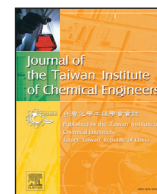




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# Synthesis and characterization of nanostructure molecularly imprinted polyaniline/graphene oxide composite as highly selective electrochemical sensor for detection of *p*-nitrophenol

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## ABSTRACT

An electrochemical sensor for the determination of *p*-nitrophenol (*p*-NP) was developed based on a molecularly imprinted polymer (MIP) on graphene oxide (GO). The sensor was prepared via precipitation polymerization of aniline and ammonium persulfate as the host molecule and initiator. The morphologies and electrochemical behavior of the imprinted sensor were characterized by Fourier transform infrared spectroscopy (FTIR), thermal gravimetric analysis (TGA), X-ray diffraction (XRD), field emission electron microscopy (FE-SEM), energy dispersive X-ray spectroscopy (EDS) and cyclic voltammetry techniques. The electrochemical investigations were conducted by screening the effects of pH, *p*-NP concentration and scan rate. Under the optimized experimental conditions, the oxidation peak current varies linearly with *p*-NP concentration, in the range  $6 \times 10^{-5}$ – $14 \times 10^{-5}$  mol/L with a detection limits of  $2 \times 10^{-5}$  mol/L (correlation coefficient of 0.991). The electrochemical sensor was also used to detect *p*-NP in tap water, where it shows the excellent recoveries. Moreover, the sensor probe revealed an excellent selectivity for *p*-NP compared to other phenol derivatives. This proposed sensor was used successfully for determination of spiked *p*-nitrophenol in water samples.

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## 1. Introduction

Increasing environmental pollution has led to quest for novel methods that can be utilized to detect the desired contaminants in the inestimable environment. Among the wide array of organic pollutants, the monitoring and determination of nitrophenols has attracted significant interest due to their toxic effect on humans, animals and plants [1]. Such pollutants will be released into the environment *i.e.* soils, lakes, rivers and ground water, during their production and application [2]. Nitrophenol derivatives are formed by photochemical reactions in the presence of nitrogen oxides [3]. *p*-nitrophenol (*p*-NP) is one of the member of this family which is cited in the list of priority pollutants by the U.S. Environmental Protection Agency (EPA) [4]. It is widely used as intermediates in the production of pharmaceutical products, dyestuffs, fungicide, insecticide, pesticides, and acid–base indicator [5–8]. *p*-NP is easily adsorbed through the skin and mucous membranes [9], and is reported as strongly potential mutagen [10], carcinogen [11,12], cyto

embryotoxic to mammals [13]. Moreover, *p*-NP is not easily degradable because of its stable structure. For these reasons, its application should be seriously controlled and it is important to extend effective and reliable methods for low level analysis of *p*-NP in environmental samples.

Conventional detection methods for *p*-NP including chromatographic techniques [14], capillary electrophoresis [15], fluorescence [16] and spectrophotometric methods [17,18] suffer generally from their need to complicated instrumentations, assay processes or long analysis time. In such circumstances, the development of simple, rapid and inexpensive methods for analysis of hazardous materials is desirable.

Electrochemical assays have received significant attentions in the detection of *p*-NP owing to their various advantages involving simple operation and instrumentation, *in situ* detection, and fast analysis. To this end a variety of electrodes such as modified glassy carbon electrodes using lithium tetracyanoethylene [19], sodium montmorillonite-anthraquinone [20], Cu<sub>2</sub>O nanoparticles [21], graphene oxide (GO) [2] and poly(methylene blue) [22], or carbon paste electrodes modified by apatite [23], crown ether/silver nanoparticles [24] have been developed for the determination of *p*-NP. However, the basic drawback of the modified

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electrodes is moderate selectivity, which leads to the interference of other phenolic compounds for the determination of the target molecule.

Imprinted polymers (MIPs) are known as promising candidates in the field of chemical sensors with well-defined recognition sites designed for a specific group of target species. Beside the high selectivity, MIPs usually show a great affinity, stability and durability against harsh chemical environments [25]. Surprisingly, there are few reports regarding the preparation of voltammetric sensors for the selective recognition and electrochemical detection of *p*-NP using molecularly imprinting technology. In this sense, molecularly imprinted poly(methacrylic acid-co-ethylene glycol dimethacrylate) [26], imprinted electrochemical sensor based on ZnO nanoparticles/carbon nanotubes doped chitosan film [27], macroporous imprinted polymer containing gold nanoparticles [28], and molecularly imprinted polyaniline-polyvinyl sulphonic acid composite [29] have been introduced.

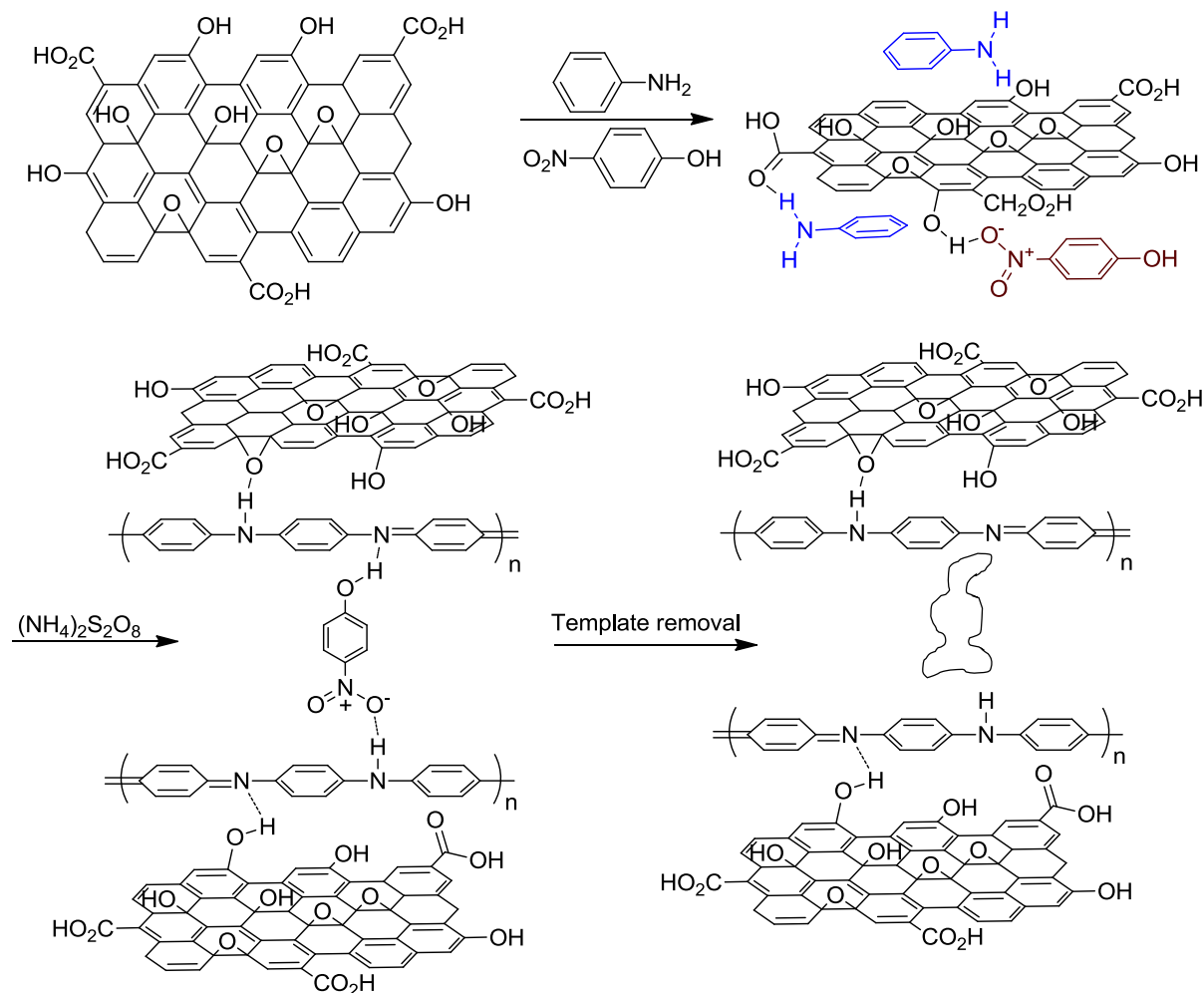
The application of conductive polymers modified using carbon materials is very attractive approach to improve the sensitivity and selectivity of imprinted electrochemical sensors [30]. Graphene oxide (GO), a single sheet of graphite oxide, has attracted great attention in material chemistry [31]. The adjustable functional groups (*i.e.* hydroxyl, carboxyl, and epoxide groups), high surface area, excellent mechanical properties, and good compatibility with polymers have made GO as promising material to synthesize nanocomposites [32].

This report intends to describe the preparation of a new MIP modified electrode based on conducting composite of polyaniline (PANI) and GO for the determination of *p*-NP. Aniline (ANI) monomer, bearing an electron rich aromatic ring, polymerized via precipitation polymerization on GO sheets in the presence of template molecule. The template molecule, *p*-NP, has electron-poor aromatic ring with hydrogen-bond donating and accepting groups. The properties of prepared sensor, such as special binding, response time, and selective recognition ability were investigated using cyclic voltammetry (CV). The proposed method was further applied to determine *p*-NP in water samples.

## 2. Experimental

### 2.1. Materials

Graphite powder, aniline, ammonium persulfate, sulfuric acid, potassium permanganate, hydrogen peroxide solution 30%, ethanol and *p*-NP were purchased from Merck (Darmstadt, Germany). Sodium dodecyl sulfate and sodium nitrate were obtained from Fluka (Hannover, Germany). All chemicals were of the highest purity available and used without any further purification. A standard stock solution (10 mmol/L) of *p*-NP was prepared daily by its dissolving in water and the working solutions were prepared by diluting the stock solution with an acetic acid/sodium acetate buffer solution (0.2 mol/L, pH 5). The buffer solution was used as the supporting electrolyte for the electrochemical experiments. All



**Scheme 1.** Illustration of the *p*-NP-MIP-PANI/GO preparation procedure.

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