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Ljubiša Balanović
Dejan Tanikić

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PREFACE

On behalf of the Organizing Committee, it is a great honor and pleasure to welcome all esteemed participants of the 54th International October Conference on Mining and Metallurgy (IOC 2023), scheduled to take place at the picturesque Bor Lake, Serbia, from October18th to 21st 2023.

The collaborative efforts of the University of Belgrade, the Technical Faculty in Bor, and the Mining and Metallurgy Institute Bor have meticulously organized this year's IOC. Our focus remains unwavering on showcasing the latest research findings and advancements in geology, mining, metallurgy, materials science, technology, environmental protection, and other engineering disciplines. Our primary objective is to foster a dynamic environment where academics, researchers, and industry professionals can come together to share their knowledge, experiences, and innovative ideas while exploring opportunities for collaborative research endeavors.

Our conference agenda is rich and diverse, encompassing plenary sessions, engaging invited lectures, technical presentations, enlightening oral and poster sessions, informative technical tours, a diverse exhibition, and memorable social gatherings. At the heart of this event lies our strong commitment to sustainable development within the mining and metallurgy sector. We are dedicated to exploring ecologically conscious methodologies, responsible resource extraction practices, and cutting-edge technologies that reduce the industry's environmental impact and enhance the well-being of local communities.

The conference proceedings comprise 129 papers authored by individuals from universities, research institutes, and industries in 22 countries. We are proud to welcome participants from Bosnia and Herzegovina, Bulgaria, Canada, China, Croatia, Germany, Greece, India, Iran, Kazakhstan, Libya, North Macedonia, Montenegro, Morocco, Romania, Russia, Slovakia, South Africa, Spain, Turkey, United States, and, of course, Serbia.

We are excited to host the 8th International Student Conference on Technical Sciences (ISC 2023) as part of IOC 2023. This event offers students from Serbia and the wider region a unique chance to showcase their research and discuss the future of their fields with experts.

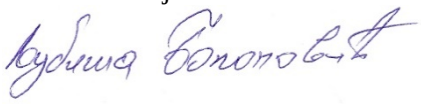
We sincerely thank the Ministry of Science, Technological Development, and Innovation of the Republic of Serbia for their generous financial support. In addition, we express our profound gratitude to all our sponsors, exhibitors, and friends of the Conference for their contributions and unwavering support for playing a pivotal role in ensuring the success of IOC 2023.

We would like to express our heartfelt thanks to all authors, committees, reviewers, speakers, and chairpersons for their invaluable contributions in shaping IOC 2023.

We look forward to welcoming you to the 55th International October Conference on Mining and Metallurgy (IOC 2024), which will be held in October 2024.

On behalf of the 54th IOC Organizing Committee,

Prof. dr Ljubiša Balanović



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ELECTROCHEMICAL METHODS FOR THE DETERMINATION OF TRYPTOPHAN AND CAFFEINE

Žaklina Tasić, Marija Petrović Mihajlović, Ana Simonović, Milan Radovanović, Maja Nujkić, Milan Antonijević

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Abstract

Electrochemical methods are widely used for the determination of various chemical compounds due to their simplicity and short time for analysis, as well as enabling low detection limits. Various electrodes have been developed over the years, including those made from carbon materials, gold, mercury, as well as various modified electrodes for detection of analytes. Carbon electrodes are particularly favored as they possess low background current and high electrical conductivity. Also, they are relatively inexpensive, easy to prepare, and generally non-toxic. In this study, the sensor characteristics of a graphite electrode prepared from spent zinc-carbon batteries for the determination of tryptophan and caffeine have been investigated. Britton-Robinson and acetate buffers were used as electrolytes. The obtained results demonstrated that this electrode is a good electrochemical sensor for detecting tryptophan alone as well as in the presence of caffeine. Additionally, the modified graphite electrode is resulting in improved sensor characteristics for the determination of tryptophan in Britton-Robinson buffer. The results obtained by differential pulse and cyclic voltammetry highlight the potential of graphite electrodes as effective electrochemical sensors for the detection of analytes.

***Keywords**: electrochemical sensor, zinc-carbon batteries, caffeine, tryptophan, modified graphite electrode*

1. INTRODUCTION

Electrochemical methods used for the analysis and detection of chemical compounds have significant advantages over other analytical methods, such as simplicity in operation, short time for analysis, and achieving low detection and low quantification limits. Over the past decades, there has been a growing trend in the development of various electrodes, such as electrodes prepared from carbon materials, as well as gold, mercury, and modified electrodes for the determination of different analytes [1,2]. Carbon electrodes possess favorable electrochemical properties, including low background current and high electrical conductivity, which make them a suitable choice for sensors. Moreover, they are relatively inexpensive, easy to prepare, and typically non-toxic. However, in light of increasing waste generation due to technological advancements, some researchers [3,4] have recommended using graphite rods from discarded batteries instead of commercial electrodes. Zinc-carbon batteries, for example, are a source of such waste. Improper disposal of such batteries can result in heavy metal pollution in the environment, which can lead to severe adverse effects on living organisms. Recycling these batteries would be an environmentally responsible measure, with significant economic benefits [5]. Considering the high electrical conductivity and large surface area of graphite rods, they have the potential to be utilized as effective electrochemical sensors [3].

The aim of this study was to investigate the sensor characteristics of a graphite electrode prepared from spent zinc-carbon batteries for the determination of tryptophan without and with the addition of caffeine in Britton-Robinson and acetate buffers using electrochemical methods, cyclic voltammetry and differential pulse voltammetry.

2. EXPERIMENTAL

For the experimental investigations, a three-electrode system consisting of a graphite electrode as the working electrode, a saturated calomel electrode (SCE) as the reference electrode, and platinum wires as auxiliary electrodes was used. The graphite electrode was rinsed with distilled water, polished with Al_2O_3, rinsed again with distilled water, and dried before each measurement.

The concentrations of the analyzed solutions of tryptophan (TRP) were $4 \cdot 10^{-4}$ M and $1 \cdot 10^{-4}$ M while for caffeine (CAF) was $1 \cdot 10^{-4}$ M. Britton-Robinson buffer (BR), with a concentration of 0.04 M, was prepared by mixing solutions of 0.04 M H_3BO_3, 0.04 M H_3PO_4, and 0.04 M CH_3COOH, while the acetate buffer with a concentration of 0.2 M was prepared by mixing solutions of 0.2 M CH_3COOH and 0.2 M CH_3COONa. The solution of 1M NaOH was used to adjust the pH value of the solutions.

Electrochemical methods used for the analysis of tryptophan and caffeine, were cyclic voltammetry (CV) and differential pulse voltammetry (DPV). The CV and DPV curves were recorded in the potential range of 0.0 to 1.2 V (vs. SCE), with a scan rate of 50 mV/s.

3. RESULTS AND DISCUSSION

Since it has been shown that a graphite electrode can be used to determine TRP in BR buffer solution at pH 4 [6], further experiments were performed in BR at pH 1.8 and acetate buffer at pH 4.35 using electrochemical methods. Figures 1a and 1b illustrate the obtained DPV curves in BR buffer (pH 1.8) and acetate buffer (pH 4.35) without and with the addition of $1 \cdot 10^{-4}$ M TRP as well as $1 \cdot 10^{-4}$ M TRP + $1 \cdot 10^{-4}$ M CAF on graphite surface.

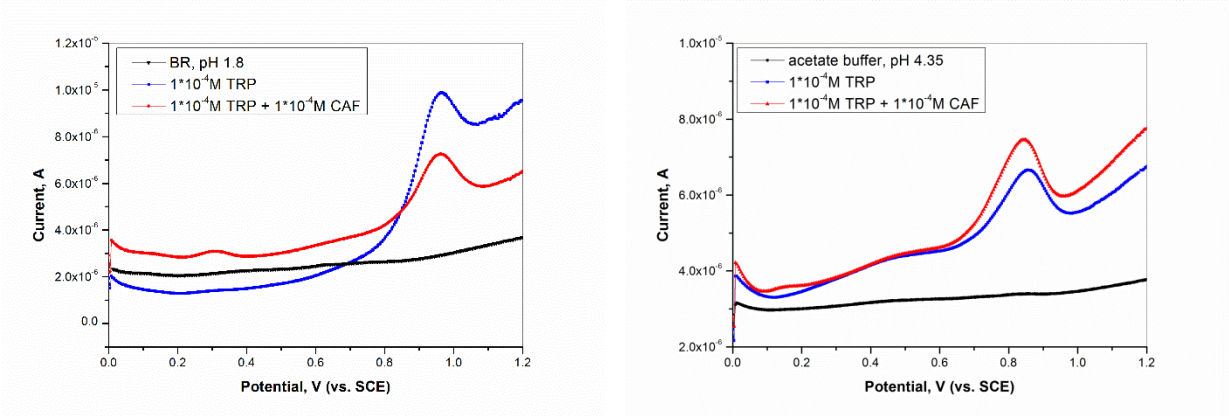

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Figure 1 - (a) Differential pulse voltammetry curves on graphite electrode in Britton-Robinson buffer solution (pH 1.8) and (b) acetate buffer solution in the absence and in the presence of $1 \cdot 10^{-4}$ M of tryptophan as well as $1 \cdot 10^{-4}$ M of tryptophan + $1 \cdot 10^{-4}$ M of caffeine, scan rate 50.0 mV/s

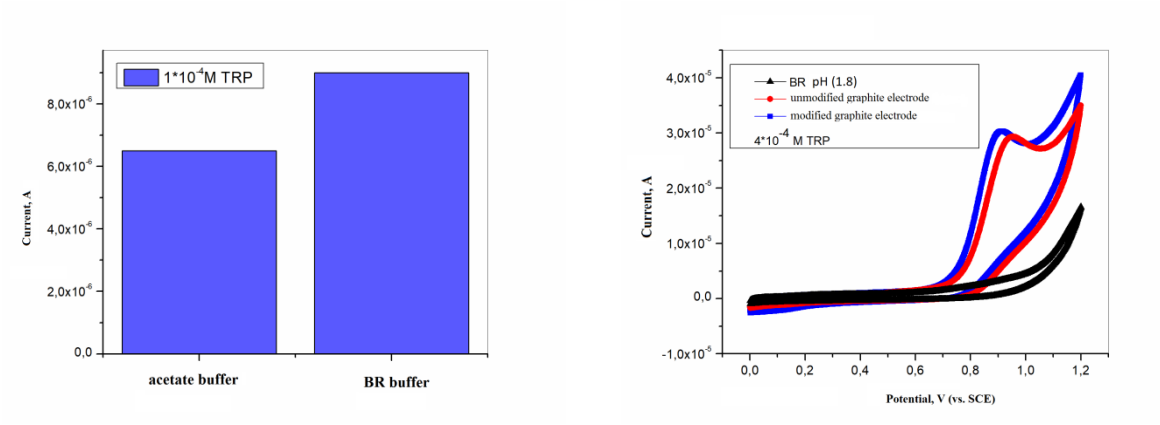

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Figure 2 - (a) dependence of the intensity of the tryptophan current peak on type of buffer solution; (b) cyclic voltammetry curves of TRP on unmodified and modified graphite electrode in Britton-Robinson buffer solution (pH 1.8)

From Figure 1a, it can be observed that there are no current peaks in the basic solution, while in the presence of TRP, a peak is observed at a potential of 0.93 V, which can be observed in the solution containing both TRP and CAF. Figure 1b shows the differential pulse voltammogram of $1 \cdot 10^{-4}$ M TRP, as well as $1 \cdot 10^{-4}$ M TRP + $1 \cdot 10^{-4}$ M CAF in acetate buffer solution. Based on the voltammogram in Figure 1b, it can be seen that there are no current peaks in the basic solution, while a peak is observed at a potential of 0.82V in the presence of TRP. Additionally, the current peak is observed at a potential of 0.83V in the solution containing both CAF and TRP at a concentration of $1 \cdot 10^{-4}$ M. It can be concluded that the graphite electrode in the acetate buffer (pH 4.35) is able to detect tryptophan. In addition, in the acetate buffer, in the presence of both tryptophan and caffeine, the intensity of the current peak was higher than the current peak of tryptophan alone. These results indicate that the graphite electrode has high sensitivity for determining TRP in both BR and acetate buffer. Additionally, it can be observed that when CAF and TRP are present in the BR solution simultaneously, the intensity of the tryptophan peak is reduced.

Based on the voltammograms (Figure 1a) and the paper by Tasic et al. [6], it can be concluded that changing the pH of the solution results in a shift in the potential of the tryptophan current peak. Decreasing the pH value leads to the appearance of an oxidation peak of TRP at higher potential values. It was also observed that caffeine leads to a decrease in the current peak of TRP in BR solution. Figure 2a shows the influence of the buffer on the intensity of the current peak of tryptophan analyzed using the graphite electrode and DPV. It can be observed that the intensity of the current peak of tryptophan in the Briton-Robinson buffer reaches a value of $9.0 \cdot 10^{-6}$ A, while in the acetate buffer, it reaches a value of $6.5 \cdot 10^{-6}$ A. Therefore, the type of buffer is an important parameter for determining the desired analytes. For improving the sensitivity of the sensor, a modification of the graphite electrode in a 0.5 M solution of H_2SO_4 was performed. The modification was carried out using cyclic voltammetry, in the potential range from 0V to 2V. The prepared electrode surface was then used for the determination of tryptophan. The behavior of the unmodified and modified graphite electrode in the Britton-Robinson solution in the presence of tryptophan is shown in Figure 2b. From the cyclic voltammogram (Figure 2b), it is evident that the TRP oxidation peak at the unmodified graphite electrode occurred at a potential of about 0.95V, while the TRP oxidation peak at the modified graphite electrode was observed at a potential of about 0.90V. The current density value was higher on the modified graphite electrode than on the

unmodified graphite electrode. It is assumed that the electrode modification has developed a larger specific surface area, which affects the intensity of the current response of tryptophan.

According to the literature data [6,7] and the results obtained in this investigation, the mechanism of TRP oxidation at the graphite electrode was proposed and shown in Figure 3.

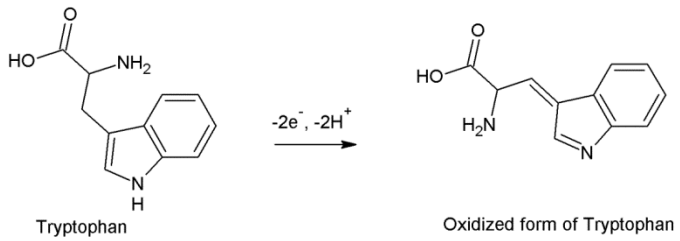

Figure 3 - The proposed mechanism of TRP oxidation on the graphite electrode

4. CONCLUSION

Based on the experimental data presented in the paper, it can be concluded that the graphite electrode has shown to be a good electrochemical sensor for the detection of tryptophan without and with the addition of caffeine. The results indicate that the graphite electrode can be successfully used for tryptophan determination at pH values of 1.8 in Britton-Robinson buffer and 4.35 in acetate buffer solutions. Additionally, it has been shown that the choice of buffer also effects the potential and intensity of the oxidation peaks of the analyzed compounds. In order to improve the sensor characteristics, the surface of the graphite electrode was modified in a simple way and it proved to be successful in determining tryptophan.

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