Gold Nanoparticle and Poly(arginine) Modified GCE for Simultaneous Determination of Hydroquinone and Catechol

Altın Nanopartikül ve Poli(arjinin) Modifiye Camsı Karbon Elektrot ile Hidrokinon ve Katekolün Eş Zamanlı Tayini

Research Article

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ABSTRACT

In this work, a novel electrochemical sensor based on gold nanoparticle (AuNP) and poly(L-arginine) (P(AG)) modified glassy carbon electrode (GCE) (P(AG)/AuNP/GCE) was designed to determine hydroquinone (HQ) and catechol (CC). Scanning electron microscopy (SEM), cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) were utilized to characterize the P(AG)/AuNP/GCE. Differential pulse voltammetry (DPV) results revealed that the modified GCE represented high selectivity for the simultaneous determination of HQ and CC. The linear working ranges of HQ and CC were in the concentration range of 5.0 to 600.0 μmol L^{-1} with the detection limits of 1.5 and 1.42 μmol L^{-1}, respectively. The prepared sensor is a suitable tool with low cost for the rapid analysis of HQ and CC in the real samples with high recoveries.

Key Words
Hydroquinone, catechol, simultaneous, poly(arginine), AuNP.

ÖZ

Bu çalışmada, hidrokinon (HQ) ve katekolün (CC) tayini için altın nanopartikül (AuNP) ve poli(L-arjinin) (P(AG)) modifiye edilmiş camsı karbon elektrot (GCE) (P(AG)/AuNP/GCE) temelli yeni bir elektrokimyasal sensör tasarlandı. Taramalı elektron mikroskobu (SEM), dönüşümlü voltametri (DV) ve elektrokimyasal impedans spektroskopisi (EİS) P(AG)/AuNP/GCE'yi karakterize etmek için kullanıldı. Diferansiyel puls voltametrisi sonuçları modifiye elektrodun hidrokinon ve katekolün eş zamanlı tayini için yüksek duyarlılık ve hassasiyet gösterdi. HQ ve CC'nin doğrusal çalışma aralığı 5.0 ile 600.0 μmol L^{-1} arasında ve gözlenebilme sınırları sırasıyla 1.5 ve 1.42 μmol L^{-1} olarak bulundu. Hazırlanan sensör hidrokinon ve katekolün gerçek örneklerde hızlı analizi için yüksek geri kazanımla düşük maliyetli uygun bir araç olarak önerildi. Hazırlanan sensör, yüksek geri kazanımlarla gerçek numunelerde HQ ve CC'nin hızlı analizi için düşük maliyetli uygun bir araçtır.

Anahtar Kelimeler
Hidrokinon, katekol, eş zamanlı, poly(arjinin), AuNP.

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INTRODUCTION

Hydroquinone (HQ) and catechol (CC) are two isomers of dihydroxybenzene that are extensively used in cosmetic, dye, antioxidants and pesticides [1, 2]. These isomers can be easily introduced into environment as pollutants and they are harmful to organisms in their very low concentrations. HQ and CC generally coexist in environment since they have similar structure and property. Several methods such as spectrophotometry [3], chromatography [4], fluorescence [5] and various electrochemical methods [6-8] have been preferred to detect HQ and CC simultaneously. Among them, electrochemical techniques have several advantages such as fast response, high selectivity, sensitivity and low cost. In the case of electrochemical analysis of HQ and CC their redox peaks can overlap because they have the same electroactive phenolic groups. Thus, these two isomers can be hardly resolved at the surface of unmodified electrodes. Therefore, composite electrodes which differentiate between HQ and CC were used as excellent resolving devices. Recently, poly(amino acids) are widely preferred to combine with nanoparticles to form a novel conducting composite for sensor applications. The use of poly(amino acids) have attracted great attention due to their various properties such as low-cost, easy preparation, functional groups and conductivity [9,10]. The poly amino acid-modified electrodes were extensively used for the determination of various substances such as pharmaceutical drugs [11-13]. Among these, poly (L-arginine) (P(AG)) as a basic amino acid has three amino groups and it can be electrostatically interact with negative groups of different molecules [14,15]. Gold nanoparticles have attracted more attention for the construction of modified electrodes due to the advantages in terms of large surface area, biocompatibility and conductivity [16,17].

Herein, a simple and cost-effective electrodeposition technique to construct the AuNP and P(AG) modified GCE (P(AG)/AuNP/GCE) for the simultaneous determination of HQ and CC was reported. The prepared sensor showed high sensitivity for the analysis of HQ and CC. In addition, the P(AG)/AuNP/GCE showed excellent stability and applicability for the HQ and CC.

MATERIALS and METHODS

Materials

L-Arginine, potassium chloride, HQ, CC, citric acid, $MgCl_2$, NaCl, KCl, $Na_2HPO_4.7H_2O$ and $NaH_2PO_4.2H_2O$ were purchased from Sigma. UA, urea, glucose and L-cysteine were supplied by Fluka. Hydrogen tetrachloroaurate (III) trihydrate ($HAuCl_4.3H_2O$) was obtained from Acros Organics (Thermo Fischer Scientific, USA). All chemicals were analytical grade. Stock solutions of HQ (0.01 M) and CC (0.01 M) were freshly prepared.

Apparatus

Electrochemical measurement experiments were conducted with an AUTOLAB PGSTAT 302N electrochemical workstation with Nova 11.1 software (Eco Chemie, The Netherlands). A conventional three-electrode system consisting of a platinum wire as auxiliary electrode a silver/silver chloride (Ag/AgCl) as reference electrode and a bare or modified glassy carbon electrode as working electrode (*d*=3 mm), was used. The surface characterization of the modified electrode was performed using a scanning electron microscope (SEM, FEI Nova NanoSEM 650 microscope (The Netherlands)). All measurements were performed at room temperature.

Preparation of the Electrochemical Sensor

Firstly, the GC electrode was carefully polished with 0.05 µmol L^{-1} alumina slurry and then washed thoroughly with ethanol and ultrapure water. Then, sonication purifying was used to remove the alumina residues. The AuNP were electrodeposited on the surface of the GCE with a 0.6 mmol L^{-1} $HAuCl_4$ solution in 0.5 M H_2SO_4 for 15 cycles in the potential range from −0.2 to +1.2 V at a scan rate of 100 mVs^{-1} [18]. The P(AG) film was electrodeposited on the surface of the GCE/AuNP electrode by the CV method in 2.5 mmol L^{-1} L-arginine (0.1 mol L^{-1} PBS, pH 7.0) solution between -2.5 V and +2.3 V at a scan rate of 100 mVs^{-1} for 7 cycles [19]. After electropolymerization, the composite electrode was washed with ultrapure water and then air-dried. For comparison, the AuNP/GCE and P(AG)/GCE were also prepared under the same condition.

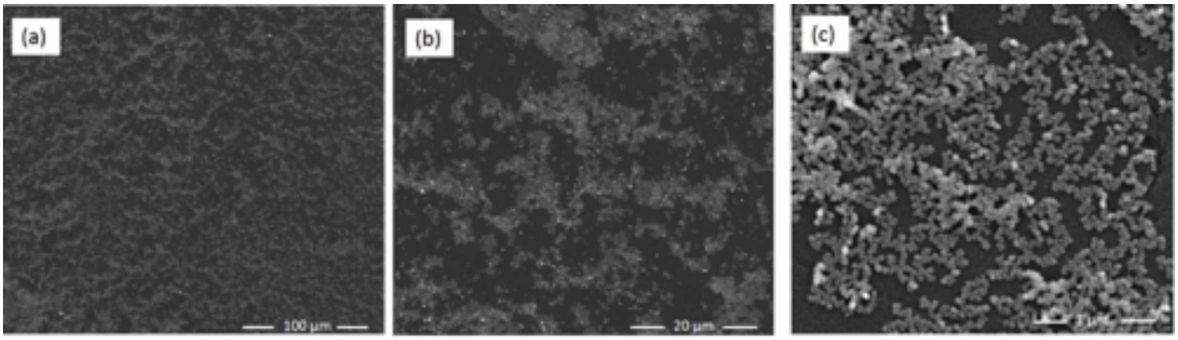

Figure 1. SEM image of P(AG)/AuNP/GCE electrode at different magnifications.

RESULTS AND DISCUSSION

SEM Images of P(AG)/AuNP/GCE

To investigate the morphology of P(AG)/AuNP/GCE, the scanning electron micrographs were taken. Figure 1 depicts that the P(AG)/AuNP/GCE is electrodeposited onto the surface of GCE and exhibits a porous structure (Figure 1). When the image was further magnified, white dots are clearly seen in Figure 1 c that indicates the presence of AuNP. These nanostructures were covered with the P(AG) thin film indicating that the P(AG)/AuNP composite was successfully obtained on the GCE by the CV technique.

Electrochemical Characteristics of Modified Electrodes

Electrochemical characterictis of the bare and the modified electrodes were performed by CV measurements. Figure 2 A exhibits the CVs of BGCE and modified electrodes in 0.1 mol L^{-1} KCl containing 5.0 mmol L^{-1} ferro-ferricyanide (Fe(CN)$_6^{3-/4-}$) redox couple (1:1). A pair of well-separated redox peaks with the lowest anodic and cathodic peak currents was observed at the BGCE (curve a) and peak-peak separation (ΔE_p) was estimated as 312 mV. Then, the surface of the electrode was modified with AuNP (curve b) or P(AG) (curve c). The anodic and cathodic peak currents both increased and ΔE_p decreased, which indicated that AuNP and P(AG) can enhance the electron transfer reaction between the electrode surface and the redox probe solution. When the AuNP were combined with the P(AG), the P(AG)/AuNP/GCE exhibited the highest redox peak currents and the value of the ΔE_p negligibly changed to 115 mV. This result might be attributed to the contribution of the AuNP to the electron transfer reaction [20].

The electrochemical impedance properties of the bare and modified electrodes were recorded in redox probe solution. The Nyquist plots for impedance measurements are given in Figure 2 B. Obviously, the BGCE showed a large semicircle, indicating the lowest rate for electron transfer. The AuNP/GCE (curve b) and P(AG)/GCE (curve c) showed smaller electron resistance value (R_{et}) compared with the BGCE. It indicates that the presence of AuNP or P(AG) could greatly increase the electron transfer reaction. Moreover, the smallest R_{et} was observed at the P(AG)/AuNP/GCE. This effect can be attributed to the synergistic effect between AuNP and P(AG).

Electrochemical Behaviour of HQ and CC at Modified GCEs

The electrochemical behaviour of HQ and CC at the bare and the modified electrodes was studied by CV. Figure 3 presents the cyclic voltammograms of the bare GCE and different electrodes containing a mixture of 50.0 µmol L^{-1} HQ and 50.0 µmol L^{-1} CC in 0.1 M PBS (pH 7.0) in the potential range of -0.2 to +0.4 V. As seen in Figure 3 a, a broad peak was observed due to the overlapping oxidation peaks of HQ and CC. However, the P(AG)/AuNP/GCE separated the voltammetric peaks into two well-separated peaks at the potentials of 0.044 V (HQ) and 0.151 V (CC). For the GCE/AuNP/P(AG) (curve d) an obvious increase in each peak current was obtained when compared with those of the AuNP/GCE (curve b) and P(AG)/GCE (curve c). The excellent electrochemical performance of the modified electrode could be ascribed to the large surface area, good electronic conductivity and synergistic effects between the AuNP and P(AG) which would be beneficial for improved electron transfer kinetics. The distinguished separation of

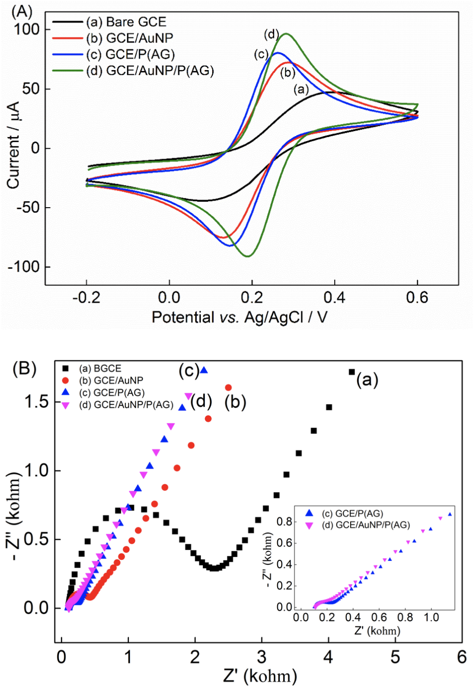

Figure 2. (A) Cyclic voltammograms and (B) the Nyquist plots of of the BGCE (a), AuNP/GCE (b), P(AG)/GCE (c) and P(AG)/AuNP/GCE (d) at the scan rate of 50 mV s^{-1} in 0.1 M KCl containing 5.0 mM $Fe(CN)_6^{3-/4-}$.

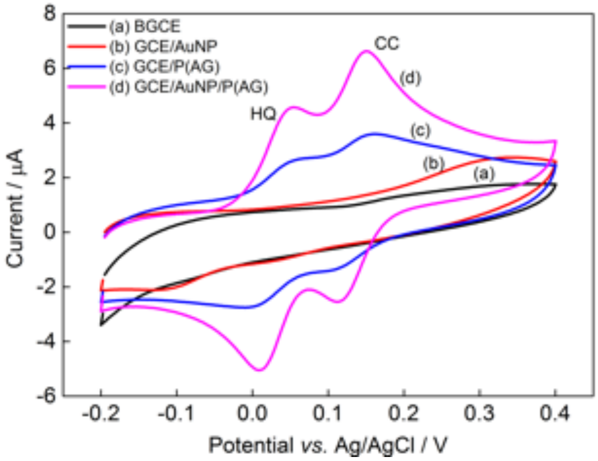

Figure 3. Cyclic voltammogram of the bare GCE (a), AuNP/GCE (b), P(AG)/GCE (C) and P(AG)/AuNP/GCE (d) in 0.1 mol L^{-1} PBS (pH 7.0) containing 50.0 µmol L^{-1} HQ and 50.0 µmol L^{-1} CC. Scan rate: 50 mV s^{-1}.

the oxidation peak potentials and the increased peak currents suggests that the simultaneous determination of HQ and CC in a mixture solution is applicable.

Effect of Solution pH

The effect of pH on the voltammetric response of the P(AG)/AuNP/GCE was performed in the pH range 5.0-8.0. As shown in Figure 4 A, the E_{pa} values shifted negatively with increasing pH from 5.0 to 8.0 for both HQ and CC. In addition, peak potential of HQ and CC were proportional to the pH value. The linear regression equations for E_{pa} with the pH for HQ and CC were E_{pa} = 614.08 pH −91.52 (R^2=0.995) and E_{pa} = 635.49 pH −95.36 (R^2=0.995), respectively. The slopes are very close to the theoretical data of −59.2 mV pH^{-1} for equal number of proton and electron process [8]. Furthermore, the oxidation peak currents of HQ and CC increased with increasing pH from 5.0 to

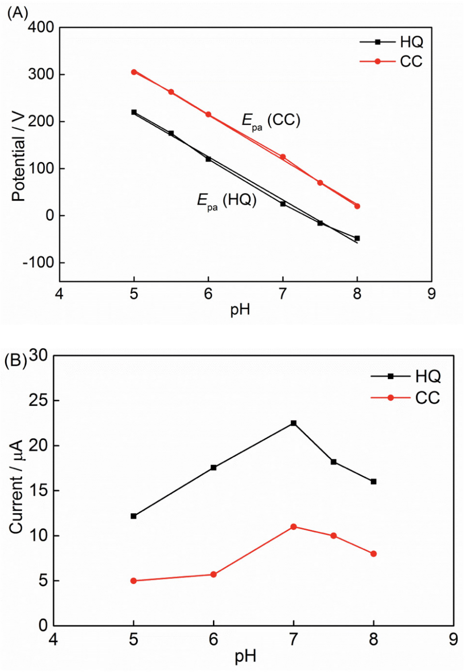

Figure 4. Effect of pH on (A) DPV peak potential and (B) DPV peak current for the oxidation of 50.0 µmol L^{-1} HQ and 50.0 µmol L^{-1} CC.

7.0 and then decreased quickly (Figure 4B). Thus, pH 7.0 was selected as optimum pH for the analysis of HQ and CC simultaneously.

Effect of Scan Rate

The influence of the scan rate of the potential sweep on the peak current of HQ and CC was studied at the P(AG)/AuNP/GCE. Figure 5 (A) and (C) show the cyclic voltammograms at the P(AG)/AuNP/GCE electrode. The peak currents increased with increasing scan rate. As shown in Figure 4 (B, D), good linear relationships were obtained between the oxidation peak current and the scan rate for HQ and CC with the linear equations (inset of Figure 5 (B) and (D)). This confirms that the electron transfer reactions for HQ and CC were adsorption-controlled processes [21,22].

Individual and Simultaneous Determination of HQ and CC

The relationship between the concentration of HQ and CC and the peak currents was investigated by differential pulse voltammetry. In these measurements, the concentration of one compound was changed while the other one was kept constant. As seen in Figure 5 (A) and (B), the peak currents of HQ and CC linearly increased in the concentration ranges of 5.0 –600 µmol L^{-1}, 5.0 –600 µmol L^{-1}, respectively. The linear fitting regression equations were I_{pa} (µA) = 0.262c (µmol L^{-1}) + 0.475 (R^2=0.986) for HQ and I_{pa} (µA) = 0.02c (µmol L^{-1}) + 0.083 (R^2=0.996) for CC. The detection limits for HQ and CC were 1.5 µmol L^{-1} and 1.42 µmol L^{-1}, respectively (3 s/m).

The comparative results between the P(AG)/AuNP/GCE with other modified electrodes was summarized in Table 1. The obtained results values were comparable with those previously reported in studies using different types of electrodes.

Figure 6 A depicts the DPV curves for the detection of HQ and CC in a mixture solution while their concentration changed simultaneously. The results show two well-defined anodic peaks for HQ and CC increased with peak potentials. Figure 6 B and (C) show the increase in peak currents with the concentrations of HQ and CC.

Repeatability, Reproducibility and Selectivity of the Prepared Sensor

To evaluate the repeatability of the sensor, differential pulse voltammograms were measured five times repetitively in PBS (pH 7.0) containing 50 µmol L^{-1} HQ and CC. The relative standard deviations (RSD) are 2.37% and 3.26%, respectively showing that the P(AG)/AuNP/GCE has good re-

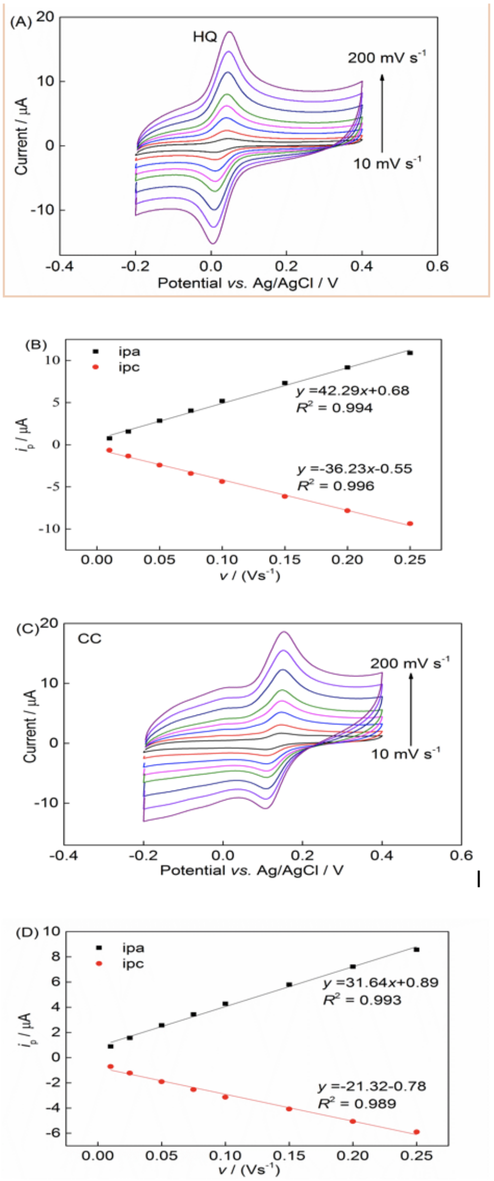

Figure 5. CV curves of 50.0 μmol L^{-1} HQ (A) and 50.0 μmol L^{-1} CC (C) in PBS (0.1 mol L^{-1}, pH 7.0) at the P(AG)/AuNP/GCE electrode with various scan rate between 10‒200 mVs‒1, and the corresponding plots of peak current versus scan rate (B, D).

peatability. In addition, the reproducibility was evaluated using five different electrodes, which were constructed independently. The RSDs were 4.22% and 5.18% for the peak measuring in 50 μmol L^{-1} HQ and CC which confirm the reliability of the fabrication process.

The effects of various substances such as ascorbic acid, dopamine, urea and phenol were investigated by DPV. Differential pulse voltammograms were recorded for 50 μmol L^{-1} HQ and CC in the presence of 50 μmol L^{-1} each of these substances. It is found that ascorbic acid, dopamine, urea and phenol did not cause any interfering effect on the voltammetric signals of the HQ and CC at the P(AG)/AuNP/GCE surface. It shows that the proposed sensor has good anti-interference ability towards the electro-oxidation of HQ and CC.

Analysis of Real Samples

The analytical applicability of the proposed method was investigated in local tap water samples at the P(AG)/AuNP/GCE. The amounts of HQ and CC in the samples were determined by DPV techni-

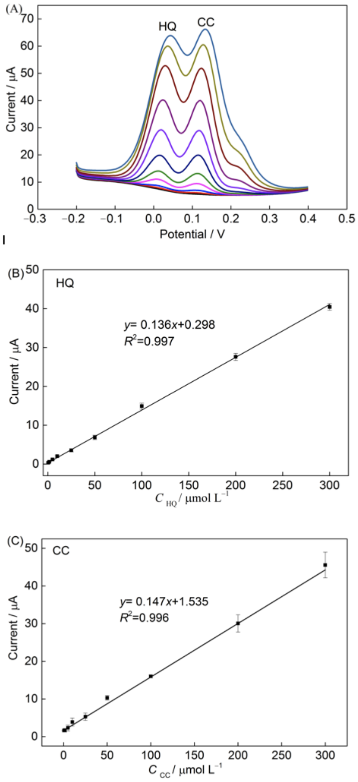

Figure 6. CV curves of 50.0 µmol L^{-1} HQ (A) and 50.0 µmol L^{-1} CC (C) in PBS (0.1 mol L^{-1}, pH 7.0) at the P(AG)/AuNP/GCE electrode with various scan rate between 10–200 mVs–1, and the corresponding plots of peak current versus scan rate (B, D).

Table 1. A comparison table of different modified electrodes for the simultaneous analysis of HQ and CC.

Electrode	Linear range (µmol L^{-1})		Detection limit (µmol L^{-1})		
	HQ	CC	HQ	CC	Reference
RGO/MWCNT/GCE	8.0–391.0	5.5 –540.0	2.6	1.8	[23]
GR-La(OH)3/GCE	5.0–300.0	5.0–300.0	0.015	0.01	[7]
Pyridine-NG	5.0–200.0	5.0–200.0	0.38	1.0	[24]
CMK-3/GCE	10.0–200.0	10.0–300.0	0.076	0.1	[25]
MWCNT/PASA//GCE	6.0–400.0	6.0–700.0	1.0	1.0	[26]
MnO_2/Pt/GCE	3.0–481.0	15.0–447.0	–	–	[6]
P(AG)/AuNp/GCE	5.0–600.0	5.0–600.0	1.5	1.42	This work

glassy carbon electrode (GCE),
reduced graphene oxide (RGO),
multi-walled carbon nanotube (MWCNT),
graphene (GR),
lanthanum hydroxide ($La(OH)_3$),
nitrogen doped graphene (NG),
mesoporous carbon material CMK-3 (CMK-3),
poly(amidosulfonic acid) (PASA),
platinum (Pt),
manganese dioxide (MnO_2).

Table 2. Simultaneous determination of HQ and CC in tap water samples (n = 3).

	Added (μmol L^{-1})		Found (μmol L^{-1})		Recovery (%)	
Sample no	**HQ**	**CC**	**HQ**	**CC**	**HQ**	**CC**
1	20	80	18.92	81.26	94.60	101.57
2	40	40	41.22	42.53	103.05	106.32
3	80	20	82.18	19.26	102.72	96.30

que. The results, which were calculated from the related calibration plot, are shown in Table 2. The recovery values were 94.60%-103.05% for HQ and 96.30-106.32 for CC, demonstrating the applicability of the developed method.

Conclusion

In summary, a sensitive method for HQ and CC determination was proposed by fabricating P(AG) and AuNP modified GCE. The modified electrode was characterized by the CV, EIS and SEM. The developed sensor has the ability to separate oxidation signals of HQ and CC with low LOD values and wide linear working ranges. In addition, the sensor shows high selectivity, reproducibility and repeatability towards HQ and CC detection. The prepared sensor was utilized to determine HQ and CC in real samples and high recovery values were obtained.

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