Chemical Methodologies 4(2020) 720-731

Chemical Methodologies

Journal homepage: http://chemmethod.com

Original Research article

Fabrication of Sulfapyridine Electrochemical Sensor Amplified with CuO/SWCNTs as High Performance Electroanalytical Tool in Real Sample Analysis

Shirin Shahraki[a], Mahboubeh Masrournia[a,*], Hassan Karimi-Maleh[b]

[a] Department of Chemistry, Mashhad Branch, Islamic Azad University, Mashhad, Iran

[b] Department of Chemical Engineering, Laboratory of Nanotechnology, Quchan University of Technology, Quchan, Islamic Republic of Iran

ARTICLE INFORMATION

Received: 10 May 2020
Received in revised: 21 July 2020
Accepted: 14 August 2020

DOI: 10.22034/chemm.2020.113387

KEYWORDS

Sulfapyridine
CuO/SWCNTs composite
Modified electrode
Electrochemical sensor

ABSTRACT

Sulfapyridine is one of the most important antibiotics drug with some side effects and determination of this drug is very important in biological and pharmaceutical samples. In this research, an electrochemical amplified sensor was fabricated as analytical tool for determination of sulfapyridine in drug samples. For fabrication of sulfapyridine sensor, a carbon paste electrode (CPE) was amplified with copper oxide decorated single wall carbon nanotubes (CuO/SWCNTs) as conductive mediator. The CuO/SWCNTs/CPE was used for voltammetric determination of sulfapyridine and results showed an increasing in oxidation current sulfapyridine about 2.35 times and decreasing 80 mV in oxidation potential in the same condition. The cyclic voltammetric (CV) investigation confirm pH dependent redox reaction for sulfapyridine in the presence of one electron and one proton with maximum sensitivity at pH=7.0. Analytical investigation showed a linear dynamic range 50 nM- 400 µM with detection limit 10 nM. In the final step, CuO/SWCNTs/CPE was used for determination of sulfapyridine in tablet, drinking water and dextrose saline sample with acceptable recovery data between 99.13% - 103.35%.

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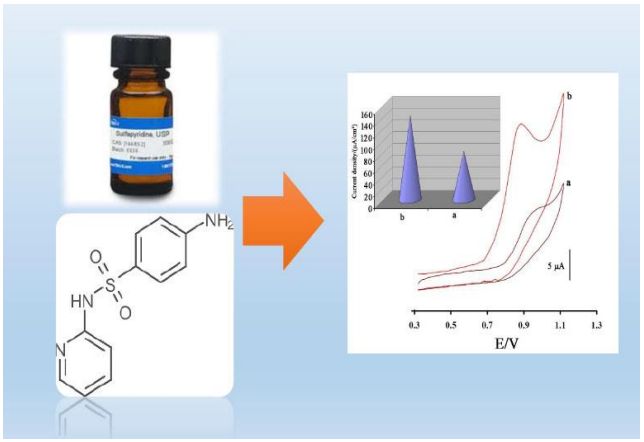
*Corresponding author: E-mail: mah.masrour@gmail.com, Department of Chemistry, Mashhad Branch, Islamic Azad University, Mashhad, Iran, Tel: +9851 3525 1317

Graphical Abstract



Introduction

Sulfapyridine is one of the sulfonamide antibiotic category drugs that divided in bacteriostatic antibiotics drug categories [1-3]. Sulfapyridine is used to treat gram-positive and negative bacteria [4]. There is an important risk of agranulocytosis after taking of sulfapyridine and determination of this drug in biological samples are very important [5]. Therefore, many of research works focused on design and fabrication high sensitive analytical tools for determination of sulfapyridine in real samples [6-10].

In between of analytical approach in determination of sulfapyridine, electroanalytical systems showed more advantages between researchers due to many advantages of them such as easy fabrication, non-toxic analysis procedure and low cost [11-20]. On the other hand, the possibility of modifying and increasing the efficiency of electrochemical sensors using different intermediaries has created a high diversity for the use of this type of analysis system [21-30]. In recent years, electrochemical sensors have rapidly replaced other analytical methods in the analysis of various pharmaceutical and food compounds [31-40]. Nanomaterials such metal nanoparticles, carbon-based nanostructures, quantum dots and nanoporous created a new approach in all of sciences [41-52]. High conductivity of nanomaterials is one of important properties of them that helps nanomaterials for using in improving sensitivity of electrochemical sensors [53-62]. In between, metal oxide decorated CNTs showed high conductivity and very interesting catalytic effect for fabrication of electrochemical sensors due to high surface area and high conductivity of CNTs [17, 63].

In this research, a new electrochemical sensor design and fabricated as analytical tool for determination of sulfapyridine as an antibiotic drug. For amplification of sensor, CuO/SWCNTs was

synthesized by a simple precipitation method and results showed powerful ability of synthesized nanocomposite for improving sensor ability in analytical application. Compared to CPE, the CuO/SWCNTs/CPE facilitated the electron transfer rate in the drug redox process and was used as a high-conductivity sensor in the analysis of the sulfapyridine in the real sample.

Experimental

Instruments and compounds

Sulfapyridine with high purity (>99%) was purchased from Sigma-Aldrich and used for preparation stock solution (0.01 M) by dissolving 0.025 g drug powder into 10 mL phosphate buffer solution (PBS) pH=7.0. Copper(II) acetate (98%), acetic acid (>99%), sodium hydroxide (97%) was purchased from Merck and SWCNTs-COOH was purchased from Sigma-Aldrich and used for synthesis of CuO/SWCNTs nanocomposite. Phosphoric acid was purchased from acros and used for preparing of PBS. For electrochemical investigation the ivium-vertex instrument was used and all of reported data are based Ag/AgCl/KCl potential as reference electrode. The CuO/SWCNTs nanocomposite was characterized by FESEM instrument model Mira-3-XMU.

Synthesis of CuO/SWCNTs nanocomposite

For synthesis of CuO/SWCNTs nanocomposite, 2.5 gr SWCNTs-COOH + 2 mL acetic acid + 600 mL copper(II) acetate (0.2 M) was added into beaker ultrasonic bath for 45 min. The mixture was heated at 100 °C under stirring condition for 45 min. After boiling of solution, 30 mL sodium hydroxide (8.0 M) was added to beaker and heating was continuous for 2 h. The sample was filtered and dried at 70 °C for 24 h.

Fabrication of CuO/SWCNTs/CPE

For fabrication of CuO/SWCNTs/CPE, 80 mg CuO/SWCNTs nanocomposite + 920 mg graphite powder were mixed in mortar and pestle in the presence of 15 mL diethyl ether. Solvent evaporated in room temperature and obtained mixture was convert to paste by adding 13 drop paraffin oil [63].

Real sample preparation

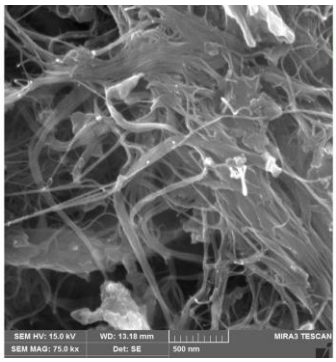
Dextrose saline and drinking water were spiked by different concentration of sulfapyridine and directly used for real sample analysis. The sulfapyridine tablet was powder in mortar and pestle and dissolved in PBS. After filtration, the solution was diluted by PBS and used for real sample analysis.

Results and discussion

Characterization of CuO/SWCNTs nanocomposite

The morphological properties and elemental ratio of synthesized CuO/SWCNTs nanocomposite was characterized by FESEM and EDS methods (Figure 1a-b).

A



B

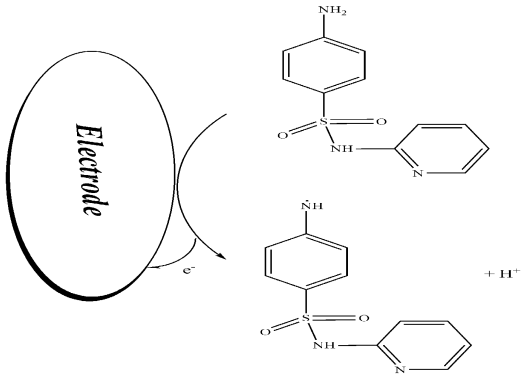
Elt	Line	Int	Error	W%
C	Ka	162.0	3.5348	46.93
O	Ka	96.4	3.5348	20.99
Cu	Ka	133.0	0.4995	32.08
				100.00

Figure 1. FESEM (A) and EDS (B) results relative to synthesized CuO/SWCNTs nanocomposite

FESEM image is presence in Figure 1a. and clearly confirm decoration of CuO nanoparticles at surface of SWCNTs. On the other hand, Figure 1b. data clearly confirm high purity of CuO/SWCNTs nanocomposite due to presence of C, O and Cu elements.

Electrochemical investigation

Scheme 1. shows redox reaction of sulfapyridine at surface of electrode [64]. According to this Scheme, pH of solution is one of the important factor in electrochemical response of sulfapyridine. Therefore, pH factor was optimized by recording cyclic voltammograms of 500 µM sulfapyridine in the 5.0<pH<9.0 (Figure 2 inset). The negative shift in oxidation potential of sulfapyridine with linear equation E = -0.0632 pH + 1.3212 (R^2 = 9971) confirm suggested mechanism in Scheme 1.



Scheme 1. Suggested mechanism for redox reaction of sulfapyridine

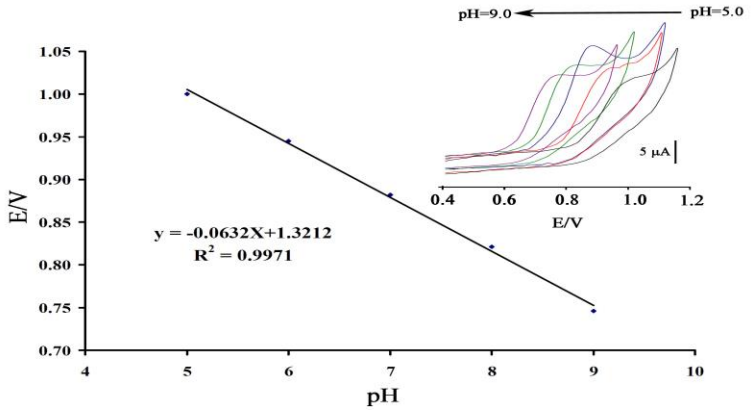


Figure 2. E-pH curve for electro-oxidation of 500 µM sulfapyridine. Inset) CV 500 µM sulfapyridine at different pH values

Redox signal of sulfapyridine was recorded at surface of CPE (Figure 3 curve a) and CuO/SWCNTs/CPE (Figure 3 curve b) and oxidation current and potential of sulfapyridine compared at surface of two electrodes. Oxidation currents 10.9 µA and 25.7 µA with oxidation potential 960 mV and 880 mV were detected for electro-oxidation of sulfapyridine at surface of CPE and CuO/SWCNTs/CPE respectively. As can be seen, with modification of CPE with CuO/SWCNTs nanocomposite the oxidation current of sulfapyridine was improved 2.35 times and oxidation potential of sulfapyridine was facilitate about 80 mV to negative values, respectively.

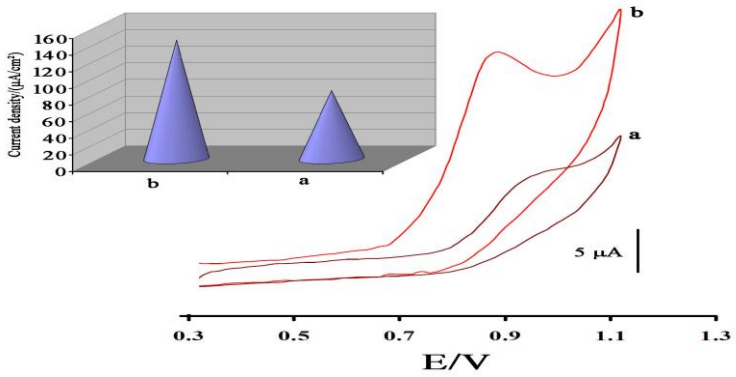


Figure 3. Cyclic voltammogram of 500 µM sulfapyridine at surface of a) CPE and b) CuO/SWCNTs/CPE. Inset, relative current density data

On the other hand, with modification of CPE with CuO/SWCNTs nanocomposite, active surface area (using solution containing $[Fe(CN)_6]^{3-/4-}$ and Randles–Sevcik equation) of CPE was increased from 0.14 cm^2 to 0.18 cm^2 and conductivity results are presence in Figure 3. inset (Current density data). This point confirm high conductivity of nanomaterials in modification of bare electrodes [65-74]

The linear sweep voltammograms of 500 µM sulfapyridine with scan rates 15.0, 30.0, 50.0 and 100 mV/s were recorded at surface of CuO/SWCNTs/CPE (Figure 4). A linear relation with equation I = 3.5025 $\nu^{1/2}$ + 10.4600 (R^2 = 0.9931) confirm diffusion control process [75-83] for electro-oxidation of sulfapyridine at surface of CuO/SWCNTs/CPE.

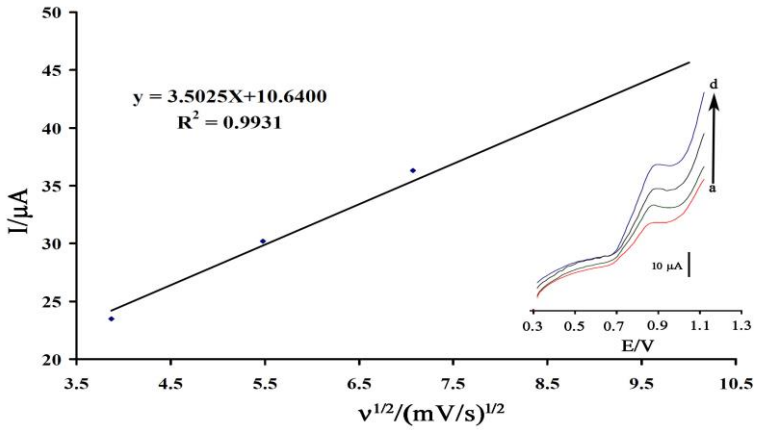


Figure 4. I- $\nu^{1/2}$ curve for electro-oxidation of 500 µM sulfapyridine. Inset) Linear sweep voltammograms of 500 µM sulfapyridine with scan rates a) 15.0, b) 30.0, c) 50.0 and d) 100 mV/s

The value of diffusion coefficient (D) of sulfapyridine at surface of CuO/SWCNTs/CPE was calculated by recording chronoamperograms of 300 µM, 400 µM and 500 µM sulfapyridine (Figure 5a). Using Cottrell equation and recording slopes in Figure 5b, the value of D was calculated 5.93 × 10^{-5} cm^2/s.

Stability of CuO/SWCNTs/CPE was check in the presence of 500 µM sulfapyridine. Results showed a decreased about 90% in initial signal of 500 µM sulfapyridine after 30 days that confirm good stability of sensor for application in one month.

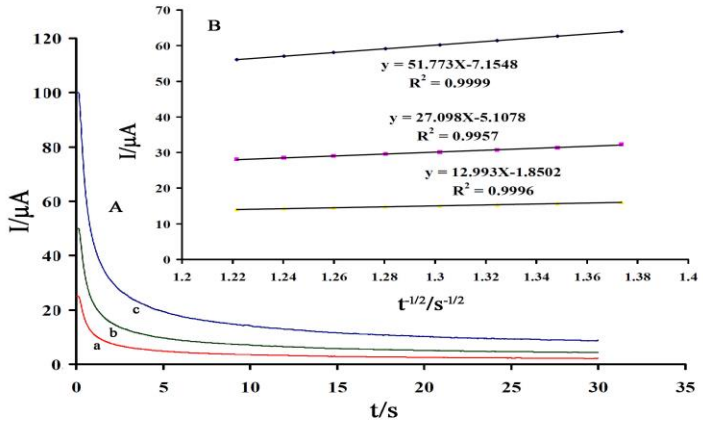


Figure 5. Chronoamperograms a) 300 µM, b) 400 µM and c) 500 µM sulfapyridine at surface of CuO/SWCNTs/CPE with applied potential 950 mV. Inset: Relative Cottrell's plots

Square wave voltammetric investigation showed a linear dynamic range (LDR) 50 nM- 400 µM for determination of sulfapyridine using CuO/SWCNTs/CPE as electroanalytical sensor with equation I = 0.1716 C + 1.0507 (R^2 = 0.9952) and detection limit (LOD) of 10.0 nM (according to the definition of $Y_{LOD} = Y_B + 3s_b$) [84]. These values of LOD and LDR compare with previous suggested electrochemical sensor in Table 1. and results display high performance ability of CuO/SWCNTs/CPE for determination of sulfapyridine compare to pervious suggested sensors.

The reproducibility of CuO/SWCNTs/CPE was examined by replicate measurements of sulfapyridine by SWV method.

Table 1. LOD and LDR results relative to suggested electrochemical sensors for determination of sulfapyridine

Electrode	Mediator	LDR (µM)	LOD (µM)	Ref.
Nickel foam	Molecularly imprinted polymer	0.59-1340	0.357	[85]
Carbon paste electrode	Multi-walled carbon nanotubes	5.96-161.07	0.049	[86]
Carbon paste electrode	ZnO nanoparticle and 1-butyl-2,3-dimethylimidazolium hexafluorophosphate	0.09-400	0.031	[64]
Carbon paste electrode	CuO/SWCNTs	0.05 - 400	0.01	This work

The recording data showed that the RSD% for 15 replicates determination of 50 µM and 100 µM of sulfapyridine were 2.2% and 3.2%, respectively. Furthermore, the repeatability of the CuO/SWCNTs/CPE was tested using four different CuO/SWCNTs/CPE for the analysis of 50 µM sulfapyridine. The results showed a RSD% of 3.8% that indicate that CuO/SWCNTs/CPE has a good repeatability.

The selectivity of CuO/SWCNTs/CPE for determination of sulfapyridine was checked by comparing the current and potential before and after the addition of the some interferences with the acceptable error 5% in current and potential. The recording data confirmed that 1000-fold Li^+, Na^+ and Br^- and also 500-fold phenylalanine, methionine and glucose don't show any interference for analysis of sulfapyridine.

In this final step, ability of CuO/SWCNTs/CPE for determination of sulfapyridine in real sample was check using tablet and dextrose saline. The results are presence in Table 2. and acceptable recovery data between 99.13%-103.35% confirm high performance ability of CuO/SWCNTs/CPE for determination of sulfapyridine in real sample.

Table 2. Real sample analysis data for determination of sulfapyridine

Sample	Added (µM)	Expected (µM)	Founded (µM)	Recovery%
Tablet	---	---	2.11±0.23	---
	10.00	12.11	12.01±0.32	99.17
dextrose saline	---	---	<LOD	---
	20.00	20.00	20.67±0.83	103.35
Drinking water	---	---	<LOD	---
	15.00	15.00	14.87±0.67	99.13

Conclusion

A new and powerful analytical tool was design and fabricated as electrochemical sensor for determination of sulfapyridine as an antibiotics drug. The CuO/SWCNTs/CPE was used for study redox reaction sulfapyridine and showed a linear dynamic range 50 nM- 400 µM with detection limit 10 nM. In addition, CuO/SWCNTs/CPE was showed good sensitivity for determination of sulfapyridine in tablet, drinking water and dextrose saline as real samples.

Conflict of Interest

We have no conflicts of interest to disclose.

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How to cite this manuscript: Shirin Shahraki, Mahboubeh Masrournia*, Hassan Karimi-Maleh, Fabrication of Sulfapyridine Electrochemical Sensor Fabrication of Sulfapyridine Electrochemical Sensor Amplified with CuO/SWCNTs as High Performance Electroanalytical Tool in Real Sample Analysis. Chemical Methodologies 4(6), 2020, 720-731. DOI: 10.22034/chemm.2020.113387.