



Glassy carbon electrode modified with a graphene oxide/poly(*o*-phenylenediamine) composite for the chemical detection of hydrogen peroxide



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ABSTRACT

Conducting poly(*o*-phenylenediamine) (POPD)/graphene oxide (GO) composites were prepared using a facile and efficient method involving the in-situ polymerization of OPD in the presence of GO in an aqueous medium. Copper sulfate was used as an oxidative initiator for the polymerization of OPD. Scanning electron microscopy and transmission electron microscopy images showed that POPD microfibrils were formed and distributed relatively uniformly with GO sheets in the obtained composites. X-ray diffraction results revealed the highly crystal structure of POPD. This composite exhibited good catalytic activity and stability. These results highlight the potential applications of POPD/GO composites as excellent electrochemical sensors. The composites were used to modify glass carbon electrodes for the chemical detection of hydrogen peroxide in aqueous media.

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1. Introduction

Graphene has high thermal conductivity [1], electrical conductivity [2, 3], specific surface area [4], and room temperature carrier mobility [5]. Graphene has attracted considerable interest for a variety of applications, such as electrochemical double layer capacitors [6], solar cells [7], fuel cells [8], rechargeable lithium ion batteries [9], and electrochromic devices [10]. However, graphene itself is insoluble in organic solvents and aggregates readily. The material is also intractable and cannot be shaped into the desired structures of composites using common processing techniques [11]. Therefore, the modification/functionalization of graphene with polymers is essential to improve the dispersibility/processability and prevent aggregation. Polymer/graphene composites have been studied extensively because the incorporation of graphene with polymers can lead to new composite materials possessing the properties of each component with a synergistic effect that would be useful in particular applications. Polymer graphene has applications in the preparation of inks [12], solar cell electrodes [13–15], and supercapacitors [7,16,17]. Hydrogen peroxide (H₂O₂) is a product of several biological and enzymatic reactions, such as oxidases and peroxidases. The compound is also an essential mediator in food, pharmaceuticals, and in clinical, industrial, and environmental processes [18]. Therefore, it is essential to detect H₂O₂. The key point in developing electrodes for detecting H₂O₂ is to decrease the oxidation/reduction

overpotentials. Many efforts have paid for detecting H₂O₂ [19–26]. In our previous work, a facile, efficient, and green route was developed to prepare GO/PANI-based sensor materials for the detection of H₂O₂ in aqueous media [27].

Owing to its conductance, electrochromic and photoelectronic properties, and permselectivity [28–32], poly(*o*-phenylenediamine) (POPD) has been used in electrocatalysis [33], electrochromic displays [34], sensors and biosensors [35–37], corrosion prevention [38], photoelectrochemistry [39] and molecularly imprinted polymers [40]. Numerous composites based on POPD have been reported [41]. The Au/POPD electrode has been chosen as a model system for studies of low-frequency capacitance of conducting polymer electrodes [42,43]. A POPD/single-wall carbon nanotube-modified electrode was prepared by electropolymerization on a glassy carbon electrode [44,45]. Gold nanoparticles supported on both the inner and outer surfaces of POPD hollow microspheres could be synthesized using POPD hollow microspheres as both a reductant and template/stabilizer. This composite showed high catalytic activity for the aerobic oxidation of alcohols in water and in air at room temperature [46]. A composite of POPD submicrosphere-supported gold nanoparticles was prepared and applied to the selective oxidation of benzyl alcohol [47]. More recently, S. Mu [48] developed an electrode consisting of POPD and reduced graphene oxide on glassy carbon. The electrocatalytic oxidative polymerization of *o*-phenylenediamine (OPD) was performed on a reduced graphene oxide (RGO)/glassy carbon (GC) electrode.

Up to now, there are no reports on preparing GO/POPD composites for electrochemical sensors. In the present study, conducting composites

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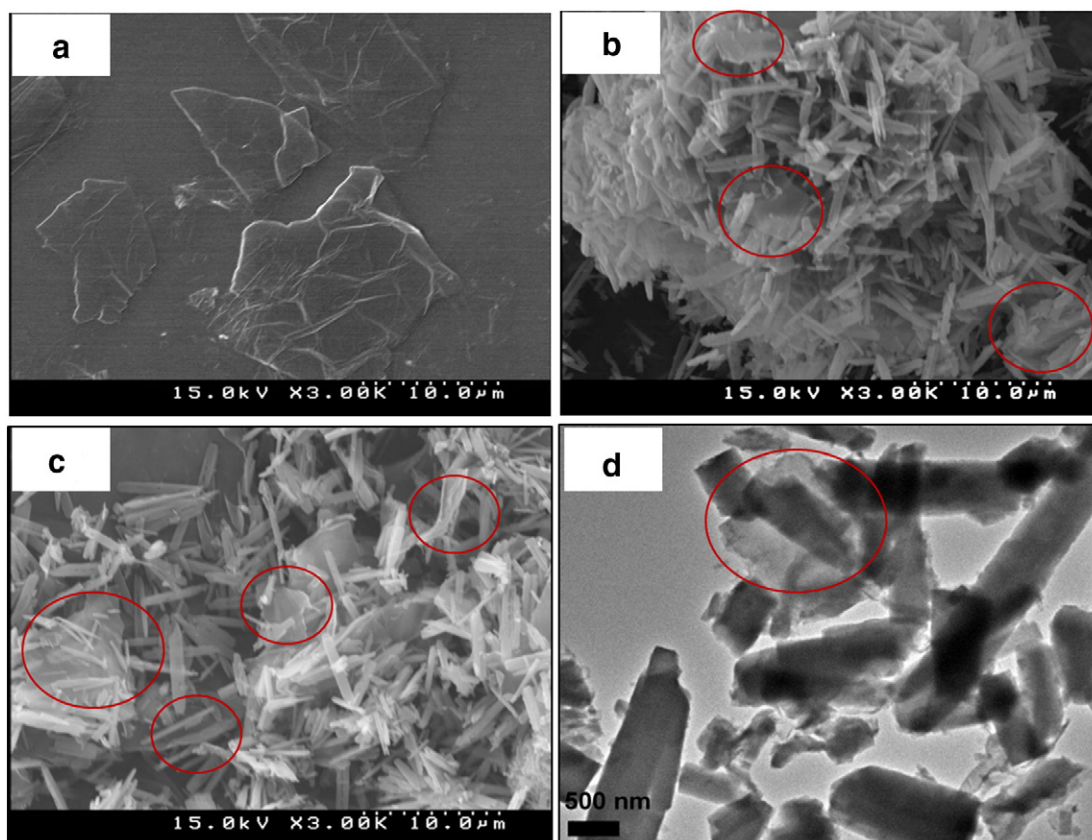


Fig. 1. SEM images of (a) bare GO and GO/POPD composite with (b) 10 wt.% and (c) 30 wt.% of GO and (d) TEM image of GO/POPD composite with 30 wt.% of GO.

of GO and POPD were prepared using a simple and efficient method. CuSO_4 was used as the oxidized initiator for the polymerization of the OPD monomer in the presence of GO sheets. The chemical structure, morphology, and electrochemical properties of the obtained composites were characterized by Fourier transform infrared (FT-IR) spectroscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD), thermogravimetric analysis (TGA), X-ray photoelectron spectroscopy (XPS), conductivity measurements, and cyclic voltammetry (CV). The composites were also used to modify glassy carbon electrodes for the chemical detection of H_2O_2 in aqueous media.

2. Experimental

2.1. Materials

OPD (Aldrich, 99.5%) and graphite powder (Alfa Aesar, 99.995%) was used as received. CuSO_4 and other reagents were of analytical grade and used without further purification. The water used in the experiments was deionized and produced in the laboratory.

2.2. Synthesis of GO

GO was synthesized from graphite powder using a modification of the Hummers method [27]. Typically, 2 g graphite powder was added to 50 ml concentrated H_2SO_4 along with 2 g NaNO_3 in a flask at 0°C in an ice-bath. Subsequently, 6 g KMnO_4 was added slowly to the solution with vigorous stirring at temperatures below 20°C . The ice-bath was then removed and the mixture was stirred at 35°C for 30 min until it became pasty brownish gray in color. The paste was diluted with 100 ml deionized water and stirred for 2 h. A 10 ml H_2O_2 (30 wt.%)

solution was added slowly to the suspension along with a 100 ml HCl (10 v/v %) solution. The mixture was centrifuged and washed with deionized water until the decant solution was neutral. The product was dried at room temperature in a vacuum oven for 24 h.

2.3. Synthesis of the GO/POPD composites

In a typical experiment, 10 mg GO was dispersed in 10 ml DI water by sonication for 30 min. 50 mg of OPD was added to the above dispersion and dissolved with magnetic stirring for 30 min. Subsequently, 2 ml of a CuSO_4 aqueous solution (0.5 M) was added rapidly to the above solution with vigorous stirring. A rapid color change was observed upon the addition of CuSO_4 . After stirring for 30 min, the product was collected from the reaction solution by centrifugation, washed five times with DI water and ethanol, and dried in a vacuum at 40°C for 24 h.

2.4. Characterization

The samples were characterized by SEM (Hitachi, S-4200), TEM (Philips, CM-200) at an acceleration voltage of 200 kV, XRD (PANalytical, X'Pert-PRO MPD) using $\text{Cu K}\alpha$ radiation, and XPS (ULVAC-PHI, Quantera SXM) using an Al X-ray source. The FT-IR (Excalibur Series FTS 3000, BioRad) spectra were recorded over the range, $400\text{--}4000\text{ cm}^{-1}$ at a resolution of 16 cm^{-1} within 32 scans using KBr pellets. The ultraviolet–visible (UV–vis) spectra were recorded over $200\text{--}800\text{ nm}$ using a UV–vis–near infrared (NIR) spectrophotometer (Cary 5000, Varian). TGA was performed on a simultaneous TGA/DSC analyzer (SDT Q600, TA Instrument) from $25\text{ to }600^\circ\text{C}$ at a heating rate of $10^\circ\text{C}/\text{min}$ under a nitrogen atmosphere.

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