

Abstract

Presented Ph.D. Thesis is focused on the use of the boron-doped diamond (BDD) electrodes for voltammetric and amperometric determination of selected nitrophenols: 2-nitrophenol (2NP), 4-nitrophenol (4NP), and 2,4-dinitrophenol (2,4DNP). These compounds are listed as “priority pollutants” by United States Environmental Protection Agency (US EPA) due to their negative impact on living organisms and are mainly used in agriculture as plant growth stimulators. BDD electrodes are used for determination of wide range of electrochemically both reducible and oxidisable organic compounds and have become a popular electrode material thanks to its commercial availability and excellent mechanical and electrochemical properties.

A differential pulse voltammetric method was developed for the determination of 2NP, 4NP and 2,4DNP at a BDD film electrode using electrochemical reduction and of 4NP and 2,4DNP using electrochemical oxidation. The method was successfully applied for the direct determination of these compounds in drinking and river water in the concentration range from 4×10^{-7} to 2×10^{-5} mol.L⁻¹. To improve the limit of quantification, a preconcentration by solid phase extraction from 100 mL (drinking and river water) and 1000 mL (drinking water) of water samples was used with limit of determination around 2×10^{-8} and 2×10^{-7} mol.L⁻¹, respectively.

The possibility to employ BDD film electrodes for amperometric detection in wall-jet arrangement in High-performance liquid chromatography (HPLC) was verified by determination of these nitrophenols based on both, electrochemical reduction and oxidation. Optimal conditions for separation at C₁₈ reverse phase column (125×4 mm, 5 µm) and amperometric detection are as follows: for cathodic detection mobile phase 0.05 mol.L⁻¹ acetate buffer pH 4.7/methanol (58/42, v/v), detection potential -1.2 V; for anodic detection mobile phase 0.05 mol.L⁻¹ phosphate buffer pH 6.75/methanol (65/35, v/v), detection potential +1.3 V. The applicability of the developed methods was demonstrated on the analysis of the model drinking and river water samples using their direct injection in the HPLC-ED setup in the concentration range from 2×10^{-6} to 1×10^{-4} mol.L⁻¹. Comparable sensitivities and limits of detection were achieved for both detection modes.

The obtained results confirm that both batch voltammetry and HPLC with electrochemical detection with unmodified BDD electrode represent reliable and sensitive analytical techniques for determination of nitrophenols with limits of detection similar to other electrodes.