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ORIGINAL ARTICLE

Electrocatalytic effect of NiO supported onto activated carbon in oxidizing phenol at graphite electrode: Application in tap water and olive oil samples

H. Hammani^{a,b}, W. Boumya^a, F. Laghrib^a, A. Farahi^c, S. Lahrich^a,
 A. Aboukas^b, M.A. El Mhammedi^{a,*}

^a Univ. Hassan I, Laboratory of Chemistry and Mathematical Modeling (LCMM), Polydisciplinary Faculty, BP 145, 25 000 Khouribga, Morocco

^b Univ. Sultan Moulay Slimane, Interdisciplinary Laboratory of Research in Science and Technology, Polydisciplinary Faculty, BP 592, 23000 Beni Mellal, Morocco

^c Univ. Ibn Zohr, Team of Photocatalysis and Environment, Faculty de Sciences, BP 8106 City Dakhla, Agadir, Morocco

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Abstract The activated carbon doped by nickel oxide (NiO/AC) was prepared using solid reaction. The obtained powder was characterized using X-ray diffraction (XRD) and infrared spectroscopy (IR). The NiO/AC was also used to catalyze the electro-oxidation of phenol at carbon paste electrode (CPE) using electrochemical approaches. Kinetic parameters of phenol oxidation (as the electron transfer coefficient, diffusion coefficient and heterogeneous rate constant) were investigated. The results confirm that phenol oxidation is catalyzed by nickel oxide, due to resonance, steric and aggregation effects.

The experimental conditions were optimized by varying chemical and electrochemical parameters, which involve the oxidation of phenol. The electrode NiO/AC–CPE exhibits a good linear range from 1.0×10^{-6} mol L⁻¹ to 8.0×10^{-5} mol L⁻¹ and 1.0×10^{-4} mol L⁻¹ to 1.0×10^{-3} mol L⁻¹ with a limit of detection of 7.09×10^{-7} mol L⁻¹ (signal (S) to noise (N) ratio, S/N = 3). This modified electrode NiO/AC–CPE offers a considerable improvement in voltammetric sensitivity toward phenol determination, compared to the unmodified electrode (CPE). The selectivity of the NiO/AC–CPE in detecting phenol was investigated for a number of ions (Cu²⁺, Cd²⁺, Ag⁺, Pb²⁺, Zn²⁺, Fe³⁺, Ni²⁺, Al³⁺, Mg²⁺, Ag²⁺ and Cr³⁺) and biomolecules (4-nitrophenol, dopamine, 2-nitrophenol, paracetamol, hydroquinone, catechol, resorcinol, 4-aminophenol and 3-aminophenol). The proposed method was successfully applied to phenol determination in tap water and olive oil.

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* Corresponding author.

E-mail address: elmhammedi@yahoo.fr (M.A. El Mhammedi).

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1. Introduction

Phenols are categorized as one of the most serious environmental contaminants, which frequently are discharged by industrial plants such as oil refineries, plastic plants, dyes, pesticides, pharmaceuticals and coal conversion. They are listed among 129 prior treatment pollutants by the United States Environmental Protection Agency (EPA) (Keith and Telliand, 1979; Zhang et al., 2011) due to their toxicity, recalcitrance, bioaccumulation, persistence in the environment and difficulty to degrade.

Various methods have been developed to control phenol in different matrices such as spectrophotometry (Zain et al., 2014; Chunli et al., 2000; Roig et al., 2003), high performance liquid chromatography (Zhou et al., 2014; Peñalver et al., 2002) and thin layer chromatography (Hofmann et al., 2008). However, the electrochemical methods have been proven as inexpensive and simple analytical methods with remarkable detection sensitivity, reproducibility and ease of miniaturization rather than other instrumental analysis methods (Karim and Lee, 2013; Mojović et al., 2011; Gao and Vecitis, 2013; Janegitz et al., 2012; Zhang et al., 2000; El-Kosasy et al., 2003).

Carbon paste electrodes (CPEs) belong to promising electrochemical or bioelectrochemical sensors of wide applicability (Dönmez et al., 2014; Tajik et al., 2013; 2014; Serpi et al., 2014). Recently, the elaboration of numerous chemically modified electrodes also demonstrates the sensitivity for the determination of phenol (Wang et al., 1998). The carbon paste electrodes modified with sepiolite (Nie et al., 2011), montmorillonite (Kurian and Sugunan, 2006), polyamide (Zou and Mo, 1997), enzyme and tissue (Bonakdar et al., 1989) were used to electrocatalyze the oxidation of phenol.

As reported in the literature (Adib et al., 1999; Badosz, 1999), activated carbon (AC) is a unique one-dimensional material. Its hollow interior space and high specific surface area can provide large amounts of electrochemical reactive sites for many applications such as super capacitor, catalysis, attaching or filling various inorganic materials, such as metal, oxide, sulfide and halide. Previous studies have shown the benefits of using activated carbon (AC) as conducting supports to confine the size of inorganic particles and thus to improve the specific capacitance. Moreover, a strong interfacial interaction between nickel oxide (NiO) and activated carbon is highly desired to facilitate the charge transfer between the two components, which can enhance the electrocatalytic performance. Nickel(II) salicylaldimine, amine, imidazole and tetraazamacrocyclic complexes have been successfully used as catalysts in the oxidation of phenol to catechol and hydroquinone (Abbo et al., 2004; Ray et al., 2007; Salavati-Niasari and Bazarganipour, 2006; Bansal et al., 2010).

In this paper, we report the synthesis and spectral characterization of activated carbon doped by nickel oxide particles (NiO/AC). Then, the NiO/AC was used as modifier onto carbon paste electrode (CPE) to evaluate their electro-catalytic oxidation of phenol. We also evaluate the analytical performance of the modified electrode NiO/AC-CPE for phenol detection in tap water and olive oil samples.

2. Experimental

2.1. Equipment and reagent

All chemicals used in this work were of analytical grade or of the highest purity available. Sodium hydroxide, sodium phosphate dibasic, monosodium phosphate and hydrochloric acid were obtained from Merck, Fluka, Riedel de Haen Chemical Companies and Sigma Aldrich and were used as received. Phenol was dissolved in $1.0 \times 10^{-1} \text{ mol L}^{-1}$ phosphate buffer (PBS) to prepare stock solutions of $1.0 \times 10^{-3} \text{ mol L}^{-1}$.

Electrochemical measurements (cyclic voltammetry and differential pulse voltammetry) were investigated using an eDAQ recorder/potentiostat EA163 controlled by eDAQChem data acquisition software with a standard three compartment cell consisting of a Pt wire as a counter electrode, an Ag/AgCl electrode as a reference and the NiO/AC-CPE as working electrode. Linear voltammetry and chronoamperometry analysis were carried out using a PGZ 100 potentiostat (Radiometer, Inc.). The pH-meter (Radiometer, SensION™, pH31 and Spain) was used for adjusting pH values.

The chromatographic systems used is constituted by a Waters 2695 pump, auto sampler and Waters 2998 photodiode array detector (PDA). The Spectra Manager software and Empower Software data registration were used for all absorbance measurements. Data acquisition was performed by the Totalchrom Software data registration.

Powder X-ray diffraction (XRD) patterns were recorded with $\text{CuK}\alpha_1$ (1.54056 Å) radiation in the range of 15° – 70° (2 θ) on a diffractometer (D 2-PHASER of BRUKER-AXS) from Germany.

The infrared spectroscopy (PerkinElmer FTIR 1600, Germany) was also used to identify the phases formed of activated carbon doped by nickel.

2.2. Analytical procedure

In this study, the modified electrodes NiO/AC were prepared according to the following procedure: Nickel nitrate was mixed in different ratios by weight (w/w) with activated carbon powder, whereas, the AC was prepared from date stone by physical activation (Hammani et al., 2017). The mixtures were ground in an agate mortar. The resulting powder was calcined at different temperatures to 200 up 800 °C for 17 h in the kiln under nitrogen flow (100 mL min^{-1}). The paste of electrode was obtained by a mixture of carbon powder and nickel particles impregnated onto activated carbon (NiO/AC) with paraffin oil. The mixture paste was then incorporated into the electrode cavity (laboratory made, 0.1256 cm^2 geometric surface area) and was polished by smooth paper. Electrical contact was established by a bar of carbon.

All measurements were carried out under ambient conditions. The appropriate solutions were transferred into the electrochemical cell. The differential pulse voltammogram were recorded in $1.0 \times 10^{-1} \text{ mol L}^{-1}$ phosphate buffer (PBS) between 0 and 1.0 V.

The proposed method was applied to determine phenol in tap water and olive oil samples. The phenolic fraction was extracted from 25 g olive oil samples and was dissolved in 25 mL hexane. Then, the polar compounds were extracted with

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