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ORIGINAL PAPER

Amperometric sensing of hydroquinone using a glassy carbon electrode modified with a composite consisting of graphene and molybdenum disulfide

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Abstract Well-dispersed composites of graphene and molybdenum disulfide were synthesized without using a mediator. The composites were characterized by scanning electron microscopy, transmission electron microscopy and X-ray diffraction. The electrochemical properties of a glassy carbon electrode (GCE) modified with the graphene/MoS_2 composite were investigated by electrochemical impedance spectroscopy and cyclic voltammetry. Hydroquinone was selected as a model target to show the sensing capability of the modified GCE. The GCE, best operated at a working voltage of 0.10 V (vs. Ag/AgCl), exhibits excellent catalytic activity towards hydroquinone, with a linear response in the 0.5 to 300 μM concentration range and a 37 nM detection limit (at an S/N ratio of 3). The superior performance of the GCE is attributed to the synergistic effects of graphene and MoS_2.

Keywords Solvothermal method · Electrochemical sensor · Differential pulse voltammetry · Dihydroxybenzene

Introduction

Hydroquinone is ubiquitous in nature since it is formed during biological degradation processes. It is one of the major raw materials generally used in manufacturing, such as coal mining, oil refining, and paint, polymer and pharmaceutical preparation [1]. Therefore, hydroquinone is released into the environment due to its applications in various industries [2]. However, hydroquinone harms the environment and devastates both marine and human life due to its high toxicity and low degradability at very low concentrations. Therefore, the development of a rapid and sensitive determination method for the analysis of hydroquinone is urgently needed. Currently, there are several analytical techniques, including liquid chromatography [3–5], chemiluminescence [6], spectrophotometry [7], for the analysis of hydroquinone. These methods have some disadvantages, such as insufficient sensitivity, high commercial price, and time-consuming procedures. However, an electrochemical analysis has the advantages of quick, simple, inexpensive, and effective analytical detection without complicated sample pretreatment [8–11].

To reduce and remove the overlap of redox peaks of phenolic compounds [12], various advanced nanomaterials have been used to modify electrodes for the determination of dihydroxybenzene isomers [13, 14]. Graphene, a two-dimensional (2D) arrangement with a honeycomb-like network, is attractive because of its superior properties, such as its suitable mechanical strength, large surface area, high conductivity and electron mobility at room temperature [15]. Because of its large π-electron structure and edges, graphene has a high electrochemical activity [16] and is

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widely applied in sensing fields [17]. However, its π-π stacking interaction also endows graphene with a hydrophobic property and causes irreversible aggregation, which limit its applications to some degree. Poly (diallyldimethylammonium chloride) (PDDA), a covalent substance, has been successfully used to modify graphene and create well-dispersed graphene in aqueous solutions [2]. Molybdenum disulfide (MoS_2), a 2D transition metal dichalcogenide, which is considered an inorganic analogue of graphene, has attracted great attention for sensor applications. However, the relatively poor electronic conductivity between two adjacent S–Mo–S layers is an obstacle to practical applications [18]. Recent efforts have been dedicated to combining MoS_2 nanosheets with conductive graphene materials to remove these disadvantages. Graphene and molybdenum disulfide are two crucial nanomaterials, and their hybrid nanomaterials are highly desirable [19]. MoS_2/reduced graphene oxide nanocomposites were created by Chekin et al. for sensitive sensing of cysteamine in human plasma [20]. Feng et al. used PDDA-functionalized graphene-MoS_2 nanoflowers to fabricate electrochemical sensors to detect eugenol [21]. Nevertheless, due to the presence of PDDA, the graphene conductivity will changed.

Well-diffused graphene was produced from graphene under alkaline conditions without non-covalent modifications and retained its conductivity. A well-dispersed graphene/MoS_2 composite was synthesized via a solvothermal route without a surfactant using a one-step method with graphene initially dispersed in ultrapure water. Then, the graphene/MoS_2-modified electrode was prepared using an effective method. The electrode exhibited a good analytical performance for hydroquinone and can be used for the determination of trace contamination.

Experimental

Materials

Graphite and hydroquinone were purchased from Sigma Aldrich (USA, www.sigmaaldrich.com). $Na_2MoO_4{\cdot}2H_2O$ and L-cysteine were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China, www.sinopharm.com). Other chemicals were analytical grade. A phosphate buffer solution (PBS, 0.1 mol L^{-1}) was prepared. Deionized water was acquired from Millipore (Bedford, MA, USA) and used in all experiments.

Preparation of graphene/MoS_2 composites

Well-dispersed graphene was synthesized as reported in our previous work [22]. Graphene (100 mg) was dispersed in 25 mL of ultrapure water, and $Na_2MoO_4{\cdot}2H_2O$ (200 mg) was added. After 5 min of ultrasonication, the pH of the solution was adjusted to 6.5 using 0.1 mol L^{-1} HCl. Then, L-cysteine (500 mg) and water (50 mL) were added and ultrasonicated for 10 min. Finally, the mixture was transferred into a 100-mL tightly closed Teflon-lined stainless steel autoclave to react at 200 °C for 36 h. After cooling at room temperature, the black graphene/MoS_2 composite was obtained via centrifugation (12,000 rpm for 30 min) three times and a water washing process followed by drying in a vacuum at 60 °C.

Characterization

Scanning electron microscopy (SEM) was carried out on a Sigma 300 microscope (ZEISS, Germany). Transmission electron microscopy (TEM) was conducted on a Tecnai F30G2 microscope (FEI, Netherlands). X-ray diffraction (XRD) patterns were collected on a D8 ADVAHCL (Bruker, Germany) instrument using Cu Ka radiation.

Fabrication of graphene/MoS_2 electrode

The glassy carbon electrode (GCE) was first polished with a 0.05 μm alumina slurry and washed with HNO_3/water (1/1, *v*/v), ethanol/water (1/1, v/v) and water via sonication. Then, the electrode was dried with N_2 before use. Subsequently, the prepared graphene/MoS_2 composites (1 mg ml^{-1}) were dropped onto the clean electrode surface and dried at room temperature.

Analytical procedure

Electrochemical measurements were carried out on a CHI 660E electrochemical workstation (Shanghai Chenhua Instrument Co. Ltd., China). A bare or graphene/MoS_2 GCE served as the working electrode in a three-electrode system. A Ag/AgCl electrode and a platinum wire were used as the reference electrode and counter electrode, respectively. The sensor was characterized using electrochemical impedance spectroscopy (EIS) in 0.1 mol L^{-1} KCl containing 5 mmol L^{-1} $[Fe(CN)_6]^{3-/4-}$ at a perturbation amplitude of −0.05 V in the frequency range from 0.1 Hz to 0.1 MHz. Differential pulse voltammetry (DPV) was performed in the potential range of −0.2 to 0.4 V.

Weihe river samples were collected in the Xi'an section. The samples were filtered to remove impurities and divided into two portions. One portion contained added standard hydroquinone and served as the spiked samples. The other portion served as the blank samples. The spiked samples were analyzed later.

Results and discussion

Characterization of graphene/MoS_2

In the process of preparing graphene, NaOH was added to obtain an aqueous homogeneous graphene sheet suspension from a precursor dispersion of GO. Because NaOH confers a large negative charge via reactions with the active functional groups on GO sheets [23], the GO is reduced by sodium borohydride. Therefore, the conductivity of the as-prepared, well-dispersed graphene was reserved.

SEM was used to investigate the morphology of the graphene/MoS_2 composites. Figure 1a shows the flower-like structure of graphene/MoS_2 and its structure can greatly increase the specific area of the composites. Figure 1b shows the TEM image of graphene, which has a wrinkled and folded sheet structure. The TEM image of the graphene/MoS_2 composites (Fig. 1c) shows that MoS_2 is supported on the graphene surface. Furthermore, the high resolution TEM image of the graphene/MoS_2 composite is shown in Fig. 1d. The typical layers of MoS_2 are displayed and the interlayer distance is 0.62 nm. Figure 2a displays the XRD pattern of graphene/MoS_2. There are three small, broad diffraction peaks that are contributed the (002), (100) and (110) planes of MoS_2. The above characterization results showed that the graphene/MoS_2 composites were successfully synthesized.

Electrochemical characterization

EIS was used to obtain a deep insight into the resistive and capacitive behaviors of the modified electrodes during the assembly process. The semicircle portion in the higher frequencies corresponds to an electron transfer limited process, and the linear portion in the lower frequencies is related to a diffusion process. The electron transfer abilities of the different electrodes are shown in Fig. 2b. The bare GCE displayed a semicircle with a Ret of 375 Ω and a linear part at low frequencies. The corresponding semicircle of the plot was reduced (130 Ω), when the GCE was modified with the graphene/MoS_2 composite material. These results indicate that the graphene/MoS_2 sensing film can enhance the electron transfer process.

The electrochemical behavior of hydroquinone was studied using differential pulse voltammetry on the bare and graphene/MoS_2-modified GCEs. Figure 2c shows the DPV signal for 1.0×10^{-4} mol L^{-1} hydroquinone for the bare GCE and graphene/MoS_2-GCE. There is a weak oxidation peak at a working voltage of 0.10 V on the bare GCE. The oxidation peak response was enhanced on the graphene/MoS_2-modified GCE. The phenomenon can be explained by the promotion of the interface electron transfer process, which is attributed to the synergistic effect of graphene and MoS_2. The results show that the graphene/MoS_2 film had a strong enhancement effect

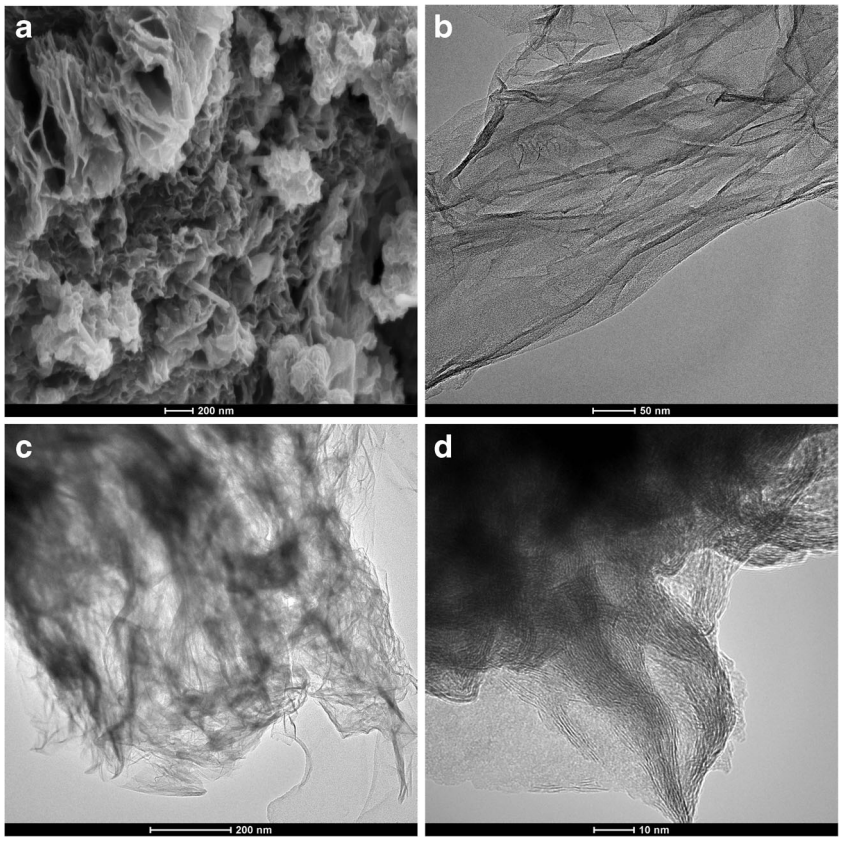

Fig. 1 **a** SEM image of MoS_2/graphene composite; TEM image of (**b**) graphene and (**c**) MoS_2/graphene composite; (**d**) HRTEM image of graphene/MoS_2 composite

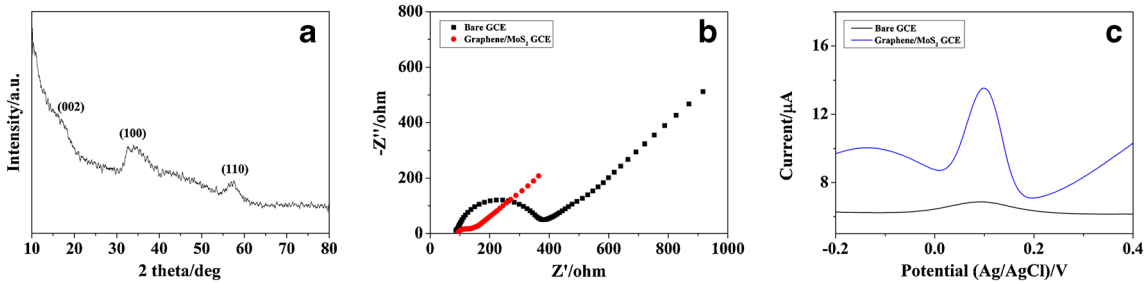

Fig. 2 **a** XRD pattern of MoS_2/graphene composite; (**b**) Nyquist plots of the bare and MoS_2/graphene GCE; (**c**) DPV obtained for the determination of hydroquinone on the bare and MoS_2/graphene GCE in a 0.1 mol L^{-1} phosphate buffer solution containing 1.0×10^{-4} mol L^{-1} hydroquinone

on the oxidation of hydroquinone, and DPV is suitable for the trace detection of hydroquinone.

Scan rate effects

To investigate the reaction kinetics, the effect of the scan rate (v) on the redox reaction of hydroquinone is shown in Fig. 3. The redox peak currents simultaneously increase with an increase in the scan rate from 30 to 300 mV s^{-1}. Both the anodic (Ipa) and cathodic (Ipc) peak currents are linearly related to the square root of the scan rate. The linear equations are Ipa (μA) = 1.035 $v^{1/2}$ (mV s^{-1}) + 1.054 and Ipc (μA) = −0.8522 $v^{1/2}$ (mV s^{-1}) − 0.7430 with R = 0.9994 and 0.9984, respectively. The results indicate that the reaction of hydroquinone on the modified electrode is a diffusion-controlled process. In addition, the oxidation (Epa) and reduction (Epc) potential have a linear relationship with the natural logarithm of the scan rate (ln$v$). The corresponding equations are Epa = 0.03526 ln$v$ + 0.02914 (R = 0.9815) and Epc = −0.02725 ln$v$ + 0.1796 (R = 0.9871). The slope of the Epa and Epc equations follow Laviron's model [24] and can be expressed as 2.3RT/n(1−α)F and −2.3RT/nαF, respectively, where n is the number of transferred electrons, α is the electron transfer coefficient, R is the gas constant, T is the absolute temperature, and F is the Faraday constant. According to the calculation, the value of α (0.56) is close to the theoretical value of 0.5 for a quasi-reversible process. The number of transferred electrons is 1.67 (approximates to 2), revealing the electrochemical reaction of hydroquinone is a two-electron transfer process in. All the results indicate that the electrochemical reactions of hydroquinone at the graphene/MoS_2 GCE are two-electron and two-proton transfer reactions.

Detection of hydroquinone

Under the optimal conditions, the graphene/MoS_2 GCE was used to detect hydroquinone at a pH of 7.5 in PBS. There is a good linear relationship between the oxidation peak current (Ip, A) and the concentration (c, mol L^{-1}) of hydroquinone over the dynamic range from 5.0×10^{-7} to 3.0×10^{-4} mol L^{-1} (Fig. 4). The linear regression equation is Ip =0.03619 c + 1.224 (R = 0.9980). The limit of detection was estimated to be 3.7×10^{-8} mol L^{-1} (S/N = 3). Compare to that of other the reported electrochemical methods (Table 1), the present method had a broad linear range and a low detection limit for hydroquinone detection. The results demonstrate that the graphene/MoS_2 composites provide a large specific surface area to increase the loading of hydroquinone and accelerate the electron transfer to amplify the electrochemical signal. Therefore, the present electrochemical method achieves a trace detection of hydroquinone.

Reproducibility, stability and interferences

The reproducibility of the electrochemical sensor was tested by repeating the detection of 1.0×10^{-4} mol L^{-1} hydroquinone. The relative standard deviation (RSD) was 5.39%,

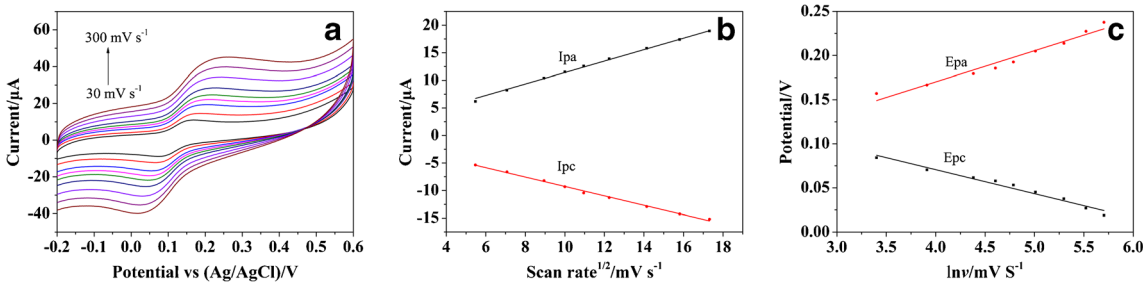

Fig. 3 **a** Cyclic voltammetry curves of 1.0×10^{-4} mol L^{-1} hydroquinone in a buffer at pH 7.5 on the graphene/MoS_2 GCE at various scan rates from 30 mV s^{-1} to 300 mV s^{-1}; (**b**) The plot of the peak currents versus the square root of the scan rate; (**c**) The relationships between the peak potential and Napierian logarithm of the scan rate

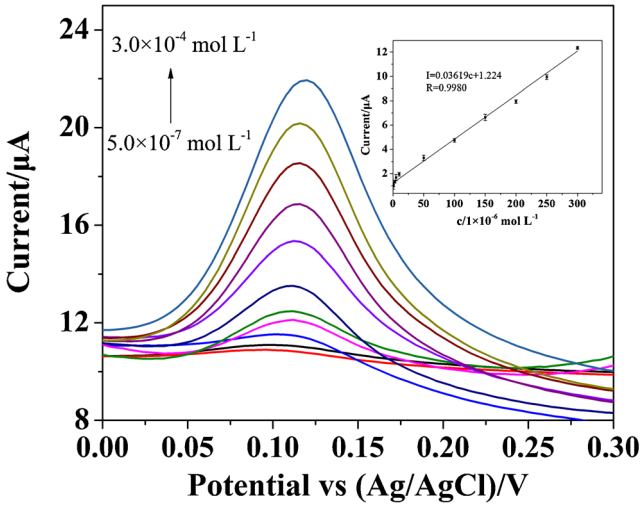

Fig. 4 DPV for the different concentrations of hydroquinone (0.5, 1.0, 3.0, 5.0, 10, 50, 100, 150, 200, 250, and 300 $\times 10^{-6}$ mol L^{-1}) on the graphene/MoS_2 GCE

which indicated an acceptable reproducibility and precision. The stability of the graphene/MoS_2 GCE was investigated by measuring the electrochemical response of 1.0×10^{-4} mol L^{-1} hydroquinone. The results indicated that the signal only decreased 4.2% when the modified GCE was stored in air for two weeks. The selectivity of the sensor was also studied by adding the interferents, e.g., some ions and species, and examining their impact on the determination of hydroquinone. The coexisting components in the sample, e.g., 200-fold sodium, potassium, copper, chloride ion, glucose, citric acid and cysteine concentrations, did not interfere with the determination of hydroquinone. Furthermore, Fig. S4 shows the DPV curves for hydroquinone in the presence of catechol and resorcinol on a graphene/MoS_2-modified GCE. Three well-distinguished anodic peaks can be seen. The results indicated that the oxidation of the dihydroxybenzene isomers in the mixed solution occurred independently at the graphene/MoS_2 electrode. All the results demonstrated that the graphene/MoS_2 electrode has a good selectivity for hydroquinone in the presence of the mentioned interferents.

Table 2 Detection of hydroquinone in tap water using the present electrochemical sensor

Samples	Added (μmol L^{-1})	Found (μmol L^{-1})	Recovery (%)
1	1	0.97	97.0
2	10	9.89	98.9
3	50	49.53	99.1
4	200	204.3	102.1

Analytical application

The present sensor was used to detect hydroquinone in Weihe water samples. Hydroquinone was not found in the Weihe water. Hydroquinone standard solutions were spiked into the Weihe water samples, and the spiked samples were analyzed with the present sensor. The results are shown in Table 2. The recovery of the spiked hydroquinone in the Weihe water samples was in the range of 97.0–102.1%. These results indicate that this method is suitable for hydroquinone determination in real samples.

Conclusion

A one step synthesis for well-dispersed graphene/MoS_2 composites was developed using a solvothermal method without any functional molecules. Evidently, layer-by-layer stacking is one of the simplest and most straightforward methods for heterostructure construction. However, interfacial contamination is an important factor that must be considered. Additionally, the adsorbates on the individual layers need to be reduced and the interface bonding needs to be improved. In this work, a sensitive electrochemical sensor was fabricated with the graphene/MoS_2 composites and used to detect hydroquinone in Weihe water samples under the optimized conditions. The present strategy provides a way to prepare graphene composites with good compatibility and dispersibility and a novel and promising platform for analytical applications of graphene materials.

Table 1 Comparison of different electrochemical methods for determination of hydroquinone

Electrode	Linear range (μmol L^{-1})	Detection Limit (μmol L^{-1})	Reference
Au-PdNF/rGO GCE	1.6–100	0.5	[1]
AuNPs/Fe_3O_4-APTES-GO GCE	3–137	1.1	[2]
Graphene GCE	1–300	0.5	[9]
GCN GCE	1–30	0.02	[12]
AuNPs-MPS CPE	10–1000	1.2	[25]
MOF-ERGO-5 GCE	0.1–476	0.1	[26]
MWCNTs-PDDA-Graphene GCE	0.5–400	0.02	[27]
ZnS/NiS@ZnS/L-Cys/AuNPs GCE	0.5–400	0.024	[28]
poly(melamine)/Graphene CPE	7–1000	0.074	[29]
Graphene/MoS_2 GCE	0.5–300	0.037	This work

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Compliance with ethical standards The author(s) declare that they have no competing interests.

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