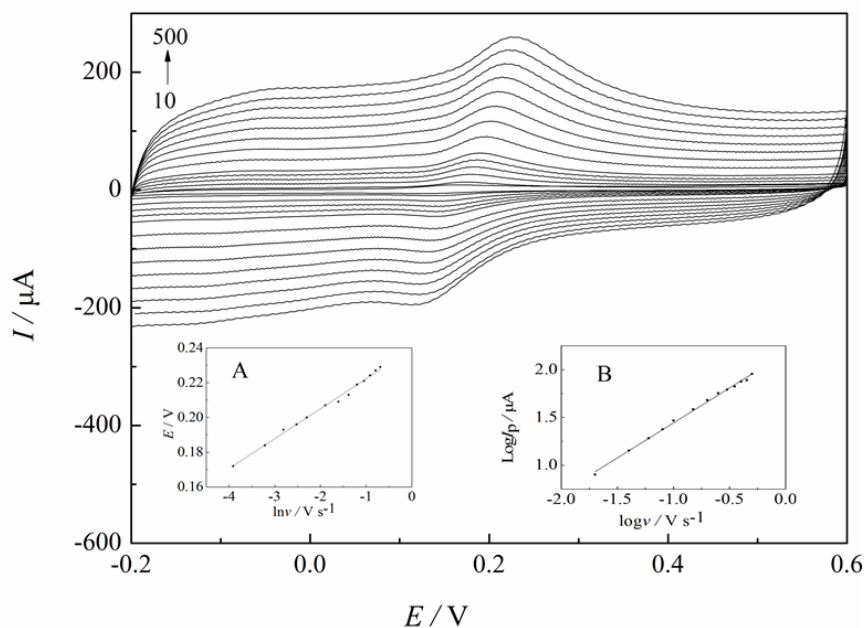


Figure 5. CV of a poly-SA/MWCNTs/CPE electrode in the presence of 0.1 mmol L⁻¹ catechol in pH 7.0 PBS at various scan rates. The scan rates are 10, 20, 40, 60, 80, 100, 150, 200, 250, 300, 350, 400, 450, 500 mVs⁻¹. Inset (A) shows a linear relationship between E_{pa} versus lnν; (B) shows a linear relationship between log I_{pa} versus log ν.

The electrochemical responses of the catechol of 0.1 mmol L^{-1} in PBS (pH 7.0) with poly-SA/MWCNTs/CPE were investigated using different scan rates (ranging from 10 to 500 mV/s), and the resultant CV curves were given in Fig.5. It can be observed that the peak current of the catechol redox reaction is increasing with scan rate increasing. And the anodic peak current (I_{pa}) was linearly dependent on the scan rate, with linear regression equation $\log I_{\text{pa}} (\mu\text{A}) = 2.1743 + 0.7317 \log v (\text{V s}^{-1})$ ($r = 0.9971$). This suggests that the electrochemical oxidation of catechol is diffusion-controlled accompanied with the absorption-controlled process [27]. The anodic peak potential (E_{pa}) shifts gradually towards positive as the scan rate increases, which was a typical characteristic of the reversible process. The logarithm of the scan rate is proportional to E_{pa} within the range of 10 to 500 mV s^{-1} with a linear regression equation of $E_{\text{pa}} = 0.2394 + 0.0272 \ln v$ ($r = 0.9940$). Then, based on the equations reported by Laviron [28], the number of transfer electron in the electrooxidation of catechol was calculated to be 1.86.

3.4 Effect of the pH on the electrochemical response of catechol at poly-SA/MWCNTs/CPE

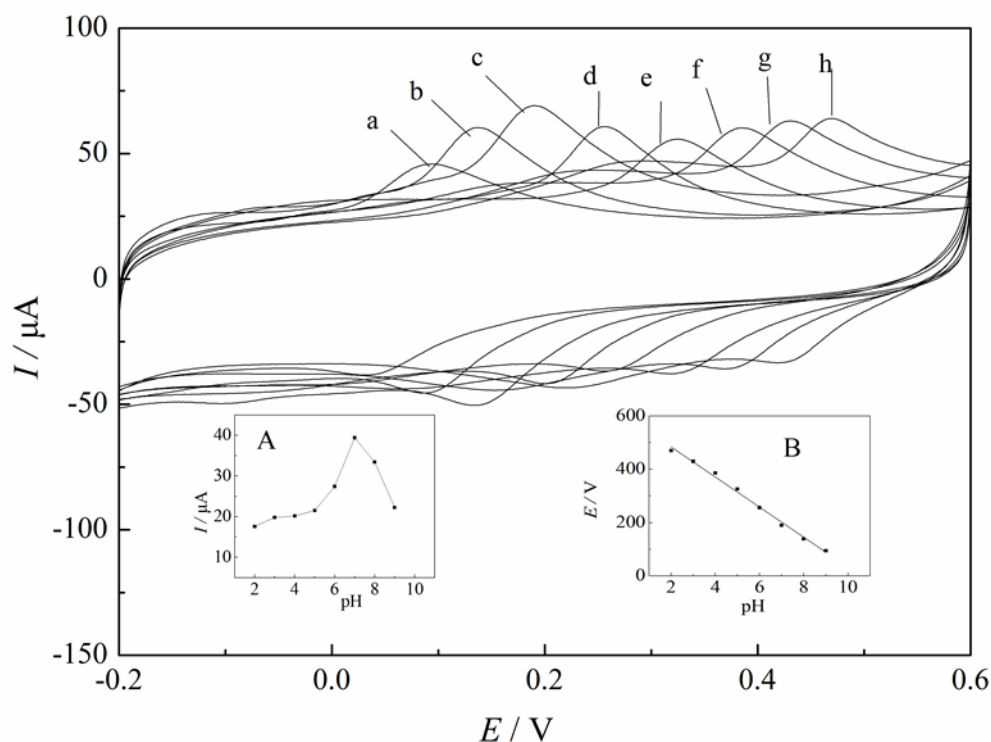


Figure 6. CV of a poly-SA/MWCNTs/CPE electrode in the presence of 0.1 mmol L^{-1} catechol at 100 mVs^{-1} in various pH: (a) 9.0, (b) 8.0, (c) 7.0, (d) 6.0, (e) 5.0, (f) 4.0, (g) 3.0, (h) 2.0. Inset (A) plots the anodic peak current versus pH; (B) shows a linear relationship between E_{pa} versus pH.

The pH of the supporting electrolyte showed a significant effect on the oxidation of catechol on the poly-SA/MWCNTs/CPE. The CVs of poly-SA/MWCNTs/CPE in PBS at different pH were plotted in Fig.6 as well as the insert Figs. 6A and 6B. As can be seen in Fig.6A, the current response firstly increases and then decreases with the decrease of pH value and the optimum pH for the electrocatalytic

oxidation of catechol at poly-SA/MWCNTs/CPE is equal to 7.0. The anodic peak (E_{pa}) shifted negatively with the increase of the pH value (see Fig. 6B), following the linear regression equation of $E_{pa} = 0.597 - 0.0565 \text{ pH}$ ($r = 0.9943$), which approximately obeys the Nernstian equation. This indicates that the involved number of electrons and protons are equal in the redox reaction [29], and the electrooxidation of catechol on the poly-SA/MWCNTs/CPE is a two-electron and two-proton process.

3.5 Amperometric determination of catechol at poly-SA/MWCNTs/CPE

Differential pulse voltammetry (DPV) was used to determine the concentration of catechol because it can discriminate against background noise and offer higher current sensitivity and better peak separation than cyclic voltammetry [30]. Under the optimized conditions, the determination of catechol at poly-SA/MWCNTs/CPE was carried out in PBS (pH 7.0) containing different amounts of catechol. A linear response was obtained between the peak current and the concentration of catechol (see Fig.7) with the linear regression equation of $I_{pa} (\mu\text{A}) = 0.13454 + 0.10007 c (\mu\text{mol L}^{-1})$ ($r = 0.9985$). The detection limit for catechol is $0.16 \mu\text{mol L}^{-1}$ ($S/N = 3$). The performance comparisons of the proposal sensor for catechol detection with other electrodes [31-36] were summarized in Table 1. As shown in Table 1, the detection limit of poly-SA/MWCNTs/CPE is not the smallest and the concentration linear range ($3\text{-}240 \mu\text{mol L}^{-1}$) is not the widest among the electrodes. However, the poly-SA/MWCNTs/CPE electrode is low-cost and easily prepared, which would be really promising for the determination of trace catechol.

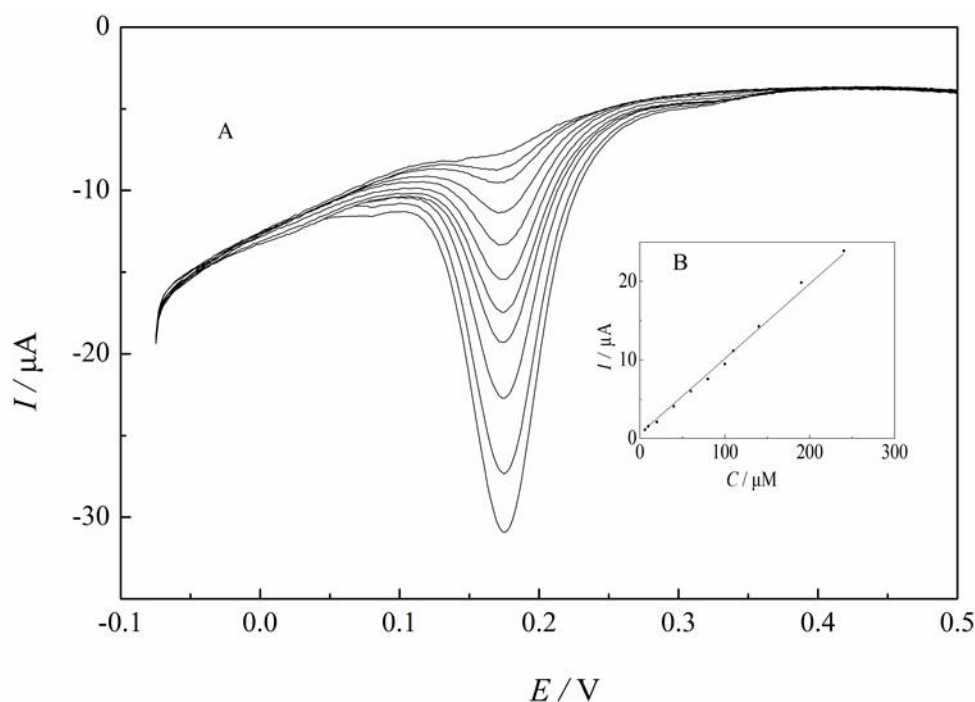


Figure 7. (A) DPVs of a poly-SA/MWCNTs/CPE electrode in pH 7.0 PBS containing different concentrations of catechol under potential scan from -0.1 V to 0.5 V at a scanning rate of at 100 m Vs^{-1} . The concentrations are 3, 10, 20, 40, 60, 80, 110, 140, 190, 240 $\mu\text{mol L}^{-1}$ inset (B) shows the calibration curve of catechol concentrations.

Table 1. Performance comparison of the proposal sensor for catechol detection with other electrodes.

Electrodes	Linear range ($\mu\text{mol L}^{-1}$)	Detection limit ($\mu\text{mol L}^{-1}$)	References
MWNTs-IL-Gel/GCE ^a	0.2-230	0.06	[31]
PIL-MWCNTs ^b	1-400	0.17	[32]
Palygorskite-modified CPE ^c	5-100	0.57	[33]
CNNS-CNT ^d	1-200	0.09	[34]
CNTs/CP ^e	1-100	0.29	[35]
ER(GO-TT-CNT) ^f	0.5-200	0.017	[36]
poly-SA/MWCNTs/CPE	3-240	0.16	This work

^aMWNTs-IL-Gel/GCE: a glassy carbon electrode modified with multi-walled carbon nanotubes (MWNTs) and ionic liquids (IL) of 1-butyl-3-methylimidazo-lium hexafluorophosphate (BMIMPF₆).

^bPIL-MWCNTs: modification of polymeric ionic liquid (PIL) on MWCNTs surfaces (PIL-MWCNTs)

^cPalygorskite-modified CPE: a palygorskite-modified carbon paste electrode

^dCNNS-CNT: three-dimensional (3D) graphitic carbon nitride nanosheets-carbon nanotube (CNNS-CNT) composite

^eCNTs/CP: the fabrication and application of carbon nanotubes/carbon paper (CNTs/CP) composite electrochemical sensors

^fER (GO-TT-CNT): in-situ polymerization of graphene oxide (GO), multi-walled carbon nanotube (CNT) and terthiophene (TT) coated glassy carbon electrode

3.6 Reproducibility, stability and selectivity

To investigate the precision of the determination and the reproducibility of the analysis, ten different electrodes were constructed using the same analytical procedure. The relative deviation (RSD) was 3.5%, indicating a good reproducibility. The modified electrode was constantly used for a week and the current response can retain 99% in the catechol detection, indicating its high stability.

The interferences of some common ions, including Na⁺, Ca²⁺, Ni²⁺, Zn²⁺, Fe²⁺, Fe³⁺, SO₄²⁺ to the determination of 0.1 mmol L⁻¹ catechol were evaluated in PBS (pH 7.0) at the modified electrode. The results exhibit no significant influence (signal change below 6%) on the signals of 0.1 mmol L⁻¹ catechol, indicating excellent anti-interference ability of the proposed electrode.

3.7 Real sample analysis

To evaluate the application of poly-SA/MWCNTs/CPE electrode for catechol determination, the real samples were detected by using DPV method as well as HPLC. The results were listed in Table 2. The amounts of catechol in the real samples were both below the detection limits. Hence, different amounts of catechol were added to the samples by spiking method. It can be seen that the results of the proposed method agreed well with those of HPLC. The recovery rate of catechol ranged from 98.7% to 101.1%, and relative standard deviations (RSD) were from 2.97 to 5.46, which all indicated poly-SA/MWCNTs/CPE electrode could be used to determinate catechol in the real samples.

Table 2. Determination of catechol in the real water samples (ND: no detection).

Water samples	Added ($\mu\text{mol L}^{-1}$)	Found ($\mu\text{mol L}^{-1}$)	Recovery (%)	RSD (% , n = 5)	Found by HPLC ($\mu\text{mol L}^{-1}$)
Ship's drinking water	0	ND	-	-	ND
	10	10.11	101.1	3.16	9.94
	20	20.20	101.0	2.97	20.03
Sea water	0	ND	-	-	ND
	10	9.89	98.9	5.46	9.92
	20	19.74	98.7	3.04	19.91

4. CONCLUSIONS

In this work, a novel poly-SA/MWCNTs/CPE electrode has been constructed by using simple preparation method and successfully employed for the determination of catechol. Compared to the bare CPE and MWCNTs/CPE, the poly-SA/MWCNTs/CPE electrode behaved the best electrical conductivity. The cyclic voltammetry measurement results reveal that the anodic peak current of catechol at the poly-SA/MWCNTs/CPE is almost 8-fold larger than that at the bare CPE. Meanwhile, the electrochemical oxidation of catechol at the poly-SA/MWCNTs/CPE electrode was diffusion-controlled accompanying with the absorption-controlled process, which is a two-electron and two-proton process. Based on the differential pulse voltammetric, the limit of measurement was found to be $0.16 \mu\text{mol L}^{-1}$ with a linear dynamic range from $3 \mu\text{mol L}^{-1}$ to $240 \mu\text{mol L}^{-1}$. The developed sensor based on poly-SA/MWCNTs/CPE electrode exhibits high sensitivity, good anti-interference ability, excellent reproducibility and long-term stability for the detection of catechol. The application could be extended to the detection of the phenolic derivatives.

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