



Electrochemical determination of fluvoxamine on mercury nanoparticle multi-walled carbon nanotube modified glassy carbon electrode



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ABSTRACT

An effective electrochemical sensor for the determination of fluvoxamine (FLV) is introduced using mercury nanoparticles multi walled carbon nanotube modified glassy carbon electrode (HgNPs/MWCNTs/GCE). The electrochemical behavior of FLV at the modified electrode was studied using cyclic and differential pulse voltammetric techniques. The peak current for the modified electrode was much larger than that on a GCE, due to the co-effect of MWCNTs and HgNPs on the electrochemical reduction of FLV. The effect of various experimental parameters including pH, scan rate, accumulation potential and time on the voltammetric response of FLV was investigated. The results indicated that the process of the reduction of this compound is irreversible and adsorption controlled. Using the optimal differential pulse voltammetric conditions, the detection limit of $0.01 \mu\text{mol L}^{-1}$, the linear response of peak current on FLV concentration in the range 0.020 to $1.750 \mu\text{mol L}^{-1}$ ($R^2 = 0.998$, $n = 6$) and the relative standard deviation of 0.9% at $1.00 \mu\text{mol L}^{-1}$ concentration level ($n = 10$) were achieved. The method was successfully applied to the determination of FLV in real samples and the results are similar to those declared by HPLC-DAD method. The effects of common interferences on the current response of FLV were investigated.

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1. Introduction

Fluvoxamine (FLV), (*E*)-5-methoxy-1-[4-(trifluoromethyl)phenyl]-1-pentanone *O*-(2-aminoethyl)oxime is an investigational serotonin reuptake inhibitor [1,2]. It has little effect on dopamine and norepinephrine reuptake systems. In addition, apart from binding to sigma 1 receptors, it has a low affinity for other receptors [3,4]. Besides the antidepressant action, FLV shows also an anxiolytic effect. Not surprisingly, several studies have demonstrated its efficacy in major depression, obsessive compulsive disorder, generalized anxiety disorder, panic disorder, social phobia, eating disorders, and post-traumatic stress disorder [5,6]. After oral administration, FLV is nearly completely absorbed from the gastrointestinal tract, and the extent of the absorption is unaffected by the presence of food. Extensive metabolism occurs in the liver, with less than 4% of the dose being excreted unchanged. The drug is excreted in the urine [7].

Several methods for the determination of FLV have been published [8], mostly based on liquid chromatography and HPLC [9–12]. Capillary electrophoresis [13], spectrophotometry [14], GC with previous derivatization step [15], and voltammetry [16,17], have been reported.

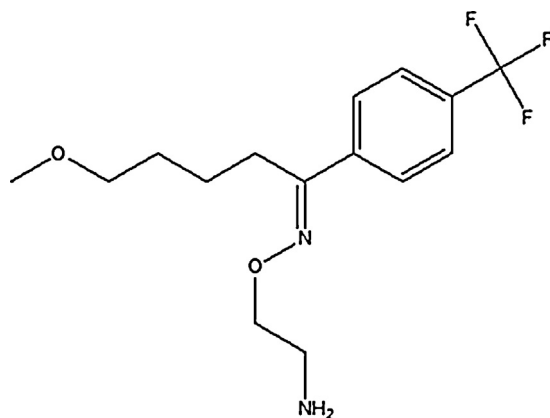
MWCNTs modified electrodes have been used to develop very sensitive adsorptive stripping methods. Adsorptive stripping pulse voltammetric techniques are effective and rapid electroanalytical techniques with well-established advantages including low detection limits and good discrimination against background currents with high sensitivities [18–20].

Metallic nanoparticles (NPs) have attracted much attention, since they show the distinctive physical and chemical properties found in neither bulk nor molecular/atomic systems. The metal NPs can in turn be utilized to modulate the properties and functions of the MWCNTs [21–23]. The metal NPs also show promise to become useful in bioelectronics devices and sensors. As metal nanoparticles, mercuric NPs can act as electron catalyst at the surface of the electrode that influences selectivity and sensitivity structure [24,25].

To the best of our knowledge, there are only two reports on the electrochemical determination of FLV which are on the basis of

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Scheme 1. Structural formula for fluvoxamine (FLV).

polarography methods [16,17] and there is no report on the determination of this compound on the electrodes modified with gold or silver nanoparticles.

Using this modified electrode, the present work found a new voltammetric procedure to trace detection of FLV based on its adsorption at the HgNPs/MWCNTs/GCE. Furthermore, the effects of a wide range of potentially interfering compounds such as sertraline, citalopram, fluoxetine, carbamazepine, some metal ions, and citrate, carbonate, oxalate, glucose and uric acid are examined.

2. Experimental

2.1. Apparatus

A Metrohm Model 827 pH lab (Herisau, Switzerland) pH-meter with a combined glass electrode was used for pH measurements. The size and morphology of the nanoparticles were characterized by scanning electron microscope (SEM114 EDX, XL30 and Philips Netherland). Voltammetric systems were conducted using a potentiostat/galvanostat (Autolab PGSTAT302 N) and it was controlled by a computer using Nova version 1.7 software. Three-electrode cell systems were used to monitor the cyclic and DP voltammograms. A saturated Ag/AgCl electrode, a platinum wire and a modified glassy carbon electrode were used as the reference, auxiliary and working electrodes, respectively. Chromatography was performed using Products HPLC (Knauer, Germany) that consist of a Knauer 1050 HPLC pump, and a Knauer 2850 PDA detector; a Rheodyne injection valve with a 20 μ l loop, was used. Separations were carried out on Macherey nagel column C₁₈ (150 mm \times 2.5 mm, particle size 4 μ m) was employed. For instrumental control, data collection and processing chromat software was employed.

2.2. Reagents

FLV (> 99.0% purity, Scheme 1) was purchased from Sigma-Aldrich. Due to low solubility of FLV in water, a stock solution of FLV (0.001 mol L⁻¹) was made in methanol-water (20–80 v/v), and more dilute solutions (1.0 \times 10⁻³–1.0 \times 10⁻⁸ mol L⁻¹) were prepared by diluting with buffer solutions. All other reagents used were of analytical grade and were purchased from Sigma-Aldrich (US) and Merck (Darmstadt, Germany) Companies. The FLV tablets (50.0 mg) were purchased from a local pharmacy. Phosphate buffers from pH 2.0 to 4.0 (0.1 mol L⁻¹) which were used for pH experiments and supporting electrolyte were prepared in double distilled water (DDW) and all the solutions were prepared with DDW.

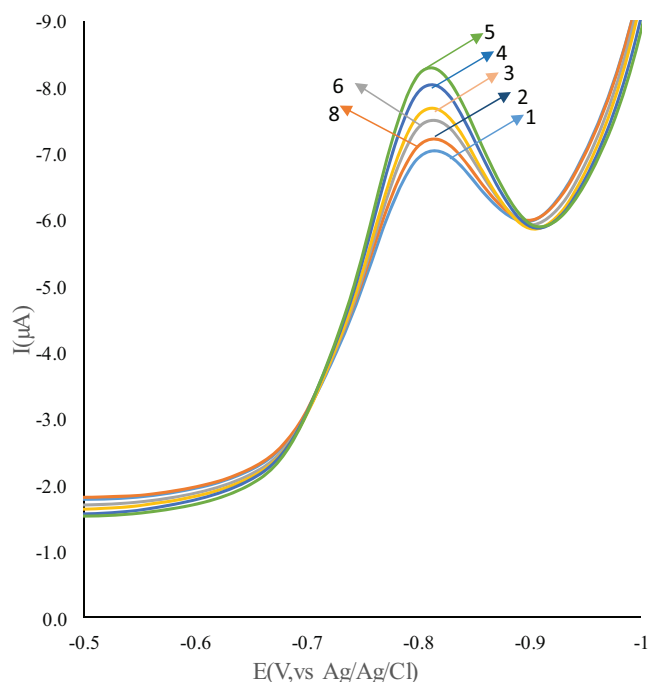


Fig. 1. Effect of deposition cycle numbers of Hg on current response of FLV (0.3 μ mol L⁻¹) in phosphate buffer solution of pH 2.0.

2.3. Preparation of modified HgNPs/MWCNTs/GCE

The mercury nanoparticles were formed in a 1.0 \times 10⁻² mol L⁻¹ Hg(NO₃)₂ solution, prepared by dissolving 0.4 g of mercury(II) nitrate in 100.0 mL of an acidified double distillate water (5% of HNO₃). Prior to modification, the glassy carbon electrode was polished successively with 0.3 μ m Al₂O₃ water slurry using a polishing cloth and it was rinsed with doubly distilled water, afterward, the electrode was sonicated thoroughly with ethanol and water then dried at room temperature. Then a 20.0 mg of MWCNTs was dispersed in a 10.0 mL of 1% sodium dodecyl sulfate (SDS) and ultrasonicated for 20 min until a homogenous suspension of MWCNTs was obtained. Then, 20.0 μ L of the suspension was carefully cast on the surface of the well-polished GCE and dried in the air [26]. The MWCNTs/GCE was thus obtained and then mounted with the help of a Teflon holder in a voltammetric cell provided with an Ag/AgCl reference electrode, a platinum auxiliary electrode, 1.0 mL of mercury(II) nitrate solution, 1.0 mL of 0.1 mol L⁻¹ potassium nitrate solution and 8.0 mL of purified water. The solution was purged with nitrogen for 300 s in order to eliminate the oxygen that was present initially. The formation of HgNPs at the MWCNTs/GCE was carried out by cyclic voltammetry (CV) scanning from 0.10 V to -1.00 V, vs. Ag/AgCl at a scan rate of 50.0 mV s⁻¹ for five cycles. The obtained electrode was named as HgNPs/MWCNTs/GCE. The modified electrode was taken out and rinsed with water. Through changing the cycle number in electro deposition process, the amount and the size of the deposited HgNPs can be controlled. Because the size of the nanoparticles significantly influenced the catalytic efficiency, five cycles was chosen as the optimum cycle number in Hg electro deposition process. Fig. 1 showed that maximum signal can be obtained by electrode which is modified by five cycles. It seems that in modification by increasing the number of cycles to 5, the electrochemical current increases and then decreases; thus, it can be concluded that those five cycles for modification are optimum. The set of electrodes was rinsed with water, and a new clean cell containing the analyte solution was fitted [27].

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