



# Simultaneous electrochemical determination of nitrophenol isomers with the polyfurfural film modified glassy carbon electrode



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## ABSTRACT

A simple and highly selective electrochemical sensor based on a polyfurfural film modified glassy carbon electrode was developed for simultaneous detection of nitrophenol isomers. The modified electrode exhibited remarkable electrocatalytic activity towards the reduction of nitro. The redox peaks for each nitrophenol isomer can be well resolved and their simultaneous determination was achieved. Under optimal conditions, the proposed sensor had wider linear ranges of 5–100  $\mu\text{mol L}^{-1}$ , 0.75–100  $\mu\text{mol L}^{-1}$  and 0.75–100  $\mu\text{mol L}^{-1}$  with lower detection limits of 0.3, 0.05 and 0.04  $\mu\text{mol L}^{-1}$  for o-nitrophenol, m-nitrophenol and p-nitrophenol, respectively. The modified electrode also showed promising selectivity, stability and reproducibility. Moreover, the proposed sensor can be applied to simultaneous determination of nitrophenol isomers in real water samples, giving satisfactory recoveries.

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## 1. Introduction

As reported by the United States Environmental Protection Agency (USEPA), the isomers of nitrophenol such as o-nitrophenol (o-NP), m-nitrophenol (m-NP) and p-nitrophenol (p-NP) are serious environmental pollutants due to their potential toxicity to human beings, animals and plants even at much low concentration [1–3]. For example, these compounds would cause severe diseases e.g., methemoglobinemia, fever, headache, drowsiness, nausea to human and animals [4–7]; and they are also hazardous to plants, for instance, 0.7  $\text{mmol L}^{-1}$  of nitrophenol in irrigating water will result in the reduction of output [8]. Moreover, nitrophenol isomers are widely applied in the chemical synthesis of dyes, pesticides, and explosive materials [8–10], and they are not only detected in soil, but also in aquatic environment due to their high stability and solubility in water [10–13]. These isomers, however, always coexist and interfere with each other due to similar structures and properties. Therefore, it has been a great challenge to separate and simultaneously detect the nitrophenol isomers, in particular, in environmental analysis.

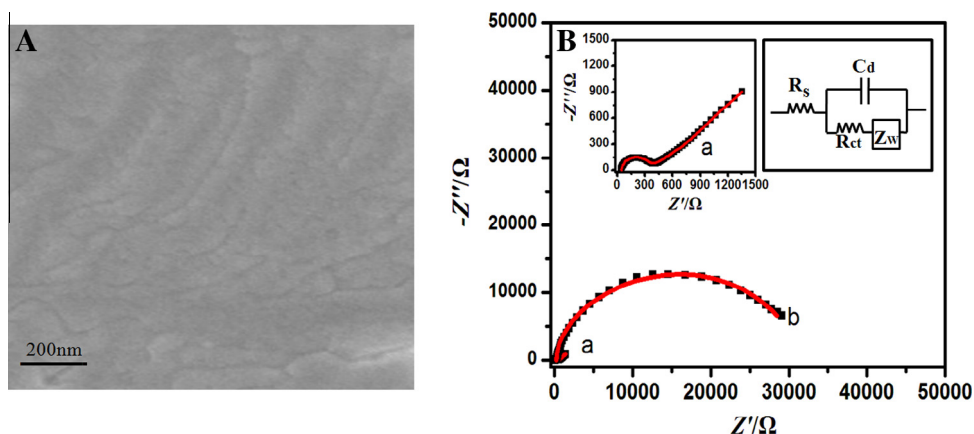
Various techniques have been utilized to determine them simultaneously, including flow injection analysis [14,15], spectrophotometry [16,17], high performance liquid chromatography [18,19], and capillary electrophoresis [20,21]. However, these methods have disadvantages of complicated operation and high

cost. Recently, electrochemical method has attracted much attention in this field due to its high-efficiency, sensitivity, low-cost, and ease of miniaturization [22,23]. In particular, nanomaterial-modified electrodes make simultaneous determination of nitrophenol isomers possible due to their unique advantages for electrochemical catalysis [24,25]. The reported examples include single wall nanotubes compound poly(4-aminopyridine) modified glassy carbon electrode (GCE) (SWNTs/POAPE/GCE) [26], multi-walled carbon nanotubes modified GCE (MWNTs/GCE) [27], nano-gold modified GCE (nano-Au/GCE) [1],  $\beta$ -cyclodextrin (CD) functionalized mesoporous silica modified carbon paste electrode (CD-SBA/CPE) [4], CD functionalized reduced graphene oxide modified GCE (CD-RCO/GCE) [11], and ordered mesoporous carbons modified GCE (OMCs/GCE) [8]. However, these electrodes are difficult to be prepared and lack of sensitivity.

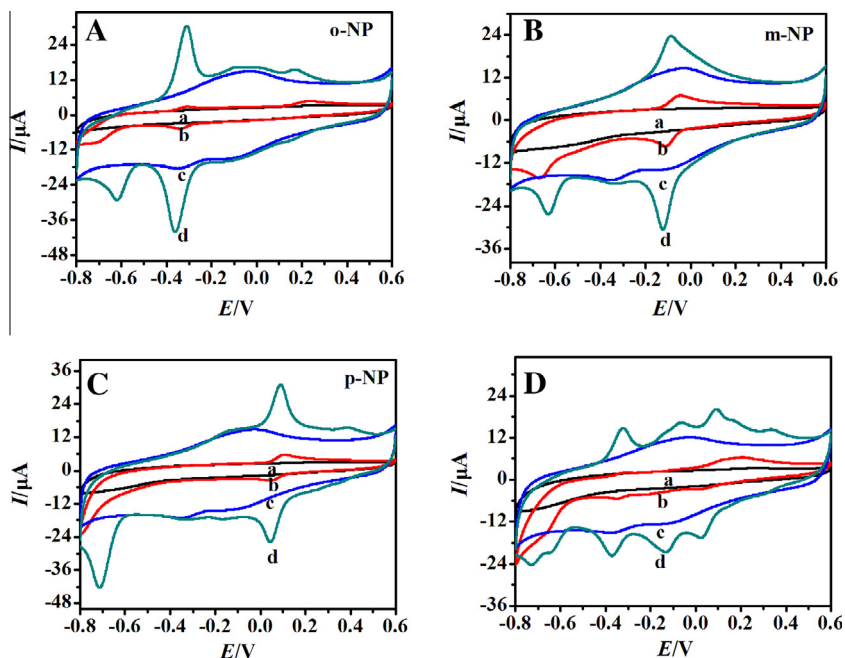
Comparing to nanomaterial-modified electrodes, polymer-modified electrodes have attracted enormous interest in the past few years, due to their good stability, containing multiple active sites, homogeneity in electrochemical deposition and strong adherence to electrode surface [28]. Electropolymerization provides advantages to immobilize polymers (e.g., poly(o-phenylenediamine) film [30], thiadiazole film [31], polytyramine film [32]) on an electrode surface as film thickness, permeation and charge transport characteristic can be easily controlled by adjusting the electrochemical parameters [29]. As reported previously, polyfurfural film can be formed on different electrodes such as Pt electrode [33,34], low carbon steel [35] and glassy carbon electrode [36], showing good adherence and electrical

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**Fig. 1.** (A) SEM image of the polyfurfural film. (B) Nyquist plots of 5 mmol L<sup>-1</sup> [Fe(CN)<sub>6</sub>]<sup>3-/4-</sup> containing 0.1 mol L<sup>-1</sup> KCl at (a) bare GCE and (b) the polyfurfural film/GCE (dotted line: recorded data and solid line: fitting curve). The frequency range is from 0.1 Hz to 100 kHz. Insets: (left) the enlarged curve of a and (right) equivalent circuit for fitting.



**Fig. 2.** CVs of (A) o-NP ( $2.0 \times 10^{-4}$  mol L<sup>-1</sup>), (B) m-NP ( $1.0 \times 10^{-4}$  mol L<sup>-1</sup>), (C) p-NP ( $2.0 \times 10^{-4}$  mol L<sup>-1</sup>) and (D) the mixture of three isomers at (b) bare GCE and (d) the polyfurfural film/GCE. CVs of 0.1 M PBS (pH 7.0) at (a) bare GCE and (c) the polyfurfural film/GCE. Scan rate was 100 mV s<sup>-1</sup>.

conductivity. In particular, Fu et al. [36] has proved that the polyfurfural film modified glassy carbon electrode exhibited a good electrocatalytic activity to the oxidation of hydroxy. We further discover that the polyfurfural film modified glassy carbon electrode also exhibits excellent electrocatalytic activity towards the reduction of nitro. Moreover, the polyfurfural film modified electrode can be used to analyze nitro contaminants, providing a guidance of the simultaneous determination of nitrophenol isomers. It would have a tremendous meaning to environmental analysis.

In this paper, the polyfurfural film modified GCE was employed to investigate the electrochemical behavior of nitrophenol isomers. The polyfurfural film showed excellent electrocatalytic activity to nitrophenol isomers. The three nitrophenol isomers were separated successfully and their simultaneous determination was realized. The proposed sensor exhibited highly sensitivity, well-defined specificity, stability and reproducibility.

## 2. Experimental

### 2.1. Chemicals and reagents

O-nitrophenol, m-nitrophenol and p-nitrophenol (analytical grade) were purchased from J&K Chemical (Beijing, China). Furfural (GC, 98%) was obtained from TCI Company. Sodium perchlorate was obtained from Fuchen Chemical Reagent Company (Tianjin, China). All other reagents were of analytical grade and used without further purification. Phosphate buffer solution (PBS, 0.1 mol L<sup>-1</sup>) was prepared by mixing the solution of K<sub>2</sub>HPO<sub>4</sub> and KH<sub>2</sub>PO<sub>4</sub>, and then adjusting the pH with NaOH or H<sub>3</sub>PO<sub>4</sub>. Stock solutions of o-NP, m-NP and p-NP were all prepared to 0.01 mol L<sup>-1</sup> in methanol and diluted to different concentrations before use by mixing with PBS (pH 7.0). The stock solutions should be stored in a dark place to avoid direct sunlight. All aqueous solutions were prepared with doubly distilled water.

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