Anal. Bioanal. Electrochem., Vol. 12, No. 7, 2020, 959-969

Analytical& Bioanalytical Electrochemistry

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Full Paper

Synthesis of Pt-SWCNTS Conductive Nanocomposite by Microwave Heated Polyol Strategy; Application for Amplification of 5-Fluorouracil Anticancer Drug Electrochemical Sensor

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Received: 6 June 2020 / Received in revised form: 21 July 2020 / Accepted: 22 July 2020 / Published online: 31 July 2020

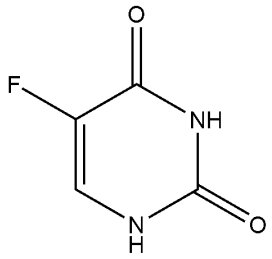
Abstract- In this study, a simple strategy was described for the synthesis of Pt-SWCNTs conductive nanocomposite by microwave heated polyol method and nanocomposite characterized by EDS, FESEM, and XRD method. The Pt nanoparticles were decorated at the surface of SWCNTs with a diameter of 22.3 nm. The synthesized nanocomposite was used for modification of the carbon paste electrode (CPE) in the presence of n-hexyl-3-methylimidazolium hexafluorophosphate (nH3MHP) and paraffin oil as binders. The Pt-SWCNTs/nH3MHP/CPE was showed a good catalytic effect for electro-oxidation of the 5-fluorouracil anticancer drug in aqueous solution. In comparison to CPE, the Pt-SWCNTs/nH3MHP/CPE increased oxidation current of 5-fluorouracil (~4.47 times) and reduce oxidation potential of this anticancer drug ~125 mV. On the other hand, Pt-SWCNTs/nH3MHP/CPE was successfully used for the determination of 5-fluorouracil anticancer drugs in injection samples with acceptable recovery data (96.13%-103.5%). According to recorded results, the sensor has a powerful tool for determination of 5-fluorouracil anticancer drug in real samples.

Keywords- 5-Fluorouracil; Anticancer drug; Microwave heated polyol method; Pt-SWCNTs conductive nanocomposite

1. INTRODUCTION

Attention to new nanostructures for use in various industries has grown significantly in recent years [1-10]. Meanwhile, conductive nanomaterials have received more attention than other nanostructures due to their widespread use in various applications such as fuel cells, batteries, electrical chips, and electrochemical sensors [11-15]. According to scientific reports, carbon nanotubes and metal compounds, especially platinum nanoparticles, have very high electrical conductivity and have played a decisive role in industrial applications [16-20]. For example, platinum nanoparticles are well used in the preparation of fuel cells [21], and carbon nanotubes are one of the most widely used nanomaterials in the preparation of electron exchange sensors [22-30]. Based on this, it can be predicted that nanocomposites based on a combination of carbon nanotubes and platinum nanoparticles can provide high electrical conductivity for the design of electrochemical sensors [31].

5-Fluorouracil (Adrucil) (Scheme 1) is anticancer that is prescribed to treat pancreatic cancer, colon cancer, cervical cancer, breast cancer, esophageal cancer, and stomach cancer over the past 20 years [32]. Due to the high side effects of this drug in the chemotherapy process, dose control used in the treatment of patients is very important and necessary [33].



Scheme 1. Structure of 5-Fluorouracil

Therefore, fast and sensitive analysis of 5-fluorouracil is a biological sample that is a major problem in the chemotherapy process [33-35]. Many analytical sensors were used for the determination of 5-fluorouracil in biological samples [36-38]. In between, electrochemical methods showed more advantages due to fast response and portable ability [39-47]. However, high over-voltage and weak redox signals of 5-fluorouracil are the most important problems in the design of electrochemical sensors for electro-analysis of 5-fluorouracil [48]. Therefore, using conductive mediators for the fabrication of a 5-fluorouracil electroanalytical sensor is very important. In between, attention to ionic liquids and carbon-metal based nanocomposite could be helps to create highly sensitive electro-analytical sensors [49-55]. Nanomaterials with incredible properties have been used as widely used materials in many chemical techniques [56-64]. High conductivity is main advantages of nanomaterials for using in electrochemical sensors [65]. Therefore, the presence study described fabrication of Pt-SWCNTs/nH3MHP/CPE as a highly sensitive electrochemical sensor for determination of 5-

fluorouracil. Two-fold amplification of CPE with Pt-SWCNTs nanocomposite and nH3MHP created a highly sensitive condition for trace level analysis of 5-fluorouracil and improved ability of sensor for determination of this anticancer drug in real samples.

2. EXPERIMENTAL

2.1. Materials and instrument

5-Fluorouracil ≥99%, chloroplatinic acid hydrate ≥99.9%, n-hexyl-3-methylimidazolium hexafluorophosphate, SWCNTs were purchased from Sigma-Aldrich. Sodium hydroxide, graphite powder 99.9%, ammonia anhydrous ≥99.98%, and paraffin oil were purchased from Across Company. A μ-Autolab PGSTAT was used for electrochemical investigation using Ag/AgCl/KClsat as a reference electrode. X' Pert Pro and Mira-3-XMU instruments were used for XRD and FESEM investigations.

2.2. Synthesis of Pt-SWCNTs/nH3MHP/CPE

The ethylene glycol solutions and H_2PtCl_6 were used as precursors for the synthesis of Pt/SWCNTs by microwave heating strategy. The 50 mL ethylene glycol + 0.8 mL potassium hydroxide (0.4 M) was mixed in an erlenmeyer flask under stirring for 10 min and 2.0 mL H_2PtCl_6 solution (0.05 M) was added into erlenmeyer flask and stirring was continued for 10 min. In the next step, 0.1 g of SWCNTs was added into the erlenmeyer flask and dispersed using an ultrasonic strategy for 30 min in distilled water. The erlenmeyer flask was placed in the center of a household microwave oven and heated for 3 min under microwave power of 600 W. The obtained powder was dried at 120 °C for 12 h.2.3. Preparation of Pt-SWCNTs/nH3MHP/CPE

The Pt-SWCNTs/nH3MHP/CPE was prepared by mixing 920 mg graphite powder + 80 mg Pt-SWCNTs in the presence of 10 cc ethanol as a solvent into mortar and pestle. After evaporation of ethanol, paraffin+ nH3MHP (80:20 v:v) was added as a binder and the resulting paste was added end of the glass tube.

3. RESULTS AND DISCUSSION

3.1. Characterization of Pt-SWCNTs nanocomposite

The Pt-SWCNTs were characterized by XRD, EDS, and FESEM methods. The XRD pattern of Pt-SWCNTs showed five planes with miller indexes [002] relative to the carbon nanotubes phase and [111]; [200]; [220] and [311] relative to Pt nanoparticles with JCPDS Card 04-0802 (Fig. 1). The diameter of Pt nanoparticle was calculated 22.3 nm using the Debye-Scherrer equation.

FESEM images of Pt-SWCNTs nanocomposite clearly showed the presence of single-wall carbon nanotubes decorated by Pt nanoparticles (Fig. 2A). According to the FESEM image, Pt

nanoparticle is a presence at the surface of SWCNTs. On the other hand, EDS analysis data show the presence of C, Pt elements that confirms the purity of synthesized Pt-SWCNTs nanocomposite (Fig. 2B).

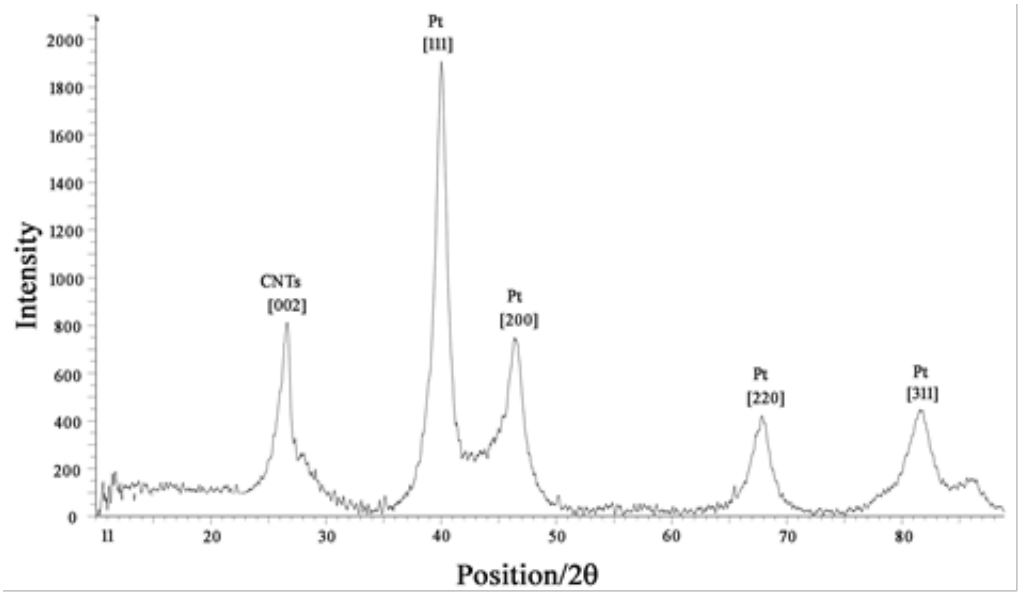
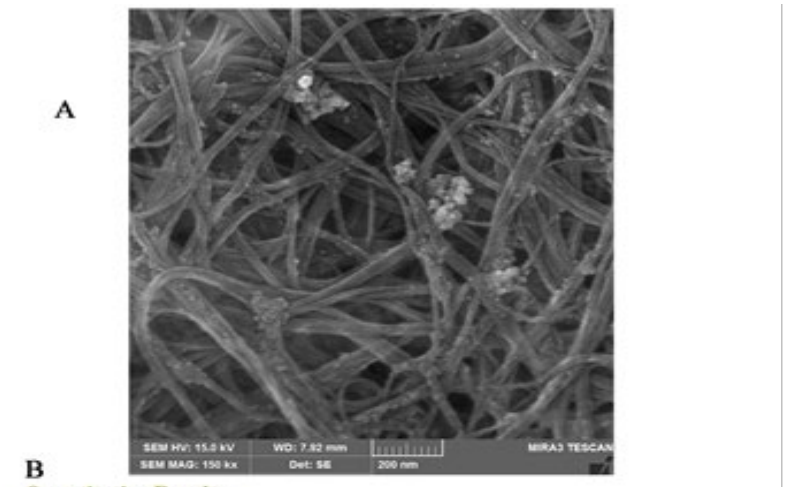


Fig. 1. XRD pattern of Pt-SWCNTs nanocomposite



Elt	Line	Int	Error	K	Kr	W%	A%	ZAF
C	Ka	516.4	9.6279	0.7088	0.3981	71.64	97.62	0.5555
Pt	La	14.0	0.5396	0.2912	0.1636	28.36	2.38	0.5767
				1.0000	0.5617	100.00	100.00	

Fig. 2. A) FESEM image and B) EDS analysis data of Pt-SWCNTs nanocomposite

3.2. Electrochemical investigation

Due to the uracil structure and reported papers, the oxidation behavior of 5-fluorouracil is dependent on pH changes [36]. Therefore, the factor of pH was optimized in the first step. Fig. 3 inset showed a square wave voltammograms of 80 µM 5-fluorouracil in the pH range of 7.5-9.5. As can be seen in fig. 3, with moving pH=7.5 to pH=9.5 the oxidation potential of 5-fluorouracil shifted to a negative value with equation E = - 0.0588 pH + 1.3012 (R^2 = 0.9982) that confirm the value of electron and H^+ in redox mechanism of this anticancer drug is equal. On the other hand, maximum oxidation current for 5-fluorouracil was detected at pH=8.5 and this condition was selected for the next steps.

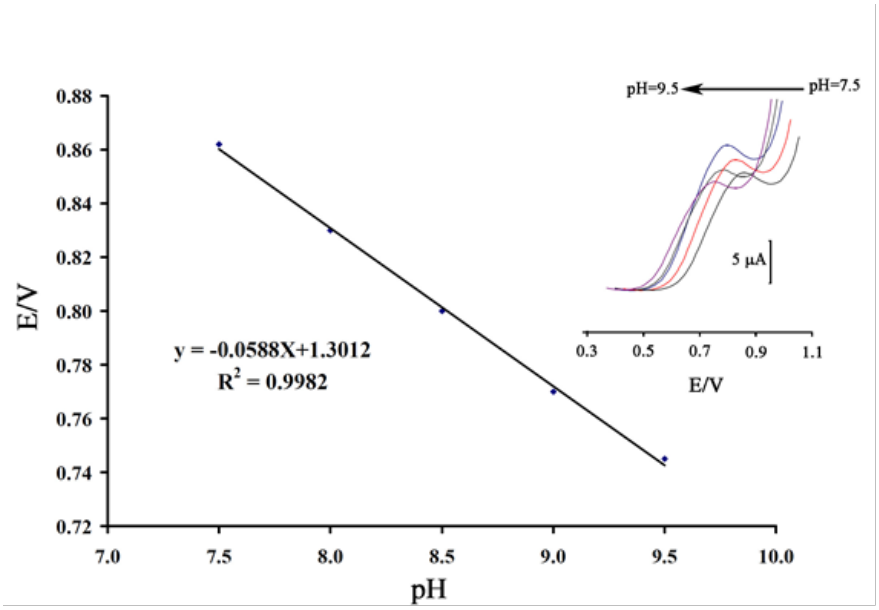


Fig. 3. E-pH curve for electro-oxidation of 80 µM 5-fluorouracil at a surface of Pt-SWCNTs/nH3MHP/CPE. Inset) Relative Square wave voltammograms for electro-oxidation of 80 µM 5-fluorouracil

For study role Pt-SWCNTs and nH3MHP on modification of CPE at the surface of Pt-SWCNTs/nH3MHP/CPE, square wave voltammograms of 80 µM 5-fluorouracil was recorded at the surface of CPE (fig. 4 curve a), Pt-SWCNTs/CPE (Fig. 4 curve b), nH3MHP/CPE (Fig. 4 curve c) and Pt-SWCNTs/nH3MHP/CPE (Fig. 4 curved).

Oxidation currents 3.78 µA, 9.44 µA, 13.5 µA, and 16.9 µA were recorded for curves a-d, respectively. As can be seen, with the modification of CPE with Pt-SWCNTs or nH3MHP, the oxidation signal of 5-fluorouracil was improved. In addition, the best oxidation signal was recorded at the surface of Pt-SWCNTs/nH3MHP/CPE that confirm the modification of CPE with Pt-SWCNTs and nH3MHP could be improved oxidation current of 5-fluorouracil ~4.47 times. Fig. 4 inset shows current density data relative to recorded square wave voltammograms

of 80 µM 5-fluorouracil at the surface of different electrodes that confirm good electrical conductivity of Pt-SWCNTs and nH3MHP as mediators.

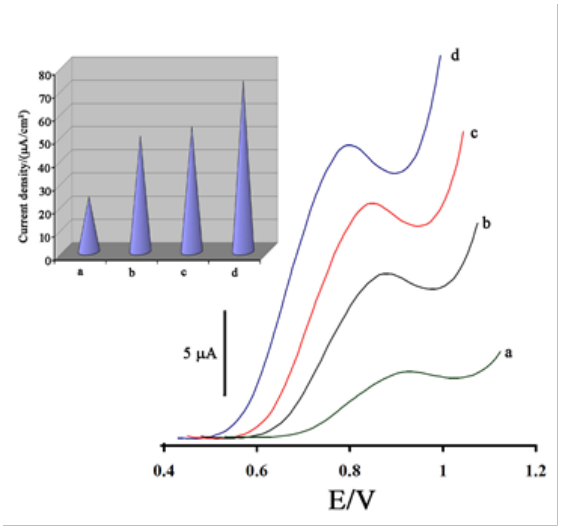


Fig. 4. SWVs of 80 µM 5-fluorouracil at surface of a) CPE, b) Pt-SWCNTs/CPE, c) nH3MHP/CPE and d) Pt-SWCNTs/nH3MHP/CPE. Inset) relative Square wave voltammograms recorded at the surface of different electrodes.

The CVs of 700 µM 5-fluorouracil at scan rate ranges 50-200 mV/s was recorded at surface of sensor (Fig. 5 inset).

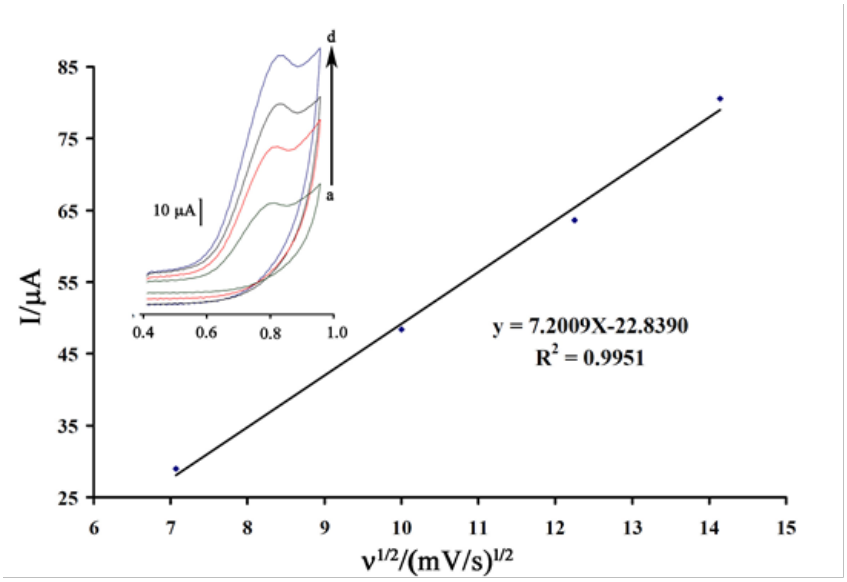


Fig. 5. Current- $\nu^{1/2}$ curve for electro-oxidation of 700 µM 5-fluorouracil at surface Pt-SWCNTs/nH3MHP/CPE. Inset) CVs of 700 µM 5-fluorouracil recorded at scan rates a) 50, b) 100, c) 150 and d) 200 mV/s.

A linear relation between oxidation current of 5-fluorouracil and v1/2 with the equation of I = 7.2009 v1/2 - 22.8390 (R^2 = 9951) was observed that confirm diffusion process [66-70] for the electro-oxidation reaction of 5-fluorouracil at surface of sensor (Fig. 5).

SWV method was used for study linear dynamic range and limit of detection 5-fluorouracil at the surface of Pt-SWCNTs/nH3MHP/CPE and results showed a linear relation between current and 5-fluorouracil concentration in the range 1.0 nM-520 µM (Fig. 6). The detection limits of 5-fluorouracil were calculated 0.4 nM using 3Sb/m equation.

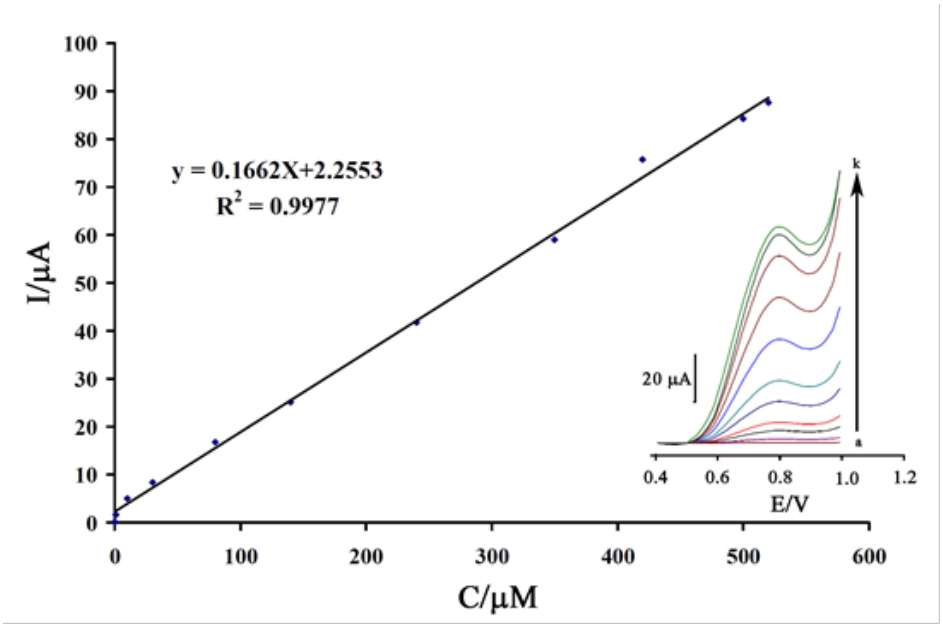


Fig. 6. Current-concentration diagram for electro-oxidation of 5-fluorouracil at surface Pt-SWCNTs/nH3MHP/CPE. Inset) Square wave voltammograms a) 0.001; b) 1.0; c) 10; d) 30; e) 80; f) 140; g) 240; h) 350; i) 420; j) 500 and k) 520 µM recorded at surface of Pt-SWCNTs/nH3MHP/CPE.

The stability of Pt-SWCNTs/nH3MHP/CPE as a new electrochemical sensor was investigated for a determination of 10.0 µM 5-fluorouracil in period time 30 days. According to recording results, a 90% initial oxidation signal of 5-fluorouracil remains after 30 days that confirm the good stability of Pt-SWCNTs/nH3MHP/CPE as a new electrochemical sensor for determination of 5-fluorouracil.

The selectivity of Pt-SWCNTs/nH3MHP/CPE as a new electrochemical sensor for determination of 10 µM 5-fluorouracil was check in the presence of some organic and inorganic interference with acceptable error 5%. Results showed nor 1000-fold of Na^+, Cl^-, K^+, and Br^-, 750-fold of vitamin B2, and glucose and 300-fold ascorbic acid did not affect the selectivity.

Table 1. Real sample analysis data for determination of 5-fluorouracil using Pt-SWCNTs/nH3MHP/CPE

Sample	Added (μM)	Expected (μM)	Founded (μM)	Recovery%
Injection	---	---	2.51± 0.21	---
	5.00	7.51	7.22± 0.46	96.13
Pharmaceutical serum (dextrose saline)	---	---	<LOD	---
	10.00	10.00	10.35± 0.76	103.5

The ability of Pt-SWCNTs/nH3MHP/CPE as a new electroanalytical sensor was a check for the determination of 5-fluorouracil in injection and pharmaceutical samples without any pre-preparation. The real sample analysis data are a presence in table 1 and confirm the high-performance ability of Pt-SWCNTs/nH3MHP/CPE for the determination of 5-fluorouracil in real samples.

4. CONCLUSION

In this research, a highly sensitive voltammetric sensor was fabricated for the determination of 5-fluorouracil. For this goal, CPE was amplified with Pt-SWCNTs and nH3MHP. The Pt-SWCNTs/nH3MHP/CPE was showed catalytic activity for the determination of 5-fluorouracil and increased oxidation current of 5-fluorouracil ~4.47 times. In addition, the Pt-SWCNTs/nH3MHP/CPE was effectively used for the determination of 5-fluorouracil in injection samples.

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