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Electrochemical Determination of Sulfapyridine using a New Approach of Modified Electrode based on Amplification with Room Temperature Ionic Liquid and ZnO Nanoparticle

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Abstract- An electroanalytical sensor amplified with ZnO nanoparticle (ZnO NPs) and 1-butyl-2,3-dimethylimidazolium hexafluorophosphate (1B23DIPF6) was fabricated as new analytical approach for determination of sulfapyridine in different real samples. The ZnO NPs was synthesized by a simple precipitation method with low calcination temperature (~ 300 °C) and characterized by XRD and FESEM methods and results confirm a particle size ~45.2 nm for ZnO NPs. In addition, the two-fold amplification of paste electrode (PE) with ZnO NPs and 1B23DIBr (PE-ZnO NPs-1B23DIPF6) created a new tool for determination of sulfapyridine in the concentration range of 0.09-400 µM with detection limit 30.1 nM. On the other hand, the PE-ZnO NPs-1B23DIPF6 showed recovery data between 99.4%-104.1% for determination of sulfapyridine in some different real samples such as urine and tablet.

Keywords- Sulfapyridine, ZnO nanoparticle, 1-butyl-2,3-dimethylimidazolium hexafluorophosphate, Electrochemical sensors

1. INTRODUCTION

Nanoscience has developed a new approach to various scientific debates and industrial applications in the new years that can be described as the scientific revolution [1-5]. The high growth of nanomaterials in human daily life is beyond doubt [6-10]. Due to the flexibility of nanomaterials for different applications and the possibility of creating different composites, many sciences use it [11-15]. Electrochemistry is one of the main sciences that has made good use of nanotechnology capabilities [16-20]. Different branch of electrochemistry filed such as super-capacitors, battery, oxygen reduction reaction and electrochemical sensors were used nanomaterials [21-30]. Many of nanomaterials especially metal-based nanomaterials showed high electrical conductivity that is very important factor for selection a conductive mediator in sensors construction filed [31-40]. As examples, copper oxide decorated reduced graphene was suggested by our group for modification of working electrode and results showed a powerful ability of sensor for determination of cholesterol (CL), ascorbic (AA) acid and uric acid in biological samples [41]. In addition, the simultaneous application of nanomaterials and other intermediates in other strategy for modification of selective and high sensitive electrochemical sensors [42-50].

Sulfapyridine is a sulfanilamide antibacterial that usually prescribed to treat linear IgA disease [51]. The high level of sulfapyridine is very dangerous for human body due to its behavior is dependent for pH of solution and it possible that crystallization within the bladder or urethra [52]. This point created a major problem for kidney health and determination its concentration is very important with portable kits.

In this research, we tried for fabrication of high sensitive sulfapyridine electrochemical sensor using synthesized ZnO nanoparticle coupled ionic liquid (1-butyl-2,3-dimethylimidazolium hexafluorophosphate in this case) modified carbon paste electrode as electroanalytical sensor. The fabricated sulfapyridine electrochemical sensor showed many advantage such as low cost and good limit of detection. Also, the sulfapyridine electrochemical sensor was showed acceptable results for determination of drug in real samples.

2. EXPERIMENTAL

2.1. Chemicals and apparatus

The analytical grade chemical materials were used in electrochemical investigation and synthesis procedure. Sulfapyridine, zinc nitrate hexahydrate, sodium hydroxide and paraffin oil were purchased from Sigma-Aldrich. 1-butyl-2,3-dimethylimidazolium hexafluorophosphate was purchased Alfa Aesar. Phosphoric acid and graphite powder were purchased from Merck.

Cyclic voltammetry, differential pulse voltammetry and chronoamperogramic studies were carried out using μ-Autolab (The Netherlands) and results analysis by Nova software. In all of

the voltammetric investigation the PE-ZnO NPs-1B23DIPF6, Ag/AgCl/KCl_{sat} and Pt wire were used as working, reference and auxiliary electrodes. The XRD model X' Pert Pro was used for ZnO nanoparticle characterization.

2.2. Synthesis of ZnO nanoparticle

A simple precipitation method using zinc nitrate hexahydrate as a metal Precursor was used for synthesis of ZnO nanoparticle. The 0.5 M zinc nitrate hexahydrate was prepared in volumetric flask (50 mL) and stirred for 30 min at room temperature. In continuous, 50 mL sodium hydroxide (1.0 M) was added to solution under stirred condition. The precipitated sample was separated by filter paper and then dried at 100 °C for 12 h. the dried samples was calcinated at 300 °C for 2 h.

2.3. Preparation of modified electrode

The PE-ZnO NPs-1B23DIPF6 was prepared by mixing the graphite powder, ZnO nanoparticle, 1B23DIPF6 and mineral oil in an agate mortar. The mixing sample hand mixing for 2 h and the resulting paste was filled into end of glass tube. The copper wire was used as an electrical contact to instrument.

2.4. Real sample preparation

Seven tablet samples was powdered in agate mortar and dissolved into water solution in erlenmeyer flask. The sample was filtered and then dilute for preparation of sulfapyridine specific concentration. Urine sample was prepared according to our previous report published paper [30].

3. RESULTS AND DISCUSSION

3.1. ZnO nanoparticle characterization

Before modification of suggested senor, we characterized the structure and diameter of ZnO nanoparticle by XRD and FESEM methods. The results relative to XRD investigation are presence in Figure 1 A and clear show the presence of 11 planes with miller indexes (100); (002); (101); (102); (110); (103); (200); (112); (201); (004) and (202) that are similar for ZnO nanoparticle with JCPDS 043-0002. The diameter of ZnO nanoparticle was determined ~45.2 nm using Scherrer equation and using data obtained at plan with miller index (101). On the other hand, the FESEM data presence in Figure 1B confirm synthesis of ZnO nanoparticle in spherical format and shape.

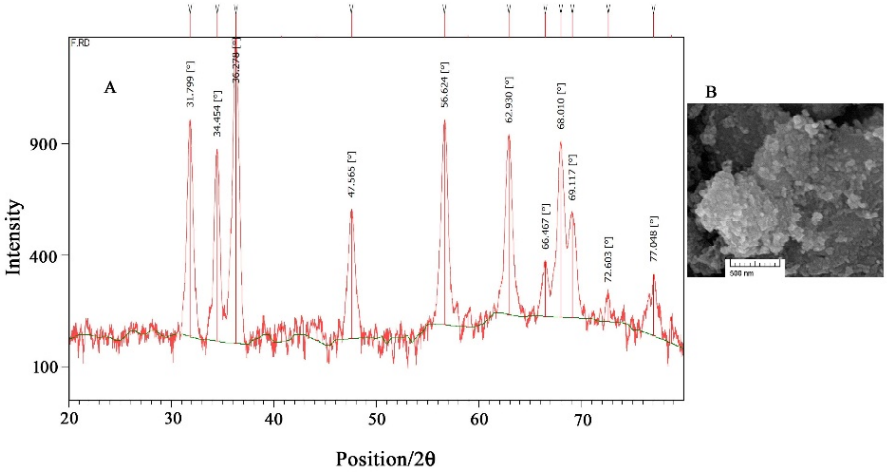
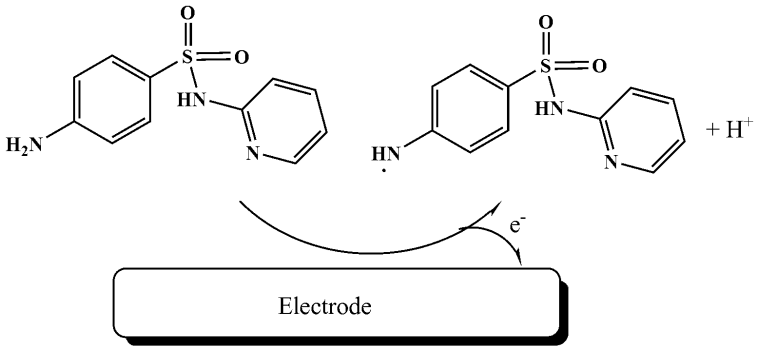


Fig. 1. XRD patter (A) and FESEM image (B) of ZnO nanoparticle

3.2. Electrochemical behavior of sulfapyridine at surface of PE-ZnO NPs-1B23DIPF6

The redox behavior and oxidation signal of sulfapyridine was recorded at surface of PE-ZnO NPs-1B23DIPF6 in the pH range 4.0-8.0 (Figure 2 inset). It is clear that oxidation signal of sulfapyridine shifted to negative value with moving pH=4.0 to pH=8.0. A linear plot with slope 73 mV/pH (Figure 2) was detected for this changing and this value confirm equal value of H^+ and e^- in electro-oxidation mechanism of sulfapyridine (scheme 1). The comparison of oxidation signals in relative voltammograms showed that maximum oxidation signal was occurred at pH=7.0 that selected as bets condition in this study.

The cyclic voltammograms of 700.0 µM sulfapyridine was recorded at surface of PE (Figure 3 curve a); PE-ZnO NPs (Figure 3 curve b); PE-1B23DIPF6 (Figure 3 curve c) and PE-ZnO NPs-1B23DIPF6 (Figure 3 curve d), respectively. The improving in current from 84.2 µA to 271.2 µA and also reduce of oxidation potential from 1060 mV to 995 mV confirm powerful ability of ZnO NPs and 1B23DIPF6 as mediators and ability of sensor for trace level analysis of sulfapyridine.



Scheme 1. Electro-oxidation mechanism of sulfapyridine

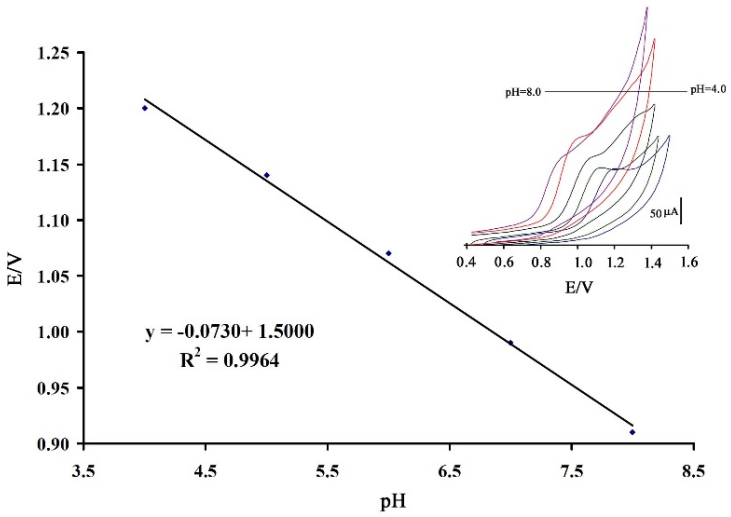


Fig. 2. Potential-pH curve for oxidation sulfapyridine at surface of PE-ZnO NPs-1B23DIPF6. Insert) The cyclic voltammograms of sulfapyridine at surface of PE-ZnO NPs-1B23DIPF6 in different pH ranges

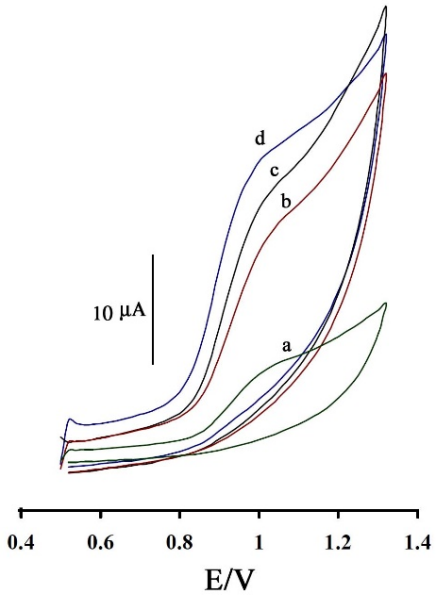


Fig. 3. The linear sweep voltammograms of 700 µM sulfapyridine at surface of PE (a); PE-ZnO NPs (b); PE-1B23DIPF6 (c) and PE-ZnO NPs-1B23DIPF6 (d) at pH=7.0

In addition, sulfapyridine showed a diffusion process oxidation at surface of PE-ZnO NPs-1B23DIPF6 due to linear relation between oxidation current of sulfapyridine an $\nu^{1/2}$ according to our recording data (see Figure 4).

After confirming the diffusion process for sulfapyridine at surface of PE-ZnO NPs-1B23DIPF6, the diffusion coefficient (D) value of sulfapyridine was determined by recording chronoamperograms of 300 and 400 µM drug using applied potential 1100 mV (Figure 5A). Using Cottrell equation and slopes of Figure 5B data, the mean value of D was determined ~3.77×10^{-5} cm^2/s.

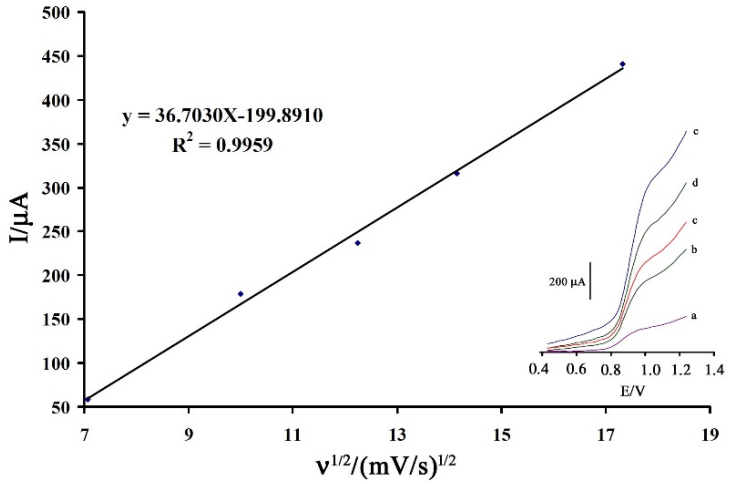


Fig. 4. Current-$\nu^{1/2}$ curve for electro-oxidation of sulfapyridine at surface of PE-ZnO NPs-1B23DIPF6. Insert) The linear sweep voltammograms of sulfapyridine at surface of PE-ZnO NPs-1B23DIPF6 at scan rates a) 50.0; b) 100.0; c) 150.0; d) 200 and e) 300 mV/s

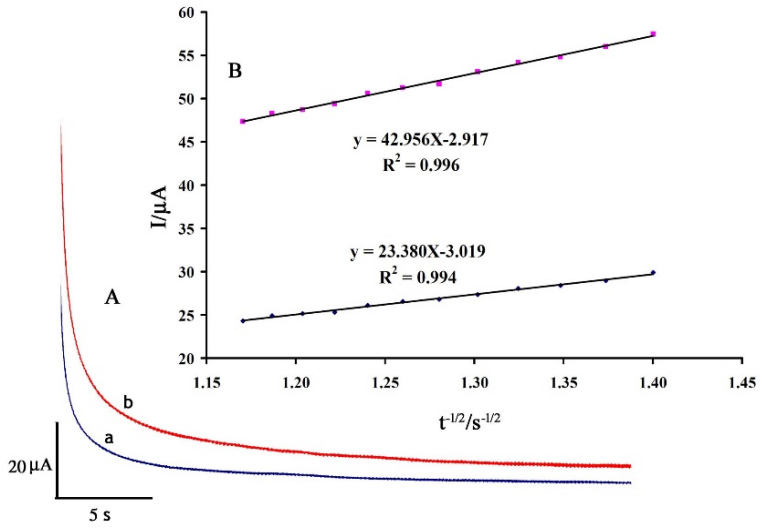


Fig. 5. Chronoamperograms obtained at PE-ZnO NPs-1B23DIPF6 in the presence of a 300, b and 400 µM sulfapyridine. Inset: Cottrell's plot from sulfapyridine

The analysis of sulfapyridine was carried out at surface of PE-ZnO NPs-1B23DIPF6. As the concentration of the sulfapyridine increases the oxidation signal increases linearly (Figure 6) in the concentration range 0.09 to 400.0 µM with regression equation: I_{pa} (µA)=0.1101 $C_{sulfapyridine}$+0.7838; R^2=0.9987. The limit of detection (LOD) for determination of sulfapyridine was found to be 30.1 nM.

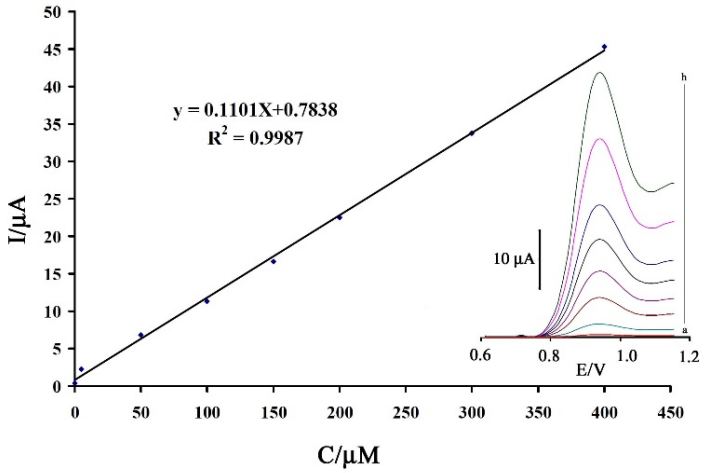


Fig. 6. Current-concentration curve for electro-oxidation of sulfapyridine at surface of PE-ZnO NPs-1B23DIPF6. Inset) differential pulse votammograms of different concentration (0.09-400 µM) sulfapyridine at surface of PE-ZnO NPs-1B23DIPF6

For successful examination of sulfapyridine on PE-ZnO NPs-1B23DIPF6, it is necessary to evaluate interfering species can be presence in the real samples containing investigated drug. Various vitamins such as vitamin B2, vitamin B6 and ionic species (potassium bromide, sodium chloride, potassium chloride, magnesium nitrate) and also biological samples (methionine, ascorbic acid and glucose) were added to the solution containing to 20.0 µM sulfapyridine to check the selectivity of sensor. The listed compounds don't show any important interference for determination of sulfapyridine at surface of PE-ZnO NPs-1B23DIPF6 that confirm good selectivity of suggested sensor.

The PE-ZnO NPs-1B23DIPF6 successfully employed for the study of sulfapyridine present in the drug and biological samples using DPV. Table 1 shows the percent recoveries of sulfapyridine and were found to be 99.4% and 104.1%, respectively. Hence PE-ZnO NPs-1B23DIPF6 exhibited the good analytical applicability towards the investigation of sulfapyridine present in the drug and biological sample.

Table 1. The analytical performance of ZnO NPs-1B23DIPF6 for determination of sulfapyridine in real samples (n=4)

Sample	Added (µM)	Expected (µM)	Founded (µM)	Recovery (%)
Tablet	---	---	5.11±0.34	---
	10.00	15.11	15.02±0.87	99.4
Urine	---	---	<LOD	---
	15.00	15.00	15.61±0.87	104.1

4. CONCLUSION

In the presence research, we tried to fabricate a high powerful and new electroanalytical sensors entitle PE-ZnO NPs-1B23DIPF6 that amplified by ZnO nanoparticle and 1-butyl-2,3-dimethylimidazolium hexafluorophosphate. The PE-ZnO NPs-1B23DIPF6 showed a powerful ability and good sensitivity for determination sulfapyridine in drug samples. The PE-ZnO NPs-1B23DIPF6 showed a good limit of detection ~30.1 nM for determination of sulfapyridine. The PE-ZnO NPs-1B23DIPF6 was successfully used as new analytical tool for determination of sulfapyridine in real samples.

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