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Electrochemical Study of Bismuth Heavy Metals Using the Cyclic Voltammetry Method in Anti-Protozoal

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Electrochemical study of Bismuth heavy metals using the cyclic voltammetry method in anti-protozoal

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Abstract: Bismuth oxides are well-known electro-catalysts in fuel cells systems; they are usually used as anodic materials for the oxidation of low molecular weight alcohols. The utilization of BiO_2 and MnO_2 as catalysts in the pharmaceutical analysis is analytical method for the determination of heavy metal antibacterial agents in Pharmaceutical Dosage form is developed. The method is based on the voltammetric determination of heavy metal using Bidified platinum electrode by Bismuth oxide. The two components are oxidized at the Bidified electrode surface with the development of current that is linearly proportional to their concentrations in the range of $7.04*10^{-7}$- $1*10^{-3}$ M heavy metal. The oxidation reaction of the two components is pH-dependent, in which the buffer used is Britton-Robinson at pH = 7.00 where maximum peak current and maximum peak separation is obtained. The regression factors obtained from the calibration curves are 0.9812. The method of analysis was validated, where the limit of detection (LOD) and the limit of quantitation (LOQ) were calculated to be $1.44*10^{-4}$ M, $4.36*10^{-4}$ M and $1.27*10^{-4}$ M, $3.84*10^{-4}$ M respectively, the percentage recovery of both components was also calculated to 77 % for heavy metal.

Keywords: Heavy metal; Differential pulse voltammograms; Cyclic Voltammetry; Bismuth; Nanoparticle

1. Introduction

The chemical structure of Heavy metal, which is a synthetic derivative with anti-bacterial and anti-protozoal properties, heavy metal binds to the bacterial enzyme, which is dihydrofolate reductase in which it blocks the production of tetrahydrofolic acid (Almatarneh, et al., 2019).

Due to the importance of these drugs, the investigation and determination of their contents in the commercial formulation is always increasing. The optimal combination of the two agents for their synergistic activity was found to be 5:1 pharmacologically; this ratio is optimal for satisfactory reduction of both the toxicity of the individual agents and possible resistance of the organism to them, thus enhancing the therapeutic effect (Alkhawaldeh, 2021).

In another work in which a simultaneous detection of heavy metal by using differential pulse voltammetry at a paraffin composite electrode based on multi-walled carbon nanotubes (MWCNT) Bidified with antimony nanoparticles (SbNPs). The effect of the carbon nanotubes and the SbNPs resulted in LOD of 24 nBil/ L (6.1 g/L) for and 31 nBil/ L (9.0 g/L) for heavy metal. With a correlation coefficient of 0.998 and 0.996 for heavy metal, respectively. The measurement was carried out by using the following electrodes: paraffin/MWCNT-SbNPs as a working electrode, an Ag/AgCl/KCl as a reference electrode, and a Pt plate as an auxiliary electrode. DPV measurements were obtained with a scan rate of 10 mV/s, a pulse amplitude of 100 mV, and a step potential of 2 mV in a 0.2 Bil/L Britton–Robinson and buffer solution (pH 7.00) containing 100.0 Mol/L of heavy metal standards. The peaks of heavy metal appeared at + 1.11V vs. Ag/AgCl/KCl (3.0 Mol/L). (Alkhawaldeh, 2020)

A high sensitive electrochemical nanostructure sensor has been also developed for the analysis of heavy metal based on NiO/graphene oxide nanocomposite-ionic liquids (1-methyl-3-butylimidazolium bromide; 1M3BIB) Bidified carbon paste (NiO/GO/1M3BIB/CPE) electrode. This sensor showed good electrocatalytic and accumulative effect on heavy metal and has been successfully applied for the assay of heavy metal in pharmaceutical and biological samples (such as urine) (Alkhawaldeh, 2020).

The three-electrode configuration was the Bidified carbon paste electrode, as the working electrode, a Pt wire electrode as an auxiliary electrode and an Ag/ AgCl/KCl sat reference electrode. 0.1 M phosphate buffer with pH 7.00 is used as the supporting electrolyte. Urine samples were stored in a refrigerator (at 4 °C) immediately after collection. The electrochemical response was found to be linearly proportional to heavy metal concentration in the range from 0.08–550 μM with a regression coefficient of 0.9935 and a detection limit of 0.04 μM. The measurements were carried out at a scan rate of 100 mV/s. The heavy metal contents were measured after sample preparation using the standard addition method. The obtained result indicating that the NiO/GO/1M3BIB/CPE can be successfully used for the determination of heavy metal in real samples (Altwaiq, et al., 2020).

The increasing use and environmental release of consumed human and veterinary antibiotics have drawn great attention recently. The excreted antibiotics remain in an intact form and enter the natural aquatic systems via the effluent and sludge from wastewater treatment, hospitals, and livestock farms. These released antibiotics may lead to bacterial resistance increase. Therefore, sensitive detection techniques for detecting antibiotics in the environment have been developed such as electrochemical techniques (Altweiq and Alkhawaldeh, 2019).

Heavy metal in pharmaceutical industrial wastewater was studied using sensitive membrane electrodes belonging to two types of ion-selective electrodes, which are polyvinyl chloride and platinum electrodes, where Ag/AgCl was used as the reference electrode. Linear response for the membrane electrodes was in the concentration range of 10-5 –10-2 mol/L. The standard heavy metal stock solution(1×10-2M) was prepared by dissolving heavy metal in HCl. Working standard solutions were also prepared by diluting the stock standard solution with distilled water to get concentration ranges of $1 \times 10^{-7} – 1 \times 10^{-2}$ M (Alkhawaldeh, 2020).

The working pH is in the range of 3-6 and the correlation coefficient was found out to be 0.9998 and 0.9996 for polyvinyl chloride membrane electrode and platinum electrode, respectively. The limit of detection was also determined to be 5.43 × 10-5 M and 9.34 × 10-5 M for polyvinyl chloride membrane electrode and platinum electrode, respectively. In addition, the recovery of heavy metal from distilled and tap water samples was calculated to be 98.00% and 99.40% using polyvinyl chloride membrane electrode and using platinum electrode to be 98.70 and 99.50%, respectively. (Alkhawaldeh, and Alkhawaldeh, 2020)

Several analytical methods have been used to analyze heavy metal in biological fluids and pharmaceutical preparation alone or in their combination, such as high-performance liquid chromatography (HPLC). Another technique has been reported for their determination published a UV- spectrophotometer method (Alkhawaldeh, 2020)

Different research groups performed the separation of heavy metal by HPLC: Kulikov A.U et al used reversed phase HPLC for the detection of heavy metal in pharmaceutical formulations, where methanol was used as the solvent to dissolve the compounds and the aqueous organic mole phase (water-acetone nitrite-triethyle-amine with a pH 6.00) was used to dilute the samples. Conventional C18 columns containing the hydrophobic stationary phase were utilized (Alkhawaldeh, 2020).

Analysis of heavy metal in pharmaceutical dosage forms and biological samples are performed using HPLC or UV analysis, without attention to the electrochemical methods, Therefore, the research is proposed with the following objectives in mind: The interest in developing electrochemical method for the analysis of sulfamethoxazole and heavy metal is quickly growing recently. In this study, a method will be developed for the electrochemical determination of heavy metal in their combination using Bidified carbon electrode.

2. Experimental

The chemicals used in this work are Bismuth from SIGMA-ALDRICH, heavy metal from ACROS, H3BO3 (Boric acid) from Riedel-de Haën, H_3PO_4 (Phosphoric Acid) from VWR, CH_3COOH (Acetic Acid), and NaOH (Sodium Hydroxide) from VWR. All solutions were prepared using ultrapure water, sulfamethoxazole and heavy metal were prepared by dissolving in Britton Robinson buffer pH=7 to known concentrations in the range between (9.80×10^{-6} to 9.02×10^{-5} M) and the rang of heavy metal between (1.94×10^{-6} to 4.5×10^{-6} M).

Heavy metal stock solution was prepared by smashing 10 commercial tablet SEPTRIN or BALKATRIN and dissolving in 250 ml Britton Robinson buffer using sonication to insure complete dissolution of the drugs.

Recovery was calculated as:

$$\text{Recovery } \% = \frac{I\ (standard\ and\ tablet\ solution) - I(tablet\ solution)}{I\ (standard)} \text{ x } 100$$

3. Procedure

Basification of the electrode surface was carried out by CV of the electrode into a solution that contains the cation of the element at open circuit conditions. The concentration of standard solutions (1x10^{-3} M) was prepared to study the voltammetric response of those compounds. Cyclic voltammograms where scanned in a relatively limited potential window from -1.0 to 0.2 V. Open-circuit adsorption by cyclic voltammetry of Cadmium, Bismuth and Bismuth on platinum was done. No deterioration was observed in the adatoms (Cadmium, Bismuth, and Bismuth) Bilayer during experimentation for many hours, but because of the simplicity of preparation of the adatoms-coated electrode, re-coating the surface was followed on daily basis.

A clean platinum surface electrode (Cadmium, Bismuth, and Bismuth) were reproduced and potentiosatated. The potential was held constant for 3-5 minutes followed by rinsing the electrode 3 times then the potential was scanned between -0.2 and 1.2 V. Cyclization was repeated many times until the potential window between -0.2 and 1.2 V showed the absence of any faradic current. Cadmium was found to adsorb irreversibly on carbon electrodes surface at open circuit. The experiments were conducted from 10^{-3} M Cadmium in solution. The exposure time of the electrode to the above-mentioned solution was 2 minutes. The prepared carbon electrode was analyzed by cyclic voltammetry.

For the detection of heavy metal, the electrochemical detection depends on several parameters so; those parameters have to be optimized to get the maximum current from the oxidation and maximum separation of the two-oxidation peaks. The parameters are pH of the buffer used and the electrode since the oxidation process of the two compounds is pH dependent because it involves the removal of an H^+ ion as seen in the following oxidation equations.

4. Results and Discussion

4.1. Modification of Platinum Electrode by Bismuth

The platinum electrode modified by Bismuth was analyzed by differential pulse voltammetry. Figure 1 shows the differential pulse voltammograms of irreversibly adsorbed Bismuth atoms on the platinum electrode. The peak observed the change of voltammograms by Bismuth.

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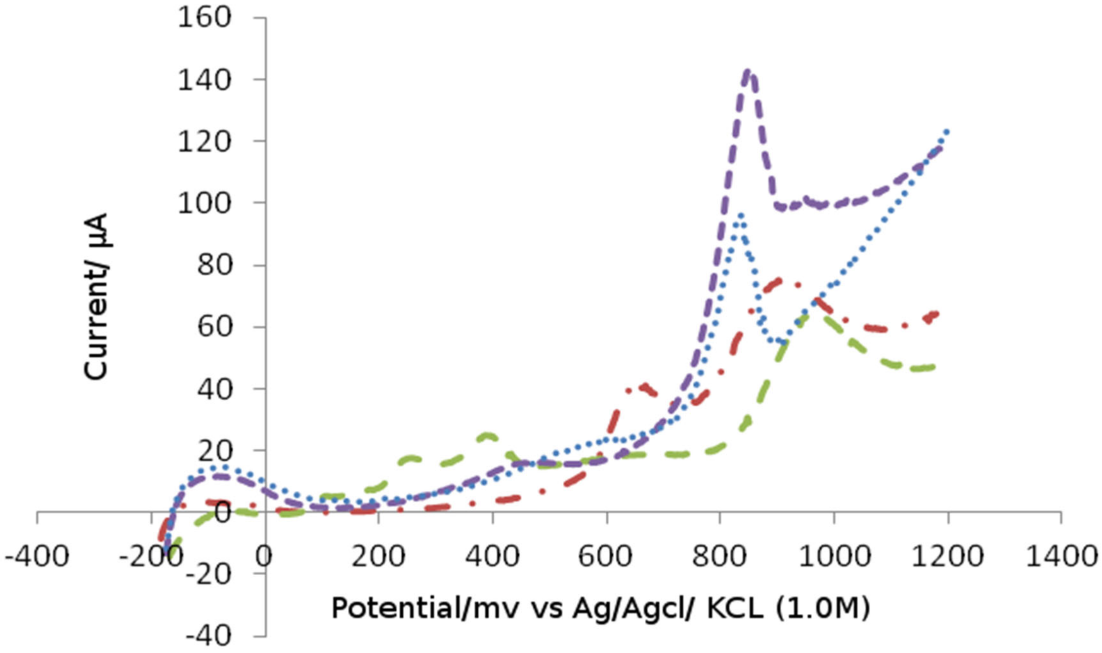

Figure 1. Differential pulse voltammograms platinum electrodes modified by Bismuth, platinum electrode. Scan rate: 10 mV/s.

In Figure 2, four voltammograms were recorded after modification of platinum electrodes by adatoms (Bismuth) in buffer a solution that contains heavy metal. After modification, the peak areas became higher and the sensitivity of the electrode increases.

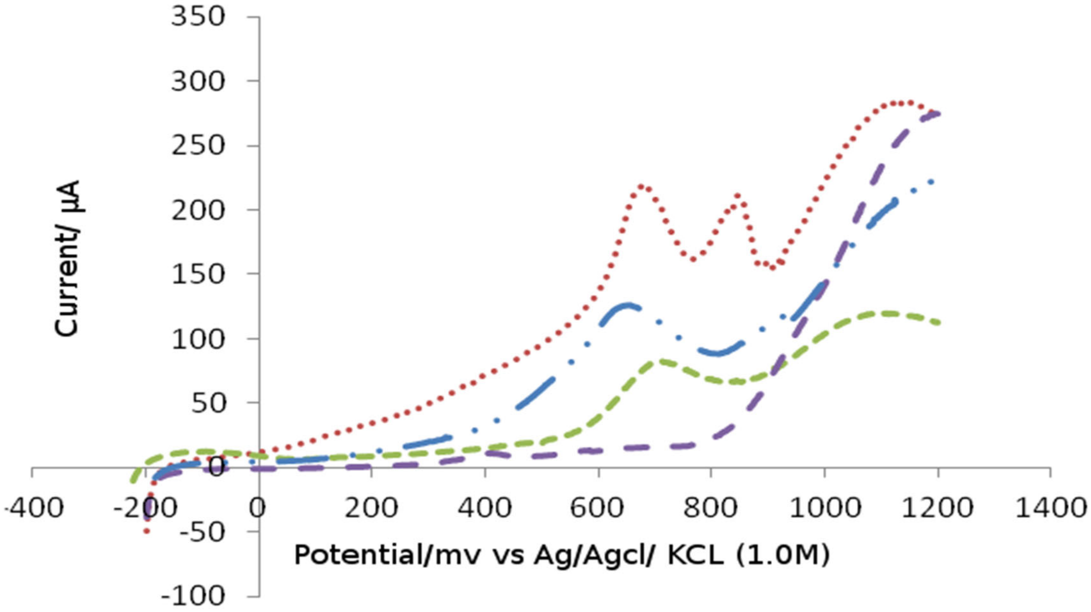

Figure 2. Differential pulse voltammograms platinum electrodes Bidified by Cadmium, platinum electrodes Bidified by Bismuth, platinum electrodes Bidified by Bismuth, and platinum electrode. Experiment parameters: potential 0.7 to 1.2 V, concentration: 1x10^{-3}M, and scan rate: 10 mV/s.

4.2. Calculation of Bismuth Coverage on the Platinum Electrode

Figure 3 explains the relation between the total surface area from differential pulse voltammograms of Bismuth and platinum electrode recorded at 10 mv/s scan rare in the buffer solution and from the bare platinum electrode (Alkhawaldeh, 2020). The area under the peak of

Bismuth and platinum electrode was calculated to be 84 μC. Moreover, the area under the oxidation peak was integrated and the corresponding charge was calculated to be 120 μC:

Figure 3 show the relation between the total surface area from differential pulse voltammograms of platinum electrode Bidified by Bismuth and a platinum electrode. This voltammograms was used for the calculation of the coverage with Bismuth atoms on the platinum electrode.

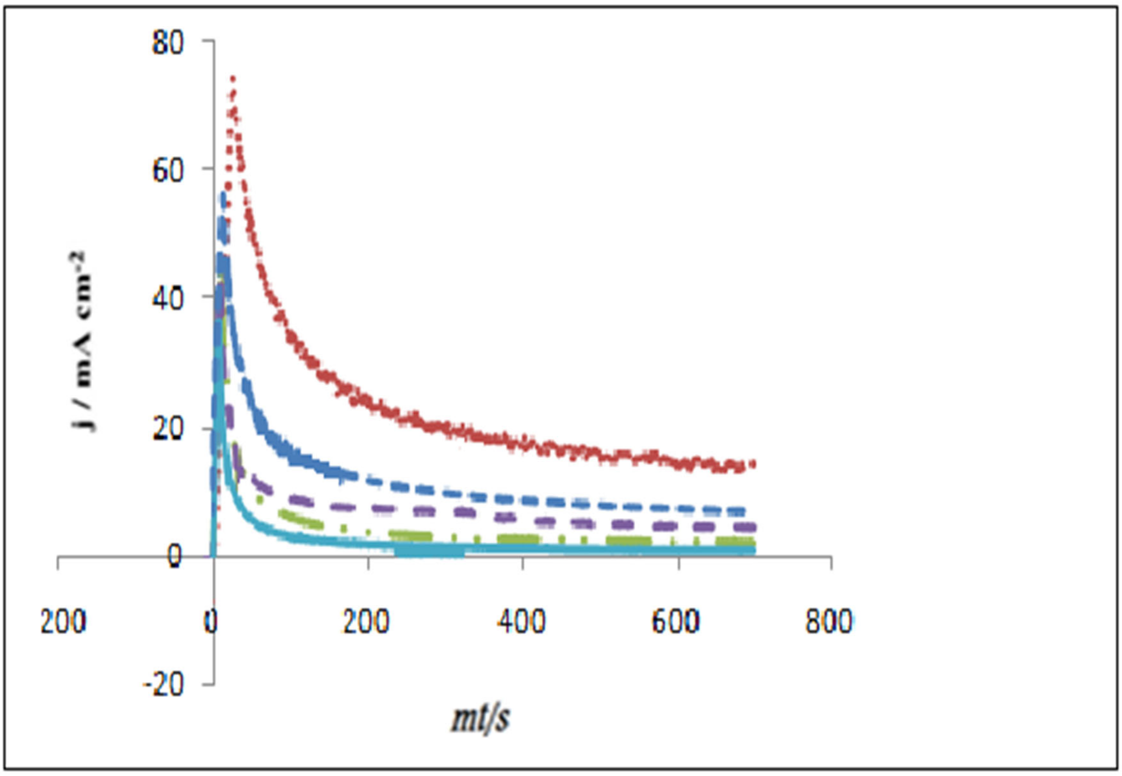

Figure 3. Differential pulse voltammograms of Bismuth Bidified platinum electrode concentration of 1x10^{-3}M and scan rate: 10 mV/s.

4.3. Validation Method of Heavy Metal

Recovery study of heavy metal was checked, as shown in Figure 4. The results were in the acceptable range of recovery from 80% to 120%. Moreover, Figure 7 shows the differential pulse voltammograms of heavy metal in Septrin and Baccarin drug formulations.

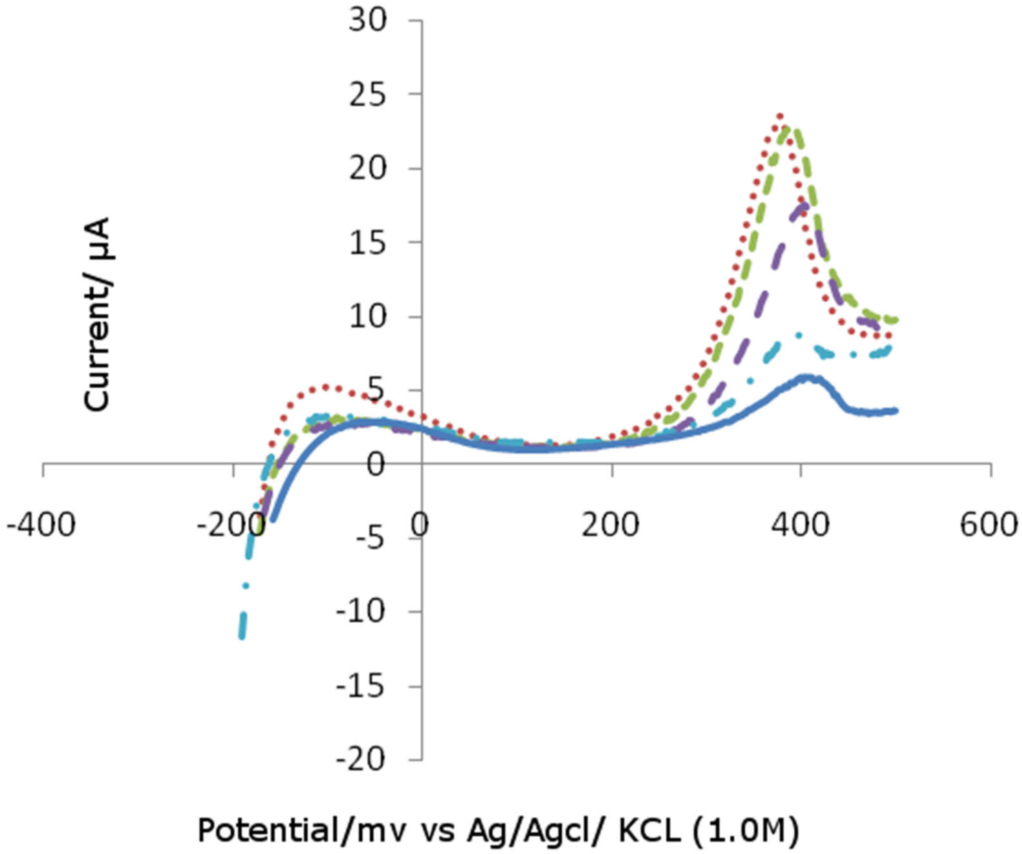

Figure 4. Differential pulse voltammograms of the standard, heavy metal in the solution using Bismuth coated platinum electrode.

5. Conclusions

In the present work, the notion of using a Bismuth -coated platinum electrode for analysis of heavy metal was introduced and applied successfully by differential pulse voltammograms and shows high selectivity to determine these molecules. The major advantage of the present method is the ease of measurement; the Bismuth coating makes the carbon electrode less prone to adsorption and less liable to the effect of impurities. It shows a linear relationship between the current peak and the concentration of heavy metal with detection limits equals 1.44 x10^{-4} M for 4.36 x 10^{-4} M for heavy metal.

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