Simultaneous Determination of Hydroquinone and Catechol Using Poly(*p*-aminobenzoic acid) Modified Glassy Carbon Electrode

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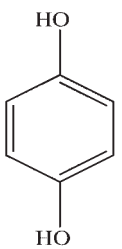
ABSTRACT: The electrochemical redox reaction of hydroquinone (HQ) and catechol (CC)was investigated with poly-(*p*-aminobenzoic acid) modified glassy-carbon electrode (poly-*p*-ABA/GCE) via cyclic voltammetry (CV) and differential pulse voltammetry (DPV). The poly-*p*-ABA/GCE has shown an excellent electrocatalytic activity for HQ and CC in 0.1 mol L^{-1} phosphate buffer solution (PBS). The oxidation and reduction separation (ΔE) has been decreased from 353 to 32 mV for HQ and from 228 to 33 mV vs. SCE for CC at the bare GCE and poly-*p*-ABA/GCE respectively. DPV curves show that the oxidation potential of HQ and CC has a separation about 105 mV at the poly-*p*-ABA/GCE. Moreover, the oxidation current of HQ and CC has been enhanced two and four times respectively at the modified electrode. Using DPV method, a highly selective and simultaneous determination of HQ and CC has been explored at the poly-*p*-ABA/GCE. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 113: 2881–2886, 2009

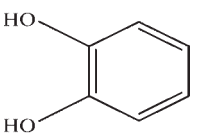
Key words: *p*-aminobenzoic acid; glassy-carbon electrode; hydroquinone; catechol; electropolymerization

INTRODUCTION

Discovered in 1880, hydroquinone (HQ) is a very important raw materials and chemical byproduct widely used in many fields such as pharmaceutical, tanning, and cosmetic industries.[1] However, as an environmental pollutant, it is toxic and can result in cancer like acute myeloid leukemia.[2] catechol (CC) is one isomer of HQ and is a model molecule for bisphenol compounds such as dopamine and L-dopa, which usually coexists in plants, fruits, wines, beers, juices etc.[3]



Hydroquinone



Catechol

The simultaneous determination of some phenolic compounds such as HQ, CC and resorcinol, becomes an interesting subject in analytical chemistry. Currently, the main methods of polyphenols determination are chromatography and spectrophotometry.[4–10] Because of the interference of bisphenol isomers coexisted in the sample. Pre-separation is required in chromatography, and complicated treatments for analytical signals are needed in spectrophotometry.

As an electroactive molecule, HQ or CC can surely be determined with the electrochemical methods.[11,12] However there are a great number of challenges for the simultaneous determination of HQ and CC. One major difficulty is that voltammetric peaks corresponding to the oxidation peaks of the two phenol isomers are seriously overlapped on most conventional solid electrodes. In addition, their competitive adsorption at the electrode surface makes the relationship between the voltammetric response and concentration in the mixtures nonlinear.[13] In order to solve these problems, many chemical modified electrodes were prepared for the simple, non-separate and simultaneous determination of isomers, such as carbon nanotubes modified electrode[14] and dual enzyme electrode.[15]

Polymer-modified electrodes (PMEs) have received attention in recent years,[16–19] due to their good stability, reproducibility, and homogeneity in electrochemical deposition and strong adherence to electrode surface.[20] Electropolymerization is a nice

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approach to immobilize polymers on PMEs by adjusting the electrochemical parameters to control the film thickness, permeation and charge transport characteristics. Brett et al. proved that the polymerization potentials of some compounds derived from aniline (such as aminobenzoic acid and aminobenzenesulphonic acid) were affected by solution pH.[21] Preechaworapun et al. developed an amperometric immunosensor using boron-doped diamond with poly(*o*-aminobenzoic acid).[22] Benyoucef et al. investigated the polymerization of aminobenzoic acid on the platinum electrode.[23] Compared with the metal electrodes, the glassy carbon electrode (GCE) has been widely used in many fields because of its good biocompatibility with the tissue, low residual current over a wide potential range, and minimal propensity to show deteriorated response as a result of electrode fouling.[24,25] Xiao et al. investigated the electrochemical properties of the polyaniline/poly(*o*-aminobenzoic acid) self-assembled film.[26] Thiemman reported a aminobenzoic acid/aniline copolymer modified GCE.[27] Poly-(*o*-aminobenzoic acid) modified GCE has been prepared to be successfully used to detect dopamine in the presence of ascorbic acid.[28] Xu et al. reported a poly-(*p*-aminobenzoic acid) modified electrode for selective determination of dopamine[29]and monoamine.[30] Chen et al. prepared a poly-(*o*-Aminobenzoic Acid) modified glassy-carbon electrode and applied it for the electrochemical determination of epinephrine.[31]

In this article, we prepared the poly-*p*-ABA modified GCE via electrochemically polymerization of *p*-aminobenzoic acid to explore the electrochemical behavior of HQ and CC. This kind of modified electrode can effectively catalyze the oxidation of HQ and CC in the 0.10 mol L^{-1} of phosphate buffer solution (PBS) at pH 7.0. The proposed method has been applied to simultaneous detection of HQ and CC in the presence of resorcinol (RC) in the samples, with satisfactory results.

EXPERIMENTAL

Apparatus and chemicals

CHI660A electrochemical workstation (Chenhua, Shanghai) was carried out for the electrochemical measurement. A conventional three-electrode system was employed with a bare GCE or poly-*p*-ABA/GCE (3.0 mm in diameter) as the working electrode, a saturated calomel electrode (SCE) as the reference electrode, and the platinum wire electrode as the counter electrode.

All solutions were prepared with redistilled water. *p*-ABA was obtained from Wulian Chemical Industry Factory (China). Other chemicals used were of analytical reagent grade. HQ, CC, and resorcinol solutions were prepared immediately before use. Artificial sewage was prepared with various kinds of common metal ions such as Fe^{2+}, Mn^{2+}, Al^{3+}, Ca^{2+}, Cu^{2+}, Mg^{2+}, Cd^{2+}, Zn^{2+} and so on, with total concentration up to 5.0×10^{-4} mol L^{-1}. The sewage also contained 1.0×10^{-4} mol L^{-1} ascorbic acid and phenol. Prior to the experiment, solutions were purged with purified nitrogen for 15 min to remove oxygen. All experiments were carried out at ambient temperature.

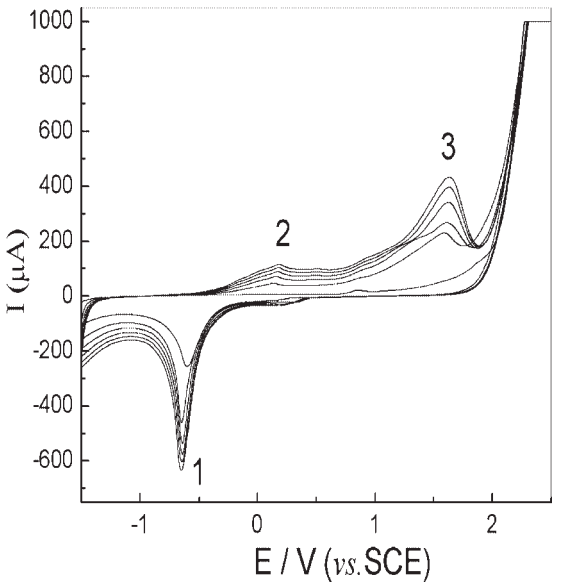


Figure 1 Repetitive cyclic voltammograms of *p*-ABA in 0.10 mol L^{-1} PBS (pH 7.0) containing 2.0×10^{-3} mol L^{-1}; initial potential: −1.5 V; terminal potential: +2.4 V, Scan rate: 100 mV s^{-1}.

Preparation of poly-*p*-ABA/GCE

The bare GCE was polished with 0.3 and 0.05 μm Al_2O_3 slurry on an emery paper and chamois leather successively, then rinsed with double-distilled water, and altrasonicated in 1 : 1 nitric acid, acetone and redistilled water for 10 min, respectively. Then the as-prepared electrode was immersed in a 0.10 mol L^{-1} PBS containing 2.0×10^{-3} mol L^{-1}*p*-ABA solution (pH 7.0) and was conditioned by cyclic sweeping between-1.5 to +2.5 V at 100 mV s^{-1} for 10 scans. Finally, the modified electrode was activated by cyclic voltammetry from −1.0 to +1.0V in 0.10 mol L^{-1} PBS (pH 7.0).

RESULTS AND DISCUSSION

Electropolymerization of *p*-ABA

Cyclic voltammetry was used to form the polymer film (shown as Fig. 1). The potential scan range,

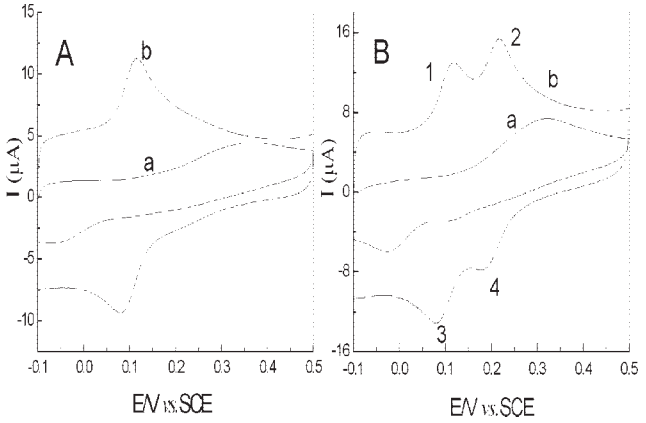


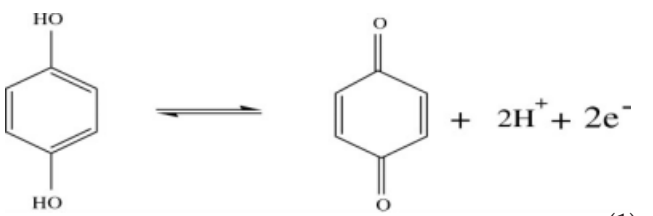
Figure 2 Cyclic voltammetric curves of 1.0×10^{-5} mol L^{-1} HQ in 0.10 mol L^{-1} PBS (pH 7.0) containing: (A) without CC and (B) with 1.0×10^{-5} mol L^{-1} CC on the bare GCE (a) and poly-(*p*-ABA) modified GCE (b). Scan rate: 100 mV s^{-1}.

especially the positive potential, is the most important factor in the preparation of the polymer film. If positive potential value for polymerization is under +1.5 V or negative one was above −0.8 V, no polymerization reaction is to occur. When the positive potential value reached 1.8 V, the electropolymerization can be observed on CVs. The experimental result showed that a better conductive polymeric film can be formed when potential scan window was from −1.5 V to +2.5 V. So we selected it for the optimum polymerization potential window in our experiments.

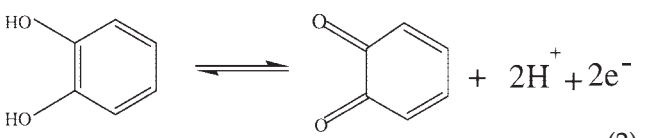
Voltammograms of *p*-ABA (2.0×10^{-3} mol L^{-1}) in pH 7.0 PBS on the GCE are shown in Figure 1. In the first scan, a weak anodic peak and a cathodic peak were observed with peak potential values at 0.90 V and −0.70 V, respectively. From the second cycle, two anodic peaks appear on the voltammograms with potentials at +0.145 and +1.588 V, respectively. The peaks' current increased upon continuous scanning, reflecting the continuous growth of the film of polymerization, and a blue polymer film is formed on the GCE surface, which is similar with the former literatues.[28,31] These facts indicate that *p*-ABA is deposited on the surface of GCE by electropolymerization. After modification, the poly-*p*-ABA modified electrode was thoroughly washed with doubly distilled water and stored in 0.1 mol L^{-1} PBS (pH 7.0) for use.

Electrochemical behavior of HQ and CC

The cyclic voltammetry study of HQ and CC on the modified electrode has been performed in 0.1 mol L^{-1} PBS (pH 7.0). Figure 2(A) recorded CV curves of 1.0×10^{-5} mol L^{-1} HQ on bare GCE and the poly-*p*-ABA modified electrode. There is a pair of redoxic peaks appearing at 0.343 V and −0.028V, with a separation (ΔE) of 0.62 V. However, a pair of well-defined redox peak of HQ appears at 0.115 V and 0.083 V on the modified electrode, with a separation of 0.032 V. These results indicate that the polymer can accelerate the electron transfer of HQ on the electrode surface. Figure 2(B) shows the cyclic voltammograms recorded at 50 mV s^{-1} containing a mixture of 1×10^{-5} mol L^{-1} HQ and CC in 0.1 mol L^{-1} PBS at the bare GCE and the modified electrode. There are a broad oxidation peak at 0.311 V and two reduction peaks at 0.111 V and −0.028 V, corresponding to the reduction of CC and HQ, respectively. This indicates that the oxidation of HQ and CC at the bare GCE is irreversible and undergoes sluggish a electron-transfer kinetic. While at the poly-*p*-ABA modified electrode, a pair of redox peak (peak 1 and 3) corresponds to the redox of HQ [eq.(1)]. Another pair of redox peaks (peak 2 and 4) appears at 0.219 V and 0.186 V with a separation (ΔE) 33 mV, which is the redox of CC [eq. (2)]. This indicates that HQ and CC oxidation overpotential is significantly lowered and fast electron transfer kinetics takes place at the poly-*p*-ABA modified electrode. The separation of oxidation peak between HQ and CC is about 104 mV. The probable reason is that the electron cloud density of HQ is lower than CC, so CC is harder to be oxidated than HQ on the polymer, which is similar with the previous report.[32] Moreover, the oxidation current of HQ and CC on the modified electrode is four times than that on the bare GCE. These results indicate that the modified electrode can accelerate the electron transfer of the electrode surface. The probable electrode reaction is as follows:



(1)



(2)

We explored the relation between the anodic peak current and the scan rate. Figure 3 shows that the anodic peak current was proportional to the square root of scan rate in the range 10–140 mV/s, with the correlation coefficients 0.998.

This result indicates that redox process of HQ and CC on the poly-*p*-ABA film modified electrode is controlled by diffusion.[33]

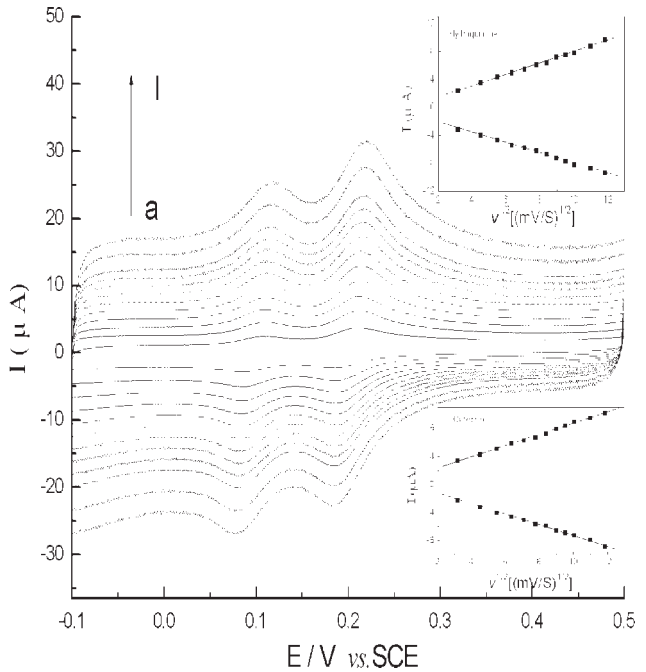


Figure 3 Cyclic voltammetric curves of 1.0×10^{-5}mol L^{-1} HQ and CC on the poly-(*p*-ABSA) modified electrode in 0.1 mol L^{-1} PBS (pH 7.0) at different scan rates: (a) 10, (b) 20, (c) 30, (d) 40, (e) 50, (f) 60, (g) 70, (h) 80, (i) 90, (j) 100, (k) 120, l 140 (mV s^{-1}), (Inset) anodic and cathodal peak currents of HQ (A) and CC (B) vs. square root of scan rate.

Effect of solution acid

In most cases, the solution pH is an important influence factor in electrochemical reactions. Cyclic voltammetry was carried out to characterize the influence of solution pH on electrochemical behavior of HQ and CC at the poly-*p*-ABA/GCE. The effect of solution pH on the response of HQ and CC was examined in the pH range from 4.0 to 8.0 (shown in Fig. 4). Figure 4(A) shows the relationship between anodic peak potential of the two bis-phenols and solution pH. The anodic peak potential (E_{pa}) is proportional with the solution pH in the range of 4.0 to 8.0. The linear regression equations of HQ [eq.(3)] and CC [eq.(4)] are described as:

$$E_{pa}(V) = 0.508 - 0.056\text{pH} \quad (3)$$

and

$$E_{pa}(V) = 0.614 - 0.057\text{pH} \quad (4)$$

with a correlation coefficient of 0.999. As a reversible electrochemical reaction:[34] $|E_p - E_{p/2}| = 2.3RT/nF$, the electron transfer number was calculated to be approximately 2 in the current study. These equations indicate that the electrode process is a two-proton coupled two-electron transfer.

Figure 4(B) shows that the anodic peak current increases with increasing solution pH until it reached 6.5. But when the pH value of solution exceeds 7.0, the anodic peak current rapidly decreased. Hence, pH 7.0 is chose for the electrochemical detection HQ and CC. The probable reason may be explained as the following: At low pH, H^+ concentration is high enough so that the nitrogen atoms of the polymer and the hydroxyl of HQ are protonated in the forms of $-NH_3^+$ and $-OH_2^+$.[35] With the increasing pH, more HQ molecules can interact with nitrogen atoms of the polymer, so the peak current becomes larger. When pH is over 7.0, HQ and CC start to be oxidized. This results in the decrease of the peak currents of HQ and CC. In addition, the relationship between the peak potential of HQ and pH of solution is also tested [Fig. 4(B)]. It can be found that the anodic potentials shift negatively with increasing pH, indicating that protons take part in the redox process of HQ and CC at the poly-*p*-ABA modified electrode.

The stability of poly-*p*-ABSA modified electrode was also examined. The modified electrode was stored in 0.10 mol L^{-1} PBS (pH 7.0) after each experiment. The cyclic voltammetric experiments were carried out using modified electrode once a day under the same operation conditions. The redox peak currents of HQ and CC did not change almost for a month, demonstrating that the modified electrode has a fairly good stability.

Simultaneous determination of HQ and CC

Differential pulse voltammetry (DPV) method is normally used for the determination of test sample because of its high sensitivity.[36–38] Determination of HQ and CC was performed with DPV at the poly-*p*-ABA modified electrode. To test the versatility of using poly-*p*-ABA/GCE for the selective determination of HQ and CC, DPVs have been recorded for

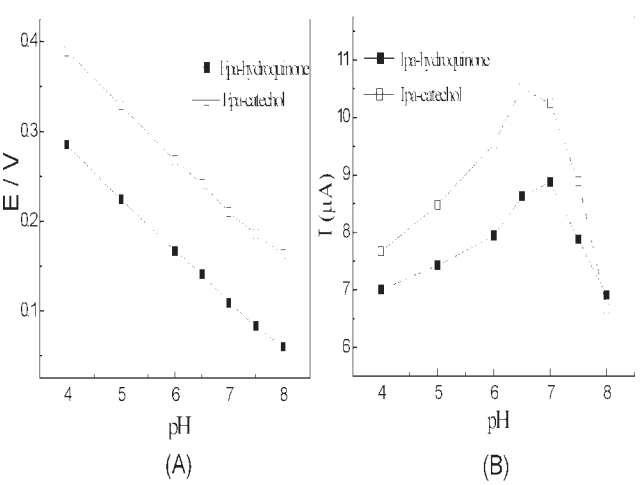


Figure 4 Effects of solution pH on the oxidic peak potential (A) and anodic peak current (B) of HQ and CC in 0.1 mol L^{-1} PBS containing 1.0×10^{-5} mol L^{-1} HQ and CC on the poly-(*p*-ABA) modified electrode, Scan rate: 100 mV s^{-1}.

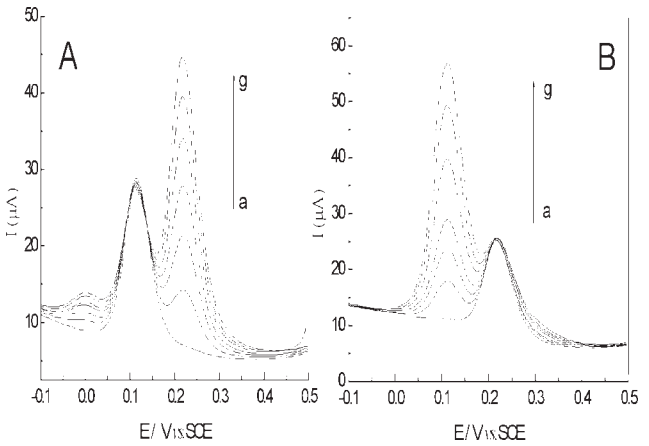


Figure 5 (A): Differential pulse voltammetry for poly-(*p*-ABA)/GCE in mixture of 6.0×10^{-5} mol L^{-1} HQ in 0.1 mol L^{-1} PBS (pH 7.0) containing different concentration of CC in the range at a→g: 0, 2, 4, 6, 8, 10, 12×10^{-5} mol L^{-1}, Scan rate: 100 mV s^{-1}. (B): Differential pulse voltammetry for poly-(*p*-ABA)/GCE in mixture of 4.0×10^{-5} mol L^{-1} CC in 0.1 mol L^{-1} PBS containing different concentration of HQ in the range at a→g: 0, 2, 4, 6, 8, 10, 12×10^{-5} mol L^{-1}, Scan rate: 100 mV s^{-1}.

different concentrations of HQ at poly-*p*-ABA/GCE in the presence of a constant concentration of CC and vice versa.

Figure 5(A) shows the DPVs recorded at different concentrations of HQ at poly-*p*-ABA/GCE in the presence of a constant concentration of CC (1.0×10^{-5} mol L^{-1}). Clearly, there is a monotonic increase in the voltammetric peak current corresponding to oxidation of HQ with the increase of the concentration and the oxidation peak current of CC almost remain constant. This response proves that the oxidation of HQ and CC at poly-*p*-ABA/GCE takes place independently.

DPVs recorded at poly-*p*-ABA/GCE for different concentrations of CC with a constant concentration of HQ of 1.0×10^{-5} mol L^{-1} are presented in Figure 5(B). Again the data show that there is a gradual increase in CC oxidation peak current with an increase in the CC concentration and the oxidation current of HQ almost remain constant. Figure 5 shows the dependence of DPV peak current on the concentration of CC and HQ. Clearly we can see that CC and HQ oxidation peak current is increased linearly with the concentration, in the range from 1.2×10^{-6} mol L^{-1} to 6.0×10^{-4} mol L^{-1} and from 2.0×10^{-6} mol L^{-1}~9.0×10^{-4} mol L^{-1} for HQ and CC respectively. The detection limits for HQ and CC are 4.0×10^{-7} mol $L^{-1}$and 5.0×10^{-7} mol L^{-1}, respectively, and the correlation factor of the straight line was 0.998 for both isomers.

Influence of interferents

Resorcinol is an isomer of HQ and CC, which normally coexists in the test sample. So to eliminate the interference of RC for detecting HQ and CC is very important.

The electrochemical response of RC was also investigated in pH 7.0 PBS. It was found that anodic peak of resorcinol appears at 0.619 V, which oversteps the applied window potential. This experimental result indicates that RC does not interfere to the simultaneous determination of HQ and CC. In theories, the electron cloud densities of HQ, CC, and RC are in the order of low to high, so HQ is harder to be oxidated than RC. The experiment result is accordant with the theory. We also examined the influence of other substances on the signals of HQ and found that no interference occurred in the present of 1000-fold sodium nitrate, 1000-fold potassium chloride, 200-fold Ca^{2+}, 100-fold Br^-, 200-fold SO_4^{2-}, with the deviations below 5%.

Analytical application

Under the optimal conditions, the poly-*p*-ABA modified electrode was used for the detection of HQ in artificial sewage samples. We added different concentrations of HQ to the electrolytic cell, then determined their contents and calculated their recoveries. The results were shown in Table I. It shows that the proposed method can be efficiently used for the determination of HQ in artificial samples. A comparison of the proposed method with the recent reports listed in Table II indicated that the proposed electrode is superior to the existing electrodes because of its working concentration range, detection limit,

TABLE I
Determination of Hq and Cc in Artificial Sewage Samples (n = 5)

Added (10^{-4} mol·L^{-1})		Found (10^{-4} mol·L^{-1})		Recovery (%)	
Hydroquinone	Catechol	Hydroquinone	Catechol	Hydroquinone	Catechol
2.0	2.0	1.95	1.96	97.5	98
2.0	2.0	2.02	2.06	101	103
3.0	3.0	2.98	3.03	99	101

TABLE II
The Working Ranges and Detection Limits of the Proposed Electrode and the Reported Methods

Reference	Linear range of HQ (mol L^{-1})	Detection limit (mol L^{-1})	Linear range of CC (mol L^{-1})	Detection limit (mol L^{-1})	Analysis real sample
[39]	1.0×10^{-5}– 1.5×10^{-3}	4.0×10^{-6}	–	–	Cosmetic
[40]	1.0×10^{-7}– 1.375×10^{-4}	1.5×10^{-8}	–	–	No
[41]	–	–	5.0×10^{-7}– 5.0×10^{-5}	1.0×10^{-7}	Tea
[42]	–	–	1.0×10^{-5}– 1.0×10^{-3}	–	No
[43]	5.0×10^{-5}– 2.0×10^{-3}	–	2.0×10^{-5}– 1.0×10^{-5}	–	No
[44]	5.0×10^{-7}– 2.5×10^{-5}	–	5.0×10^{-7}– 2.0×10^{-5}	–	No
This work	1.2×10^{-6}–6.0×10^{-4}	4.0×10^{-7}	2.0×10^{-6}– 9.0×10^{-4}	5.0×10^{-7}	Sewage

and ability to effectively determine HQ and CC in samples.

CONCLUSION

The electrochemical behaviors of HQ and CC at the poly-(*p*-ABA) modified electrode have been investigated and compared with that obtained on bare GCE. The poly-(*p*-ABA) modified GCE shows well an attractive recognized response towards HQ and CC electrooxidation. HQ and CC can be completely separated and can be simultaneously determined at the poly-(*p*-ABA) modified GCE at a relatively low potential in pH 7.0 phosphate buffer solution. The proposed method can also be applied to the simultaneous determination of HQ and CC in the presence of resorcinol in sewage samples, with satisfactory results.

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