

## Abstract

# Rapid and Simple Determination of Nitrite in Soil by Using Portable and Miniaturized Electrochemical Tools <sup>†</sup>

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Nowadays, the rapid and easy determination of nitrite in the environment and foods represents a necessity since its chemical importance has been highlighted in many fields, especially in food and agriculture development. Nitrite is part of the nitrogen cycle, and eutrophication, through its accumulation, can lead to highly toxic effects on plants and animals. Moreover, nitrites are used as additives in food processing and as inhibitors of microorganism growth, an overexpression of them in the body being able to affect the transport of oxygen in the blood and generate methemoglobinemia, known to cause death [1,2]. Thus, electrochemical sensors based on carbon nanomaterials and different matrices of dispersion were developed for the sensitive and rapid determination of nitrite in soil samples extracted with low-volume suction lysimeters. Commercial screen-printed carbon paste electrodes (SPEs) on a PVC support were modified with nanocomposite material based on multiwalled carbon nanotubes (MWCNTs) and different matrices of dispersion (polymers, sol-gel, etc.). Electrochemical (cyclic voltammetry, electrochemical impedance spectroscopy and amperometry) and morpho-structural (SEM, FTIR) studies were performed in order to characterize and optimize the analytical parameters of the developed sensors. The determination of nitrite in real samples of soil solutions was carried out at room temperature by using low-volume suction lysimeters for root-level soil monitoring. The electrocatalytic behavior of the modified sensors toward the oxidation of nitrite was studied in acetate and phosphate buffer solutions of 0.1 M, respectively, with a pH ranging from 4 to 9. An enhanced electrochemical behavior and a decrease in the potential used for nitrite oxidation were obtained by using chitosan (CS) as a dispersion matrix for MWCNT. Completing the electrochemical studies with the morpho-structural analysis revealed a synergistic effect between MWCNT and chitosan. Optimization of the working potential, as well as the buffer's pH, was performed in order to achieve a sensitive and selective determination of nitrite by using MWCNT-CS-based sensors. Thus, a sensitive and selective determination of nitrite was achieved in acetate buffer 0.1 M, pH 5, at an applied potential below +0.6 V vs. Ag/AgCl. The detection of nitrite was performed with a specific sensitivity of 204.4 mA·M<sup>-1</sup>·cm<sup>-2</sup> in a linear range up to 1.7 mM in acetate buffer 0.1 M, pH 5, at an applied potential of +0.58 V vs. Ag/AgCl. The limit of nitrite detection achieved by using MWCNT-CS-based sensors was 2.3 μM (S/N = 3), and the sensors showed good stabilities and reproducibilities. A miniaturized portable system using the developed MWCNT-CS-based SPEs was dedicated to the detection of nitrite in soil solution samples extracted using suction lysimeters. A simple and miniaturized electrochemical sensor based on MWCNT-CS nanomaterial was developed for the sensitive, selective and rapid determination of nitrite in soil solutions. The detection of nitrite in real



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samples was performed by using a portable electrochemical detector with the developed sensor inserted directly into the suction lysimeters.

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