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Nickel oxide/carbon nanotube nanocomposites prepared by atomic layer deposition for electrochemical sensing of hydroquinone and catechol

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ABSTRACT

The fabrication of nickel oxide/carbon nanotube (NiO/CNT) nanocomposites by atomic layer deposition (ALD) and their electrochemical performances for sensing hydroquinone (HQ) and catechol (CC) were investigated. NiO nanopaticles with a size of ~4.9 nm formed by ALD were highly dispersed on the wall of pristine CNT. Then, the prepared NiO/CNT nanocomposites were used to modify glassy carbon electrode (NiO/CNT/GCE) for electrochemical detection, which presented enhanced electrochemical performances towards detecting HQ and CC. Not only were the redox peak currents of HQ and CC greatly enhanced, but also the oxidization peaks of the both phenolic isomers could be well separated. Under the optimized conditions, the current responses are linear for HQ and CC in the range of 10–500 μM and 10–400 μM, with detection limit of 2.5 μM (S/N = 3) for both compounds.

1. Introduction

As two important isomers of phenolic compounds [1], hydroquinone (HQ) and catechol (CC) have been widely exploited in medicines, cosmetics, and so forth [2]. However, the excessive intake of HQ and CC may lead to different diseases including but not limited to fatigue, headache, and liver function lesion [3]. HQ and CC are officially regarded as environmental pollutants in US and in European Union because they are highly toxic and low degradable in the ecological system [4]. Therefore, the quantitative detection of HQ and CC is of great importance. Various analytical techniques, such as liquid chromatography [5], fluorescence [6,7], gas chromatography–mass spectrometry [8], chemiluminescence [9,10], and electrochemical methods [2,4] have been widely used for the measurement of HQ and CC. Compared with other analytical methods, electrochemical techniques have recently drawn much more attention due to their characteristics of fast, simple and sensitive. However, because of the similar structures of these twophenolic isomers and the coexistence of other interferences [11], the selective and sensitive electrochemical determination of HQ and CC still remains a challenge. To overcome these issues, many nanomaterials with intriguing electrochemical properties have been successfully applied to realize the selective and sensitive detection of HQ and CC [4,12].

Nickel oxide (NiO) is a very promising transition metal oxide for the characteristics of non-toxicity, low cost, high catalytic activity as well as easy preparation [13]. However, the poor stability and low electronic conductivity of NiO limit its wide application in electrochemical fields. To overcome these problems, some NiO-based nanocomposites with suitable supports (e.g. CNT, grapheme and other carbons nanomaterials) have been designed and prepared via various methods for electrochemical applications. For example, Goulart et al. fabricated NiO/multiwalled carbon nanotube (NiO/MWCNT) nanocomposites by electrodeposition to detect HQ, CC and BPA simultaneously [13]. Zhang et al. prepared the NiO/MWCNTs nanocomposite by magnetron sputtering deposition method, which was directly used as an electrode to detect the concentration of glucose [14]. Liu et al. fabricated flower-like NiO/nitrogen-doped carbon hollow spheres via chemical bath deposition followed by calcination at 350 °C under nitrogen atmosphere as an appealing electrode material for supercapacitor [15]. However, the precise control over the size and distribution of NiO nanoparticles onto conductive supports still remains challenging.

Atomic layer deposition (ALD) has emerged as an important and powerful vapor-phase deposition technique since 1970s. Due to the sequential, self-saturating, gas-surface reaction control nature of ALD processes, it possesses several distinct advantages over other vapor-phase counterparts, such as the very precise thickness control at the atomic level, excellent uniformity and conformality, and low growth temperature, and so forth [16–18]. ALD has been widely considered as

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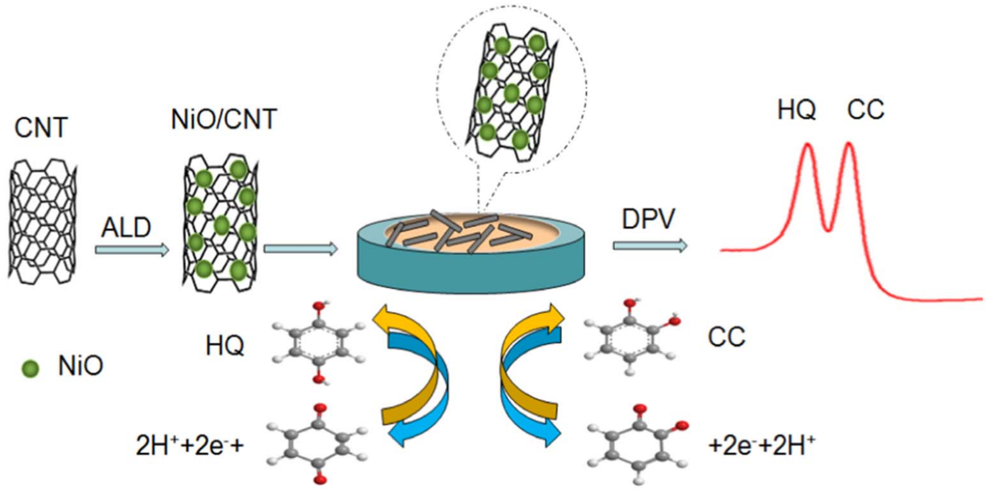

Scheme 1. The schematic illustration of fabrication of NiO/CNT nanocompistes and their application in electrochemical detection of HQ and CC.

a promising technology for surface modification and fabrication of nanostructured materials, and has been applied in many areas because of its precise control over particle size and distribution [19]. For instance, several NiO-based nanocomposites have been successfully fabricated by ALD method, which presented very promising electrochemical performances [20–22]. Although ALD technology has been extensively used in electrocatalysis and electrochemical energy storages [23–26], it has been rarely used in the field of electrochemical detection. Therefore, it will be very important and meaningful to extend the applications of ALD into electrochemical detection. In this paper, we reported the fabrication of NiO/CNT nanocomposites by ALD method and their application as electrode material for electrochemical detecting of HQ and CC (Scheme 1). Because of numerous active sites, the prepared NiO/CNT nanocomposites show a very remarkable performance in detecting HQ and CC. As far as we know, this is the first time that NiO/CNT nanocomposites prepared by ALD have been applied to electrochemical detection of electroactive biomolecules.

2. Experimental section

2.1. Reagents

HQ, CC, uric acid (UA), ascorbic acid (AA), hydrogen peroxide (H_2O_2), $NaH_2PO_4 \cdot 2H_2O$, $Na_2HPO_4 \cdot 12H_2O$, bis(cyclopentadienyl)nickel (II) were purchased from Aladdin, and Nafion (5 wt% in ethanol) was purchased from Sigma-Aldrich. CNT sample (40–60 nm in outer diameter and 5–15 μm in length) was obtained from Shenzhen Nanotech Port Co. (Shenzhen, China). All the other chemicals are of analytical grade and used without further purification. The 0.1 M phosphate buffer solution (PBS) with various pH were prepared by mixing the stock solution of 0.2 M NaH_2PO_4 and 0.2 M Na_2HPO_4.

2.2. Fabrication and characterization of NiO/CNT

The fabrication of NiO on CNT was performed in a hot-wall, closed chamber-type ALD reactor (D100-4P8C8H2F). First, ~30 mg CNT powder was dispersed in pure ethanol. Then, the suspension was dispersed on the Si substrate by spin-coating and dried at room temperature [20]. Finally, the Si wafer was transferred to the ALD instrument. During reaction process, nitrogen plays as the protective gas, O_3 acts both as reagent to form the defect site and as a source of oxygen element, and bis(cyclopentadienyl)nickel(II) acts as precursor of Ni element. The pulse, exposure, and purge times for the bis(cyclopentadienyl)nickel(II) precursor were 1, 2, and 2 s, respectively, and for O_3 they were 1, 10, and 5 s, respectively. The typical deposition cycles were 400, and the duration time of ALD process is about 2.3 h. For the characterization of the prepared NiO/CNT composites, TEM and selected area electron diffraction (SAED) experiments were performed with JEM-2100 (JEOL Co., Japan), and XPS analysis was conducted on a Thermo Scientific VG ESCALAB 200i-XL spectrometer.

2.3. Preparation of NiO/CNT/GCE and electrochemical tests

Before modification, the glassy carbon electrode (GCE, 3.0 mm in diameter) was carefully polished, and then 5 μL of NiO/CNT suspension (5 mg/mL with 0.25 wt% Nafion) or CNT suspension (5 mg/mL with 0.25 wt% Nafion) was dropped onto the GCE carefully and dried at room temperature. Cyclic voltammetry (CV), differential pulse voltammetry (DPV) and electrochemical impedance spectroscopy (EIS) were carried out on a CHI-660C electrochemical workstation (Chen Hua instruments Co., Shanghai). The conventional three electrode cell was applied with a bare GCE or a modified GCE as working electrode, platinum wire and saturated calomel electrode (SCE) as auxiliary electrode and reference electrode, respectively.

3. Results and discussion

3.1. Characterization of NiO/CNT

NiO/CNT nanocomposites were firstly characterized by TEM. We can clearly find that pristine CNT are curving tube with smooth wall (Fig. 1(A)). After ALD treatment, NiO nanoparticles homogenously formed on the wall of CNT (Fig. 1(B)), and the size of NiO particles is quite uniform (4.9 ± 0.2 nm). The particle size can be controlled by ALD process parameters during the nanoparticles nucleation and growth process [20]. Fig. 1(C) is the corresponding high-resolution TEM image of NiO/CNT nanocomposites. It shows two well-defined lattice fringe spacings of 0.209 and 0.243 nm, which could be ascribed to the (200) and (111) crystal planes of NiO, respectively [27]. In Fig. 1(D), the SAED pattern further confirmed the polycrystalline characteristic of NiO nanoparticles on CNT through a multiple concentric rings pattern.

The chemical compositions and states of the NiO/CNT composites were characterized by XPS. As shown in Fig. 2(A), the survey spectrum of NiO/CNT demonstrates the existence of carbon, oxygen, and Ni species. In Fig. 2(B), the high-resolution spectrum of Ni 2p shows two obvious peaks located at 856.4 and 874.4 eV, which are ascribed to Ni $2p^{3/2}$ and Ni $2p^{1/2}$, respectively [28]. Besides, the binding energy peaks at 861.6 and 880.2 eV are assigned to the satellite peaks of Ni $2p^{3/2}$ and Ni $2p^{1/2}$, respectively. In Fig. 2(C), the C 1 s peak centered at 284.6 eV is the typical graphitic carbon peak. The XPS spectra of C 1 s can be further fitted to four peaks located at 284.16, 285.06, 286.96 and

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