

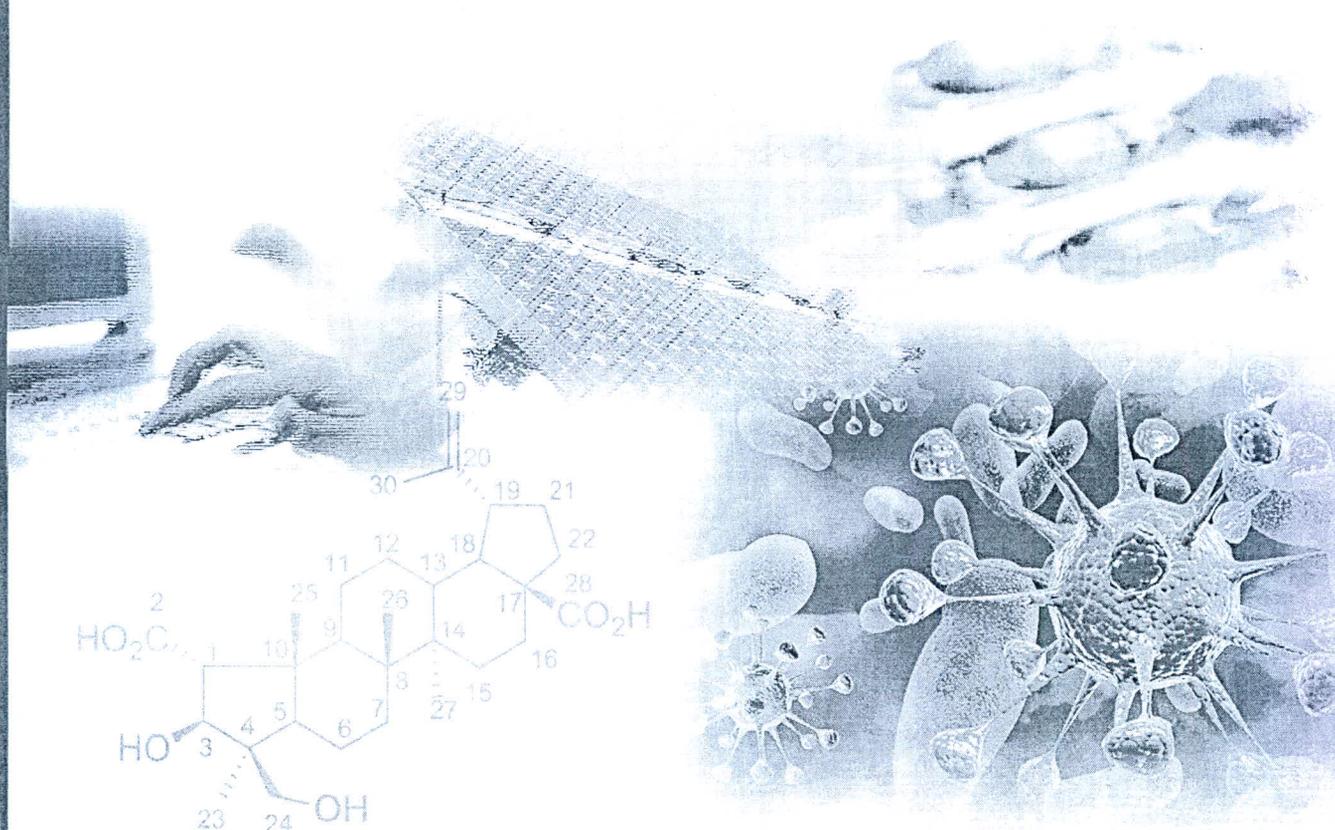
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การเผยแพร่ 1

วารสารมหาวิทยาลัยศรีนครินทรวิโรฒ



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ความเป็นมา

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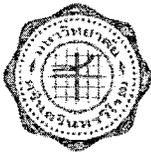
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- บทความที่ส่งถึงกองบรรณาธิการ ขอสงวนสิทธิ์ที่จะไม่ส่งคืน ผู้เขียนจะต้องทาสีเนาส่วนตัวไว้



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Editorial

The purpose of the Srinakharinwirot Science Journal is to present the research work from the researcher who want to present their research work to the interested people in science and technology field. Now, this issue is a special one for ceremony with the 60 th years of Srinakharinwirot University. However, all papers were reviewed by the the great peer reviewer from the Pure and Applied Chemistry International Conference (PACCON 2011) committee. PACCON 2011 which was held during January 5-7, 2011 at the Miracle Grand Hotel in Bangkok, Thailand. It is my great honor and pleasure to get those papers from the conferences to present as a special issue of Srinakharinwirot University Science Journal.

Sincerely yours

Associate Professor Dr.Pornpimol Muangthai

Editor in Chief

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AMPEROMETRIC DETERMINATION OF NITRITE ON A PENCIL CARBON ELECTRODE USING $K_4Fe(CN)_6$ MEDIATOR

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Abstract

A low cost amperometric nitrite sensor was developed using a pencil carbon working electrode with a $K_4Fe(CN)_6$ mediator. The pencil carbon electrodes were characterized with cyclic voltammetry in an $K_4Fe(CN)_6$ solution. The $K_4Fe(CN)_6$ mediator was added into 0.05 M phosphate buffer solution (pH 7.5). It compared to without the mediator, it was found that the presence of the $K_4Fe(CN)_6$ mediator enhanced the sensitivity and selectivity of nitrite sensor. Amperometric potential reduction was applied at 0.0 V. An analysis of the system showed a detection limit as 1 mM, a linear range as 4.5 to 65 mM, and a correlation coefficient as 0.99.

Keywords: Amperometric, Nitrite, Pencil carbon electrode, $K_4Fe(CN)_6$

Introduction

Nitrite is a precursor in the formation of nitrosamines, many of which have been shown as potent carcinogens in human bodies. It is an important contamination in environment, beverages, and food products as a preservative. Thus, its determination is very crucial for the environmental reasons and for public health. In the recent year, many methods have been developed such as spectrophotometry [1-5], ion chromatography [6-12], gas chromatography-mass spectrometry [13-18], chemiluminescence [19-25], capillary electrophoresis [26-29], and flow injection analysis [30-31]. The potential

low cost, portability rapid response and simple use of electrochemical devices provide a number of attractive options for nitrite determination [32-48].

Many researcher groups studied electrochemical for nitrite analysis such as Jiang, L. [39] study nitrite sensor by development of chitosan-carboxylated multiwall carbon nanotube at a glassy carbon electrode for study the oxidation behavior of nitrite using cyclic voltammetry and differential pulse voltammetry modes. Liu, S. and Ju, H. [48] investigated of nitrite biosensor based on the direct electron transfer of hemoglobin (Hb) and a sensing mechanism was combined the

advantageous features of colloidal gold nanoparticle and carbon paste technology. Zhu, N. [47] describes the nitrite determination based on poly(aminoamine)-modified carbon nanotubes with covalent bonding. Nitrites are electroactive species at carbon electrodes. However, their reduction requires undesirably high overvoltage. As a result, voltammetric determination of nitrite suffers from interference by other readily reducible compounds. In this work the author would have to improve the selectivity nitrite determination, by lowering the applied potentials which can be achieved by adding $K_4Fe(CN)_6$ electrocatalyst as a mediator in phosphate buffer solution.

Materials and Methods

All the electrochemical measurement, including cyclic voltammetry (CV) and amperometry were carried out with a CHI1230A electrochemical analyzer. A conventional three electrode system was used, consisting of a pencil electrode (STAEDTLER; id. 0.5 mm) as a working electrode, platinum wire as an auxiliary electrode and an Ag/AgCl (3 M) as a reference electrode. The cyclic voltammetric method of the pencil electrode in solution of 0.1 M KCl containing $K_3Fe(CN)_6/K_4Fe(CN)_6$ were recorded. The scan rates were set as 5–1000 mV/s. The amperometric method of a pencil electrode in solution of 0.05 M phosphate buffer (pH 7.5) with 20 mM $K_4Fe(CN)_6$ with the stirred solution were measured for the nitrite determination. Amperometric experiments were carried out in a stable system operated at 0.0 V.

Results

Figure 1 shows CV curves at the pencil electrode at different scan rates in 0.1 M KCl electrolytes containing 0.005 M $[Fe(CN)_6]^{3-/4-}$ redox couple.

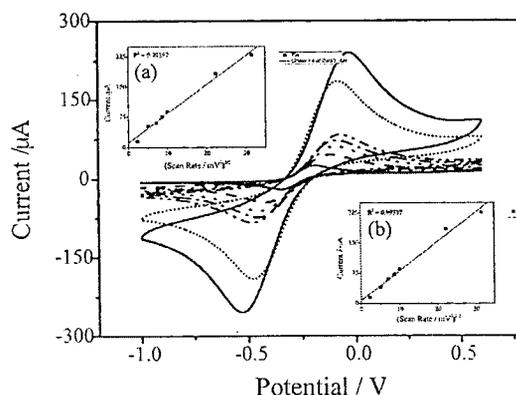


Figure 1 CV curves at a pencil electrode in 0.1 M KCl + 0.005 M $[Fe(CN)_6]^{3-/4-}$ at the scan rates of 5, 25, 50, 75, 100, 500, and 1,000 mV/s (from inner to outer). The insets are relative to a square root of scan rate of anodic current (a) and cathodic current (b).

The symmetrical anodic and cathodic peaks associate with the oxidation and reduction of the ferricyanide-ferrocyanide couple at the pencil-solution interface. The ratios of anodic/cathodic peak currents approach one, which indicates that both ferrocyanide and ferricyanide are stable in solution and the reversibility of the electrode process. The insets of Figure 1, that relative of a square root of scan rate of anodic current (a) and cathodic current (b) are shown with $r^2 = 0.99387$ and 0.99337 , respectively. The anodic peak potential shifts positively with the rising of the scan rate, while the peak

current increases.

In figure 2 shows the amperograms of the three additions of KNO_2 (at 200, 250 and 300 s) in different concentration 0.00 mM, 9.68 mM, 19.06 mM and 28.19 mM of $\text{K}_4\text{Fe}(\text{CN})_6$ in 0.05 mM phosphate buffer (pH 7.5) solution at the pencil electrode by fixing potential 0.0 V.

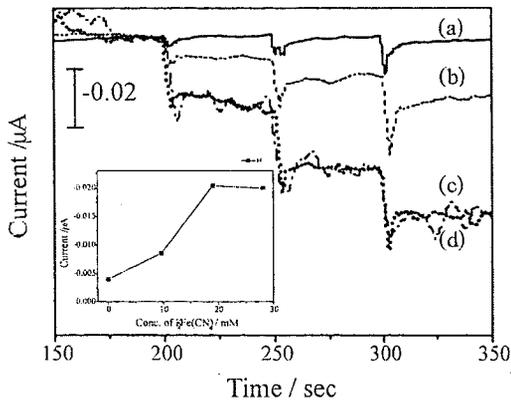


Figure 2 Amperograms of the each addition 10 μL of 15 mM KNO_2 in 0.05 M phosphate buffer (pH 7.5) with different concentration of (a) 0.0 mM, (b) 9.68 mM, (c) 19.06 mM, and (d) 28.15 mM $\text{K}_4\text{Fe}(\text{CN})_6$ at a pencil electrode and fixed potential 0.0 V. The inset was relative to concentration of $\text{K}_4\text{Fe}(\text{CN})_6$ and average cathodic current KNO_2 ($n=3$).

The current was increased from $-0.004 \mu\text{A}$, $-0.0086 \mu\text{A}$, $-0.0204 \mu\text{A}$, and $-0.02 \mu\text{A}$ while increased the concentration of $\text{K}_4\text{Fe}(\text{CN})_6$ as 0.00 mM, 9.68 mM, 19.06 mM and 28.19 mM (Figure 2a, 2b, 2c and 2d), respectively. The highest current of KNO_2 was occurred at the 19.06 mM $\text{K}_4\text{Fe}(\text{CN})_6$

(Figure 2c), that higher than without $\text{K}_4\text{Fe}(\text{CN})_6$ about 5 times and the reaction was responded time less than 2 seconds. This phenomena proved that the $\text{K}_4\text{Fe}(\text{CN})_6$ have been potent to catalyze the KNO_2 . The NO_2^- was reduced to NH_2OH by Fe^{2+} and then Fe^{2+} change to Fe^{3+} . The Fe^{3+} was reduced to Fe^{2+} by getting one electron from the electrode and change to Fe^{2+} for oxidized the next NO_2^- . The electrocatalysis reaction would be expressed as equation (1) and (2) [49].

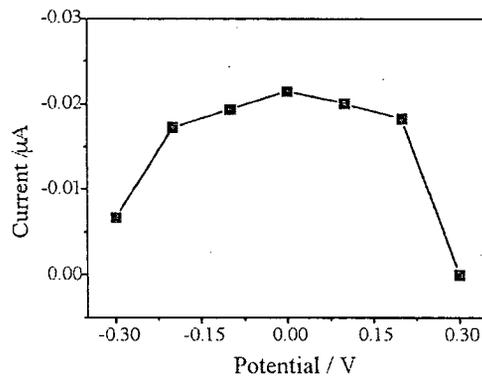
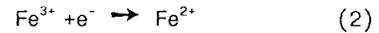
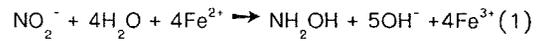


Figure 3 Effect of potential on determination 5 mM KNO_2 in 0.05 M phosphate buffer (pH 7.5) + 20 mM $\text{K}_4\text{Fe}(\text{CN})_6$ solution at a pencil electrode using amperometric determination.

Figure 3 shows a typical amperometric response (the steady-state current curve) during successively 5 mM nitrite to pH 7.5 phosphate buffer with 20 mM catalyst of $\text{K}_4\text{Fe}(\text{CN})_6$. Under the various applied potentials of $-0.3-0.3 \text{ V}$

(vs. Ag/AgCl), we found that the current increased from -0.3 V and reach the maximum at 0.0 V. When the applied potential higher than 0.1 V, the current quite stable until 0.25 V and its immediately dropped at 0.3 V. The potential at -0.3 and 0.3 V were giving the lowest current due to these potentials were not optimum potential for the electrocatalytic reduction of nitrite by the $K_4Fe(CN)_6$. The potential at 0.0 V was selected for further experiments.

Amperometric curve for determination KNO_2 in 0.05 M phosphate buffer (pH 7.5) mixed 20 mM $K_4Fe(CN)_6$ were recorded by polarizing the pencil working electrode at 0.0 V in a stirred solution. The linear curve was presented in Figure 4.

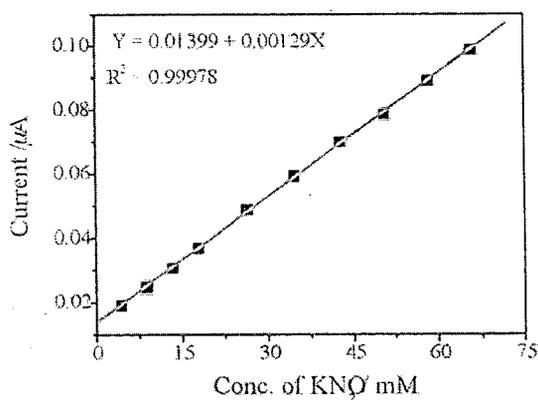


Figure 4 The linear correlation curve of cathodic current at 0.0 V vs. the concentration of KNO_2 ($4.5 - 65$ mM).

The KNO_2 could be determinates in the concentration of $4.5-65$ mM by the amperometric method. Sensitivity was $0.00129 \mu A/mM$ and a detection limit of 1 mM was calculated at a signal-to-noise ratio of 3.

Conclusions and Discussion

Pencil electrode for KNO_2 determination was investigated on $K_4Fe(CN)_6$ catalyst solution. The resulting sensor was used amperometric method that received the linear $4.5-60$ mM, the sensitivity $0.00129 \mu A/mM$ and the detection limit 1 mM.

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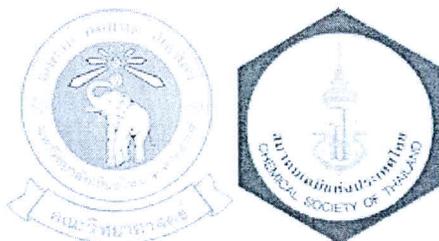
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A LOW COST OF NITRITE DETERMINATION BY SQUARE WAVE VOLTAMETRIC METHOD USING A GOLD NANOPARTICLE MODIFIED PENCIL CARBON ELECTRODE

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Abstract: A sensitive and stable nitrite sensor was developed using Au nanoparticles modified a pencil carbon electrode. The scanning electron microscopy (SEM) and electrochemical characterization confirmed the electrodeposited Au nanoparticles on a pencil carbon electrode. The Au nanoparticles modified pencil carbon electrode providing an excellent catalytic ability for NO₂⁻ determination exhibited the oxidation peak at 0.85 V by square wave voltammetry. The nitrite sensor gave a low detection limit of 30 μM and a long linearity range between 100 μM – 2.5 mM with a high sensitivity of 7.60 μA mM⁻¹.

1. Introduction

Voltammetry has been used widely in the investigation of redox reactions and the analysis of organic or inorganic substances using various available electrodes. Among these, the pencil carbon electrode (PCE) provides a lot of advantages such as high electrochemical reactivity, commercial availability, good mechanical rigidity, disposable, low costs, low technology and easy modification. Additionally it was reported that pencil electrodes offer a renewal surface, which is simpler and faster than polishing procedures, the results is reproducible for the individual surfaces. It can also be miniaturized. Therefore PCEs have been increasingly utilized in many different electroanalytical applications recently, such as, application to determine of trace metals[1], DNA[2,3,4,5,6,7], sulfide[8], immunoassay and some drugs in pharmaceutical formulations[9,10,11]. The PCE, when combined with a more highly sensitive and accurate voltammetric technique such as square-wave or differential pulse voltammetry, can lead to an attractive trace analysis technique.

Nitrite is a precursor in the formation of nitrosamines, many of which have been shown as potent carcinogens in human bodies. It is an important contaminant in environment, beverages, and food products as a preservative. Thus, its determination is very important for environmental reason and for public health. In recent year, many methods have been developed such as spectrophotometry [12,13,14], ion chromatography, gas chromatography-mass spectrometry [15], chemiluminescence, and capillary electrophoresis [16,17,18]. The alternative electrochemical devices providing the potential of low cost, portability, rapid

response and simple use are attractive for nitrite determination.

In this research, the higher sensitivity of nitrite determination can be achieved by using AuNPs electrodeposition as electrocatalyst in phosphate buffer solution

2. Materials and Methods

2.1 Reagents

Potassium nitrite was obtained Carlo Erba Reagents, France. Hydrochloric acid 37%, copper(II)sulfate pentahydrate and sodium sulfate were purchased Merck (Germany). Potassium chloride was from Australia. Potassium hexacyanoferrate(II)trihydrate and potassium hexacyanoferrate(III) were obtained Riedel-dehaen. Sulfuric acid 98% was purchased from Lab scan analytical science Thailand. Deionized water was obtained from a Milli-Q-gradient system (Millipore, $R \geq 18.2 \text{ M}\Omega \text{ cm}$).

2.2 Apparatus and electrochemical measurements

Electrochemical measurements were conducted using an Electrochemical Analyzer (Model 1230A, CH Instruments) at room temperature. Pencil rods (2B), id. 0.5 mm and length 5 cm, were purchased from STAEDTLER, Germany. A conventional three electrode system consisted of pencil carbon, a Ag/AgCl (3 M) and a platinum wire as working, reference, and auxiliary electrodes, respectively.

2.3 Preparation of the AuNPs on a pencil electrode

The preparation of PCE was carried out as described earlier [11]. In brief, a mechanical pencil 0.5 (Pilot, Japan), was used as a holder for pencil lead. All leads had a total length of 60 mm and a diameter of 0.5 mm. Electrical contact to the lead was achieved by wrapping a metallic wire around the metallic part of the pencil. 10 mm of lead was immersed in solution for the measurement. The length of 10 mm of lead corresponds to an active electrode area of 15.9 mm².

The AuNPs-modified pencil carbon (AuNPs/PC) electrode was prepared by electrodeposition of cycling the potential between -0.6 to 1.2 V for 1 cycle at scan rate of 50 mV s⁻¹. The deposition solution contained 10 ppm of HAuCl₄ in 0.1 M HCl/0.1 M KCl. The AuNPs modified indium-tin oxide (ITO) in the same condition was characterized by the Scanning Electron Microscope (SEM; LEO, Model 1400VP)



3. Results and Discussion

The characterization of AuNPs/ITO surface was shown in Figure 1. Figure 1A shows a typical SEM image of bare ITO surface and the AuNPs/ITO surface in Figure 1B. The surface of bare ITO is clear while the surface of AuNPs/ITO deposited by AuNPs shows the spotted dispersion on the ITO surface. The average size of AuNPs is 100 nm.

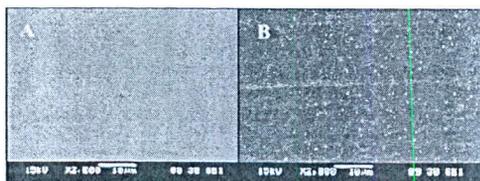


Figure 1. SEM image of (A) Indium-Tin Oxide (ITO) and (B) ITO after AuNPs electrodeposition by cyclic voltammetry.

The electrochemical behavior of nitrite was investigated at the AuNPs/PCE in 0.05 M PBS buffer solution from pH 2.0 to 9.0 by cyclic voltammetry (Figure 2). It was found that increasing pH effected the oxidation peak to higher potential. The best respond was received at pH 4.0 (Figure 2C). Au provides the oxidation peak at 1.25 V. When compared with the oxidation peak of nitrite shown at 0.85 V, it was found that the oxidation peak of nitrite was not overlape with the Au oxidation peak. This result can be explained that AuNPs function as an electrocatalysts for nitrite oxidation. The From these result, pH 4.0 was used for the rest of experiments.

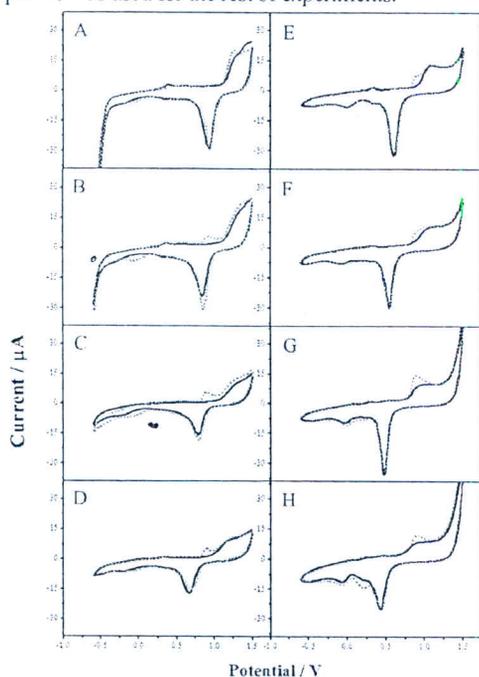


Figure 2. Cyclic voltammograms of 0 mM (solid lines) and 1 mM (dotted lines) Nitrite in pH 2(A), pH 3(B), pH 4(C), pH 5(D), pH 6(E), pH 7(F), pH 8(G), and pH 9(H) 0.05 M PBS buffer using the AuNPs modified pencil carbon electrodes at 50 mV / s

AuNPs were electrodeposited on pencil carbon electroded by cyclic voltammetry at 1 cycle and 10 cycles. The electrodeposited AuNPs voltammograms are shown in Figure 3A. Figure 3B shows the square wave voltammograms of 1.5 mM nitrite in 0.05 M PBS buffer (pH 4.0) at bare PCE and AuNPs electrodeposition on PCE for a cycle and 10 cycles. It was found that both of electrodeposition provide the similar anodic current and higher than 2 times anodic current compare to the bare PCE, so a cycle was selected for nitrite determination.

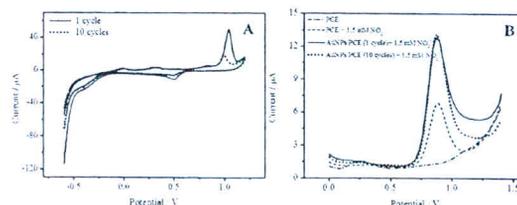


Figure 3 (A) Cyclic voltammograms of 10 ppm Au deposited on pencil carbon electrodes at 1 cycle and 10 cycles using scan rate of 0.05 V/s. (B) Square wave voltammograms of 1.5 mM Nitrite in 0.05 M PBS buffer pH 4.0

The electrocatalytic mechanism of AuNPs toward the oxidation of nitrite is:



Figure 4 shows the calibration curve of various nitrite concentrations (0 to 2.5 mM) obtained from square wave voltammetry. The linear range is found to be between 100 µM to 2.5 mM, and the detection limit ($S/N=3$) is 30 µM. The excellent sensitivity of 7.60 µA/mM was received at $R^2 = 0.99915$. The detection limit and sensitivity are lower and higher than the immobilization of nanometer-sized gold colloid attached to an ethylenediamine monolayer modified electrochemically pretreated glassy carbon electrode, respectively [19].

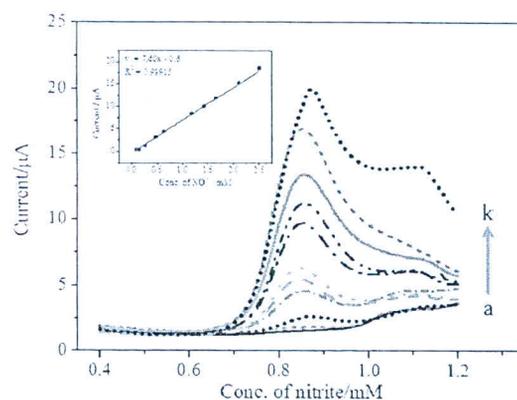


Figure 4. Square wave voltammograms of various nitrite concentrations (0 to 2.5 mM (a to k)). The inset shows a calibration plot of oxidation peak current versus nitrite concentration using square wave voltammetry.

4. Conclusions

AuNPs modified pencil carbon electrode for nitrite determination was investigated in PBS buffer solution. The proposed sensor was used by square wave voltametric method. The linearity between 0.1 – 2.5 mM was received. The sensitivity of 7.60 $\mu\text{A}/\text{mM}$ and the detection limit of 30 μM were obtained by the proposed electrode.

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