

BioCop Fellowship Report 2006

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Universidade de Coimbra, Departamento de Quimica, Coimbra 3004-535 (Portugal).

Host scientist Professor Christopher M.A. Brett

Interval 2-16 July 2006.

Since Professor Christopher M.A. Brett has broad knowledge concerning the modified electrochemical sensors, the goal of my sponsored visit was to learn more about Anodic Stripping Voltammetry techniques coupled with modified sensors applied to heavy metal analysis in real samples.

My visit to the Department of Chemistry University of Coimbra gave me the opportunity to meet a friendly and dedicated group of people who were enthusiastic about my work in the BioCop Project. This activity gave me the chance to talk to each one of them and find out more about the electrochemical techniques specially the anodic stripping analysis, the electrochemical modified sensors and the impedance technique.

In the first day of my visit (2 July) I had a meeting with the Professor Christopher M.A. Brett. We discussed about my previous work in Italy and the obtained results. Also we defined the activities to be developed during my presence in his laboratory.

I began my work (3 July) by carrying out some experiments concerning the subject of comparative study between Screen Printed Electrodes and Carbon Film Electrode using the Square Wave Anodic Stripping voltammetry.

Since we have some problems employing our sensors at high concentrations of acid [0.44 M] we decide to apply the Square Wave Anodic Stripping Voltammetry (SWV) at hydrochloric acid 0.44 M employing the sensors from Portugal laboratory (Carbon Film Electrodes). The SWV conditions were: Conditioning potential – 0.6 V for 30 seconds, potential of deposition of bismuth and lead equal to -1.1V for 300s, equilibration time 15 s, Frequency 50 Hz and pulse potential equal to 0.019V). From the results we noted that the background increase with repetition but the background of carbon film remains stable. In order to understand this

difference between the two experiments we decide to apply the impedance spectroscopy technique.

On Thursday July 6 I attended the seminar given by PhD student Carla Gouveia which discussed the influence of nafion coatings and surfactant on the stripping voltammetry of heavy metals at bismuth-film modified carbon film electrodes. The seminar was very helpful for learning about the Modified electrode because it focused on the influence of surfactants on the determination of lead and cadmium by impedance spectroscopy using modified electrode with nafion.

In second week, my work was focused on the study of the characterisations of Screen Printed Electrodes modified and unmodified with nafion by electrochemical impedance spectroscopy at different media and potentials.

Characterisation of the SPE using spectroscopy impedance technique

The Screen printed electrodes with and without nafion film have been characterized by electrochemical impedance spectroscopy (EIS) with a view to better understanding of their advantages and limitations in electroanalysis, particularly in anodic stripping voltammetry of metal ions.

The parameters of impedance technique are initial frequency equal to 60000 and the final frequency equal to 0.1 HZ and amplitude equal to 10 mV.

The characterisation of the SPE by the EIS was done at different potential and different electrolyte support using unmodified and modified Screen Printed electrode with 0.5 % of nafion.

I- Unmodified SPE

I.1. Hydrochloric acid (0.44 M) combined with unmodified SPE

I.1.a Potential = 0 V

I.2. Acetate buffer 0.1 M pH = 4.2

I.2.a Potential = 0 V

II. Modified SPE with 0.5% of nafion

II. 1. Hydrochloric acid (0.44 M) combined with SPE modified with 0.5% of nafion

II. 1.a Potential = 0 V

II. 2 Acetate 0.1 M pH = 4.2 buffer

II.2a Potential = 0V

III. Comparison between modified and unmodified SPE at 0 V using HCl 0.44 M

IV. Comparison between modified and unmodified SPE at 0 V using Acetate buffer

V. Comparison between electrolyte support using unmodified SPE

VI. Comparison between electrolyte support using modified SPE with 0.5 % of nafion

It seems that the resistance decreased with the repetition in the case of hydrochloric acid and acetate buffer it means that some reactions are working at the surface of the electrode (some charge transfer reaction). Transfer reaction in HCl is more pronounced than in the case of acetate buffer with and without KCl. In general the electrode must be more capacitive without any reaction at the surface (not under going any reaction). The decrease in the resistance means that the surface of the electrode is not stable.

The impedance plots for SPE unmodified and modified with 0.5 % of nafion in HCl 0.44 M at 0 V showed that the resistance increase with Nafion film. Which can be due to the limitation imposed to charge transfer by the polymer coating, the same behaviour was previously observed in Nafion-coated glassy carbon electrodes [1]. The nafion film protects the surface of the electrode. In the case of acetate buffer we didn't note any change using nafion film.

All these experiments could not be carried out with successful results without the supervision and guidance of laboratory people. I'm very grateful to everybody that helped me. Special thanks to Professor Christopher M.A. Brett Who agreed to collaborate with us and accepted my presence to his team. Thanks are due to Dr Rasa Pauliukaite and the PhD student Carla Gouveia. Also many thanks to Professor Ana Maria Oliveira Brett for her kindness and advice.

Thanks are due to Dr. C. Situ and the BioCop Management Group for their technical assistance.

[1] C.M.A. Brett, D.A. Fungaro, J.M. Morgado, M.H. Gil, J.Electroanal. Chem. 1999, 468, 26.