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Electrocatalytic oxidation of hydrazine at a glassy carbon electrode modified with nickel ferrite and multi-walled carbon nanotubes

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Abstract A hydrothermal technique was used to synthesize nickel ferrite nanoparticles (NF-NPs) deposited on multi-walled carbon nanotubes (MWCNTs). The material was characterized by scanning electron microscopy, energy dispersive spectrometry, and X-ray powder diffraction which showed that the NF-NPs are located on the surface of the carboxylated MWCNTs. The material was used to modify a glassy carbon electrode which then was characterized via cyclic voltammetry, electrochemical impedance spectroscopy, and amperometry. The electrode displays strong electrochemical response to hydrazine. A potential hydrazine sensing scheme is suggested.

Keywords Nickel ferrite · MWCNTs · Modified electrode · Electrocatalysis · Hydrazine

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Introduction

Since the first synthesis of hydrazine from diazoacetic ester [1], it has been greatly interested in both chemical and pharmaceutical industry. Also, it has been widely used as rocket fuel, emulsifiers, catalysts, weapons for mass destruction, corrosion inhibitors, insecticides and plant-growth regulators, etc. In the meanwhile, it can threaten people's health from injection, inhalation of vapors, or skin contact. The threshold value range from 1 μg ml^{-1} to 10 μg ml^{-1} according to the regulation of the American Conference of Governmental Industrial Hygienists (1963). Thus, sensitive and fast detection and determination of hydrazine is becoming more and more important. Numerous methods have been developed to determine hydrazine, such as chromatography [2], spectrometry [3, 4], chemiluminescence [5], flow injection analysis [5–7] and electrochemical technique [8–12]. Among them, electrochemical technique is the most development potential method because of its fast response, high sensitivity, low cost, etc.

Unfortunately, hydrazine exhibited irreversible oxidation required large overpotential at bare carbon electrode. As a result from this, most of the modified electrodes have been fabricated and applied in the detection and determination of hydrazine, which can dramatically lower the overpotential and increase the oxidation current response to hydrazine. Such as reduced graphene sheets modified glassy carbon electrode (GCE) [13], polypyrrole/GCE [14], palladium-modified microdisk array electrode [15], Ag/MWCNTs/GCE [16], ZnO/GCE [17], Ni(II)-baicalein complex modified MWCNTs paste electrode [18], etc.

Nickel ferrite, with the formula of NiFe$_2$O$_4$, has a face-centered cubic structure with a large unit cell containing

eight formula units [19]. And it is not only quite commonly used as a kind of magnetic material [20], but also used as the catalyst in the decomposition of carbon dioxide. What's more, because of the properties of nano nickel ferrite, such as its large specific surface area, various redox states, electrochemical stability, pseudocapacitive behavior [21] and so on, it has been used in the preparation of semiconducting gas sensors [22, 23], glucose biosensor [24], etc. Besides, the search for techniques for the immobilization of redox mediators, which would provide a stable catalytic response for the analyte at the electrode surface is quite reasonable [25]. To enhance the attachment of nano-nickel ferrite onto the glassy carbon electrode, MWCNTs have been used, which are suitable materials for electrode modification and support in biosensor applications because of the high accessible surface area, low electrical resistance, extremely high mechanical strength and stiffness, outstanding charge-transport characteristics and high chemical stability [26–29].

Herein, we, for the first time, have successfully synthesized $NiFe_2O_4$/MWCNTs via one-step process using hydrothermal technique, as confirmed directed by scanning electron microscopy (SEM), energy dispersive spectrometry (EDS), X-ray powder diffraction (XRD). Glassy carbon electrode was modified using $NiFe_2O_4$/MWCNTs to form $NiFe_2O_4$/MWCNTs/GCE as an amperometric sensor for the electrocatalytic oxidation of hydrazine. The electrochemical sensor exhibited wide linear range and low detection limit, excellent stability and reproducibility.

Experimental

Reagents and apparatus

Carboxylated multi-walled carbon nanotubes (MWCNTs) (diameter 30–50 nm) were purchased from Chengdu Institute of Organic Chemistry of Academy of Sciences (http://www.timesnano.com) and synthesized by chemical vapor deposition. $Ni(NO_3)_2 \cdot 6H_2O$ was obtained from Shanghai Hengxin Chemical Reagent Co., Ltd. (China). Other chemicals were obtained from Shanghai Chemical Reagent Co. Ltd. (Shanghai, China, http://www.china-reagent.com), such as hydrazine, $Fe(NO_3)_3 \cdot 9H_2O$, NaH_2PO_4 and Na_2HPO_4, etc. All of them were of analytical grade and used as received without further purification. Doubly distilled water was used throughout this research. Phosphate buffer saline (PBS, 0.1 M) were prepared by varying the ratio of NaH_2PO_4 to Na_2HPO_4. And all experiments were conducted at 20 °C

SEM and EDS were performed with a Hitachi S-4300 (Japan); XRD study was carried out on an X-ray diffractometer (Shimadzu, Japan) using Cu Kα source (λ=1.5406

Angstroms) at 40 kV, 30 mA in the range of $10° < 2\theta < 70°$ at the scan rate of 6.0°/min. The electrochemical experiments were performed on CHI660B electrochemical workingstation (CH Instruments, Chenhua Corp. Shanghai, China, http://chi.instrument.com.cn). All electrochemical experiments were carried out by three-electrode system with bare glassy carbon electrode or the modified electrode as the working electrode, a platinum wire as the auxiliary electrode, and a saturated calomel electrode (SCE) as the reference electrode.

Synthesis of $NiFe_2O_4$/MWCNTs nanocomposites

$NiFe_2O_4$/MWCNTs nanocomposites were synthesized via a facile hydrothermal method refered from [30]. The procedure of the synthesis was performed as follows: 0.04 g MWCNTs were dispersed into 2 mL ethylene glycol to form a turbid liquid. And then 10 mL solution, containing 0.15 g nickel nitrate, 0.40 g ferric nitrate, 3.60 g sodium acetate and 0.20 g polyethylene glycol, was rapidly poured into the turbid liquid and vigorously stirred for 30 min at room temperature. After that, the mixture was transferred into a Teflon-lined stainless steel autoclave. The clave was maintained at 200 °C for 12 h, and finally cooled to room temperature naturally. The product was collected by a magnet and washed with distilled water and absolute alcohol alternately for several times and dried at 70 °C.

Fabrication of $NiFe_2O_4$/MWCNTs/GCE

Prior to modification, the bare glassy carbon electrode was polished to a mirror-like surface with alumina slurry followed by rinsing thoroughly with doubly distilled water. After that, the electrode was sonicated in nitric acid (1:1), absolute alcohol and doubly distilled water for 5 min, successively, in order to remove any adsorbed substances on the surface of the electrode. Finally, it was dried under nitrogen atmosphere ready for use.

5 mg the synthesized $NiFe_2O_4$/MWCNTs nanoparticles were suspended in 0.5 mL doubly distilled water under ultrasonic to form a suspension. 6 μL above suspension were dipped onto the pretreated glassy carbon electrode and the electrode was dried in air to obtain the $NiFe_2O_4$/MWCNTs modified GCE ($NiFe_2O_4$/MWCNTs/GCE).

Results and discussion

Characterization of $NiFe_2O_4$/MWCNTs nanocomposites

Figure 1 shows the SEM images of the MWCNTs before and after $NiFe_2O_4$ deposition. The pristine MWCNTs are curved and twisted with each other with very smooth surfaces (Fig. 1a). After the deposition of $NiFe_2O_4$, the

Fig. 1 SEM images of MWCNTs (**a**) and NiFe$_2$O$_4$/MWCNTs (**b**)
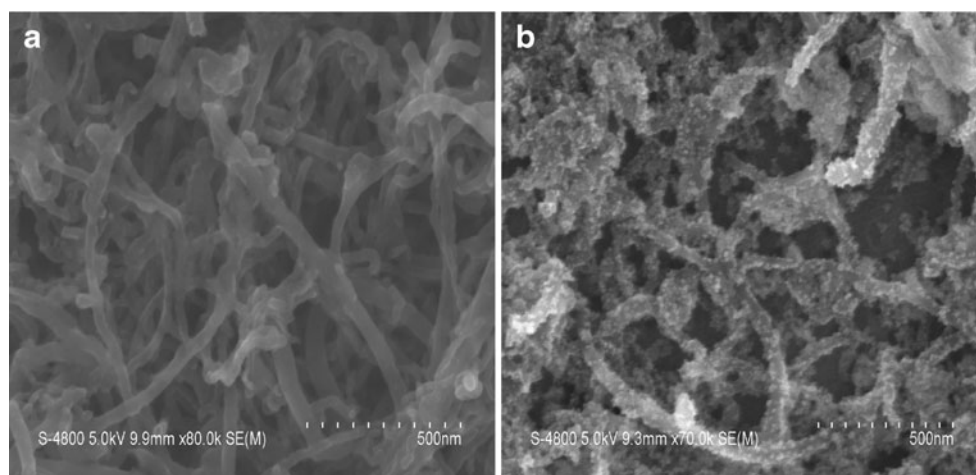
MWCNTs became visibly hackly (Fig. 1b), which indicating that nanoparticles adsorbed onto the surface of MWCNTs.

To make sure the nanoparticles were nickel ferrite, EDS and XRD were investigated. The EDS result (Fig. 2a) shows that the weight content of C, O, Fe, Ni is 36.14%, 25.19%, 25.30%, 13.37% and the atom content of C, O, Fe, Ni is 57.16%, 29.91%, 8.61%, 4.32%. From the data given above, it can be easily calculated that the nickel-to-iron atomic ratio is 1:2, which goes well with the nickel-to-iron ratio in NiFe$_2$O$_4$. However, iron-to-oxygen atomic ratio is smaller than 2:4, which was ascribed to carboxylated MWCNTs. Figure 2b shows the XRD pattern of the NiFe$_2$O$_4$/MWCNTs powders. (111), (220), (311), (222), (400), (422), (511), (440) belong to NiFe$_2$O$_4$ according to the standard JCPDS (Card No. 10–0325). The diffraction angle at $2\theta=26.24°$ corresponds to the characteristic peak of MWCNTs. The results indicate that NiFe$_2$O$_4$/MWCNTs composites are successfully synthesized.

Electrochemical behavior of the NiFe$_2$O$_4$/MWCNTs/GCE

To study the electrochemical properties of NiFe$_2$O$_4$/MWCNTs/GCE, the cyclic voltammetries (CVs) and Nyquist plots of impedance measurements for 5 mM [Fe(CN)$_6$]$^{3-/4-}$ solution containing 0.1 M KCl were investigated, as shown in Fig. 3. The NiFe$_2$O$_4$/GCE (curve b) showed a sluggish voltammetric response, with a ΔE_p value of 160 mV, indicating that the NiFe$_2$O$_4$ on the electrode surface hinders the electron transfer kinetics. However, a significant increase in the peak current associated with a decrease in the ΔE_p value was noticed at NiFe$_2$O$_4$/MWCNTs/GCE (curve c). The ΔE_p value observed at the NiFe$_2$O$_4$/MWCNTs/GCE are very close to that of the bare GCE (curve a), suggesting nearly reversible or quasireversible electron transfer kinetics for these electrode interfaces. In the case of the NiFe$_2$O$_4$/MWCNTs/GCE, a significant increase in the peak currents were observed (curve c) because of the increase of the surface area. In Fig. 3b, the semicircular portion at higher

Fig. 2 EDS (**a**) and XRD (**b**) pattern of NiFe$_2$O$_4$/MWCNTs
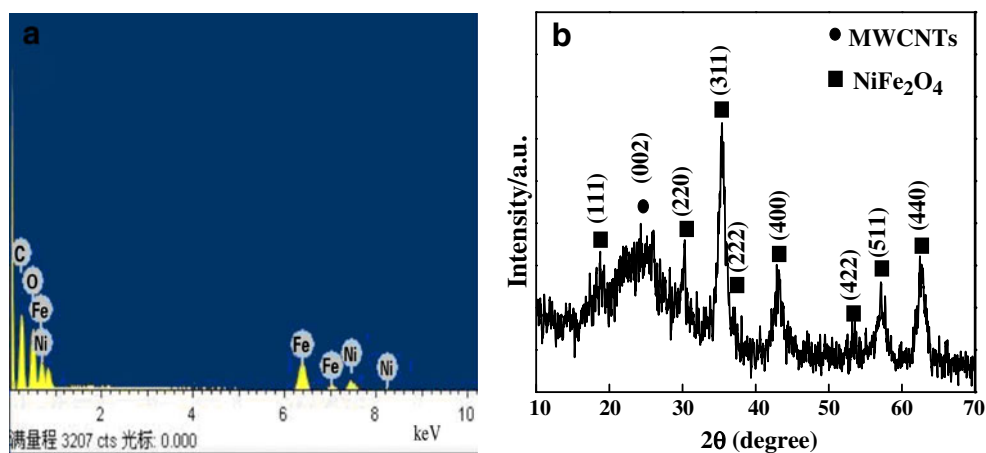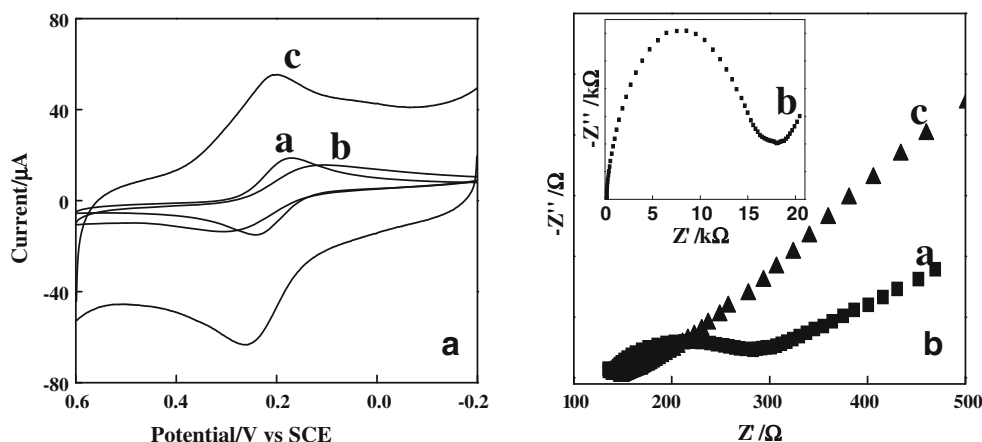
Fig. 3 **a** Cyclic voltammograms of different electrodes in 5 mM [Fe(CN)$_6$]$^{3-/4-}$ solution containing 0.1 M KCl at a scan rate of 50 mVs^{-1} and **b** Nyquist plots of 5 mM [Fe(CN)$_6$]$^{3-/4-}$ in 0.1 M KCl from 10 kHz to 0.1 Hz at an ac amplitude of 10 mV under open-circuit potential conditions, obtained for (**a**) bare GCE, (**b**) NiFe$_2$O$_4$/GCE and (**c**) NiFe$_2$O$_4$/MWCNTs/GCE

frequencies represents the electron transfer-limited process The NiFe$_2$O$_4$/MWCNTs/GCE (curve c) shows lower interfacial electron transfer resistance than that of the NiFe$_2$O$_4$/GCE (curve b) and of bare GCE (curve a). These facts suggest that the electron transfer is faster at the NiFe$_2$O$_4$/MWCNTs/GCE in comparison with that of the NiFe$_2$O$_4$/GCE and the bare GCE, which facilitates the arrival of the electrochemical probe on the surface of the electrode. This illustrates that MWCNTs not only help to moor the NiFe$_2$O$_4$ nanoparticles onto the surface of the glassy carbon electrode, but also to increase the electron transferrate.

Electrocatalytic activity of hydrazine
at NiFe$_2$O$_4$/MWCNTs/GCE

In order to investigate the electrocatalytic activity of hydrazine on the NiFe$_2$O$_4$/MWCNTs/GCE, the responses of bare GCE (curve a), MWCNTs/GCE (curve b), NiFe$_2$O$_4$/MWCNTs/GCE (curve c) were explored with 0.5 mM hydrazine in N$_2$-saturated 0.1 M PBS (pH=7.4) at 50 mVs^{-1}, shown in Figure 4. It is obvious that hydrazine couldn't be oxidized on the bare GCE (curve a). Under the
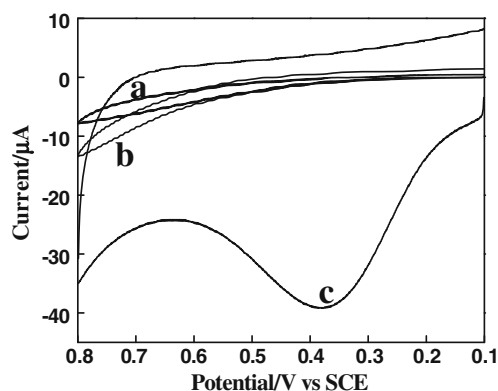
same experimental conditions, the oxidation of hydarzine on MWCNTs/GCE demostrated increase background current, but no defined anodic peaks were observed (current b) [31–33]. Nevertheless, with the addition of NiFe$_2$O$_4$, NiFe$_2$O$_4$/MWCNTs/GCE (curve c) demostrated an anodic peak at around 0.38 V. The electrochemical response of hydrazine on NiFe$_2$O$_4$/MWCNTs/GCE is irreversible, as no cathodic current is observed during the reverse sweep. According to previous reports [34], a possible electrochemical reaction for hydrazine is suggested to be N$_2$H$_4$+4OH$^-$→N$_2$+4H$_2$O+4e$^-$. As faster electron transfer leads to a well-defined peak, the substantial increasement in the peak current reflects a faster electron transfer process. Therefore, hydrazine is effectively detected by oxidation on the suggested electrode.

Amperometric response for the determination of hydrazine

Figure 5 shows a typical amperometric response of the NiFe$_2$O$_4$/MWCNTs/GCE upon the successive addition of hydrazine into continously stirred 0.1 M PBS (pH=7.4) at

Fig. 4 Cyclic voltammograms of (**a**) bare GCE, (**b**) MWCNTs/GCE and (**c**) NiFe$_2$O$_4$/MWCNTs/GCE with 0.5 mM hydrazine in N$_2$-saturated 0.1 M PBS(pH=7.4) at 50 mVs^{-1}
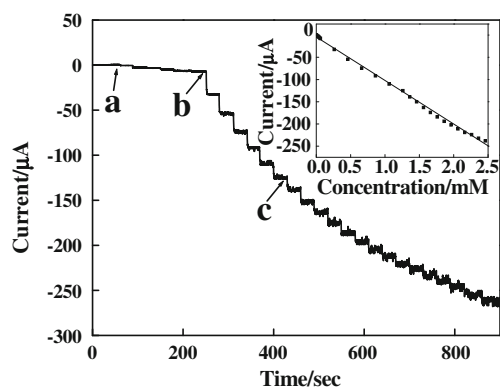

Fig. 5 Amperometric response of the NiFe$_2$O$_4$/MWCNTs/GCE with successive addition of hydrazine into 0.1 M PBS (pH=7.4); the amount of hydrazine addition: **a**→**b**, 0.01 mM steps; **b**→**c**, 0.2 mM steps; **c**→, 0.1 mM steps. The applied potential was 0.40 V. Inset: plot of I$_{pa}$ vs. concentration

Table 1 Comparison of the responses of some amperometric hydrazine sensors constructed based on different modified electrode materials

Electrode	Method	Detection limit (μM)	Linear range (μM)	Ref.
BiHCF/CC	Amperometric	3	7.0–1010.0	[34]
Ru-polymer/GC	FIA	8.5	10–10000	[35]
NiHCF/CC	CV	8.0	20–2000	[36]
Pd/BDD	LSV	2.6	24.2–85	[37]
FePc/MP/Au	CV	11	13–92	[38]
$NiFe_2O_4$/MWCNTs/GC	Amperometric	1.5	5.0–2500.0	This work

an applied potential of 0.40 V. The amperometric sensor exhibited a linear response in the concentration range from 5.0 μM to 2.5 mM, with $r^2 = 0.9982$. Based on the signal-to-noise ratio (S/N) of 3, it has a very low detection limit, which was estimated to be 1.5 μM. Besides, the detection limit was lower than BiHCF-modified CCEs [34], Ru-complex films [35], etc. (shown in Table 1). Our hydrazine electrochemical sensor exhibited a rapid response (within 3 s) and sensitive response (0.1 μA μM^{-1}) to the change of hydrazine concentration, and an obvious increasement in the oxidation current upon successive addition of hydrazine was obtained. The modified electrode achieved 96% of the steady-state current within 3 s.

Stability, reproducibility and interference

To investigate the reproducibility of the $NiFe_2O_4$/MWCNTs/GCE, ten modified electrodes made independently, showed an acceptable reproducibility with a relative standard deviation of 4.2% for the current determined at the hydrazine concentration of 250 μM. With one sensor, the mean steady-state current was −25.0 μA with a relative standard deviation of 4.1% for six determinations at a hydrazine concentration of 250 μM. When the modified electrode was not in use, it was stored in air at room temperature. The peak currents almost unchange with cyclic voltammetry at the optimum conditions for 100 cycles. Also, the peak currents of the modified electrode could remain 95% after 30 days in air at room temperature.

Table 2 Results of the recovery test

No.	Hydrazine added (μM)	Hydrazine found (μM)[a]	Recovery (%)
1	50	49±1	98
2	100	101±3	101
3	150	152±2	101
4	200	198±4	99
5	250	250±2	100

[a] Average of three determinations

Quantitative determination of hydrazine from tap water sources spinked with known quantity of hydrazine

The results demonstrated that the mentioned sensor has outstanding stability.

The effect of various substances, as the potential interference compounds, was studied on the determination of 250 μM hydrazine under the optimum conditions. Test results showed that 100-fold quantities of common cations and anions (Na^+, Ca^{2+}, Fe^{3+}, K^+, Mg^{2+}, Cl^-, NO_2^-) did not obviously interfere with the determination of hydrazine. There was no or little interference of 40-fold quantities of ascorbic acid, dopamine, L-tryptophan, glucose, guanine, adenine.

Application to real sample analysis

The validity of the mentioned method for the determination of hydrazine was evaluated. Five tap water samples were spiked with different concentrations of hydrazine and were analyzed under optimized conditions using the above technique. The results were shown in Table 2. The results were satisfactory, exhibiting that the mentioned methods could be efficiently used for the determination of hydrazine in sample analysis.

Conclusions

A sensitive hydrazine sensor has been fabricated by dipping $NiFe_2O_4$/MWCNTs nanoparticles synthesized by a facile hydrothermal method. The $NiFe_2O_4$/MWCNTs/GCE possesses better response to the electrocatalytic oxidation of hyrazine than any other electrode mentioned in the manuscript. A low detection limit, wide linear range, excellent selectivity, good stability and repeatablility make it potential candidate for the design and invention of hydrazine sensors.

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