DETERMINATION OF Cu AND Fe IN REAL SAMPLE OF WATER AND ETHANOL USING SQUARE WAVE ANODIC STRIPPING VOLTAMMETRY WITH A MICRO/NANO BORON-DOPED DIAMOND ELECTRODE

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Resumo

Searching for a more cost-effective alternative with adequate selectivity, sensitivity, and reproducibility in the determination of metallic ions, proves to be important to develop reliable methodologies in the determination of these contaminants using a micro/nano boron-doped diamond electrode for copper and iron analysis in aqueous and ethanolic environments.

Introduction

Numerous toxic metals are released into the environment through industrial activities every year. It is necessary to monitor their presence in different matrices and ecosystems [1] since metals as copper and iron, can interfere with the nervous, gastrointestinal, cardiovascular, renal, and hematopoietic systems in humans and other animals, even at low concentrations. Electrochemical methods have been employed for the determination of compounds at trace levels in industrial, biological, and environmental product monitoring studies with adequate sensitivity, precision, and accuracy. Boron-doped diamond (BDD) electrodes appear as a new material, with advantages favored the evolution of the use of diamond to detect a variety of analytes, including traces of heavy metals [6].However, there are no studies in the literature regarding the use square wave anodic stripping voltammetry (SWASV) with a micro/nano BDD electrode for determination of copper and iron metals in water and fuel ethanol samples.

In this way, the development and validation of a voltammetric method for these metals becomes important. In the present work, a laboratory validation study was developed for quick, simple, and low-cost quantification of copper and iron metals in aqueous and ethanolic media using an electrode containing a mixed morphology that until now has not been addressed in the literature.

Materials and Methods

The materials used three electrodes: The working electrode (BDD); The reference electrode (Ag/AgCl) and the counter electrode (Pt). The electrode BDD was obtained with a doping level of 15,000 ppm.

The Morphological and structural characterization of BDD films were carried out through SEM, AFM, XRD, and Raman scattering spectroscopy techniques. The electrochemical measurements required to study the reversibility kinetics of BDD electrode were performed by cyclic voltammetry (CV), with 50 mL of 0.5 mol L^{-1} H_2SO_4 solution as support electrolyte, in a potential range of -3 to +3V with a scanning speed of 30 mV s^{-1}. The study the kinetics of BDD electrode reversibility were performed in $K_3Fe(CN)_6$ 1 mmol L^{-1} solution were obtained in a potential scan in the range of -0.2V to 1V by CV, tests were performed at scanning speeds found in the range of 5 to 100 mV s^{-1}. BDD electrode was used as anodic material for electroanalytical methodology development for metals detection in aqueous and ethanolic matrices. The electroanalytical methodology was validated.

Results and Discussion

The BDD film was formed by diamond grains with randomly oriented crystals, having a microcrystalline grain morphology, as can be seen in Fig.1a. In addition, the roughness, for this film, obtained by AFM (Fig. 1b) analysis was 492.5 nm, showing that it has a roughness higher than many electrodes used in electrochemistry.

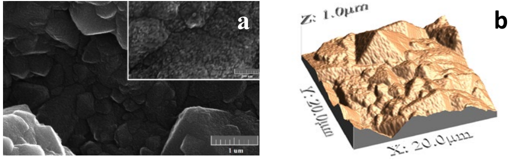

Fig. 1 – (a) Image obtained by SEM from the surface of the BDD diamond film; (b) AFM image of the BDD sample.

In this work the selectivity was evaluated through the standard addition method. Two analytical curves of the metals (0.001 to 0.006 mg L^{-1} of Cu^{2+} and Fe^{2+}) were constructed from a standard solution of these metals. The results obtained for the study of the curves parallelism showed that for the studied metals both matrices showed good selectivity of the technique. Precision was assessed by repeatability estimates. The results obtained for the accuracy test in terms of percent recovery were within the predicted range, it is concluded that the method presents satisfactory precision and accuracy.

Conclusion

In this work a micro/nano BDD electrode was produced, characterized, and applied as anodic material for the detection of Cu^{2+} and Fe^{2+} ions, in aqueous and ethanolic matrices, by SWASV. With this purpose, electroanalytical methodology was intra-laboratory validated. Renucleated BDD electrode was found to be feasible in metal trace analysis.

References

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