

Electrochemical detection of sulfasalazine in real samples by GO/Fe₃O₄@SiO₂ nanostructure and ionic liquid modified carbon paste electrode

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A core-shell nanocomposite (GO/Fe₃O₄@SiO₂ in this case) and ionic liquid have been used to improve the sensory properties of carbon paste electrode (CPE) entitle GO/Fe₃O₄@SiO₂/IL-CPE as new analytical approach. We validated the applications of GO/Fe₃O₄@SiO₂/IL-CPE for the determination of sulfasalazine by using different electrochemical techniques. Under the optimized experimental condition, the linearity range and the detection limit (LOD) of GO/Fe₃O₄@SiO₂/IL-CPE were 1.0– 300.0 and 0.4 μM for sulfasalazine, respectively. The recording data showed that GO/Fe₃O₄@SiO₂/IL-CPE could be fabricated easily fabricated with outstanding stability. The sensor was applied to the detection of drug in real samples.

Keywords: GO/Fe₃O₄@SiO₂ nanocomposite, Sulfasalazine, Voltammetry, Carbon paste electrode

1. INTRODUCTION

Sulfasalazine is an azo-salicylic acid derivatives with anti-inflammatory effect that was synthesized and used for treating rheumatoid arthritis from 1938 [1-3]. During treatment of autoimmune diseases the sulfasalazine prescribed as mainly drug in this process. Molecule of sulfasalazine has make by connection of pyridyl benzene sulfonamide and salicylic acid (SA) groups by azo bond. The mesalazine part was act as active section in drug structure and reduces inflammation by blocking the activity of lipoxygenase and cyclooxygenase. On the other hand, this drug can created adverse reactions which may lead to hemopoietic disorders and cancer. Multidisciplinary clinical trials have demonstrated

the adverse effects of 3–20% of sulfasalazine use in the treatment of patients. This has led researchers to suggest the use of the drug in the treatment of patients by controlling its dose. Studies by medical researchers have confirmed the presence of between 6–32 $\mu\text{g}\cdot\text{mL}^{-1}$ of sulfasalazine in the serum of patients undergoing treatment [4-10]. For this purpose and to control the health of patients treated with this drug, determination of sulfasalazine could enhance the therapeutic application of the drug.

Several analytical techniques like liquid chromatography [13-16], optic based method [17, 18], capillary electrophoresis-MS [19, 20] and redox-based methods [21-23] have been reported to determine sulfasalazine. While some of these methods have many benefits, such as wide range of applications and ease of operation, important problems such as the use of toxic solvents, the inability to be kitable, as well as the long analysis time of their use have faced many problems. Compared to methods such as chromatography and optical, Compared to methods such as chromatography and light, in recent years serious attention has been paid to electron exchange methods, especially electrochemical methods, as an important alternative method [24-44]. Several benefits have been reported for electron exchange methods, including simplicity, high sensitivity, easy modification for selective analysis and wide linear concentration range [45-65].

Carbon paste electrode (CPE) is preferred over other electrodes for electrochemical analysis due to its easy applicability and processing, wide potential window and high sensitivity [81-90]. CPE can be chemically modified with other materials to form conductive matrixes which also increase active position in electrode surface compared to inert substrate electrodes. Further, they display low background current and enhance electrochemical sensitivity [66-75].

Nanostructured carbon-based materials and metallic nanoparticles [76], due to their increased surface area-to-volume ration have been widely used as electrode modifiers. It has also been reported that the sensitivities of electrochemical sensors can be greatly enhanced by using nanostructured materials [77-82]. In recent years, graphene has attracted much attention for construction of modified electrodes for electrochemical sensors due to their low residual current, high potential range and good chemical stability. Graphene oxide (GO) has been considered as a versatile scaffold for nanomaterials to form hybrid nanocomposite with improved properties. [83-85].

Magnetic nanoparticles, on other hand, provides high surface area for conjugating functional ligands, exhibits low toxicity and can be easily functionalized. However, bare iron nanostructures (without any coating) gets oxidized and aggregated due to high surface energy. Hence, many approaches have been put forth for preventing the oxidation and aggregation of the nanoparticles. One of the main approaches is the formation of magnetic core-shell structure [84-87]. These core-shell structure not only helped in overcoming the problems of aggregation and oxidation, it also displayed better functions than the bare nanoparticles.

Ionic liquids are class of conductive chemical moieties which are promising mediators in sensor applications [88-93]. Due to their high ionic conductivity, they promote fast ion mobility in electrolyte and thus are envisaged to be used as electrolyte in voltammetric studies [94].

In this project, the synthesis of $\text{GO}/\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell nanocomposite and its use to modify the carbon paste electrode surface was reported. The $\text{GO}/\text{Fe}_3\text{O}_4@\text{SiO}_2/\text{IL}$ -CPE exhibited high electrocatalytic activity and sensitivity towards sulfasalazine. The $\text{GO}/\text{Fe}_3\text{O}_4@\text{SiO}_2/\text{IL}$ -CPE has a many advantage such as high conductivity and large specific surface area.

2. EXPERIMENTAL

2.1. Instruments and materials

Electrochemical measurements were achieved on a PGSTAT 302N Autolab potentiostat/galvanostat, controlled using an electrochemical system software. pH were performed using a Metrohm 710 pH-meter. An Ag/AgCl/KCl, GO/Fe₃O₄@SiO₂/IL-CPE and a platinum wire suggested as the reference, working and auxiliary electrodes, respectively.

All chemicals, including sulfasalazine were obtained from Merck Co., and n-hexyl-3-methylimidazolium hexafluoro phosphate was obtained from Sigma CO., with analytical grade purity. The phosphate buffer solutions (PBS) was used for adjust the conditions and prepared using orthophosphoric acid and its salts. The GO/Fe₃O₄@SiO₂ nanocomposite was synthesized according to recommend procedure by Beitollahi et al. group by carefully maintaining the conditions [95]. The surface morphologies of GO/Fe₃O₄@SiO₂ were investigated by SEM and depicted in Fig. 1.

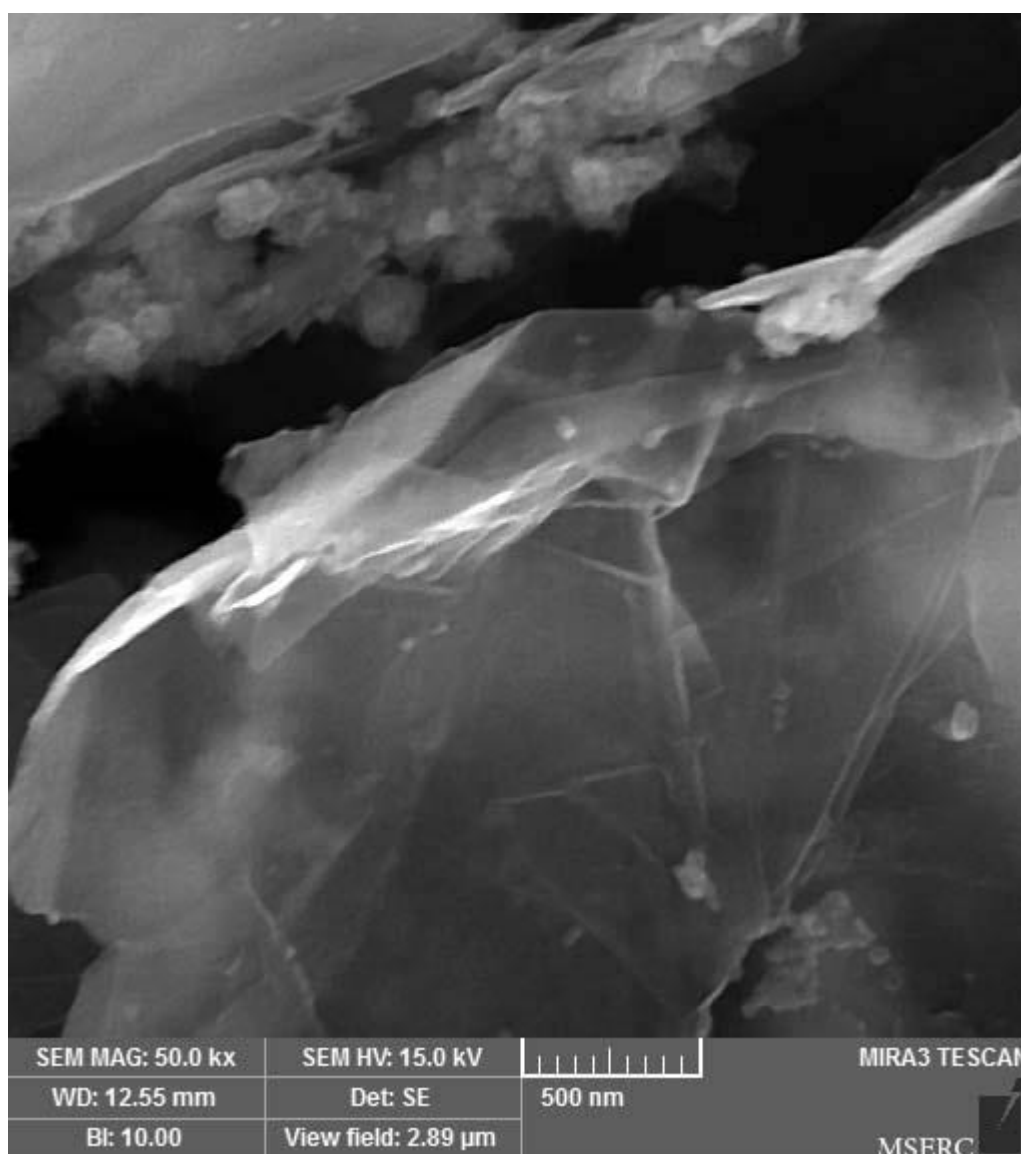


Figure 1. SEM image of GO/Fe₃O₄@SiO₂

2.2. Preparation of GO/Fe₃O₄@SiO₂/IL-CPE

GO/Fe₃O₄@SiO₂/IL-CPE were ready by mixing 0.04 g of GO/Fe₃O₄@SiO₂ + 0.96 g graphite powder and approximately, ~0.8 mL of ionic liquid with a mortar and pestle. The obtained paste was packed into the end of a glass tube connected with copper wire for electrical connection.

2.3. Preparation of Real Samples

Several of sulfasalazine tablets (500 mg/tablet, Mehr Darou Company, Iran) were subjected to mechanical grinding. The 500 mg of the powder obtained from sulfasalazine tablets dissolving in 25 mL water under stirring and ultrasonication. After dilution with PBS in different ration, the results solutions were used for real sample analysis by the standard addition method. Urine samples were prepared according results procedure by Beitollahi et al. group [95].

3. RESULTS AND DISCUSSION

3.1. Redox behavior of sulfasalazine

The electrochemical reported papers confirm that redox behaviour of sulfasalazine is pH dependent [96]. Therefore, it was essential to improve the solution pH to highly sensitive detection of sulfasalazine [97]. The electrochemical behaviour of sulfasalazine was examined by voltammetry using GO/Fe₃O₄@SiO₂/IL-CPE modified electrode at different pH ranging from 2-9. The results indicated better electroactivity of sulfasalazine at GO/Fe₃O₄@SiO₂/IL-CPE surface neutral pH than the basic or acidic medium. Thus, the neutral conditions (pH 7.0) was designated as an optimal pH for sulfasalazine determination at GO/Fe₃O₄@SiO₂/IL-CPE surface.

Figure 2 shows responses of CV to redox reaction 200.0 μM sulfasalazine at a CPE (curve b) and GO/Fe₃O₄@SiO₂/IL-CPE (curve a). The oxidation potential of sulfasalazine observed around 870 mV that showed negative shift about 130 mV compare to CPE. Additionally, with moving CPE to GO/Fe₃O₄@SiO₂/IL-CPE, the oxidation current of sulfasalazine dramatically increased. These improvement in potential and current with moving CPE to GO/Fe₃O₄@SiO₂/IL-CPE showed a high electrical conductivity at surface of modified sensor that is relative to presence of GO/Fe₃O₄@SiO₂ and IL at CPE surface [95, 98].

3.2 Effect of Scan Rate

To get a better insight on the reaction kinetics of the oxidation of sulfasalazine, scan rates were varied (Figure 3). A noteworthy enhancement in the oxidation signal in linear sweep voltammetry was observed with an increased scan rate. Moreover, diffusion controlled oxidation processes can be inferred as evident from the linear plot of I_p versus $v^{1/2}$ towards the analyte [84-86].

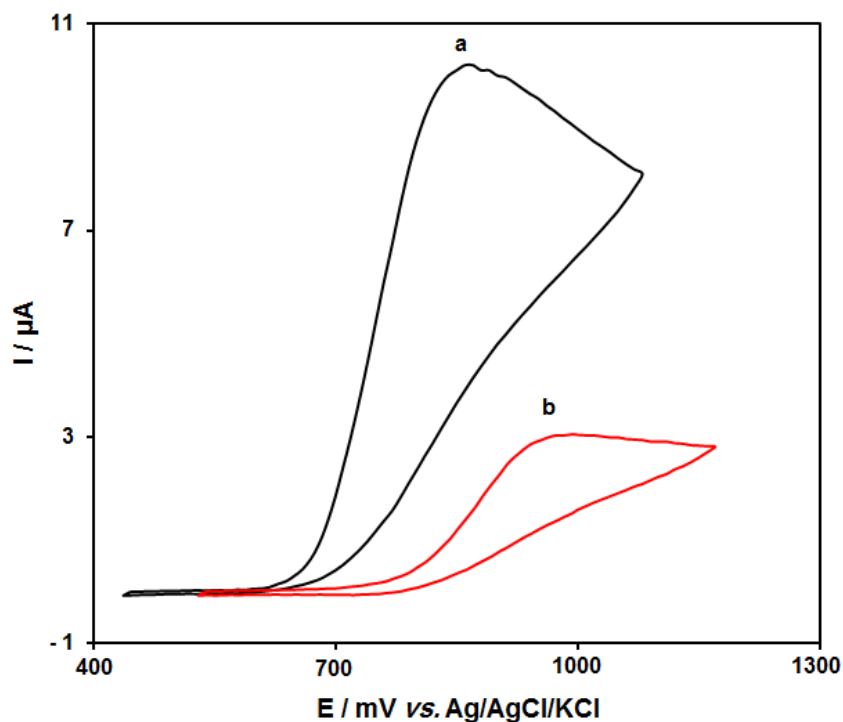


Figure 2. CVs of a) GO/Fe₃O₄@SiO₂/IL-CPE and b) CPE in the presence of 200.0 μM of sulfasalazine. Condition; pH 7.0 and scan rate 50 mV s⁻¹.

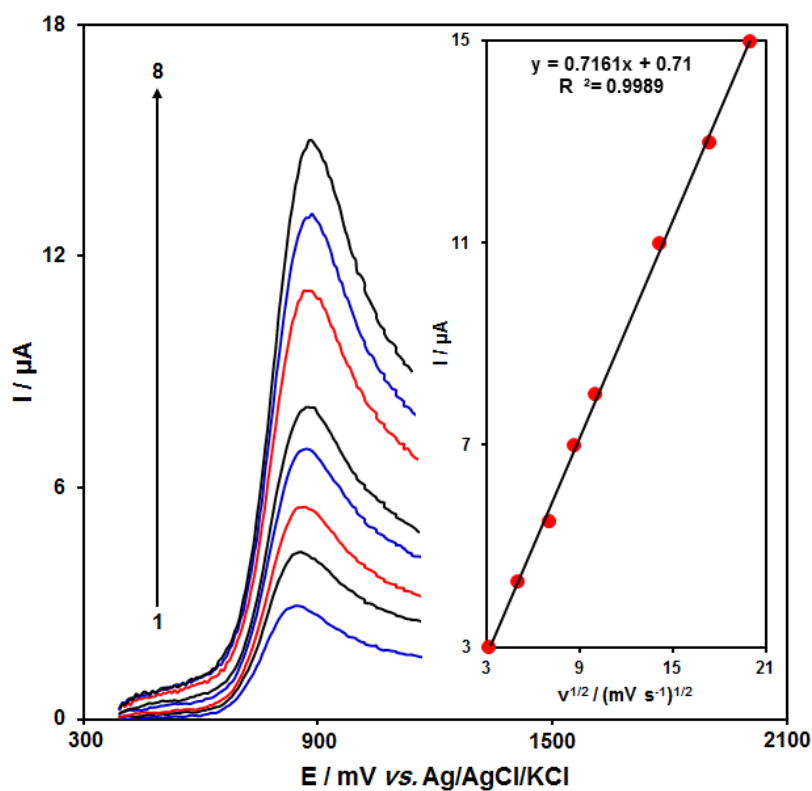


Figure 3. LSV of GO/Fe₃O₄@SiO₂/IL-CPE containing 100.0 μM sulfasalazine at scan rates 1) 10, 2) 25, 3) 50, 4) 75, 5) 100, 6) 200, 7) 300, and 8) 400 mV s⁻¹, respectively. Inset: variation of I_{pa} current vs. $v^{1/2}$.

The ascending section of the LSV, which have been selected at a scan rate of 10 mVs^{-1} for sulfasalazine, was selected for drawing Tafel plots (Figure 4). Electron transfer kinetics between $\text{GO}/\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{IL-CPE}$ and substrate (sulfasalazine) were found to influence these sections and are designated as Tafel region. The Tafel slope was calculated to be 0.1258. This finding confers one electron process at electrode, in which the charge transfer coefficients was found to be $\alpha = 0.53$ for sulfasalazine.

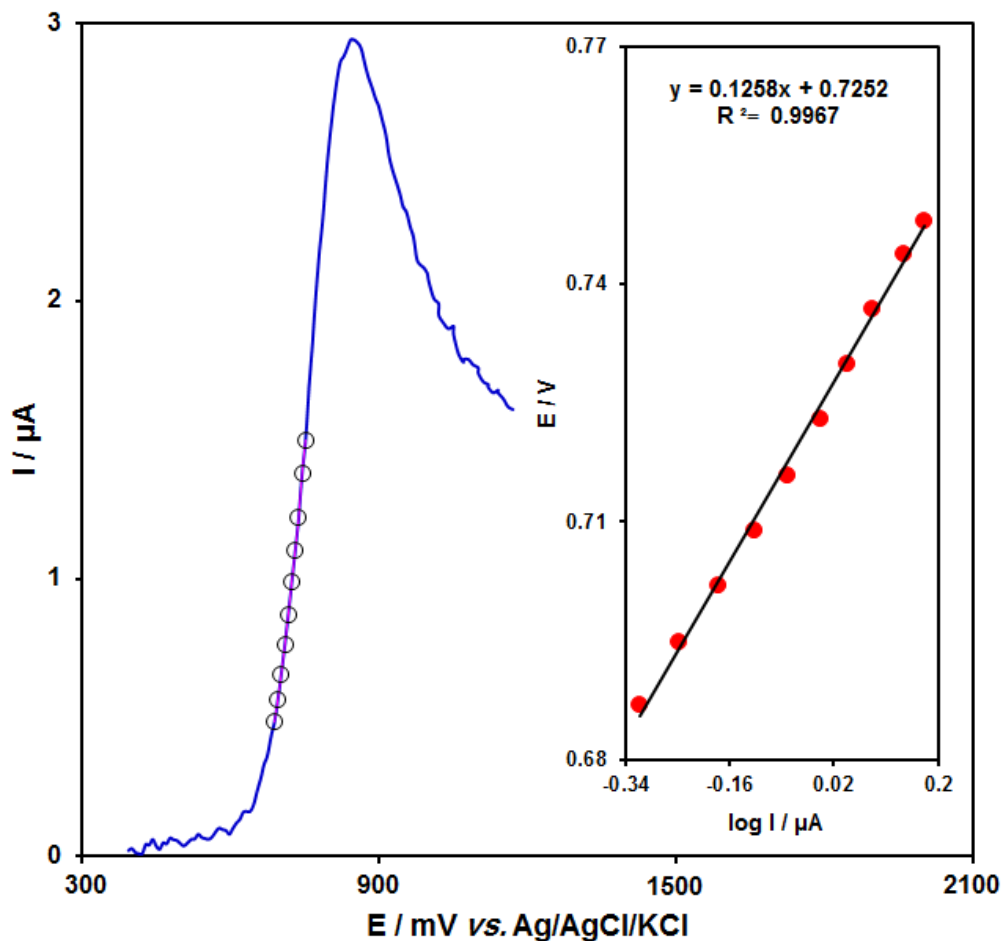


Figure 4. LSV of $\text{GO}/\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{IL-CPE}$ containing $100.0 \mu\text{M}$ sulfasalazine at scan rate 10 mV/s . Inset) Tafel plot of $\text{GO}/\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{IL-CPE}$ derived from the LSV.

3.3. Chronoamperometric study

Chronoamperometric behaviour of sulfasalazine at $\text{GO}/\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{IL-CPE}$ were conducted by adjusting the step potential at 0.92 V for different concentrations of sulfasalazine (Figure 5) and recorded signals were used for next step investigation.

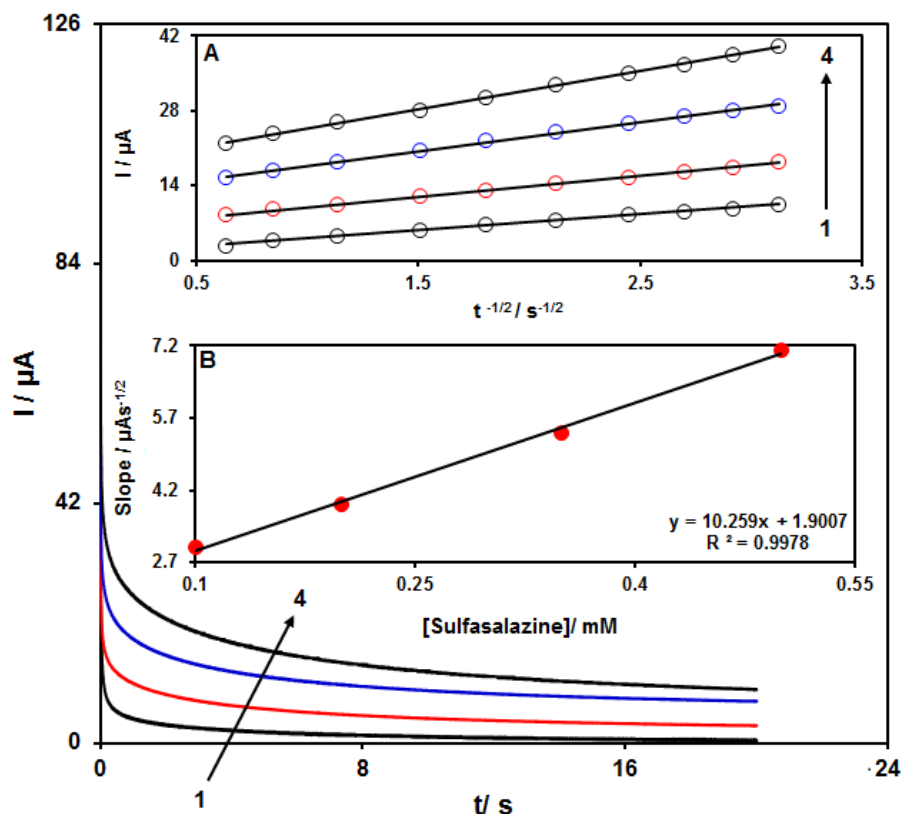


Figure 5. Chronoamperograms obtained at GO/Fe₃O₄@SiO₂/IL-CPE in for 1) 0.1, 2) 0.2, 3) 0.35 and 4) 5.0 mM of sulfasalazine. Insets: (A) Plots of I vs. $t^{-1/2}$ obtained from chronoamperograms 1–4. (B) Plot of the slope of the straight lines against sulfasalazine concentration.

The value of sulfasalazine speed in solution constant (diffusion coefficient; D) was calculated by the Cottrell equation ($I = nFAD^{1/2}C_b\pi^{-1/2}t^{-1/2}$) in a mass transport limited condition. Based on Cottrell equation, the plots of I versus $t^{-1/2}$ for various concentrations of sulfasalazine were useful determination of sulfasalazine diffusion coefficient (Figure 5A and B). Based on concluded slopes in Figure 5 B that derived from Figure 5A data, mean values of D were $1.1 \times 10^{-6} \text{ cm}^2/\text{s}$ for sulfasalazine that is an acceptable data for aqueous solution [99].

3.4. Calibration curves

Using the peak currents recorded with the GO/Fe₃O₄@SiO₂/IL-CPE, the analyte concentration in aqueous samples were determined through differential pulse voltammetric (DPV) studies. DPV has an edge over other techniques in terms of sensitivity and performance (Fig. 6) [100].

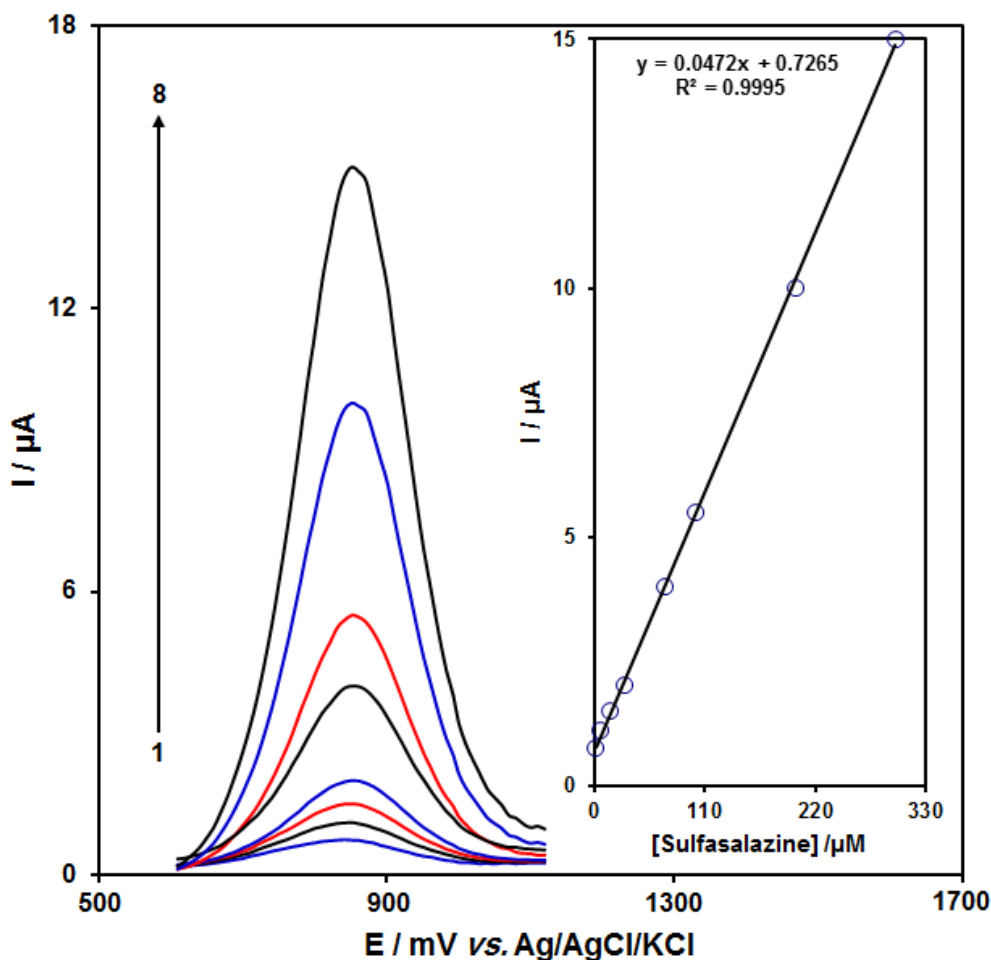


Figure 6. DPVs of GO/Fe₃O₄@SiO₂/IL-CPE containing 1) 1.0, 2) 5.0, 3) 15.0, 4) 30.0, 5) 70.0, 6) 100.0, 7) 200.0 and 8) 300.0 μM sulfasalazine. Inset: Plot of the peak current as a function of sulfasalazine concentration.

The results indicated a good linearity relation between the DPV currents and sulfasalazine concentration with LDR of 1.0-300.0 μM (correlation coefficient of 0.9995) and the limit of detection 0.4 μM.

3.5. Analysis of sulfasalazine in real sample

The DPV strategy was used to evaluate the practical performance of GO/Fe₃O₄@SiO₂/IL-CPE in determining sulfasalazine in real samples viz., sulfasalazine tablet and urine. The obtained results relative to standard addition technique for determination of sulfasalazine displayed in Table 1.

The recovery values between 97.8 and 102.1 are acceptable for confirming ability of GO/Fe₃O₄@SiO₂/IL-CPE as new electroanalytical sensor for determination of sulfasalazine in real samples.

Table 1. Determining sulfasalazine in real samples through GO/Fe₃O₄@SiO₂ /IL-CPE All the concentrations are in μM (n=3).

Sample	Spiked	Found	Recovery (%)	R.S.D. (%)
sulfasalazine tablet	0	4.0	-	3.2
	2.5	6.6	101.5	2.4
	5.5	8.8	97.8	1.9
	7.5	11.4	99.1	3.5
	10.0	14.3	102.1	3.2
	0	-	-	-
Urine	5.0	4.9	98.0	1.9
	10.0	10.2	102.0	3.3
	15.0	14.8	98.7	2.2
	20.0	20.2	101.0	2.8

4. CONCLUSION

A drug electrochemical sensor entitle GO/Fe₃O₄@SiO₂/IL-CPE was fabricated for determination of sulfasalazine. The high conductivity of GO/Fe₃O₄@SiO₂ nanocomposite and ionic liquid helps to GO/Fe₃O₄@SiO₂/IL-CPE that showed good catalytic activity for sulfasalazine. The proposed sensor involves high sensitivity, very low detection limit, and easy handling. The GO/Fe₃O₄@SiO₂/IL-CPE as analytical tool has been effectively employed to measurement of sulfasalazine in rea; samples with acceptable recovery data. The GO/Fe₃O₄@SiO₂/IL-CPE sensor showed outstanding response for determining sulfasalazine in the range, 1.0-300.0 μM, with a detection limit of 0.4 μM respectively.

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