

A non-enzymatic glucose sensor based on electrodepositing Cu/ graphene nanocomposite film modified electrode

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Abstract: A novel non-enzymatic glucose sensor was developed by electrodepositing copper film onto the graphene(GR) substrate. The morphologies and structures of the nanocomposites were characterized by field emission scanning electron microscope (FESEM) . The electrochemical performances of the Cu/GR nanocomposite film were investigated. The Cu/GR nanocomposite film showed good electrocatalytic activity towards glucose oxidation in alkaline solution. The Cu/GR nanocomposite film - based sensor displayed a linear concentration range from 8×10^{-6} to 9.4×10^{-4} mol/L with the sensitivity of $0.225 \text{ A} \cdot \text{L} \cdot \text{mol}^{-1}$ and the detection limit ($S/N=3$) of $2.5 \mu\text{mol/L}$ for the detection of glucose. With an enhanced electrocatalytic property ,high sensitivity and good stability. The as-prepared sensor is promising for the future development of nonenzymatic sensors.

Key words: graphene; copper; nanocomposite film; glucose; electrocatalysis

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1 Introduction

Graphene ,emerging as a true 2-dimensional material has received increasing interest due to its interesting properties such as high surface area ,low cost ,high conductivity ,and ultra-high Youngp's modulus^[1-5]. Graphene holds similar stable physical properties compared with carbon nanotubes ,which can be considered as an unrolled carbon nanotubes^[6]. It has shown great promise in many applications ,such as polymer composites^[7] , mechanical resonators^[8] ,liquid crystal devices^[9] and so on. Recently ,some studies have demonstrated that graphene is a good candidate of advanced materials for electrochemical applications^[10-14] ,especially for the construction of electrochemical sensors and biosensors^[11-13].

The enzyme sensors based on glucose oxidase have been widely used for the detection of glucose^[15-18]. But it is well known that the activity of enzyme can be easily affected by temperature ,pH ,humidity ,and toxic chemicals^[19]. Therefore ,several non-enzymatic electrochemical glucose sensors have been gradually explored to improve the electrocatalytic activity towards the oxidation of glucose for example ,noble metal-based (containing Pt^[20-22] and Au^[23-24]) ,and alloys -based (containing Pt ,Ru ,Pb ,and Cu^[25-30]) amperometric glucose sensors. However ,most non-enzymatic sensors suffer from low sensitivity ,high cost of rare metal precursors ,

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poor specificity to glucose and so on. It is attractive to develop graphene-based nanocomposite films as enhanced sensing platform for constructing electrochemical sensors and biosensors, because these kinds of nanocomposite films may enhance the sensitivity of the sensors.

Electrochemical deposition of copper for applications in electrocatalysis is an active research area^[30-32]. We anticipate that the Cu film, with its thin native oxide layer on the highly exposed surface, may be employed as an alternative electrode material, and Cu-graphene composite films are favorable for electrofriction applications^[33]. In this paper, we described an electrodeposition method to prepare novel nanocomposite film of Cu and graphene for glucose sensing. Modification of graphene with Cu film greatly improved the electrocatalytic properties of glucose oxidation and detection. Compared with the graphene or Cu film modified electrode, Cu/GR nanocomposite film exhibited much better electrochemical and electrocatalytic activities to glucose, which could be attributed to the synergistic effects of Cu film and graphene. The novel Cu/GR nanocomposite film could have promising potential in the electrochemical biosensing applications.

2 Experimental

2.1 Reagents

Graphite powder (spectrum pure), copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) and sodium sulfate (Na_2SO_4) were purchased from Shanghai Chemical Reagent Co. (China). D-(+)-glucose was purchased from Sigma. All other chemicals were of analytical grade and all the solutions were prepared with double-distilled water. Glucose solution (0.1 mol/L) was prepared and allowed to mutarotate at room temperature for at least 24 h before measurements.

2.2 Apparatus and measurements

JEOL 2100 transition electronic microscopy was used for transmission electron microscopy (TEM) analysis. Raman spectra were measured on Super Labram II Confocal microscopic Raman spectrometer Instrument (Dilor, France). Field Emission Scanning Electron Microscope (FESEM) image was obtained using a JSM-840 field emission SEM system (Japan).

Electrochemical experiments were performed with a CHI 660B electrochemical workstation (CH Instruments, Shanghai, China). All experiments were conducted using a three-electrode electrochemical cell. Bare GCE or its modified electrode was used as the working electrode. A platinum wire electrode and an Ag/AgCl (3 mol/L KCl) electrode were used as counter and reference electrode, respectively. Prior to the electrochemical measurements, the solutions were deoxygenated by bubbling pure nitrogen gas for at least 20 min and a nitrogen atmosphere was kept over the solution during the experiments to eliminate the interference.

2.3 Synthesis of graphene

Graphene Oxide (GO) were synthesized from graphite powder by the modified Hummers method^[34-35]. Then GO was dispersed in double-distilled water and sonicated for 30 min to form a homogenous mixture. And the resulting graphene oxide dispersion was mixed with hydrazine and ammonia. After being vigorously stirred for a few minutes, the solution was stirred for 1 h at the temperature of 95°C. The product was isolated by centrifugation, washed with double-distilled water until the pH value descended to 7.0. Finally, the graphene (GR) product was obtained after dried in oven.

2.4 Preparation of Cu/GR nanocomposite film modified electrode

Prior to modification, the GC electrode was polished with 0.3 and 0.05 μm $\alpha\text{-Al}_2\text{O}_3$ powder to obtain a mirror-like surface, sonicated in ethanol and doubly distilled water, and then dried. GR (1.0 mg) was dispersed in 1 mL Dimethyl sulphoxide (DMSO) and sonicated to form a homogenous mixture. Then 3 μL of

1.0 mg · mL⁻¹ mixtures were dropped on the surface of GCE and dried under an infrared lamp. Electrodeposition of Cu film on the surface of GR modified GC electrode was carried out by cyclic voltammetry (CV) method. It was performed by consecutive CV over a suitable potential range from -0.60 to +0.60 V at a scan rate of 100 mV · s⁻¹ in 10 mmol/L CuSO₄ containing 0.1 mol/L Na₂SO₄^[36]. After electrodeposition, the electrode was rinsed with double-distilled water and dried in air. The Cu film or GR-modified GCE electrode was prepared as comparison.

3 Results and discussion

3.1 Characterization of the graphene sheets and Cu/GR nanocomposite film

The morphologies and the structures of the prepared graphene were characterized using Raman spectra and TEM. Raman spectroscopy is a powerful nondestructive tool to distinguish ordered and disordered crystal structures of carbon. G band is usually assigned to the E_{2g} phonon of C sp² atoms, while D band is a breathing mode of κ-point phonons of A_{1g} symmetry^[37]. Fig. 1(A) shows the Raman spectra of graphene oxide (curve a) and prepared graphene (curve b). The frequencies of the G and D bands in the GR are similar to that observed in the GO. However, GR have an increased D/G intensity ratio relative to GO. This change suggests a decrease in the size of the in-plane sp² domains and a partially ordered crystal structure of GR^[3-4]. It can be observed from TEM image (Fig. 1B) that the prepared graphene sheets are rippled and resemble crumpled silk veil waves. Therefore, the data from Raman spectra and TEM clearly demonstrate that GR were successfully prepared. Furthermore, FESEM image (Fig. 2) clearly shows that Cu film have successfully assembled onto the graphene matrix to form the Cu/GR nanocomposite film by electrodepositing method.

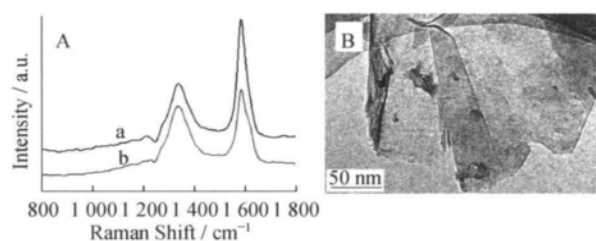


Figure 1 (A) Raman spectra of graphene oxide (a) and graphene (b) (B) TEM image of graphene

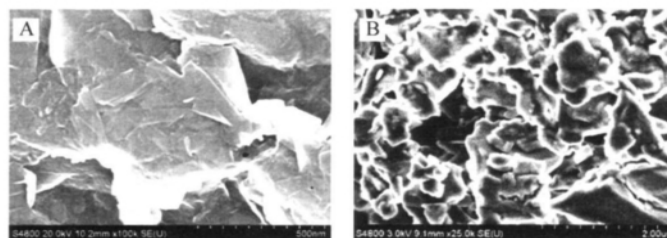


Figure 2 FESEM image of graphene (A) and Cu/GR nanocomposite film (B)

3.2 Electrochemical behaviors of Cu/GR nanocomposite film

Fig. 3 shows the Cyclic voltammograms of Cu/GR (a), Cu (b), and GR (c) modified electrodes by sweeping the potential between -1.0 and 0.4 V at a scan rate of 50 mV · s⁻¹ in 0.1 mol/L NaOH solution. As shown in Fig. 3, the GR modified electrode did not show any obvious redox peak over measured potential range (curve a). For Cu film and Cu/GR modified electrodes, well-defined peaks are observed in the positive

and negative scans, which indicated the evidence of Cu oxidation and reduction states.

However, the peak current at the Cu/GR nanocomposite film modified electrode is much larger than that at the Cu film modified electrode. Anodic peaks at -0.35 and -0.10 V are attributed to the conversion of Cu(0) to Cu(I) and that of Cu(I) to Cu(II), respectively. The cathodic peaks at -0.41 and -0.80 V are ascribed to the transition of Cu(II) to Cu(I) and that of Cu(I) to Cu(0), respectively. These peaks were in good agreement with those of the Cu electrodes previously reported in strongly alkaline solutions^[38]. According to Faraday's law $Q = nFA\Gamma$ (where n is the electron-transfer number, F is Faraday's constant and A is the geometrical surface area of electrode), the surface coverage of Cu film onto the graphene modified electrodes was estimated to be $8.05 \times 10^{-8} \text{ mol} \cdot \text{cm}^{-2}$, much larger than that obtained at Cu film modified electrode ($1.25 \times 10^{-8} \text{ mol} \cdot \text{cm}^{-2}$), it may be ascribed that graphene with large specific surface area could facilitate more Cu film depositing on it.

3.3 Electrocatalysis of glucose at the Cu/GR nanocomposite film modified electrode

Fig. 4 shows the Cyclic voltammograms of Cu/GR nanocomposite film modified electrode at $100 \text{ mV} \cdot \text{s}^{-1}$ in 0.1 mol/L NaOH at various concentrations of glucose. It can be seen that with the addition of glucose into the electrochemical cell, the oxidation peak current increased dramatically, indicating a typical electrocatalytic oxidation process of glucose. At the same time, only a single oxidation peak and no reduction peak indicate that glucose could be irreversibly oxidized at the nanocomposite film modified electrode.

Fig. 5 displays cyclic voltammograms of Cu/GR (a), Cu (b), and GR (c) modified electrodes in 0.1 mol/L NaOH in the presence of $1.0 \text{ mmol/L Glucose}$ at a scan rate of $100 \text{ mV} \cdot \text{s}^{-1}$. The GR modified electrode does not exhibit a single oxidation peak around 1.2 V , but the Cu/GR nanocomposite film and Cu film modified electrodes show the oxidation peak current for glucose at around 1.2 V . This indicates that Cu film plays an important role in oxidizing glucose. Besides, it can be obviously investigated that the electrocatalytic oxidation peak current at the Cu/GR nanocomposite film modified electrode is much higher than that of the Cu film modified electrode, indicating that the graphene sheet could be used as a good platform for incorporation of Cu film.

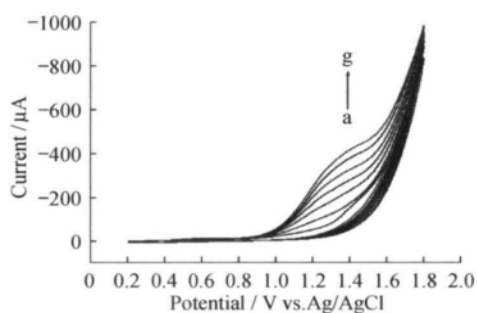


Figure 4 Cyclic voltammograms of Cu/GR nanocomposite film modified electrode at $100 \text{ mV} \cdot \text{s}^{-1}$ in 0.1 mol/L NaOH containing: (from a to g) $0, 1, 2, 3, 4, 5, 6 \text{ mmol/L}$ of Glucose

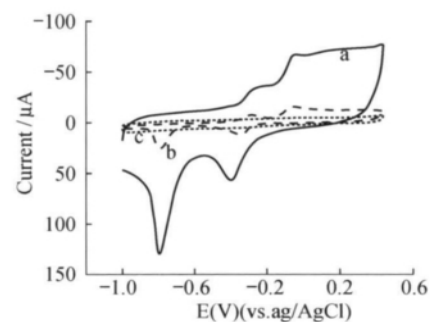


Figure 3 Cyclic voltammograms of Cu/GR (a), Cu (b) and GR (c) modified electrodes by sweeping the potential between -1.0 and 0.4 V at a scan rate of $50 \text{ mV} \cdot \text{s}^{-1}$ in 0.1 mol/L NaOH solution

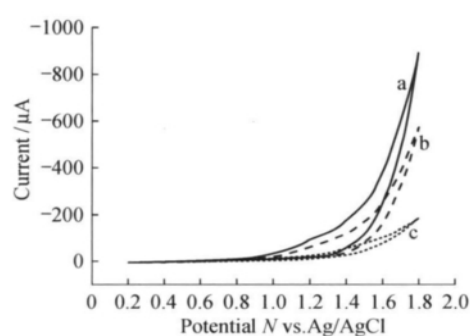


Figure 5 Cyclic voltammograms of Cu/GR (a), Cu (b) and GR (c) modified electrodes in 0.1 mol/L NaOH in the presence of $1.0 \text{ mmol/L Glucose}$ at a scan rate of $100 \text{ mV} \cdot \text{s}^{-1}$

The amperometric responses of Cu/GR nanocomposite film modified electrode to successive additions of glucose are shown in Fig. 6. A linear dependence of response current on glucose concentration is observed in the range from 8×10^{-6} to 9.4×10^{-4} mol/L with a correlation coefficient of 0.9967 (inset of Fig. 6). The detection limit ($S/N=3$) can be estimated to be $2.5 \mu\text{mol/L}$. The sensitivity of Cu/GR nanocomposite film modified electrode is $0.225 \text{ A} \cdot \text{L} \cdot \text{mol}^{-1}$, which is much higher than those reported at Cu nanocluster/multi-walled carbon nanotubes (MWCNTs) ($17.16 \text{ mA} \cdot \text{L} \cdot \text{mol}^{-1}$)^[36], oil/MWCNTs/Cu-NPs composite ($59.2 \mu\text{A} \cdot \text{L} \cdot \text{mol}^{-1}$)^[39], copper nanoparticles/zinc oxide composite ($0.16 \pm 0.02 \text{ A} \cdot \text{L} \cdot \text{mol}^{-1}$)^[40] - modified electrodes. The enhanced electrochemical performances of Cu/GR nanocomposite film could be mostly attributed to the unique 2D nanostructure and high specific surface area of GR nanosheets.

3.4 Stability reproducibility and selectivity of the Cu/GR nanocomposite film modified electrode

The stability of Cu/GR electrode was investigated. It was found that the biosensor remained about 87% of the initial response after 2 weeks. 8 successive measurements to $50 \mu\text{mol/L}$ glucose by the same electrode were carried out to investigate the repeatability of the biosensor, the relative standard deviation (RSD) was found to be 4.58%.

The oxidizable compounds such as ascorbic acid (AA) and uric acid (UA) are normally co-existed with glucose in real samples. In view of the physiological levels of glucose ($3-8 \text{ mmol/L}$) and the interfering agents (0.1 mmol/L)^[41], The effects of common interfering species on Cu/GR nanocomposite film modified electrode were examined. The amperometric response of the biosensor to the consecutive injection of 1 mmol/L glucose and interfering species including ascorbic acid (AA), uric acid (UA), dopamine (DA), and l-cysteine (molar ratio of glucose: interferences = 20:1) at an applied potential of 1.1 V . The Cu/GR nanocomposite film modified electrode exhibited high sensitivity to glucose oxidation and depressed responses towards these interferences.

4 Conclusions

We have demonstrated a simple and facile strategy to construct a novel non-enzymatic glucose sensor by electrodepositing Cu film onto the graphene modified glassy carbon electrode. The Cu/GR nanocomposite film exhibited high electrocatalytic activities toward glucose. The newly prepared non-enzymatic glucose sensor presents high sensitivity, fast response and good stability. Therefore, the new Cu/GR nanocomposite film could have promising potential in the electrochemical biosensing applications.

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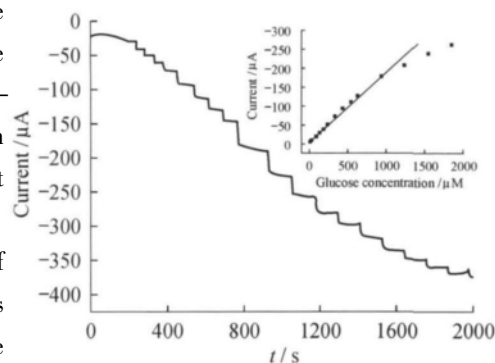


Figure 6 Amperometric response of Cu/GR nanocomposite film modified electrode at 1.1 V with successive addition of glucose in 0.1 mol/L NaOH solution. Inset: relationship of the catalytic current and the concentration of glucose

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基于铜-石墨烯纳米复合膜修饰电极的无酶葡萄糖传感器

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摘要: 采用电沉积的方法在石墨烯表面修饰一层铜膜,对铜/石墨烯纳米复合膜进行了表征. 研究显示铜/石墨烯纳米复合膜修饰电极对葡萄糖有较好的电催化活性,并且在 $8 \times 10^{-6} \sim 9.4 \times 10^{-4}$ mol/L 范围内呈线性关系,检测限为 $2.5 \mu\text{mol/L}$ ($S/N=3$),灵敏度为 $0.225 \text{ A} \cdot \text{L} \cdot \text{mol}^{-1}$. 实验表明该修饰电极对葡萄糖有较好的选择性.

关键词: 石墨烯; 铜; 纳米复合膜; 葡萄糖; 电催化

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