

**Área: ANA**

## Electrochemical sensor based on poly(phenanthroline) and oxygenated carbon nanotubes for determination of lead

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### Highlights

Development of an electrochemical sensor using nanocomposite based on poly(phenanthroline) and oxygenated carbon nanotubes to detect lead, aiming at forensic application.

### Resumo/Abstract

Approximately 71% of violent deaths in 2023 were committed with firearms, reaching approximately 32,749 homicide victims<sup>1</sup>. When a firearm is fired, various organic and inorganic compounds, such as heavy metals, are released into the environment. These released compounds can be retained in various parts of the shooter such as the hands, face, and hair. Therefore, techniques that could detect a variety of compounds at low concentrations are extremely useful for verifying firearm discharge<sup>2</sup>. Therefore, this work aims to develop a nanocomposite sensor for lead detection aiming at forensic application. The proposed methodology involves electrode modification, selection and optimization of the electroanalytical technique, and evaluation of the sensor's performance for lead determination. The graphite electrode surface was modified by the deposition of a nanocomposite film composed of oxygenated carbon nanotubes (O-CNT) and electropolymerized poly(phenanthroline) using 5 cycles of cyclic voltammetry in the potential range from 0.0 V to -1.25 V vs. Ag/AgCl in a solution of phenanthroline ( $6.3 \times 10^{-3}$  mol L<sup>-1</sup>) in sulfuric acid. After deposition, the film was activated to increase its electroactivity. The activation step was performed using 5 cycles of cyclic voltammetry in the potential range from 0.0 V to 1.2 V vs. Ag/AgCl in a 0.1 mol L<sup>-1</sup> sulfuric acid solution<sup>3</sup>. To characterize the film, scanning electron microscopy (MEV) images, electrochemical impedance spectroscopy measurements, and cyclic voltammetry measurements were carried out. For the analysis of lead, the square wave voltammetry technique was applied in the potential range from 0.0 V to -1.2 V vs. Ag/AgCl, and the following parameters were adopted: potential increment of 8 mV; Frequency of 100 Hz; pulse amplitude of 100 mV; deposition potential of -0.8 V, and deposition time of 90 seconds (lead concentration < 1 μmol L<sup>-1</sup>) and 270 seconds (lead concentration > 1 μmol L<sup>-1</sup>). The electrolyte solution that gave the best results for lead analysis was a KCl-HCl solution (0.1 mol L<sup>-1</sup>; pH 4). To prevent lead from impregnating the electrode, a cleaning method was used, which consisted of a voltammetric measurement in a lead-free solution. The sensor exhibited promising results, obtaining a linear response in the range of 0.5 to 7.5 μmol L<sup>-1</sup> and a limit of detection of 0.038 μmol L<sup>-1</sup> for lead analysis.

<sup>1</sup>CERQUEIRA, Daniel; BUENO, Samira (coord.). Atlas da violência 2025. Brasília: Ipea; FBSP, 2025. 174 p. Disponível em: <https://repositorio.ipea.gov.br/handle/11058/17165>.

<sup>2</sup>WONGPAKDEE, Thinnapong et al. The development of screen-printed electrodes modified with gold and copper nanostructures for analysis of gunshot residue and low explosives Forensic Science International, v. 364, p. 112243, 2024. Disponível em: <https://doi.org/10.1016/j.forsciint.2024.112243>.

<sup>3</sup>SHUL, Galyna; WEISSMANN, Martin; BÉLANGER, Daniel. Electrochemical Formation of an Ultrathin Electroactive Film from 1,10-Phenanthroline on a Glassy Carbon Electrode in Acidic Electrolyte. Langmuir, v. 30, n. 22, p. 6612–6621, 2014. Disponível em: <https://doi.org/10.1021/la500349t>.

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