

FULL PAPER

Novel electrochemical sensing platform for caffeine using three dimensional NiO nanowrinkles modified glassy carbon electrode

Effat Sharifi Pour^a | Hadi Beitollahi^{b,*} ^aDepartment of Chemistry, Graduate University of Advanced Technology, Kerman, Iran^bEnvironment Department, Institute of Science and High Technology and Environmental Sciences, Graduate University of Advanced Technology, Kerman, Iran

We addressed electrochemical behaviors of caffeine at the modified sensor (three dimensional NiO nanowrinkles modified glassy carbon electrode (3D NiO NWs/GCE)) in an aqueous medium with cyclic voltammetry, differential pulse voltammetry and chronoamperometry. The modification of sensor was performed by drop-casting process. Electrochemical investigations showed that the fabricated 3D NiO NWs/GCE sensor exhibited excellent catalytic performance for the oxidation of caffeine. Based on the optimal conditions of pH=7.0 in CV, caffeine oxidation happens at a potential ~200 mV less positive than that of the unmodified GCE. The current response of 3D NiO NWs/GCE sensors presents a remarkable sensitivity based on CV. The calibration plot is linear (R^2 : 0.9991) over the concentrations ranging between 0.1 and 800.0 μM of caffeine. Limit of detection (LOD) was 0.03 μM for caffeine.

***Corresponding Author:**

Hadi Beitollahi

Email: h.beitollahi@yahoo.com

Tel.: +983433776611

KEYWORDS

Caffeine; three dimensional NiO nanowrinkles; glassy carbon electrode; electrochemical sensor.

Introduction

Caffeine has been introduced as one of the natural alkaloids, which is a member of the class of compounds known as xanthenes. In fact, theobromine and theophylline are other known members of the above class [1]. Caffeine has been considered as a heterocyclic nitrogen that consists of organic compounds with the fundamental features of an amine. Actually, caffeine naturally occurs in numerous plant materials like coffee beans, cola nuts and beans, tea leaves, etc. as one of the main constituents that functions as one of the natural pesticides in plants [2]. It is a psycho-active substance with the extensive uses worldwide because of its stimulant features. Researchers have reported stimulation of the central nervous system (CNS) and cardio-vascular system as one of

the effects of caffeine [3]. Moreover, its moderate use of Caffeine is correlated with lower risks of numerous chronic illnesses like Parkinson's, diabetes, dementia, colon cancer as well as liver disease [4]. Nonetheless, higher volumes of caffeine may result in nausea, trembling, seizure and nervousness [5]. Moreover, this material causes cardiovascular diseases, kidney malfunction, as well as hyper-activity [6]. Therefore, researchers actively investigate the construction of a simplified, reliable and inexpensive method to detect and quantify caffeine. Analytical methods have attracted more attention in recent years in separation and detection systems [7-13].

Several analytical techniques are available to detect caffeine in dosages and human fluids accurately like HPLC [14,15], gas

chromatography mass spectrometry [16], spectrometry [17], spectrofluorometry [18], and electrochemistry [19-21]. Some of these methods are expensive, require utilization of numerous organic solvents, and involve skillful workforce and laborious preparation process of the samples.

Among the various analytical techniques [22,23], researchers have performed many studies on the electrochemical techniques because of their faster responses, higher sensitivities, simple operations, and possible miniaturization [24-28]. In addition to the absence of the preparation process of the samples, appropriateness for real-time monitoring with the inexpensive devices has provided the other major benefit of electrochemical techniques. Nonetheless, successfulness of electrochemical techniques relies upon the function of the working electrode materials due to dependence of analytical factors on the responses of working electrode. Moreover, researchers have illustrated working electrode substances with various modification instructions to detect analyte [29-37]. Hence, experts in the field must focus on the design of the modified electrodes because of the gradual passivation or pollution of the unmodified solid working electrodes as a result of absorbing the electro-chemically generated products.

The unique properties of NPs make them an extremely valuable modifying material, being used for electrochemical applications [38-42]. Experts in the field have largely applied metal oxide NPs in building electrochemical sensors because of their promising physico-chemical features [43,44]. Moreover, they have constructed metal oxide NPs with various morphologies using versatile techniques. The keys to the performance of the metal oxide NPs in electro-analysis are larger ratio of surface to volume, higher surface reaction activities, as well as higher catalytic efficiencies [45,46]. In addition, researchers have demonstrated that using metal oxide NPs enhance the response

duration, linear ranges, LOD, prolonged stability as well as reproducibility of the electrochemical sensors [47-49]. In fact, it is possible to achieve strong affinity of metal oxide NPs to the surfaces of the working electrode using diverse procedures like physical absorption, electro-deposition, electro-polymerization, and chemical covalent bonding. Besides, researchers have been specially interested in Nickel oxide (NiO) that is one of the p-type semiconductors with a wider bandgap equal to 3.7 eV under room temperature and higher isoelectric point (IEP) of ~ 10.7 , as the modifying agents in the field of electrochemical sensing due to higher catalytic activities in basic media nickel redox centers for oxidizing several organic compounds with few costs in comparison with other catalysts, in particular, noble metal [50-53].

In the current work, an enhanced sensor based on 3D NiO NWs/GCE was devised for sensitive detection of caffeine. The 3D NiO NWs/GCE exhibited obvious electrocatalytic activities for caffeine oxidation.

Experimental

Apparatus and chemicals

In this step, electrochemical measurements were done using an Autolab potentiostat/galvanostat. Measurements were performed at room temperature, with a single component 3-electrode cell that had a platinum auxiliary electrode and an Ag/AgCl (3 M KCl) reference electrode. 3D NiO NWs/GCE was used as the working electrode and a Metrohm 827 pH-meter was used for controlling the pH of solutions. Each chemical was of analytical reagent grade, bought from Merck Company in Darmstadt, Germany. In addition, we applied doubly distilled-water. Caffeine and all other reagents were of analytical grade and were prepared from Merck chemical company.

Electrode preparation

For preparing the 3D NiO NWs modified GCE, 1 mg of 3D NiO NWs was dispersed in 1 mL distilled water and ultra-sonicated for nearly thirty minutes. Then, 4 μL of the suspension was coated on the GCE surface and finally dried at the room temperature.

Results and discussion

Electrocatalytic oxidation of caffeine at 3D NiO NWs/GCE

The effects of the pH of solution on the oxidation responses of 3D NiO NWs/GCE for 200.0 μM caffeine was carefully investigated by CV in 0.1 M phosphate buffer solution (PBS) in various pH-values ($2.0 < \text{pH} < 9.0$). It

is notable that under pH 7.0 of PBS, we observed the best voltammetric responses; i.e., the largest peak current. Finally, we chose pH 7.0 in 0.1 M PBS for other analytical experimentations.

The cyclic voltammograms (CVs) recorded by bare GCE, as shown in Figure 1, curve b, and CuO NFs/GCE as shown in curve a, were carried out using 50 mVs^{-1} scan rate in 0.1 M PBS at a pH of 7.0, including 200.0 μM caffeine. The anodic peak potential for caffeine oxidation at the bare GCE was ~ 1500 mV in comparison to ~ 1300 mV for on the 3D NiO NWs/GCE. Moreover, the current value increased in modified electrode (3D NiO NWs/GCE) which revealed that the 3D NiO NWs/GCE had electrocatalytic behavior to the caffeine oxidation.

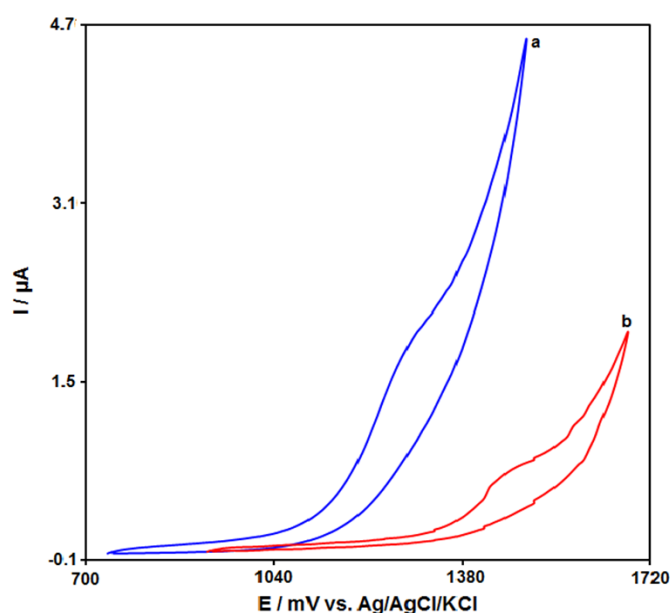


FIGURE 1 Cyclic voltammograms of (a) 3D NiO NWs/GCE and (b) bare GCE in 0.1 M PBS (pH 7.0) in the presence of 200.0 μM caffeine the scan rate 50 mVs^{-1}

Effect of scan rate

We determined the effects of the potent scan rate on the oxidation current of caffeine on the surface of 3D NiO NWs/GCE (Figure 2). Enhancing the potential scan rate elevated

the peak current. Additionally, the oxidation process was diffusion-controlled that was due to the linear dependence of the anodic peak current (I_p) on the square root of the potent scan rate ($v^{1/2}$) within a wider ranges between 10 and 400 mVs^{-1} (Figure 2.inset).

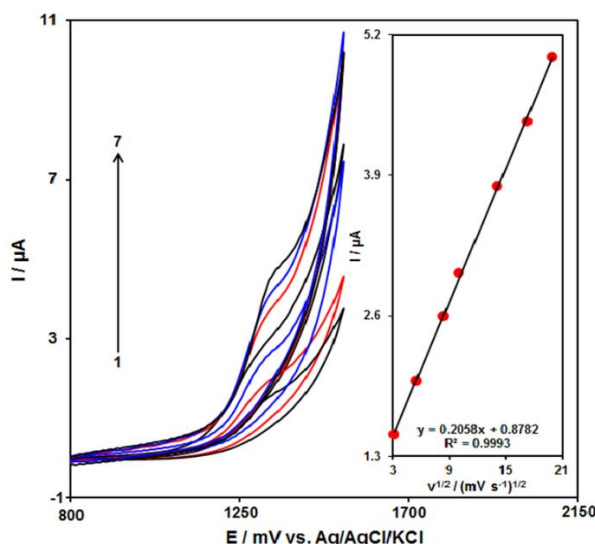


FIGURE 2 (A) Cyclic voltammograms of 3D NiO NWs/GCE in 0.1 M PBS (pH 7.0) containing 200.0 μM of caffeine various scan rates; numbers 1–7 correspond to 10, 30, 70, 100, 200, 300, and 400 mV s^{-1} . Inset: Variation of anodic peak current vs. square root of scan rate

Chronoamperometric measurements

With the confirmation of diffusion process during the caffeine oxidation process on the surface of 3D NiO NWs/GCE, the chronoamperometric method with applied potential of 1350 mV was employed for determining diffusion coefficient (D) of caffeine (Figure 3). Experimental outputs of I

versus $t^{-1/2}$ were drawn by Figure 3A, with the best fits for various concentrations of caffeine. In the next step, we plotted final slopes that corresponded to the straight lines in Figure 3A, plotted against caffeine concentration (Figure 3B). Finally, mean value of D has been computed $1.3 \times 10^{-6} \text{ cm}^2/\text{s}$ based on the Cottrell equation and final slope [54].

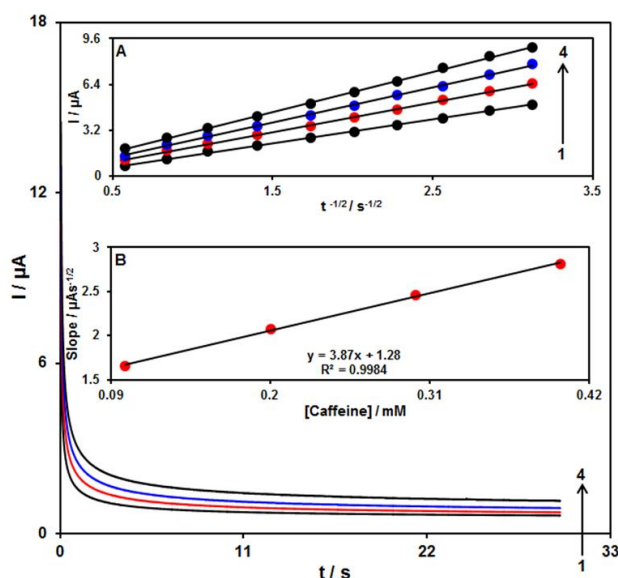


FIGURE 3 Chronoamperograms obtained at 3D NiO NWs/GCE in 0.1 M PBS (pH 7.0) for different concentration of caffeine. The numbers 1–4 correspond to 0.1, 0.2, 0.3 and 0.4 mM of caffeine. Insets: (A) Plots of I vs. $t^{-1/2}$ obtained from chronoamperograms 1–4. (B) Plot of the slope of the straight lines against caffeine concentration.

DPV detection of caffeine on the surface of the new sensor

In this step, we used DPV for investigating voltammetric sensor of 3D NiO NWs/GCE towards detecting caffeine detection. Figure 4 showed the differential pulse voltammograms of caffeine with diverse concentrations in 0.1 M PBS at a pH of 7.0 (Step potential=0.01 V and pulse amplitude=0.025 V). We showed linear

enhancement of the oxidation current with caffeine concentrations in ranges from 0.1–800.0 μM (Figure 4, inset). In addition, the detection limit, C_m , of caffeine was obtained using the following equation.

$$C_m = 3S_b/m$$

The detection limit is 0.03 μM . The comparison of the results for the detection of caffeine with different modified electrodes in the literature is listed in Table 1.

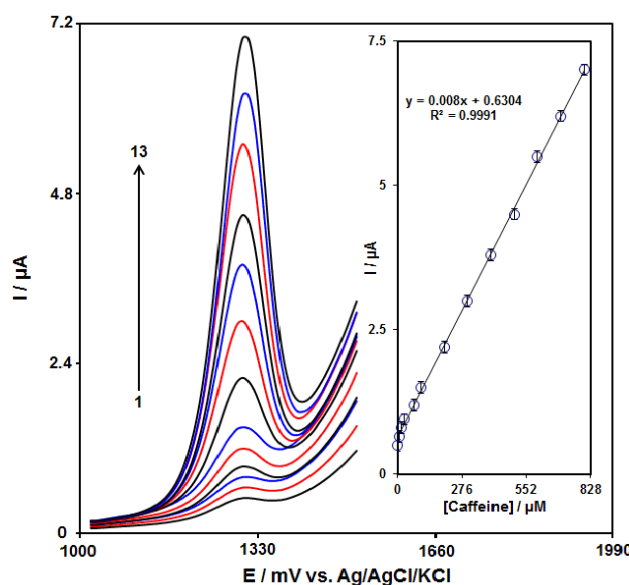


FIGURE 4 Differential pulse voltammograms of 3D NiO NWs/GCE in 0.1M PBS (pH 7.0) containing different concentrations of caffeine. Numbers 1–13 correspond to 0.1, 5.0, 15.0, 30.0, 70.0, 100.0, 200.0, 300.0, 400.0, 500.0, 600.0, 700.0 and 800.0 μM of caffeine. Inset: the plots of the electrocatalytic peak current as a function of caffeine concentration in the range of 0.1–800.0 μM .

TABLE 1 A comparison of electrochemical techniques for the detection of caffeine at the prepared electrode in this work and some other works

Ref.	Limit of Detection	Linear Range	Method	Electrochemical Sensor
[55]	1.2×10^{-7} M	2.5×10^{-7} – 1.0×10^{-4} M	DPV	Single-walled carbon nanotubes/carbon ceramic electrode
[56]	2.0×10^{-7} M	4.0×10^{-7} – 8.0×10^{-5} M	DPV	Nafion and graphene oxide/glassy carbon electrode
[57]	18×10^{-9} M	2 - 120 μM	DPV	CuS nanoparticles/carbon paste electrode
[58]	0.00194 mg/L	0.00388–4.85 mg/L	DPV	Electrodepositing zinc oxide on multiwalled carbon nanotube/glassy carbon electrode
This work	0.03 μM	0.1- 800.0 μM	DPV	3D NiO NWs/GCE

Conclusion

The present study demonstrated constructing a 3D NiO NWs/GCE and its application in the determination of caffeine. Finally, this new sensing method displayed the better functions in electrochemical detection of caffeine in the wider linear ranges (0.1-800.0 μM) and lower LOD (0.03 μM). Considering the very good results, it seems that the 3D NiO NWs/GCE can be considered as a new method in the development of accurate and inexpensive electrochemical sensors.

Orcid:

Hadi Beitollahi: <https://orcid.org/0000-0002-0669-5216>

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