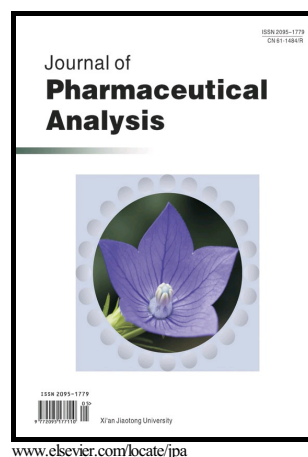


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# Electrooxidation of sulfanilamide and its voltammetric determination in pharmaceutical formulation, urine, and human serum on glassy carbon electrode

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## Abstract

For the first time, the sulfanilamide was determined in otologic solution, urine and human serum by electroanalytical techniques on glassy carbon electrode. The CV experiments showed an irreversible oxidation peak at +1.06 V in 0.1 mol/L BRBS (pH = 2.0) at 50 mV/s. Different voltammetric scan rates (from 10 to 250 mV/s) suggested that the oxidation of SFD on the GCE was a diffusion-controlled process. Square-wave voltammetry (SWV) optimized conditions showed a linear response to SFD from 5.0 to 74.7  $\mu\text{mol/L}$  ( $R = 0.999$ ) with detection and quantification limits of 0.92 and 3.10  $\mu\text{mol/L}$ , respectively. The developed square-wave voltammetric method showed better results for detection limit, and linear range than the CA method, being successfully applied to determine SFD concentration in pharmaceutical formulation, urine and serum human samples with recovery next to 100%.

## Keywords

Square-wave voltammetry; Sulfanilamide; Glassy carbon electrode; Pharmaceuticals; Biological fluids

## Introduction

Sulfonamides were the first drugs with a selective effect on bacteria, and which could be systemically used against bacterial infections [1]. They are commonly applied for human and veterinary use, due to their ability to inhibit gram-positive and gram-negative bacteria, as well as protozoa [2]. In humans, common infections treated by sulfanilamide (SFD) drug include urinary tract infections, vaginal infections, strep throat and some staph infections [3]. Recently, sulfonamide residues in the aquatic environment have become one of the most concerning issues in public health. They exhibit potential toxicity to human beings and aquatic organisms, and are responsible for the emergence of antibiotic resistant bacteria [4]. Although relevant, few methods have been developed for quantification of SFD in pharmaceuticals and other matrices, including chromatographic methods [5–8] and fluorescence [9]. These methods are usually expensive, time consuming, require sample pre-treatments in some cases, and involve great labor [10]. However, the electrochemical methods present good advantages for drugs detection, such as high sensitivity, accuracy, precision, simplicity, low cost, and tedious work during sample preparation procedures [11]. Tadi, Motghare and Ganesh [10], describe a method for SFD determination using a pencil graphite electrode chemically modified with molecular imprinting technology. This sensor, under optimized conditions, has very low detection limit of 0.02 nmol/L and two linear ranges from 0.05–1,100 nmol/L and 1.1–48  $\mu\text{mol/L}$  with sensitivity values of 1.168 and 0.012  $\mu\text{A}/\mu\text{mol/L}$ , respectively. The sensor was applied successfully in analysis of SFD in spiked human serum and ground water samples. Wei *et al.* [12] developed a novel sensor based on glassy carbon electrode (GCE) modified with molecularly imprinted polymer and graphene oxide for SFD determination. The sensor was characterized using scanning electron microscopy, cyclic voltammetry, and electrochemical impedance spectroscopy and square-wave voltammetry. Under optimized conditions, the intensity of the oxidation peak current of SFD showed two linear dynamic ranges from 10 to 1000 ng/mL. Although these studies showed good results, the use of bare GCE has some advantages, such as dispensing tedious steps modification, low cost, and ease of use [13–21]. In this sense, to the best of our knowledge, this is the first time that an electrochemical sensor based on bare GCE is applied to the quantification of SFD in pharmaceuticals and fluid biologics.

## 2. Experimental

### 2.1 Chemicals

The entire chemicals were analytical grade and were used without further purification. A stock solution of 10.0 mmol/L SFD was prepared in a medium of Britton-Robinson Buffer Solution (BRBS, 0.1 mol/L), which was prepared by mixing equimolar amounts of phosphoric acid (85.0%), acetic acid (99.8%), boric acid (99.5%) and then its pH was adjusted with 1.0 mol/L sodium hydroxide solution.

### 2.2 Apparatus

The voltammetric measurements were carried out on an Autolab PGSTAT 128 N (Metrohm Autolab B.V., Utrecht, and The Netherlands) potentiostat/galvanostat controlled by NOVA 1.10.4 electrochemical software. The three-electrode electrochemical cell was set with GCE ( $A = 0.07 \text{ cm}^2$ ) as a working electrode, an Ag/AgCl 3.0 mol/L KCl electrode as a reference electrode, and a platinum wire as a counter electrode. The pH measurements were done with a calibrated pH meter with standard buffers at room temperature.

### 2.3 Electrode preparations procedure

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