Área: ANA

Sample preparation and electrochemical detection: combining gas diffusion extraction with miniaturized electrochemical devices for nitrite quantification

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Highlights

Gas-diffusion extraction and electrochemical detection in a single step. Inexpensive analytical system for nitrite determination.

Resumo/Abstract

Nitrite is used as a food additive and preservative (E249–E252), mainly for meat products, inhibiting the growth of microorganisms. However, exposure to excessive amounts of nitrite can cause serious health problems. Most conventional analytical methods are time-consuming, high-cost, and require several sample preparation steps. This study presents a simple and low-cost 3D-printed analytical device for quantifying nitrite coupling gas-diffusion extraction and electrochemical detection in a single step. The miniaturized electrochemical sensor was fabricated using micropipette tips and metallic wires (Pt and Ag). The device was assembled by inserting a 3D printed and miniaturized electrochemical cell (acceptor reservoir) in the glass sample reservoir, which was filled with 10 mL of the nitrite standard or sample solutions and 1 mL of HCl 12 mol L^{-1}. PTFE (Polytetrafluoroethylene) membranes (pore size of 1 µm; Amanco, Brazil) allowed gas diffusion of the generated NO_2 and separation between the sample reservoir (donor solution) and the electrochemical cell containing the acceptor solution (supporting electrolyte). Figure 1-A showed that good analytical performance (LOD = 58 µmol L^{-1} and R^2 = 0.99596) was obtained for the analytical curve constructed using 500 µL of nitrite standard solutions without extraction by gas diffusion, indicating that the sensor has great potential for nitrite determination. Next, we evaluated the proposed system's potential to detect nitrite after gas extraction.

Figure 1: A) Analytical curve constructed in the linear range of 100 to 1100 µmol L^{-1}. **B)** Real-time extraction curve for 100 µmol L^{-1} of the nitrite in donor solution.

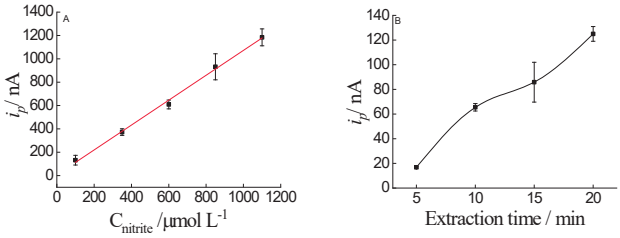

Figure 1-B shows a sharp increase in the nitrite signal during the first 20 min of the extraction, indicating that nitrite can be extracted and collected by the supporting electrode. Further optimization will be performed to improve the analytical performance of the method. The preliminary results demonstrated that the approach described here is a promising way to fabricate a simple, efficient, and cost-accessible analytical device that couples the sample preparation step and electrochemical detection for quantifying nitrite in food samples.

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