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Case Study

ELECTROCHEMICAL BEHAVIOUR OF ACETAMINOPHEN ON GLASSY CARBON ELECTRODE MODIFIED WITH BISMUTH COMPOSITES

Iram Yasmin[1], Uzma Sattar[2] and Bin Qi[3]*

[1,3]School of Chemistry, Northeast Normal University
[2]University of Sargodha, Pakistan

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A B S T R A C T

An electrochemical method for the quantitative detection of acetaminophen at a glassy carbon modified with Bismuth composites (Bi/GCE) is presented for the first time. In this research, Bismuth composite were effectively prepared by ultrasonic method, and a best electrochemical sensor-Bi/GCE was formed for analysis of Acetaminophen (AC). Scanning electron microscopy (SEM), transmission electron microscopy (TEM) were used to define the morphology of modified electrode. The electrochemical behavior of AC on the modified electrode was studied using cyclic voltammetry (CV) and differential pulse method (DPV) in pH 7.0 phosphate buffer. The results presented that in the neutral environment, AC had a clear electro catalytic effect on the modified electrode, and the reaction of AC on the modified electrode was controlled by adsorption. Sensor is used to investigate the utility of the proposed method for the determination of Acetaminophen in real samples.

INTRODUCTION

Different solid electrodes have been used in differentvoltammetry methods over the years. Of the many different solid materials that can be used as working electrodes, the best is; gold, carbon and platinum [1-3]. Electrodes based on carbon commonly have large application than the other solid electrodes because of their large use, large surface area, low background current, high stability, low cost and suitability for various sensing and detection applications [4]. However, electron transfer rates study atcarbon surfaces are slow than noble metal electrodes.

The most popular electrode is Glassy carbon because of its best electrical and mechanical properties, good working potential range, greater chemical inertness and relatively reproducible performance [3, 5]. Other good activity such as electrochemical, heat, chemical, or laser treatments have also been used to increase the performance [3, 6]. Among these, the electrochemical activation is more considerable and faster in situ pretreatment process. Recently. Bismuth film electrodes have been studied as an unconventional mercury electrode for determination of trace metals (7-9). Bismuth is an environmentally good material with very low toxicity and have good performance to mercury in stripping analyses.

The best and greatervoltammetry performance of bismuth film modified glassy carbon electrode is due to the fused alloys which they form with heavy metals (10-11).

Corresponding author:* **Bin Qi
School of Chemistry, Northeast Normal University

Bismuth/glassy carbon electrodes were formed on several substrates, including gold, platinum (12,13), carbon materials (14,15), glassy carbon (16,17,18) and carbon fibers by a plating method. Carbon electrodes have been successfully mixed with pure metals and used for electrochemical study. The Electroanalytical performance of these electrodes was based on the properties of the carbon material and electro catalytical characteristics of doped metals (Pt, Pd, Bi, etc.) in oxidation of some biological and organic materials. In compare with these researches, we prepared a bismuth-glassy carbon (Bi/GC) composite electrode in which bismuth powder is used to provide bismuth ions into the supporting electrolyte for the deposition of bismuth on the glassy carbon electrode. The Bi/GCE was electrochemically oxidized and from bismuth powder bismuth ions were produced to the supportive electrolyte. Formation of sensors for the qualitative and quantitative determination of drugs is one of the best methods in this research.

They must to be very specific, accurate with extended durability. Sensitivity and specificity are the leading important factors that help to determine a target sample. From the study of many pharmaceutical analysis, laboratory analysis of the drug shows a significant role in quality control of drug and quality declaration of pharmaceutical preparations and biological solutions (such as serum and urine) in the medical field. The Quality of drugs additional has ahugeinfluence on human health. Acetaminophen (N-acetyl-p-aminophenol; paracetamol) (AC) is a generally arranged and over the best drug used as painkiller and antipyretic all over the world with slightly imperfect anti-inflammatory properties. Usually, AC

does not show any injurious side effects at suggested dosage, however, irregular use of AC, primarily, is related with acute liver and kidney. It is an operative and fine-tolerated agent working to decrease fever and lessen mild-to-reasonable pain related with backache, muscular aches, arthritis, headache, toothache and postoperative pain [19, 20]. Normally, the perfect use of acetaminophen is harmless and has no risky side effect.

Though, overdose or long-term use of acetaminophen may cause adverse effects on health because of the accumulation of toxic metabolites, which can lead to harmful and occasionally fatal hepatotoxicity and nephrotoxicity [21]. Therefore, it is most important to improve simple, fast, sensitive, and accurate analytical methods for the detection of acetaminophen in pharmaceutical preparations and biological fluids.

After study the literature it shows that there are different methods that have been performed for the determination of AC. The studied methods are very classy, advanced and delicate i.e. titrimetric (22), colorimetric (23), spectrophotometry and spectroscopy (24), gas chromatography, near-infrared transmittance spectroscopy, High Performance Liquid Chromatography (25) Present work is good step in providing a highly sensitive detection of AC. Here in a simple Bi/GCE electrode was established for the electrochemical oxidation of AC. The Bismuth composite material shows anoutstandingelectro catalytic activity and electrical conductivity. The Bismuth composite-based sensor showed noticeable sensing ability and selectivity for determination of AC in real samples.

Experimental Design

Reagents

Acetaminophen(AC)was purchased from FLUCK company, Nafion (5 wt%) was supplied by Sigma-Aldrich. though, Bismuthpowder, nitricacid, methanol, ethanola supportive electrolyte K3Fe2(CN6) were purchased from Beijing Chemical Company Limited. The phosphate buffer solution (PBS) is made up of Na_2HPO_4, NaH_2PO_4 and deionized water, and all the reagents were of analytical grade.

Equipment's

All electrochemical measurements and to check the behavior of acetaminophen were carried out by the best three-electrode system with a CHI 660D electrochemical workstation (Changhua, Shanghai, China). The modified glassy carbon electrode with Bismuth composite was used as the working electrode, and Ag/AgCL and a Pt plate with a surface area of 4 cm used as the reference electrode and the counter electrode, respectively.

The scanning electron microscope (SEM, XL-30 ESEM, Philips Company,) and Transmission electron microscope (TEM, JEM-2100F, Japan) was used to detect the morphology.

Preparation of real samples

The acetaminophen was obtained from a FLUCK company. Five tablets (equivalent to 250 mg of acetaminophen in each tablet) of acetaminophen were precisely weighed and excellently powdered in an agate mortar. Asuitable amount of the powder was weighed and distributed in 100 mL of deionized water with ultrasonic sonication for 60 min. Then the mixture was filtrated. After filtration, the filter cake was washed five times with 10mL deionized water, and all filtrates were collected in a 100 mL volumetric flask and diluted to the designated volume with ultrapure water for further analysis.

Preparation of the Sensor

The GCE was polished with Alumina powder and then cleaned carefully with nitric acid (1:1), the distilled water and ethanol is an ultrasonic bath for 10 minutes, respectively. The electrode was preserved in phosphate buffer (pH 7.0) containing 5.0_10_3 mol L Bismuth by cyclic voltammetry between _0.6 and 2.0V. Then the film was washed with ethanol and double distilled water to eliminate physically adsorbed material. After that, the film electrode was transmitted to an electrochemical cell, and electrochemical measurements were carried out.

Characterization of Bi/GCE Electrode

The dissolution of bismuth composite was performed at 0.3 V (Ag/AgCl) for 1 min in 0.1 M phosphate buffer solution (pH.7). For the reduction of the dissolved bismuth ions, acathodic potential of -1.2 V (Ag/AgCl) was applied to the working electrode for 5 min, without stirring. After deposition of the bismuth, Acetaminophen solutions were added to the cell as required, and a preconcentration potential of -1.2 V was applied to the working electrode for 5 min while stirring. After an equilibration time of 10 s, square wave anodic stripping voltammograms were recorded between -1.2 V and -0.2 V without stirring. [Fig.2.5] For repetitive measurements, the electrode was cleaned at -0.3 V for 30sunder stirring conditions to remove the residual Bismuth ions. Atroom temperature experiment was done.

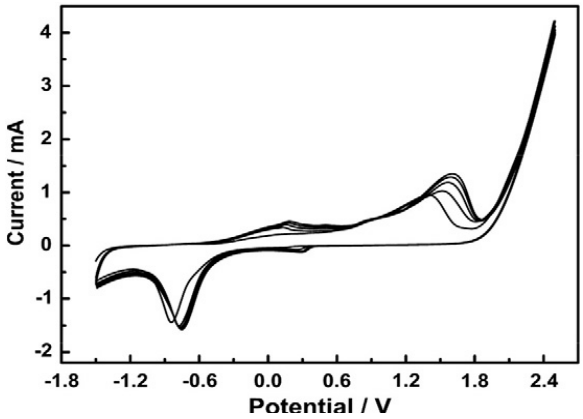

Fig 1 Cyclic voltammograms of 1.0x1-3M AC in pH 7 phosphate buffer at Sscan rate(-1.2v and -0.2)

RESULTS AND DISCUSSION

Morphological characterization of ELECTRODE

The morphological characterization of RGO Bi/GCE was studied out by using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). As can be seen in Fig. 1 (A), shows the bare surface of glassy carbon electrode. Fig. 2 (B) indicate the morphology of Bi/GCE, which is describing that a large number of spindles or rod shapestructure of Bi composite were incorporated on the surface of GCE which is very beneficial for the adsorption of acetaminophen The size of partials are about 100nm. Fig. 3 (C) this structure is advantageous to offer a large surface area. Such structures prevent the aggregating of GCE and improve the conductivity of Bismuth film, which is advantageous for enhancing the electrochemical detection of AC.

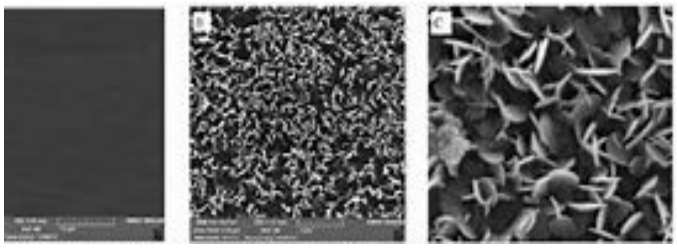

Figure 2 a) SEM image, bare GCE b) SEM image, Bi/GCE and (c) TEM image of Bi/GCE composite

Procedure for Electrochemical Measurements

15mL phosphate buffer (pH 7) containing a specific amount of acetaminophen was added to an electrochemical cell. Electrochemical measurements were carried out after 2min accumulation under the open-circuit conditions while stirring, and cyclic voltammograms were recorded in the potential range of _0.2–2.0V at a scan rate of 0.2Vs_2 after 10s quiet time. Electrochemical behavior of acetaminophen at the modified electrodes was studied. The electrochemical behavior of 0.1 mM acetaminophen in 0.1 MPBS (pH 7.0) at the bare GCE and the modified glassy carbon electrodes were studied by cyclic voltammetry. As shown by (curve A) in Fig. 2, the CVs obtained at the bare GCE display an irreversible oxidation peakwith quite small peak current. In contrast, the CVs of acetaminophenat the 4-ABA/GCE (curve b) show a pair of well-defined redox peaksin potential range between 0.1 V and 0.6 V, with the greatly enhancedpeak currents. The appearance of the reversible redox peaks indicates that Bismuth composite film can significantly accelerate the oxidation reaction process.

At the same time, it is also observed that Bi/GCE can catalyze moreeffectively the oxidation process of acetaminophen through promoting the rate of electron transfer between the electrode and species in the electrolyte, which is exemplified by a pair of reversible redox peaks with the larger peak currents (curve B). It is worth noting that, the peak currents obtained at modified electrode is the higher values in comparison with those obtained at the bare GCE.

The comparison of voltammetry results clearly demonstrates that Bi/GCE composite films exhibit good conductivity, prominent electronictransport property, and excellent catalytic activity towards the oxidation acetaminophen.

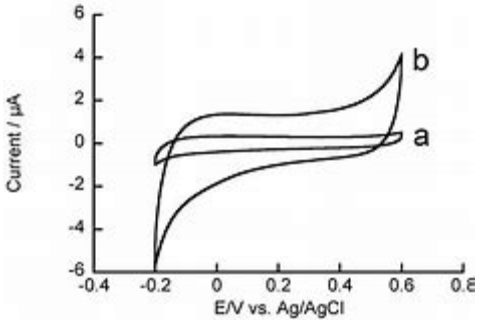

Fig 3 Cyclicvoltammetry behavior of acetaminophen (a) bare Glassy carbon electrode and (b) Bi/GCE in phosphate buffer(pH 7.0) at -0.2-0.6 Scan rate

Quantitative determination of AC

The scope of enhancement in the electrochemical response of Bi/GCE was examined through the determination of AC in 0.1 M PBS (pH7) using DPV technique. From Fig. 3, the peak current was found to increase linearly with the successive addition of AC.

To evaluate this feature, measurement parameters were optimized and a calibration curve was constructed. Figure3.3 shows the square wave voltammograms obtained for different concentrations of acetaminophen. The electrical signals of AC gradually increase as their concentration increases, and the peak current and Acetaminophen concentration showed a good linear relationship in the range of 6μM~120μM, the equation of linear regression as (Y=0.383+92.8μM) and coefficient of correlation R=(0.9996), and detection limit is 3.2 x 10^{-7}mol/l (s/n=3). This indicates that the AC has a significant co-operation effect to electrochemical sensor.

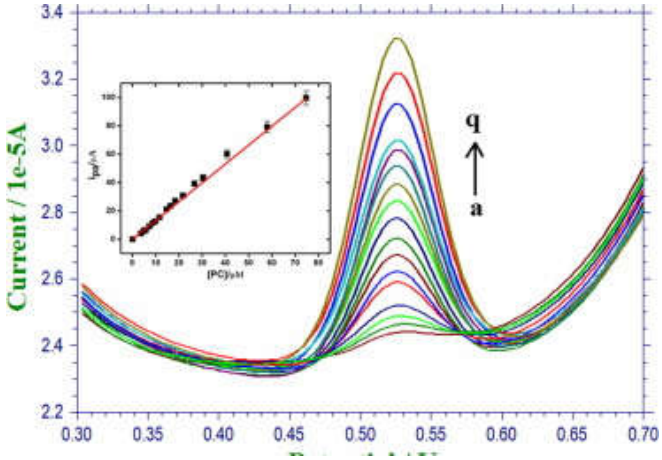

Fig 4 linear Volta gram is obtained at different concentration of acetaminophen

Stability and selectivity of the electrochemical sensor

In order to investigate the selectivity of this method for AC detection, I-t measurements were performed on similarly structural compound and some inorganic ions in organisms . first, Fixed acetaminophenconcentration as 0.9μM in the pH=7.0 pbs and then continue to add Ba^{2+}, Zn^{2+}, Al^{3+}, Bi^{3+}, and Ni^{3+}, their concentration is 100 times AC .in the fig 5,we found that there is no inference ,When 10 μL acetaminophen was added, the oxidation peak current continued to increase. The results show that the modified electrode has good selectivity, the use of the modified electrode for detecting 10μMAC about 10 times that use the same technique, the relative standard deviation (RSD) was 2.80%, the results show that the modified electrode has good reproducibility .and then The Bi/GCE should be save at 4 ℃ a week, that modified electrode were determined10μMAC by DPV, the oxidation current can still reach 94% of the original current, the results show that the modified electrode has good stability .

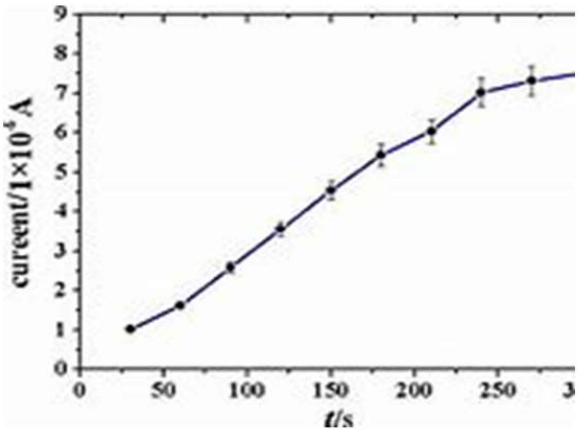

Fig 5 stability and selectivity curve at different concentration of acetaminophen

CONCLUSION

Amodified electrode Bi/GCE, was invented by modifyingthe surface of the glassy carbon electrode with bismuth composite film by cyclic voltammetry for electrochemicalstudy of acetaminophen. The Bi/GCE exhibits an excellentelectrocatalytic activity towards the oxidation of acetaminophen. Voltammetry determination of acetaminophen at the Bi/GCE presents lower detection limit and wider linear range. Moreover, acetaminophen can be selectively determined without the interference of nickel and other trace elements by using the Bi/GCE. The as-prepared electrodeis employed to the determination of acetaminophen in real samples and the satisfying results are achieved. Therefore, Bi/GCEprovides a new way to determine acetaminophen sensitively and selectively in real samples.

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