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Application of Fe_3O_4@SiO_2/MWCNT Film on Glassy Carbon Electrode for the Sensitive Electroanalysis of Levodopa

Mohammad Reza Ganjali[1,2,*], *Hadiseh Salimi*[3], *Somayeh Tajik*[4], *Hadi Beitollahi*[4], *Morteza Rezapour*[5], *Bagher Larijani*[6]

[1] Center of Excellence in Electrochemistry, School of Chemistry, University of Tehran, Tehran, Iran
[2] Biosensor Research Center, Endocrinology & Metabolism Molecular-Cellular Sciences Institute, Tehran University of Medical Sciences, Tehran, Iran
[3] Department of Chemistry, Graduate University of Advanced Technology, Kerman, Iran
[4] Environment Department, Institute of Science and High Technology and Environmental Sciences, Graduate University of Advanced Technology, Kerman, Iran
[5] IP Department, Research Institute of the Petroleum Industry (RIPI), P.O. Box 14665-137, Tehran, Iran
[6] Diabetes Research Center, Endocrinology & Metabolism Clinical Sciences Institute, Tehran University of Medical Sciences, Tehran, Iran
[*]E-mail: ganjali@khayam.ut.ac.ir

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A Fe_3O_4@SiO_2/MWCNT (FSCNT) film was coated on a glassy carbon electrode , and then electrochemical oxidation behavior of levodopa was considered in PBS (pH 7.0) by cyclic and differential pulse voltammetry (CV and DPV). The obtained results showed that the new electrode has an electrocatalytic activity in oxidation of levodopa which causes a remarkable enhancement in its oxidation current.. In the optimum conditions, the anodic peak current indicated a linear relation versus levodopa concentration in the range of 1.0×10^{-5} to 6.0×10^{-4} M with detection limits of 2.0×10^{-6} M (signal-to-noise = 3). This method was used to the measurement of levodopa in urine samples.

Keywords: Levodopa; Fe_3O_4@SiO_2/MWCNT nanocomposite; Glassy carbon electrode; Voltammetry

1. INTRODUCTION

There are several human diseases that have been identified to be caused by protein deposition both extracellularly and intracellularly. These diseases are caused by formation of insoluble amyloids in tissues. Amyloids are toxic species. Indirect toxicity arises when amyloids trigger a series of lethal events leading to cell death. They interact with membranes leading to permeabilization and cell

damage [1-4]. Some important protein deposition diseases include Alzhimers, Parkinsons, diabetes and systemic amyloidosis. Dopaminergic medications, such as levodopa (L-3,4-dihydroxyphenylalanine) and dopamine receptor agonists, are prescribed in Parkinson's patients with the therapeutic goal of ameliorating motor deficits. Dopamine fails to pass the blood-brain barrier, so it cannot be directly injected to replenish the insufficient of dopamine [5-7]. Levodopa is chosen as a remedial drug for the treatment of Parkinson because it is able to penetrate the blood-brain barrier, convert to dopamine and preferentially accumulate in the striatum. However, the auto-oxidization of levodopa occurs in the peripheral system, which can produce serious side effects, such as nausea, vomiting and dyskinesia [8-11]. Therefore, it is important to have a simple and precise analytical method for the determination of levodopa in biological fluids.

Among many analytical techniques, voltammetric methods are very promising method to detect levodopa due to the electroactive nature of this biomolecules [12-15]. Electrochemical sensors have been extensively applied over the others because of advantages such as simplicity of sample preparation, low cost of instrumentation, miniaturization, high sensitivity, and selectivity [16-27].

For biosensing applications, other important characteristics are simplicity, robustness and portability. Regarding this matter, the development of glassy carbon electrodes has met this demand, offering a system of electrode designed with great simplicity and economy [28-30]. To develop their electrochemical performance, electrodes have been improved [31-33].

With increasing development of nanotechnology, nanoparticles have attracted special attention during recent decades due to their unique electrical, large surface-to volume ratio, optical and catalytic feature, and so their applications in electrochemistry have increased greatly [34-48]. Among many nanomaterials, nano-oxides due to their remarkable properties have involved in many various applications [49-55]. As sample, magnetic Fe_3O_4 nanoparticles have been used as modifier because of remarkable magnetic responsivity, relative large surface area and ease of surface functionalization. However, these nano-oxides are normally in colloidal forms. Recently, such point has caused a wide interest in the synthesis and application of new nanomaterials by loading a nano-oxide on another solid such as carbon nanotubes, resin and polymer or modification with Fe_3O_4 as the core and metal or metal oxide as the shell [56-59].

Carbon nanotubes (CNTs) are a novel nanoscale material, mainly consisting of SWCNTs and MWCNTs. These nano-scale materials have attracted considerable interest owing to their extraordinary mechanical and unique electrochemical properties. The subtle electronic behavior of CNTs demonstrates the ability to stimulate electron-transfer reactions when used as electrode materials [60-62].

Recently, the silica (SiO_2) has been widely used due to the excellent properties. The SiO_2 shell can enhance the storage stability and can enhance the dispersibility of the nanoparticles in solution. Furthermore, a large number of -OH groups on the surface of SiO_2 also can make sites for surface modification [63-66]. Hence, the Fe_3O_4@SiO_2/MWCNT (FSCNT) nanocomposite can be an interesting material in biosensing applications.

Based on the above mentioned, it is important to create suitable conditions for determination of levodopa in biological fluids. In this study, we describe application of Fe_3O_4@SiO_2/MWCNT nanocomposite as a nanostructure sensor for voltammetric determination of levodopa. The proposed

sensor showed good electrocatalytic effect on levodopa. The modified electrode shows advantages in terms of selectivity, reproducibility and sensitivity. Eventually, we study the analytical performance of the suggestion sensor for levodopa determination in urine samples.

2. EXPERIMENTAL

2.1. Apparatus and chemicals

Electrochemical measurements were done by an Autolab potentiostat/galvanostat (PGSTAT 302N, Eco Chemie, the Netherlands). Experimental conditions were controlled through General Purpose Electrochemical System (GPES) software. A three-electrodes cell was used at 25 ± 1 °C. An Ag/AgCl/KCl (3.0 M) electrode, a platinum wire, and the FSCNT/GCE were used as the reference, auxiliary and working electrodes, respectively. A Metrohm 710 pH meter was employed for pH measurements. Fe_3O_4@SiO_2/MWCNT nanocomposite was synthesized in our laboratory as described elsewhere [67].

The solutions were freshly prepared with double distilled water. Levodopa and all other reagents were of analytical grade and were purchased from Merck chemical company (Darmstadt, Germany). The buffer solutions were prepared from orthophosphoric acid and its salts in the pH range of 2.0-9.0.

2.2.Modification of the electrode

A bare glassy carbon electrode was coated with Fe_3O_4@SiO_2/MWCNT nanocomposite as follows. Stock solution of Fe_3O_4@SiO_2/MWCNT nanocomposite was prepared by dispersing 1 mg Fe_3O_4@SiO_2/MWCNT nanocomposite in 1 mL aqueous solution with the aid of ultrasonication for 1 h. Then, a 5 µl aliquot of the Fe_3O_4@SiO_2/MWCNT/H_2O suspension solution was casted on to the carbon working electrode surface, and waiting the surface to dry in the room temperature.

3. RESULTS AND DISCUSSION

3.1. Electrocatalytic oxidation of levodopa at a FSCNT/GCE

Fig. 1 depicts the cyclic voltammetric responses for the electrochemical oxidation of 600.0 µM levodopa at FSCNT/GCE (curve a) and bare GCE (curve b). The anodic peak potential for the oxidation of levodopa at FSCNT/GCE (curve a) is about 190 mV compared to 450 mV the amount for the bare GCE (curve b). Also, by comparing the oxidation peak of levodopa at the FSCNT/GCE (curve a) and bare GCE (curve b), an extensive enhancement of the anodic peak current at FSCNT/GCE relative to the value obtained at the bare GCE (curve b) can be seen. In fact, the obtained results

obviously indicate that the Fe_3O_4@SiO_2/MWCNT nanocomposite improve the levodopa oxidation signal.

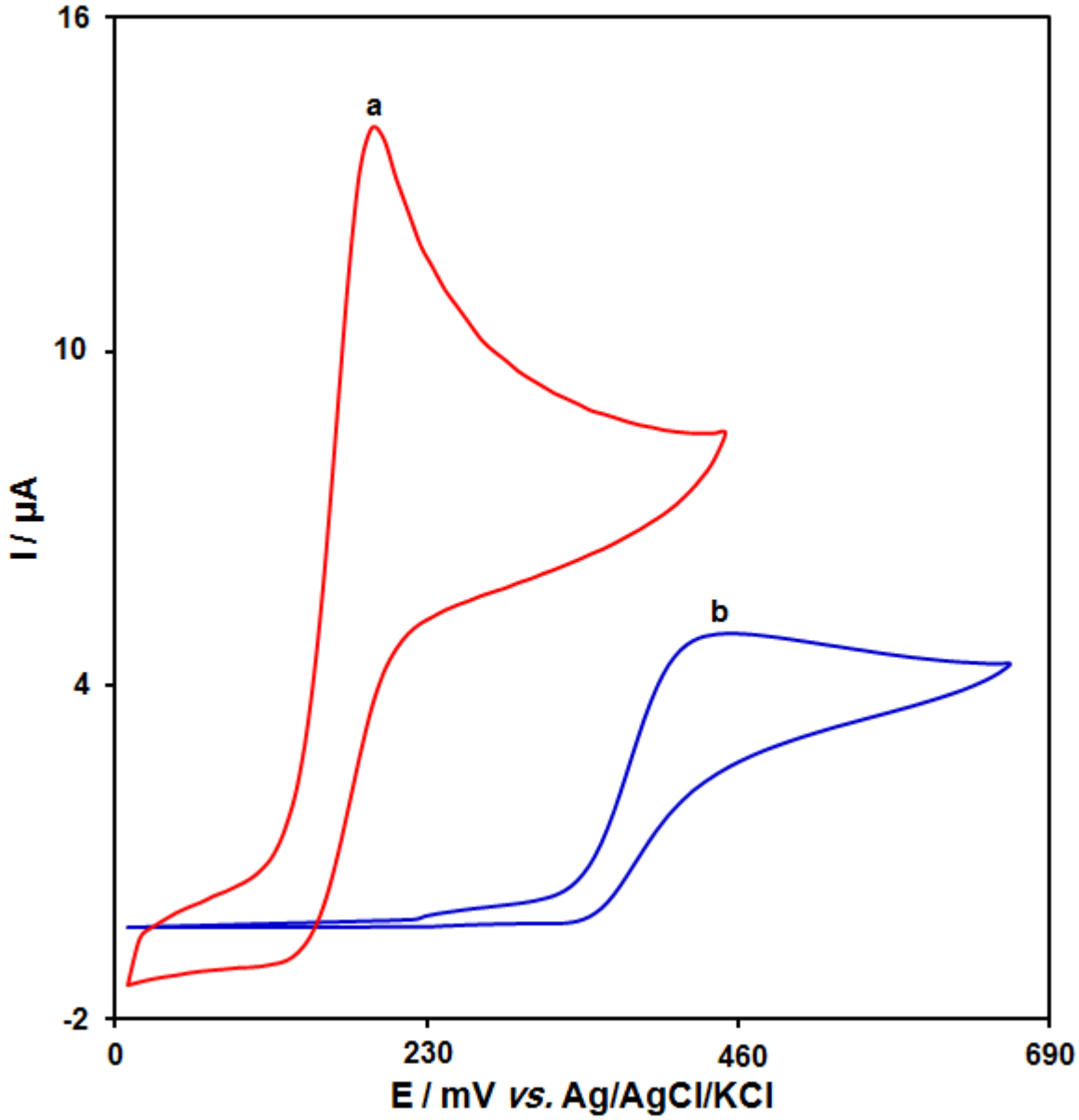

Figure 1. Cyclic voltammograms of (a) FSCNT/GCE and (b) bare GCE in 0.1 M PBS (pH 7.0) in the presence of 600.0 µM levodopa at the scan rate 50 mVs^{-1}.

The effect of scan rates on the oxidation of levodopa has been considered too (Fig. 2). The results revealed that growing in the potential scan rate enhanced the oxidation peak current. Also, the oxidation process is diffusion controlled as deduced from the linear dependence of the anodic peak current (I_p) on the square root of the potential scan rate ($v^{1/2}$) over a wide range of 5 - 500 mV s^{-1}.

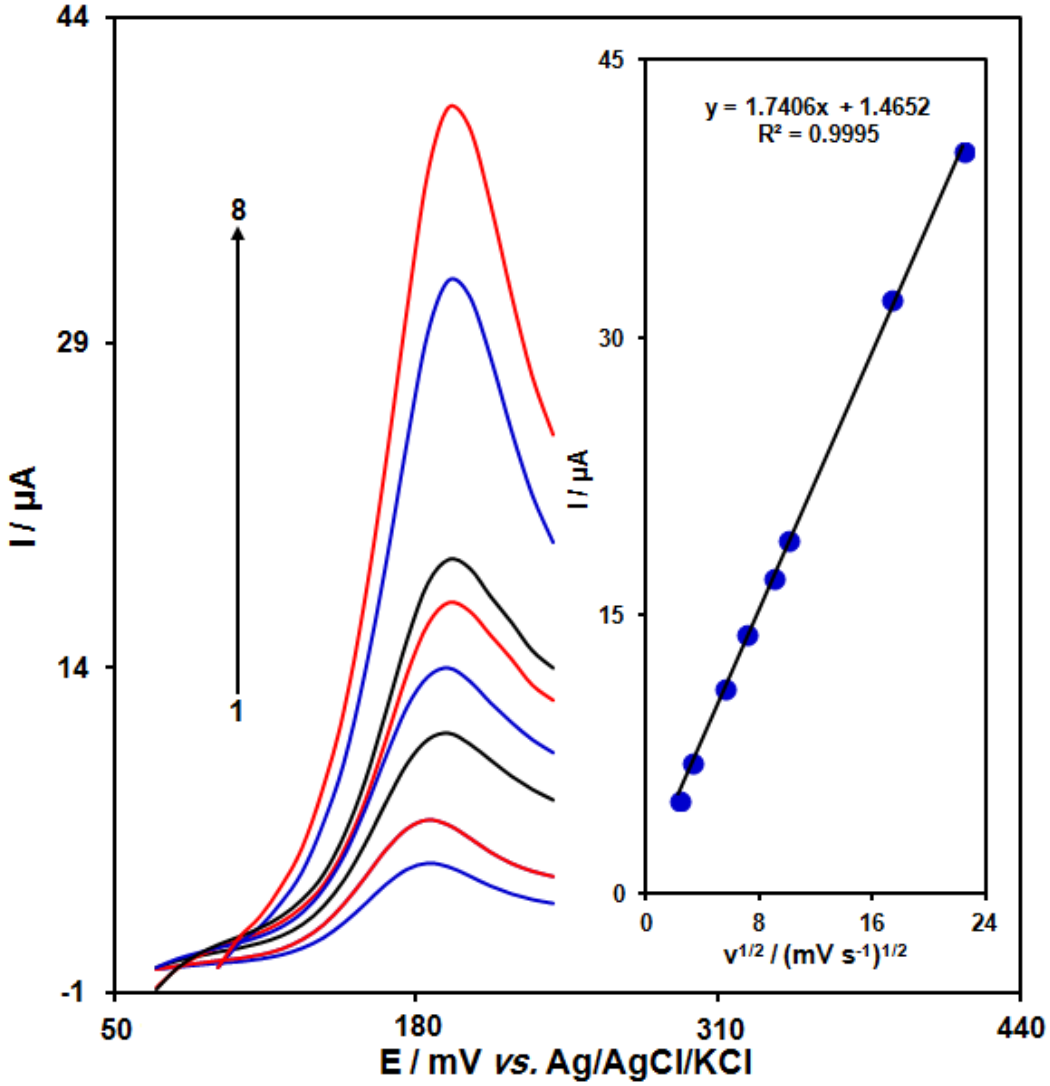

Figure 2. LSVs of FSCNT/GCE in 0.1 M PBS (pH 7.0) containing 600.0 µM levodopa at various scan rates; numbers 1-8 correspond to 5, 10, 30, 50, 80, 100, 300 and 500 mV s^{-1}, correspondingly. Inset: variation of cathodic peak current vs. $v^{1/2}$.

3.2. Chronoamperometric study

Chronoamperometric study of levodopa at FSCNT/GCE were done by setting the working electrode potential at 0.25 V for the several concentrations of levodopa in PBS (pH 7.0) (Fig. 3).

For an electroactive material such as levodopa which have a diffusion coefficient of D, the observed current for the electrochemical reaction at the mass transport limited condition is obtained by the Cottrell equation [68].

$$I = nFAD^{1/2}C_b\pi^{-1/2}t^{-1/2} \quad (1)$$

Where D and C_b are the diffusion coefficient (cm^2 s^{-1}) and the bulk concentration (mol cm^{-3}), respectively. Experimental curves of I vs. $t^{-1/2}$ were used, with the best fits for various concentrations

of levodopa (Fig. 3A). The slopes of the resulting straight lines were then drawn vs. levodopa concentration (Fig. 3B). From the resulting slope and Cottrell equation the mean value of the D was calculated 1.8×10^{-5} cm^2/s.

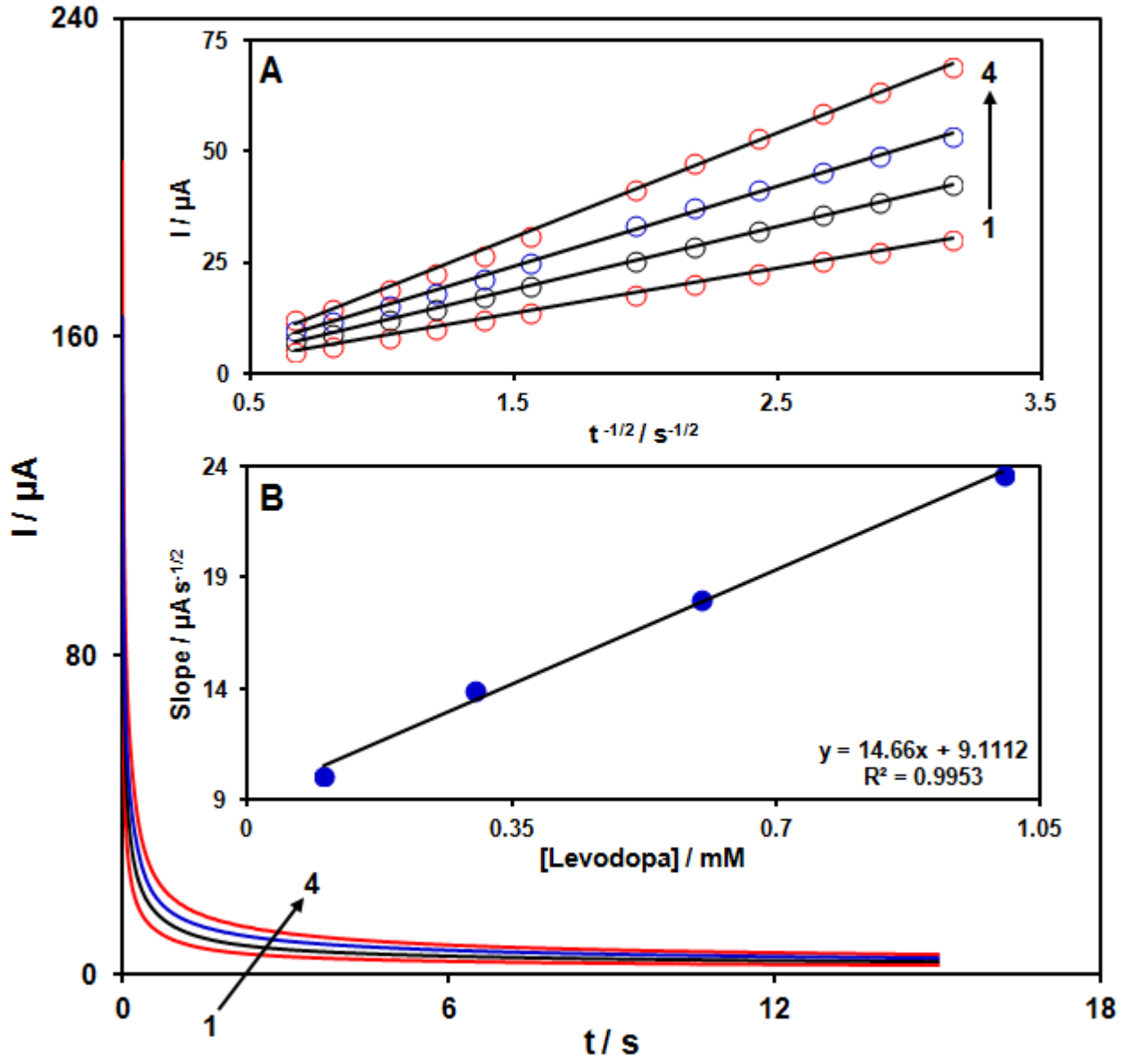

Figure 3. Chronoamperograms gotten at FSCNT/GCE in 0.1 M PBS (pH 7.0) for several concentrations of levodopa. The numbers 1–4 correspond to 0.1, 0.3, 0.6 and 1.0 mM of levodopa. Insets: (A) Plots of I vs. $t^{-1/2}$ obtained from chronoamperograms 1–4. (B) Plot of the slope of the straight lines against levodopa concentration.

3.3. Calibration curve and limit of detection

The oxidation peak current of levodopa at the electrode surface can be applied for the determination of levodopa in the solution. Hence, differential pulse voltammetry (DPV) experiments were used for different concentrations of levodopa (Fig. 4).

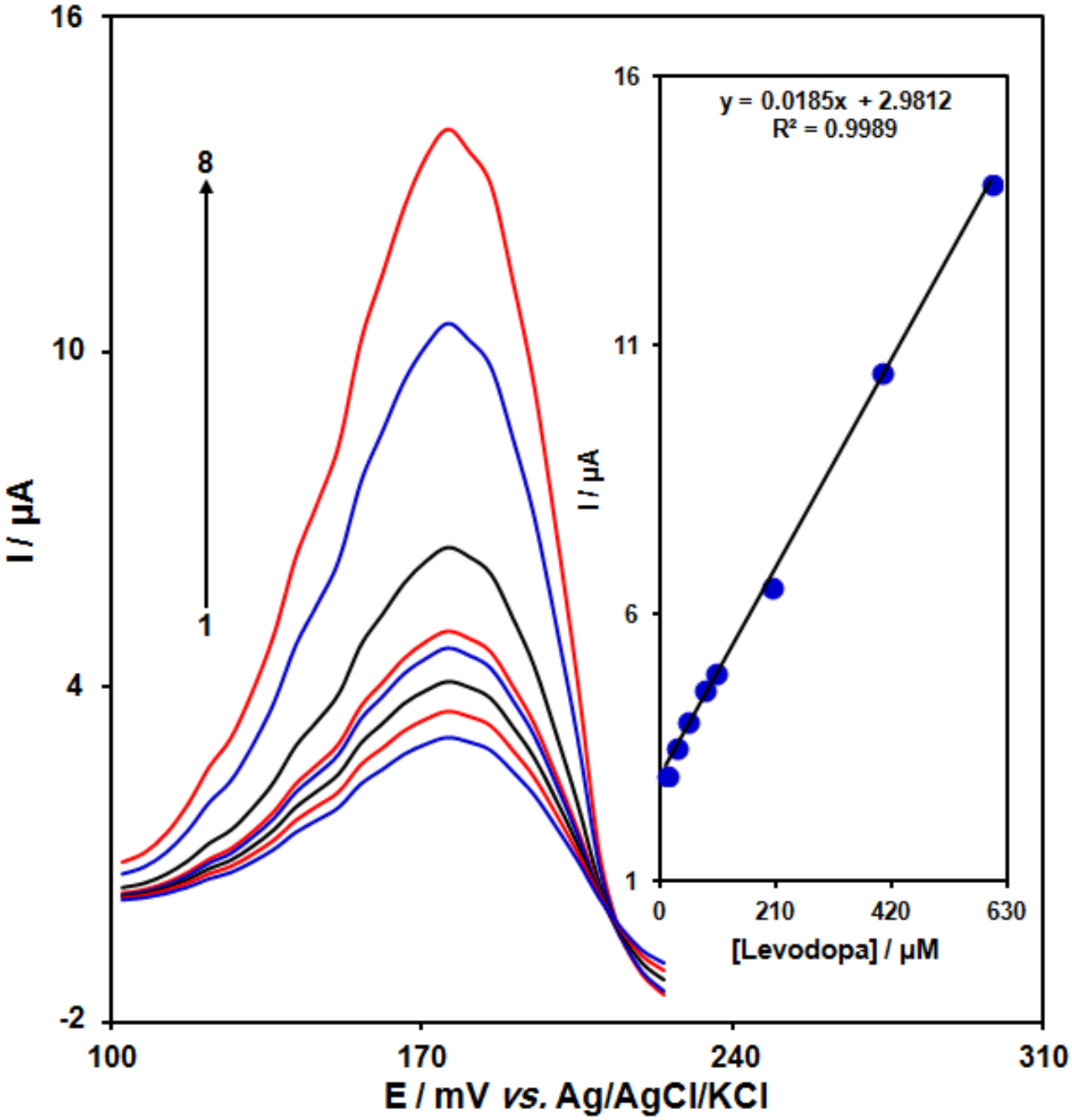

Figure 4. DPVs of FSCNT/GCE in 0.1 M (pH 7.0) containing different concentrations of levodopa. Numbers 1–8 correspond to 10.0, 30.0, 50.0, 80.0, 100.0, 200.0, 400.0 and 600.0 µM of levodopa. Inse: Plot of the electrocatalytic peak current as a function of levodopa concentration in the range of 10.0-600.0 µM.

The oxidation peak currents of levodopa at the surface of the proposed modified electrode were corresponded to the concentration of the levodopa within the ranges 10.0 to 600.0 µM. The detection limit (3σ) of levodopa was calculated to be 2.0×10^{-6} M. These values are comparable with the values obtained previously for determination of levodopa (Table 1).

Table 1. Comparison of the efficiency of some modified electrodes used in the determination of levodopa

Electrode	Modifier	Method	LDR (M)	LOD (M)	Ref.
Carbon Paste	Meso-tetrakis(3-methylphenyl) cobalt porphyrin (CP) and TiO2 nanoparticles	Voltammetry	1.0×10^{-6} – 1.0×10^{-4}	6.9×10^{-8}	[5]
Carbon Paste	Graphene nanosheets and 1-(4-bromobenzyl)-4-ferrocenyl-1H-[1,2,3]-triazole	Voltammetry	5.0×10^{-8} -8.0×10^{-4}	1.5×10^{-8}	[9]
Graphite Screen Printed	Graphene Oxide/ZnO	Voltammetry	1.0×10^{-6} -1.0×10^{-3}	4.5×10^{-7}	[12]
Glassy Carbon	Fe_3O_4@SiO_2/MWCNT	Voltammetry	1.0×10^{-5} -6.0×10^{-4}	2.0×10^{-6}	This work

3.4. Determination of Levodopa in urine sample

for studying the analytical applicability of the proposed method in the complex matrix, it was applied to the measurement of levodopa in some urine samples. The obtained results of the determination of levodopa in spiked urine samples are given in Table 2. Satisfactory recovery of the experimental results was found for levodopa. The reproducibility of the method was stated by the mean relative standard deviation (R.S.D.).

Table 2. Analysis of levodopa in spiked urine samples (n=5) by FSCNT/GCE. The concentrations are in µM.

Spiked	Found	Recovery (%)	R.S.D. (%)
0	-	-	-
10.0	10.2	102.0	2.7
20.0	19.8	99.0	1.9
30.0	29.1	97.0	3.2
40.0	41.4	103.5	2.5

4. CONCLUSIONS

A Fe_3O_4@SiO_2/MWCNT nanocomposite is fabricated and used as an electrochemical electrode modifier for a sensitive and selective analysis of levodopa. The MWCNT with interconecting network has excellent electrical conductivity and large surface areas. The grown Fe_3O_4@SiO_2 nanocomposite further increase the electro-active surface area of adsorption and redox reactions of levodopa.

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