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Short communication

Fabrication of ultra-sensitive and selective dopamine electrochemical sensor based on molecularly imprinted polymer modified graphene@carbon nanotube foam

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ABSTRACT

A new type of three-dimensional (3D) electrochemical sensor was prepared by combining carbon nanotubes on graphene foam (GF/CNT) with molecularly imprinted polymer (MIP), which affords simultaneous identification and quantification towards target compound. The hybrid sensor shows ultralow detection limit of 6.67×10^{-16} M (S/N = 3), wide range of 2×10^{-15} M to 1×10^{-12} M, and superb selectivity for dopamine (DA) detection.

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1. Introduction

High sensitivity, among others, is one featured merit of electrochemical sensor in quantification of substances [1–6]. However, target analyte possessing electrochemical activity is a prerequisite of this approach as most of electrochemical determination is based on redox reaction of analyte on the interface between electrode and electrolyte [7–9]. One way to circumvent this requirement is to cover the electrode surface with a non-conductive membrane which owns on/off gates in response to analyte and apply probe media to indicate electrical signals. Thereafter, concentration of target molecules can be calculated. By carefully molding the gates on the membrane, selective recognition of sensor toward targets can also be realized.

Our groups, as well as others, have developed such a sensing strategy by using molecularly imprinted polymer (MIP) as the insulating film [10–16], where commercial electrodes are often adopted. Besides, in these reports, additional materials with good conductivity and high surface area are usually involved in order to enhance sensitivity and electron transfer rate [14–17]. Recently, we fabricated gold microwire with nanoporous surface as supportless working electrode and used it for accurate, sensitive and selective assay of metronidazole [18]. With the aim of exploring sensing materials with more desirable performance, we turn to the widely distributed element-carbon, and employed three dimensional (3D) carbon nanotube at graphene foam as the electrode substrate. Preparation of such hybrid materials have been reported [19,20] and they are mainly applied as high-performance energy storage devices [21,22]. Herein, we construct a novel electrode by coupling the carbon hybrid with MIP film and this free-standing sensor proves its outstanding performance in dopamine (DA) detection.

2. Experimental

2.1. Reagents and apparatus

Dopamine (DA), ascorbic acid (AA) and uric acid (UA) were purchased from Adamas Reagent Co. Ltd. (Shanghai, China). *L*-adrenaline bitartrate (AD), *o*-phenylenediamine and α-phenylethylamine (PEA) were from Sigma-Aldrich Co. (Shanghai, China) and phosphate buffer solution (PBS) was prepared from KH_2PO_4 and K_2HPO_4. 3-O-methyldopamine hydrochloride and caffeic acid were obtained from Adamas Reagent Co. Ltd. All the reagents are of analytical grade and the solutions were prepared with double distilled water. All electrochemical measurements were carried out using a CHI 760E electrochemical workstation (Shanghai, China) with a three-electrode system (prepared electrodes as working electrode; saturated calomel electrode (SCE) and platinum wire as reference and counter electrodes, respectively). Scanning electron microscopy (SEM)

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images were taken with a Zeiss Supra 55VP microscope operating at 10 kV.

2.2. Preparation of GF/CNT/MIP

Graphene foam (GF) was grown in the way similar to previous report [5,23,24]. Nickel foam (NF) was placed in a horizontal quartz tube furnace and heated to 1000 °C at 100 mTorr under Ar flow of 500 sccm and H_2 flow of 100 sccm for 10 min. Then, 100 sccm methane was supplied into the chamber under 800 sccm Ar and 100 sccm H_2 for 10 min. After that, the gases were purged and the chamber was cooled down to room temperature.

The vertically aligned CNTs was grown on GF using plasma-enhanced chemical vapor deposition (PECVD) similar to the method reported in [17,25,26]. GF with Ni template was treated with O_2 plasma (80 W) for 90 s and coated with 3 nm Al via atomic layer deposition. Fe–Co bimetallic catalyst was synthesized via spraying iron acetate and cobalt acetate ethanol solution on carbon fiber paper at 90 °C and subsequently heated at 500 °C for 10 min. The GF was put in the PECVD chamber, which was first evacuated to 10^{-5} Pa, and then 20 sccm H_2 was supplied at 133 Pa. The chamber was ramped to 500 °C (100 °C min^{-1}), and then a radio frequency alternating magnetic field (30 W) was switched on to treat the Fe–Co bimetallic catalyst using H_2 plasma for 2 min. As the 20-sccm H_2 flow was maintained, 40 sccm of C_2H_2 was introduced and kept for 20 min to grow CNTs.

The resulting material was then submerged in 1 M $FeCl_3$ and 2 M HCl to completely remove NF. The obtained GF/CNT (5 × 5 mm) pieces were fixed onto a glass wafer and electrical lead was made by silver paint.

MIP film was prepared on GF/CNT surface by cyclic voltammetry (CV) in solution containing 2 mM DA and 6 mM *o*-phenylenediamine in PBS (pH = 7.0), which was implemented in the potential range of 0.0~0.8 V for 200 cycles at 50 mV s^{-1}. The embedded DA were then extracted by CV from −0.5 to 0.5 V in 0.1 M NaOH until a pair of stable redox peaks emerged in probe solution containing 5 mM $Fe(CN)_6^{3-/4-}$ and 0.1 M KCl. Non-imprinted polymer decorated sensor (GF/CNT/NIP) was fabricated via the same procedure without adding DA.

2.3. Electrochemical measurements

The differently modified electrodes were incubated in analyte solution at certain concentration for 10 min, and then rinsed with water and ready for CV and electrochemical impedance spectroscopy (EIS) analyses. A washing step was followed after detecting one kind of analyte to extract sorbed compounds by CV in 0.1-M NaOH at −0.5~0.5 V until stable peaks reappeared.

3. Results and discussion

Typical cyclic voltammograms during MIP preparation are displayed in Fig. 1A. The currents decreased with continuous scanning until no more changes can be observed at the last several cycles, suggesting that an insulating polymeric film formed on GF/CNT and led to restraint of the voltammetric response. In Fig. 1B, the Raman spectrum of GF illustrates two characteristic peaks at ~1584 cm^{-1} (G-band) and ~2726 cm^{-1} (2D-band) [5,23], and the G/2D ratio suggests typical graphene structure with few-layer. There is no D band at 1347 cm^{-1} [25], which corresponds to the defects and disorderly carbon, indicating the high quality of graphene. In comparison, distinct peaks of D, G and 2D bands were observed from GF/CNT, of which the D-band is related to defects of CNTs [25]. The SEM image in Fig. 1C shows that GF/CNT after etching off NF keeps interconnected structure with slightly rough surface. At high resolution, we can observe that the CNTs on GF are complex intertwinement with the diameter around 30~50 nm. MIP modification barely results in apparent change on surface morphology of GF/CNT, but with a little growth in diameter of CNT to about 40~65 nm (Fig. 1D). EDS spectra confirm the successful extraction of DA from

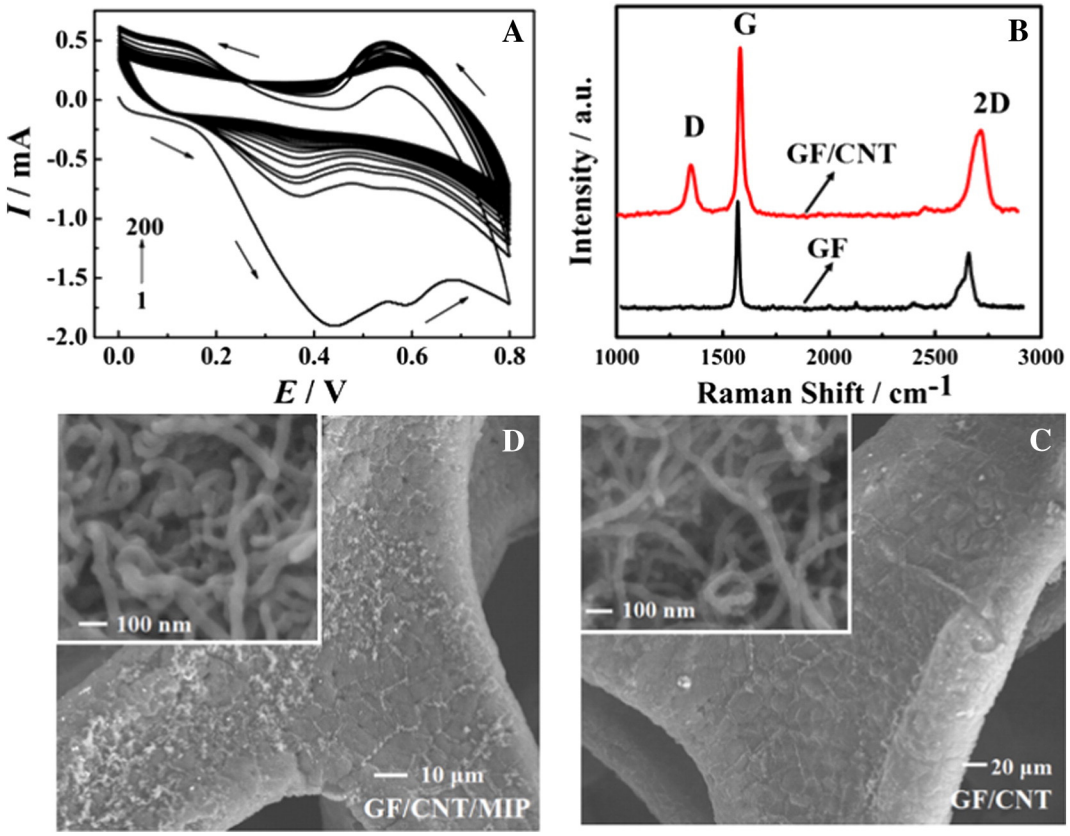

Fig. 1. Cyclic voltammograms for electrochemical preparation of DA-imprinted MIP (A); Raman spectra of GF and GF/CNT (B); SEM for GF/CNT (C) and GF/CNT/MIP (D).

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