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ORIGINAL PAPER

Electrochemical properties and simultaneous determination of dihydroxybenzene isomers at ordered mesoporous carbon-modified electrode

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Abstract The voltammetric behaviors of dihydroxybenzene isomers were studied at an ordered mesoporous carbon-modified glassy carbon (OMC/GC) electrode. Compared to the bare electrode, the electrocatalytic activity of the modified electrode toward dihydroxybenzenes is evidenced by the increase of the peak current and the decrease of the peak separation (ΔE_p) in 0.1 M pH 5.0 phosphate buffer solution (PBS). Furthermore, at the OMC/GC-modified electrode, the three isomers could be separated entirely. The oxidation peak potential difference between hydroquinone and catechol is 154 mV, whereas that difference between catechol, and resorcinol is 370 mV. In the amperometric detection, the peak currents of dihydroxybenzene increased linearly with increasing dihydroxybenzene contents. The detection limits were 7.6×10^{-8} M, 1.0×10^{-7} M, 9.0×10^{-8} M for hydroquinone, catechol and resorcinol, respectively, which are the lowest values ever reported for dihydroxybenzene isomers. These make OMC/GC electrode a promising candidate for the simultaneous determination of isomers.

Keywords Ordered mesoporous carbon · Voltammetry · Dihydroxybenzene isomers · Modified electrode

1 Introduction

The *o*-, *m*-, and *p*-dihydroxybenzene isomers are important environmental pollutants and they are difficult to degrade. Due to their similar structures and properties, they usually coexist and interfere with each other during their determination. Their detection has become one of the important studies for environmental analysis [1]. Therefore, it is very important to develop simple and rapid analytical methods for the determination of dihydroxybenzene isomers. At present, various methods have been used, including chromatography [2, 3], spectrophotometry [4, 5], and electrochemical method [6, 7]. In the last few years, the electrochemical method has received more attention due to its high efficiency, low cost, and easy operation. However, the unmodified electrodes such as glassy carbon (GC) have limited selectivity, and the simultaneous determination of dihydroxybenzene isomers is then difficult. Moreover, the competition between the phenolic isomers at the electrode surface results in the non-linear dependence of the voltammetric response with the isomer concentrations [8].

Recently, much effort has been devoted to the electrochemical detection and separation of dihydroxybenzene isomers at new electrode materials, including surfactant [9], carbon-based materials [10, 11], etc. Besides the materials mentioned above, there has been significant interest in the development of one such novel carbon material, i.e., ordered mesoporous carbons (OMCs). Since they have been discovered in 1999, ordered mesoporous carbons (OMCs) [12, 13] have attracted much attention owing to the extremely well-ordered pore structure, electrical conductivity, and high specific surface area [14, 15]. Recently, our group has successfully used OMC-modified electrode to catalyze some important molecules, such as nitrite, bromate, hydrogen peroxide [16], L-cysteine [17], epinephrine, ascorbic acid, glutathione, uric acid, dopamine [18], etc. These studies demonstrated that OMC can promote electron-transfer reactions when used as an electrode in an

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electrochemical reaction. Therefore, OMC may have more interests and potential advantages for many advanced applications than other materials [19–21].

In this study, the electrochemical properties of dihydroxybenzene isomers are investigated using a glassy carbon (GC) electrode modified with OMC and the simultaneous determination of dihydroxybenzene isomers at the OMC-modified GC electrode is reported. The three dihydroxybenzene isomers are identified and separated.

2 Experimental

2.1 Reagents

OMC was synthesized according to the previously reported study [22]. Hydroquinone, catechol and resorcinol (purchased from Tianjin, China) were used without further purification. All the chemicals used were of analytical reagent grade. Aqueous solutions were prepared with doubly distilled water and stored in the shade. Phosphate buffer solution (0.1 M, PBS) was used as supporting electrolyte solution, which was made from K_2HPO_4, KH_2PO_4, and adjusting the pH with H_3PO_4 or KOH.

2.2 Apparatus

All the electrochemical experiments were performed with a CHI 830b Electrochemical Analyzer (CH Instruments, Shanghai Chenhua Instrument Corporation, China) in a conventional three-electrode cell. A conventional three-electrode cell was used, including an Ag/AgCl (KCl saturated) electrode as reference electrode, a platinum wire as the counter electrode, and a bare or modified glassy carbon disk (GC) as working electrode. All potentials in this paper were measured and reported versus Ag/AgCl electrode. The sample solutions were purged with purified nitrogen for at least 15 min to remove oxygen prior to the beginning of a series of experiments. All the measurements were carried out at room temperature.

2.3 Preparation of the modified electrodes

Prior to the modification, the GC electrode of 3-mm diameter was polished with 1.0, 0.3, and 0.05 μm alumina slurry, respectively, and sonicated successively in 1:1 nitric acid, absolute alcohol, and double-distilled water. The cleaned electrode was dried with high-purity nitrogen steam. The specific procedure for the preparation of the modified electrode was as follows: 5 mg of the OMC was dispersed in 10 mL of *N*, *N*-dimethylformamide (DMF) with the aid of ultrasonic oscillation to give a 0.5 mg mL^{-1} black suspension, then casting 7 μL of OMC suspension, and the solvent was evaporated under an infrared lamp to obtain the OMC/GC electrode.

3 Results and discussion

3.1 Electrochemical properties of OMC

The presence of the oxygen-containing functional groups on the surface of OMC has been reported in the literature [17]. Figure 1 shows the cyclic voltammograms of OMC/GC and GC electrodes in 0.1 M pH 2.0 PBS at 50 mV s^{-1}. No peak was observed at the bare GC electrode (curve a). At OMC/GC electrode (curve b), a couple of well-defined reversible redox peak was observed with a formal potential of +0.20 V in the potential range from –0.20 to +0.80 V. The appearance of redox peaks may be attributed to the protonation/depronation of the oxygen-containing functional (possibly carboxylic acid) groups on the surface of OMC. A similar electrochemical behavior has been observed for various carbon nanotubes [23]. Compared to the bare GC electrode, the background current of OMC-modified electrode is larger, indicating that the effective surface area of OMC/GC electrode is larger.

The cyclic voltammograms (CVs) of ferricyanide were investigated at different electrodes (data not shown). The potential difference (ΔE_p) between the anodic and cathodic peaks at OMC/GC electrode is 77 mV, whereas at the bare GC electrode, this difference is 122 mV. The peak-to-peak separation ΔE_p is a measure of the standard rate constant for electron transfer, the lower the ΔE_p, the higher is the electron-transfer rate. This implies that OMC exhibits faster electron rate kinetics compared with the bare GC electrode [24]. Furthermore, the response current at the OMC/GC electrode is much larger than that at the bare GC electrode, indicating that the OMC/GC electrode has higher electroactive surface area. The result clearly shows that OMC/GC electrode has relatively better electrochemical

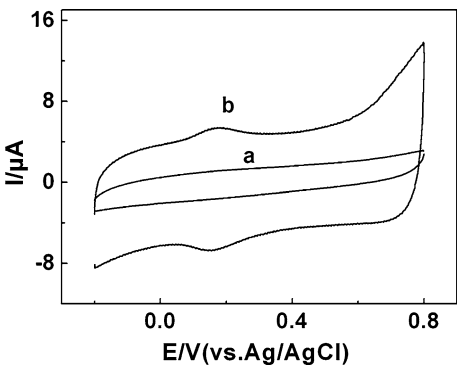

Fig. 1 Cyclic voltammograms at bare GC (*a*) and OMC/GC (*b*) electrodes in 0.1 M PBS (pH 2.0), scan rate 50 mV s^{-1}

reaction ability. The improved electrochemical behavior of OMC may be attributed to a large number of edge plane-like defect sites on the surface of OMC accessible for the electrolyte [25].

3.2 Electrochemical behaviors of dihydroxybenzene isomers

The cyclic voltammograms recorded in the presence of 0.5 mM hydroquinone in 0.1 M PBS (pH 5.0) at 50 mV s^{-1} on both OMC/GC (i) and bare GC electrodes (ii) are shown in Fig. 2a. At the bare GC electrode, the anodic and cathodic peaks of hydroquinone are at 0.372 and 0.108 V, respectively. The potential separation (ΔE_p) of 264 mV indicates that hydroquinone exhibits an irreversible electrochemical behavior. However, at the OMC/GC electrode, the reversibility of hydroquinone is significantly improved together and the current signal is increased. The oxidation peak potential negatively shifts to 0.238 V, and the reduction peak potential positively shifts to 0.173 V with a peak separation (ΔE_p) of 65 mV. The peak current is threefold higher than that at the bare GC electrode surface.

The electrochemical behavior of catechol was also studied (Fig. 2b). At the bare GC electrode (ii), the oxidation and reduction of catechol exhibit broad waves with peak potentials of 0.477 and 0.211 V. The ΔE_p of catechol at the bare GC electrode is 266 mV. The proportion between the oxidation peak current and the reduction peak current (i_{pa}/i_{pc}) is about 2.5, indicating an irreversible electrochemical process. However, as shown in Fig. 2b (i), the redox of catechol at the OMC/GC electrode shows a reversible process and a significant increase in current signal. The oxidation peak potential negatively moves to 0.350 V and the reduction peak positively shifts to 0.294 V with a ΔE_p value of 56 mV. Furthermore, i_{pa}/i_{pc} is close to 1. The small ΔE_p indicates that the redox overpotential of catechol is significantly lowered at the OMC/GC electrode, and the electrochemical redox activity of catechol is also highly improved.

Figure 2c shows the voltammetric responses of 0.5 mM resorcinol at bare GC and OMC/GC electrodes in 0.1 M PBS (pH 5.0). At the bare GC electrode, a small anodic peak of resorcinol is observed at 0.844 V. Compared with the bare GC electrode (Fig. 2c, ii), the oxidation peak current of resorcinol increases significantly and the anodic peak potential of resorcinol shifts negatively to 0.806 V at the OMC/GC electrode (Fig. 2c, i). The electrocatalytic oxidation of resorcinol by OMC as a mediator is evidenced by the increase of the anodic peak current and the shift of the anodic peak potential compared to the bare GC electrode.

At the bare GC electrode, the dihydroxybenzene isomers exhibit irreversible redox processes. However, at the OMC/GC electrode, there is a small peak separation and the peak current is higher for hydroquinone and catechol. For the resorcinol, the oxidation peak potential is improved and the peak current increases significantly. This means that OMC plays an important role as a promoter which enhances the electrochemical reactions of dihydroxybenzene isomers. Due to its high porosity, the real surface area of the OMC-modified GC electrode is far greater than that of the bare GC electrode. Therefore, the adsorption of dihydroxybenzene isomers on the OMC/GC-modified electrode surface is much stronger. Therefore, the peak current increases evidently with the background voltammetric response at the OMC-modified GC electrode.

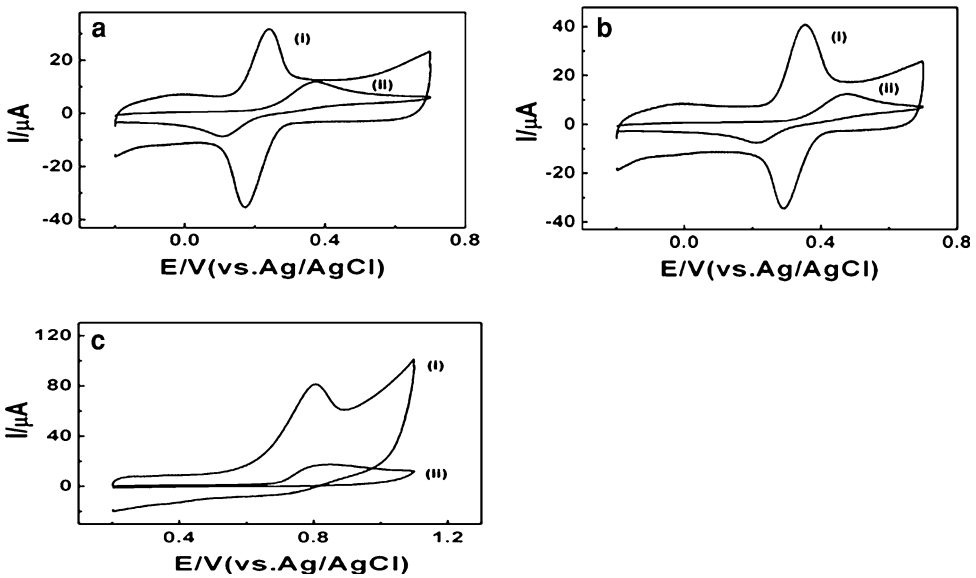

Fig. 2 Cyclic voltammograms at a scan rate of 50 mV s^{-1} for 0.5 mM hydroquinone (**a**); 0.5 mM catechol (**b**) and 0.5 mM resorcinol (**c**) at OMC/GC (i) and bare GC (ii) electrodes in 0.1 M PBS (pH 5.0)

3.3 Optimization of conditions

3.3.1 Effects of scan rate

The electrochemical behaviors of dihydroxybenzene isomers were also studied at various scan rates. From 5 to 90 mV s^{-1}, the oxidation peak currents of dihydroxybenzene isomers increase in 0.1 M PBS. The oxidation peak potential positively shifts when the scan rate is increasing, and the oxidation peak currents exhibit a linear relationship with the scan rates (Fig. 3), suggesting that the electrode processes are limited by adsorption [26].

3.3.2 Effect of solution pH

As a proton has an influence on the electrode reaction, the acidity of electrolyte has a marked effect on the electrochemical behaviors of dihydroxybenzene [27]. Figure 4 shows the relationship between the pH values and the anodic peak potentials of dihydroxybenzene isomers. It was found that the oxidation peak potential of hydroquinone shifts negatively with the increase of pH values. The behaviors of catechol and resorcinol are similar to hydroquinone. The potential separation at about pH 5.0 is larger than that at other pHs, indicating the higher resolution at pH 5.0. The effect of pH on the potential separation ($\Delta E_{p/o}$) between the anodic peak potential of hydroquinone and catechol and the potential separation ($\Delta E_{o/m}$) between catechol and resorcinol at the OMC/GC electrode is shown in inset of Fig. 4. In the pH range from 2.0 to 9.0 the potential separations ($\Delta E_{p/o}$, $\Delta E_{o/m}$) are ca. 120–150 and 260–400 mV, respectively, which means that the anodic peaks of three dihydroxybenzene isomers can be separated successfully in the pH range of 2.0–9.0. Therefore, the simultaneous determination of dihydroxybenzene isomers is possible in a broad pH range at OMC/GC electrode. However, at low pH values, the oxidation potentials are much positive and the interference is obvious. Above pH 9.0, the oxidation peaks of hydroquinone and catechol overlap to form a broad hump. Therefore, a weak acidic solution is better for the experiments. In this study, pH 5.0 was chosen as the optimum pH.

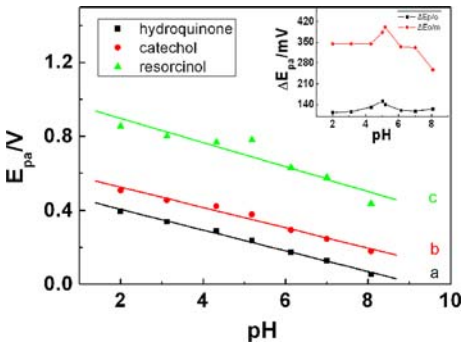

Fig. 4 The influence of pH on the anodic peak potentials (each 0.5 mM, scan rate 50 mV s^{-1}). Inset shows the relationship between peak potential separation and pH

3.4 Electrochemical behaviors of a mixture of dihydroxybenzene isomers

In order to evaluate the sensitivity and selectivity of the OMC/GC electrode for the simultaneous determination of dihydroxybenzene isomers, the electrochemical behaviors of mixed components of dihydroxybenzene isomers (each concentration of 0.5 mM) were investigated using CVs (as shown in Fig. 5). At the bare GC electrode, there are only two oxidation peaks at potentials of 0.444 and 0.831 V, which indicates that the voltammograms of hydroquinone and catechol overlap to form a wide peak, and the peak current is low. Therefore, the bare GC electrode cannot separate the voltammetric signals of hydroquinone and catechol (Fig. 5a). It is impossible to use the bare GC electrode for the voltammetric determination of the mixed system of dihydroxybenzene isomers. However, at the OMC/GC electrode, three well-defined oxidation peaks are present at potentials of 0.247, 0.401, and 0.771 V for the

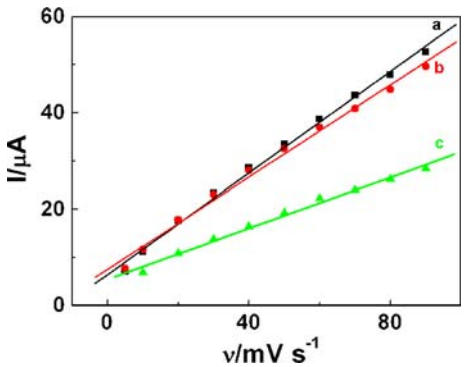

Fig. 3 The relationship between oxidation peak current and scan rates, hydroquinone (*a*), catechol (*b*), and resorcinol (*c*)

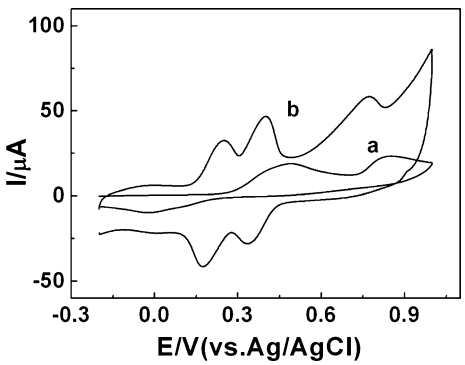

Fig. 5 Cyclic voltammograms of the bare GC (*a*) and the OMC/GC (*b*) electrodes in dihydroxybenzene isomers (each 0.5 mM)

oxidation of hydroquinone, catechol and resorcinol, respectively (Fig. 5b). The OMC-modified electrode resolves the mixed voltammetric signals into three well-defined voltammetric peaks. The three well-defined anodic peaks observed at the OMC/GC electrode clearly indicate the essential role of OMC in the electrochemical behaviors. The oxidation peak potential difference between hydroquinone and catechol is up to 154 mV, and the peak separation between catechol and resorcinol is 370 mV. The peak potential separation between hydroquinone and catechol is larger than the other electrodes reported (100 mV) [28] (105 mV) [29], (111 mV) [10], (125 mV) [30]. Thus, the *o*-, *m*-, and *p*-dihydroxybenzene isomers could be well separated at the OMC-modified electrode. Moreover, the oxidation peak currents are remarkably increased (two times) as compared to that at the GC electrode. Thus, the simultaneous determination of dihydroxybenzene isomers is feasible at the OMC/GC electrode. It is clear that the increase in oxidation current, shift in oxidation potential, and well separation of the oxidation peaks at the OMC/GC electrode can offer special approach for the simultaneous electrochemical determination of dihydroxybenzene isomers in mixture solution. Moreover, the oxidation peak potentials of dihydroxybenzene isomers in the mixture solution are found at the same positions as in separated solutions (Fig. 2a–c), which proves that the oxidation of dihydroxybenzene isomers in the mixture solution takes place independently at the OMC/GC electrode. It is also important to note that the electrochemistry of hydroquinone and catechol are reversible at the OMC/GC electrodes, but the electrochemistry of resorcinol is irreversible.

These advantages of OMC/GC electrode could be mainly attributed to the unique properties of OMC. First, there are plenty of edge plane-like defects and oxygen-containing functional groups on the surface of OMC, which may provide many favorable sites for electron transfer to molecules [17, 31, 32], resulting in a large, increasing peak current and low oxidation overpotential. The presence of the oxygen-containing functional groups on the surface of OMC, e.g., –COOH, –C=O, and –OH, has been confirmed by Zhou et al. [16]. These active groups can form hydrogen bonds with the –OH groups of the dihydroxybenzenes, which weaken the –OH bond energies, and the electrons would be transferred through O···H–O. Second, the porous structure of the OMC has different space resistances to the different dihydroxybenzenes which affect the rates of dihydroxybenzene to the electrode surface. They have then different peak potentials and could be separated. Third, due to the unique electronic structures of the OMC, they could act as a promoter to enhance the electrochemical reaction. The huge specific surface area of OMC can also increase the effective area of the electrode. Therefore, the peak currents of dihydroxybenzene increase, and their oxidation potentials shift negatively. Nevertheless, the charge distribution of dihydroxybenzene isomers is quite different, and the isomers exhibit different conformation at the especial charged OMC electrode interface. In theory, the density of the electron cloud orderly lowers from hydroquinone to catechol to resorcinol; therefore, their electroactivity decreases. It was found that the conjugation effect of dihydroxybenzene isomers with the OMC electrode interface is also different. Therefore, the dihydroxybenzenes show different oxidation–reduction potentials at this especial electrode interface.

3.5 Simultaneous determination of dihydroxybenzenes isomers

On the basis of the voltammetric results described above, the redox of dihydroxybenzene isomers can be catalyzed at the OMC/GC electrode, and their voltammetric signals are well separated. The interfering effect on the amperometric determination of dihydroxybenzene isomers was studied (as shown in Fig. 6). An increase in the oxidation current was obtained with each addition of 10 μM hydroquinone at +0.25 V. However, no significant response was observed with injection of 10 μM of catechol (a) and 10 μM of resorcinol (b). This behavior shows that catechol and resorcinol do not interfere in the determination of hydroquinone because the difference of peak potentials of the three isomers is large enough when using the OMC/GC electrode. Therefore, it appears likely that amperometric detection of dihydroxybenzenes isomers at OMC/GC electrode is possible.

Amperometric responses recorded at the OMC/GC electrode for different concentrations of hydroquinone at constant concentrations of catechol (0.1 mM) and resorcinol (0.1 mM) at a controlled potential of +0.25 V are presented in Fig. 7. In inset of Fig. 7, the modified electrode exhibits good linear ranges from 10 to 200 μM for

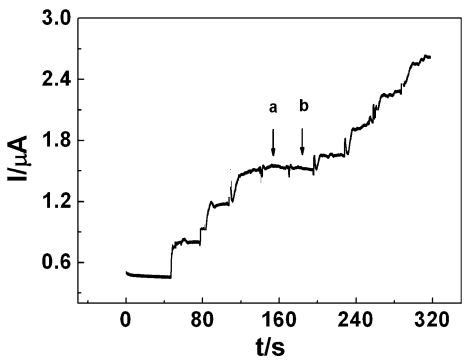

Fig. 6 Amperometric response of the sequential additions of hydroquinone and (10 μM) catechol (*a*) and (10 μM) resorcinol (*b*) with applied potential at +0.25 V in 0.1 M pH 5.0 PBS

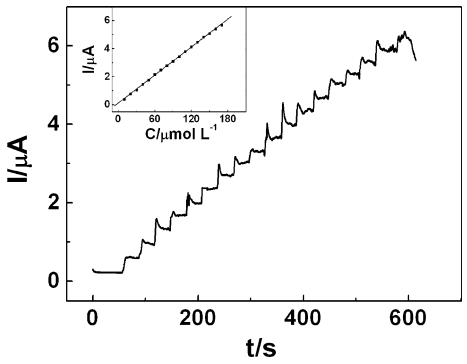

Fig. 7 Current–time curves obtained at the OMC/GC electrode to successive addition of 1.0×10^{-5} M hydroquinone into 0.1 M PBS in the presence of 0.1 mM catechol and 0.1 mM resorcinol with applied potential at +0.25 V. Inset is the calibration curve for hydroquinone at OMC/GC electrode

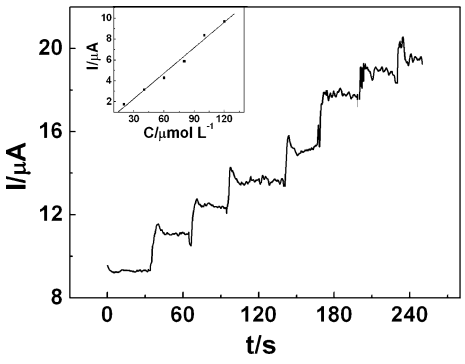

Fig. 9 Current–time curves obtained at the OMC/GC electrode to successive addition of 2.0×10^{-5} M resorcinol into 0.1 M PBS in the presence of 0.1 mM hydroquinone and 0.1 mM catechol with applied potential at +0.75 V. Inset is the calibration curve for resorcinol at OMC/GC electrode

the detection of hydroquinone in the presence of 0.1 mM catechol and 0.1 mM resorcinol with a regression equation of $i = 1.20 \times 10^{-7} + 0.03324c$ (A, M, $R = 0.999$). The detection limit is 7.6×10^{-8} M ($S/N = 3$), lower than 1.0×10^{-6} M [29], 6.0×10^{-7} M [9]. Therefore, the sensor can be used to detect hydroquinone in the presence of its isomers catechol and resorcinol.

We also investigated the electrocatalytic activity of OMC/GC electrode toward the oxidation of catechol (Fig. 8) and resorcinol (Fig. 9). The linear ranges is from 1.0×10^{-5} to 3.0×10^{-4} M ($i = 1.155 \times 10^{-7} + 0.025c$, A, M, $R = 0.999$) for catechol in the presence of 0.1 mM hydroquinone and 0.1 mM resorcinol and 1.0×10^{-5} to 1.2×10^{-4} M ($i = -1.507 \times 10^{-7} + 0.081c$, A, M, $R = 0.993$) for resorcinol in the presence of 0.1 mM hydroquinone and 0.1 mM catechol. The detection limits for $S/N = 3$ are 1.0×10^{-7} M for catechol, 9.0×10^{-8} M for resorcinol.

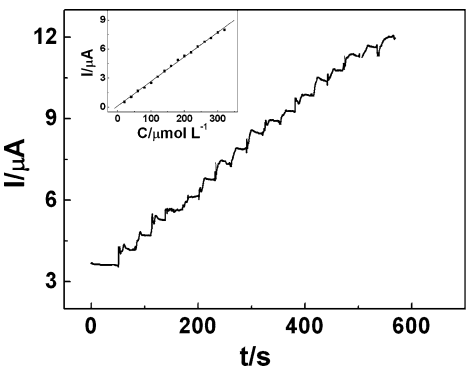

Fig. 8 Current–time curves obtained at the OMC/GC electrode to successive addition of 2.0×10^{-5} M catechol into 0.1 M PBS in the presence of 0.1 mM hydroquinone and 0.1 mM resorcinol with applied potential at +0.4 V. Inset is the calibration curve for catechol at OMC/GC electrode

3.6 The reproducibility and stability of OMC/GC electrode

Usually, a poor reproducibility is encountered when carbon electrodes are applied to the determination of molecules. To further ascertain the reproducibility of the results, five different GC electrodes were modified with OMC, and their responses to the oxidation of dihydroxybenzene isomers were tested. The separation between the voltammetric signals of dihydroxybenzene isomers and the sensitivities remained the same at all the five modified electrodes. These results suggest that by using the recommended method, the fabricated OMC-modified electrode has a good stability and accuracy for the determination of dihydroxybenzene isomers.

3.7 Interference of coexisting substances

The possible interferences of some species in the wastewater were also tested. A great number of cations and anions, such as K^+, Na^+, Al^{3+}, Cu^{2+}, NO_3^-, Cl^-, SO_4^{2-}, Ac^-, Ca^{2+}, and Mg^{2+}, (each of $c = 1 \times 10^{-2}$ M), had no influence on the signals of the dihydroxybenzenes, with deviations below 5%. Only a high concentration of phenol was found to interfere markedly with the determination of dihydroxybenzenes and this could be removed by distillation (the phenol content is on a comparative level with dihydroxybenzenes; therefore, it will not interfere markedly with the determination of dihydroxybenzenes).

4 Conclusions

The redox behavior of dihydroxybenzene isomers was demonstrated at the OMC/GC electrode. The OMC-modified electrode showed an excellent electrocatalytic activity

toward dihydroxybenzene isomer systems. Moreover, in a mixture solution, the isomer oxidation peaks become well resolved and are separated by about 150 mV for hydroquinone and catechol, 370 mV for catechol and resorcinol. This enables a highly selective and a simultaneous determination of dihydroxybenzene isomers at the OMC/GC electrode. The electrocatalytic enhancement, stability, and selectivity for dihydroxybenzene isomers oxidation demonstrate the potential application of OMC/GC electrode for further investigation and simultaneous determination of other isomer systems.

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