

A hydrothermally prepared reduced graphene oxide-supported copper ferrite hybrid for glucose sensing

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Received 19 June 2015; received in revised form 20 June 2015; accepted 20 June 2015

Available online 30 June 2015

Abstract

A stable enzyme-free glucose sensor was fabricated via a facile in situ hydrothermal route by the formation of CuFe_2O_4 nanoparticles into the graphene oxide sheets. The contents of graphene oxide in composites were varied from 10 to 30 wt%. The morphology of formed $\text{CuFe}_2\text{O}_4/\text{rGO}$ nanocomposite was found by TEM analysis. The electrocatalytic activity of different $\text{CuFe}_2\text{O}_4/\text{rGO}$ samples towards glucose oxidation was studied by employing cyclic voltammetry and chronoamperometry techniques. $\text{CuFe}_2\text{O}_4/\text{rGO}(30 \text{ wt}\%)$ sample showed the highest oxidation current. An excellent sensitivity value of $1824.22 \mu\text{A mM}^{-1} \text{cm}^{-2}$ with a detection limit of $1.0 \mu\text{M}$ was gained by $\text{CuFe}_2\text{O}_4/\text{rGO}(30 \text{ wt}\%)$. The sensor also exhibits good reproducibility and long term stability as well as high selectivity with insignificant interference from ascorbic acid and uric acid.

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Keywords: Magnetic materials; Nanostructures; Copper ferrite; Graphene oxide

1. Introduction

Despite of great advantages of enzymatic glucose sensors (high selectivity, good sensitivity, fast and reversible) which are based on the immobilization of glucose oxidase (GOx), the lack of stability remains the main problem in the application of enzymatic sensors [1,2]. Since a sensor must be stable in high temperature and aggressive environments, development of glucose sensors without using enzymes has been the subject of many studies [3–5]. Sensing applications based on magnetic nanoparticles can play a significant role in modifying an electrode surface by increasing its surface area, high catalytic efficiency, enhancing the mass transport and strong adsorption ability [6–8]. In spite of having unique abilities, the agglomeration of the magnetic nanoparticles is a big problem since they tend to reduce the energy associated with the high surface area to volume ratio of the nano-size particles. Protecting the

magnetic nanoparticles by various types of coatings is a solution to overcome this problem [9]. In addition, coating provides a new platform to enhance the properties of the magnetic nanoparticles. This paper is devoted to elaborate and characterize a non-enzymatic glucose sensor using the magnetic nanoparticles which are coated with reduced graphene oxide protective layer; the composite possesses both the electrical conductive and superparamagnetic properties due to excellent electronic, mechanical and thermal properties of graphene and superior magnetic properties of CuFe_2O_4 nanomagnetic particles.

2. Materials and methods

2.1. Chemicals and reagents

$\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, ethanol ($\text{C}_2\text{H}_5\text{OH}$, 99.7% purity), D-(+)-glucose, uric acid (UA), ascorbic acid (AA) sucrose and fructose were obtained from Aldrich. All other reagents were of analytical grade and were used as

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received. Deionized water was used for all experiments which were carried out at room temperature.

2.2. Synthesis of graphene oxide (GO)

Graphene oxide (GO) was synthesized from graphite flakes (purchased from Sigma Aldrich) according to the modified Hummer method [10,11]. In brief, 5 g of graphite flakes and 2.5 g of NaNO_3 were mixed together followed by the addition of 108 ml concentrated H_2SO_4 and 12 ml H_3PO_4 . After 10 min stirring in an ice bath, 15 g of KMnO_4 were slowly added to keep the temperature of the mixture below 5°C to prevent overheating and explosion. The mixture was stirred at 35°C for 12 h and the resulting solution was diluted by adding 400 ml of water under vigorous stirring. To ensure the completion of reaction with KMnO_4 , 15 ml of H_2O_2 was added to the mixture. The reaction product was centrifuged and washed with deionized water and 5% HCl solution repeatedly. Finally, the product was dried at 60°C .

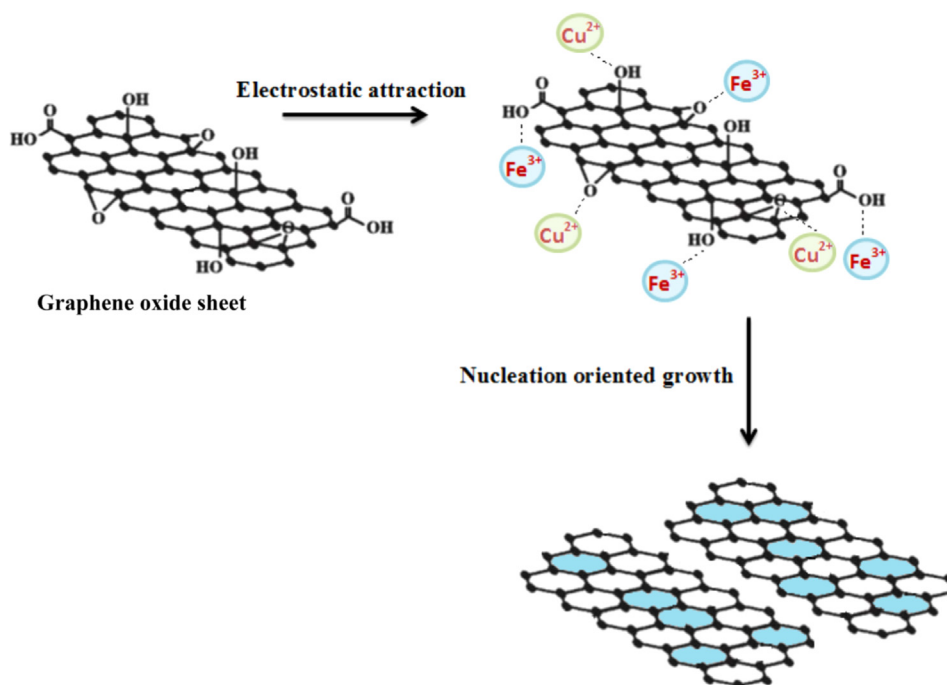
2.3. Synthesis of magnetic $\text{CuFe}_2\text{O}_4/\text{rGO}$ composite

$\text{CuFe}_2\text{O}_4/\text{rGO}$ with differing graphene content (10, 20, 30 wt%) were synthesized via hydrothermal method. A typical experiment procedure for the synthesis of $\text{CuFe}_2\text{O}_4/\text{rGO}$ with 30 wt% graphene content is as follows: 192 mg of GO was dispersed into 144 ml of ethanol with sonication for 1 h. Then, 0.5798 g of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ and 1.9392 g of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were added to 36 ml of ethanol with stirring for 30 min at room temperature. The above two solutions were then mixed together and stirred for 30 min. After that, the mixture was adjusted to a pH of 10.0 with 6 M NaOH solution and stirred for 30 min, yielding a stable dark-green homogeneous emulsion. The resulting mixture was transferred into

a 200 mL Teflon-lined stainless steel autoclave and heated to 180°C for 24 h under autogenously pressure. The reaction mixture was allowed to cool to room temperature, and the precipitate was filtered, washed with distilled water five times, and dried in a vacuum oven at 60°C for 12 h. The product was labeled as $\text{CuFe}_2\text{O}_4/\text{rGO}$ (30 wt%). For comparison, the same method was used to synthesize pure CuFe_2O_4 without adding rGO. The synthesis process of the nanocomposite is shown in Sch. 1.

2.4. Characterization and instrumentations

Scanning electron microscopy (SEM) images and transmission electron microscopy (TEM) images were captured using Hitachi SU 8000 model instrument. X-ray diffraction patterns (XRD) measurements were recorded using Cu $K\alpha$ radiation to analyze the nanoparticles structures. Fourier transform infrared spectra (FTIR) of the samples were recorded on a Perkin-Elmer RX1FT-IR spectrometer with a wavenumber resolution of 2 cm^{-1} as potassium bromide (KBr) pellets at a weight ratio. The electrochemical glucose detection performance was investigated by cyclic voltammetry (CV) at scan rate of 10 mV s^{-1} , chronoamperometry ($I-t$) and electrochemical impedance spectroscopy (EIS) techniques using AUTOLAB model PGSTAT in a conventional three-electrode cell where glassy carbon electrode (GCE, 3 mm diameter), Pt foil and saturated calomel electrode (SCE) in 0.1 M phosphate buffer solution at pH 7.4 at room temperature. The surface of bare glassy carbon electrode (3 mm diameter) was polished with 1.0 and $0.3\ \mu\text{m}$ alumina slurry on polishing cloth to eliminate any traces which effect on the rate of electron transfer. The electrode should be held in a vertical position while making Fig. 8 motions on the polishing pad. The electrode surface was rinsed with distilled water to remove all traces of the polishing



Scheme. 1. The synthesis process of the $\text{CuFe}_2\text{O}_4/\text{rGO}$ nanocomposite.

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