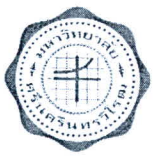
ภาคผนวก

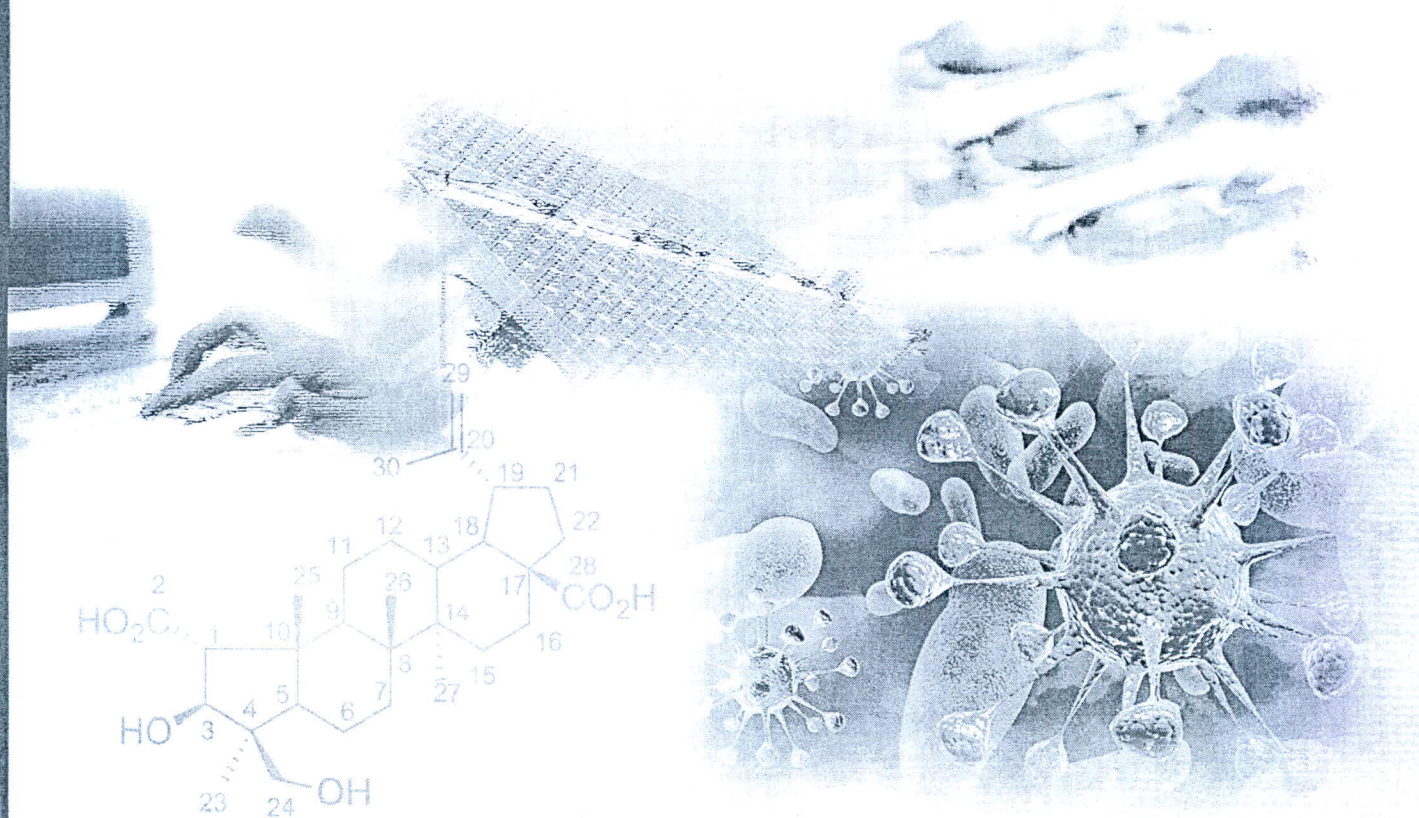
การเผยแพร่ 1

วารสารมหาวิทยาลัยศรีนครินทรวิโรฒ



Journal of Srinakharinwirot University
(สาขาวิทยาศาสตร์และเทคโนโลยี)

ปีที่ 3 (ฉบับพิเศษที่ 1) มกราคม 2554 Volume 3 (Supplement 1) January 2011 ISSN 1686-9311



PACCON
PURE AND APPLIED CHEMISTRY INTERNATIONAL CONFERENCE 2011

January 5-7

มหาวิทยาลัยศรีนครินทรวิโรฒ

ปีที่ 3 (ฉบับพิเศษที่ 1) มกราคม 2554 Volume 3 (Supplement 1) January 2011 วารสารมหาวิทยาลัยศรีนครินทรวิโรฒ (สาขาวิทยาศาสตร์และเทคโนโลยี) ISSN 1686-9311

ความเป็นมา

ด้วยมหาวิทยาลัยศรีนครินทรวิโรฒได้มีนโยบายสนับสนุนในการเผยแพร่ผลงานวิชาการในรูปแบบของวารสาร เพราะเห็นว่าจะเป็นประโยชน์ในการพัฒนาความรู้ทางวิชาการแก่สังคม และประเทศชาติ จึงได้จัดทำวารสาร

1) วารสารมหาวิทยาลัยศรีนครินทรวิโรฒ (สาขาวิทยาศาสตร์และเทคโนโลยี)

2) วารสารศรีนครินทรวิโรฒวิจัยและพัฒนา (สาขามนุษยศาสตร์และสังคมศาสตร์)

เพื่อเป็นการเผยแพร่บทความทางวิชาการและวิจัยด้านวิทยาศาสตร์และเทคโนโลยี และด้านมนุษยศาสตร์และสังคมศาสตร์ โดยจัดทำเป็นวารสารราย 6 เดือน (ปีละ 2 ฉบับ/สาขา)

วัตถุประสงค์ของการจัดพิมพ์วารสาร

1) เพื่อเผยแพร่ผลงานวิชาการที่มีคุณภาพของบุคลากรทั้งภายในและภายนอกมหาวิทยาลัย

2) เพื่อเป็นสื่อกลางในการแลกเปลี่ยนความคิดเห็นทางวิชาการ

3) เพื่อส่งเสริมและพัฒนาศักยภาพทางวิชาการของบุคลากร

การเผยแพร่ มอบให้หอสมุดสถาบันการศึกษาและหน่วยงานของภาครัฐ

จำนวนพิมพ์ 500 ฉบับ (ฉบับพิเศษที่ 1)

- บทความวิชาการและวิจัยทุกเรื่องได้รับการพิจารณากลั่นกรองโดยผู้ทรงคุณวุฒิ (Peer reviewer) จากภายในและภายนอกมหาวิทยาลัย จำนวน 3 ท่าน/บทความ
- บทความ ข้อความ ภาพประกอบ และตาราง ที่ลงตีพิมพ์ในวารสารเป็นความคิดเห็นส่วนตัวของผู้เขียน กองบรรณาธิการไม่จำเป็นต้องเห็นด้วยเสมอไป และไม่มีส่วนรับผิดชอบใดๆ ถือเป็นความรับผิดชอบของผู้เขียนแต่เพียงผู้เดียว
- บทความจะต้องไม่เคยตีพิมพ์เผยแพร่ที่ใดมาก่อน และไม่อยู่ระหว่างการพิจารณาของวารสารฉบับอื่น หากตรวจสอบพบว่ามีการตีพิมพ์ซ้ำซ้อน ถือเป็นความรับผิดชอบของผู้เขียนแต่เพียงผู้เดียว
- บทความใดที่ผู้อ่านเห็นว่าได้มีการลอกเลียนหรือแอบอ้างโดยปราศจากการอ้างอิง หรือทำให้เข้าใจผิดว่าเป็นผลงานของผู้เขียน กรุณาแจ้งให้ศูนย์สำนักพิมพ์ มศว ทราบ จะเป็นพระคุณยิ่ง
- บทความที่ส่งถึงกองบรรณาธิการ ขอสงวนสิทธิ์ที่จะไม่ส่งคืน ผู้เขียนจะต้องทำสำเนาส่วนตัวไว้

วารสารมหาวิทยาลัยศรีนครินทรวิโรฒ
Journal of Srinakharinwirot University
(สาขาวิทยาศาสตร์และเทคโนโลยี)

ปีที่ 3 (ฉบับพิเศษที่ 1) มกราคม 2554 Volume 3 (Supplement 1) January 2011 ISSN 1686-9311

Editorial office

Journal of Srinakharinwirot University

Publishing Center Srinakharinwirot University

Office of the President, 5th Floor.

114 Sukhumvit 23, Wattana, Bangkok 10110, Thailand.

Tel. (662) 649-5000 ext. 5911, 5912

Fax: (662) 258-8938

E-mail: swujournal@swu.ac.th

http://pubcenter.swu.ac.th

Published by Sahamitrprinting & Publishing co,.ltd.

59/4 Moo.10 Kanchanapisek Road.

Bangmuang Bangyai, Nonthaburi 11140

Tel. (662) 903-8257-9 Fax: (662) 921-4587

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

Table of Contents

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

A. Rukkajeekuln[1], *S. Taweemanoon*[1], *S. Kongchey*[1], *T. Tangkuaram*[2], *A. Preechaworapun*[1]*

[1]*Pibulsongkram Rajabhat University, Phitsanulok, Thailand.*
[2]*Maejo University, Chiang Mai, Thailand.*

**Corresponding Author E-Mail: anchanaph@yahoo.com*

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 reducable 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.

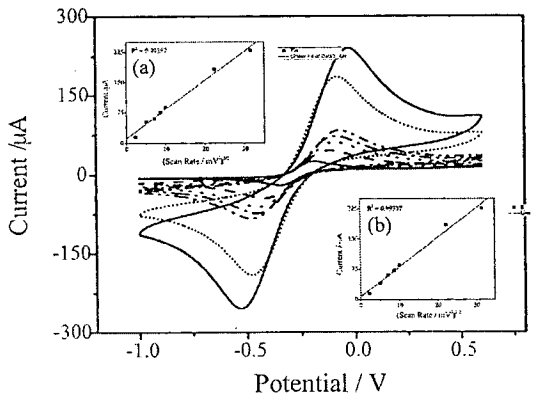


Figure 1 CV curves at a pencil electrode in 0.1 M KCl + 0.005 M $[Fe(CN)_6]^{3-/4-}$ at the scan rates of 5, 25, 50, 75, 100, 500, and 1,000 mV/s (from inner to outer). The insets are relative to a square root of scan rate of anodic current (a) and cathodic current (b).

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 $K_4Fe(CN)_6$ in 0.05 mM phosphate buffer (pH 7.5) solution at the pencil electrode by fixing potential 0.0 V.

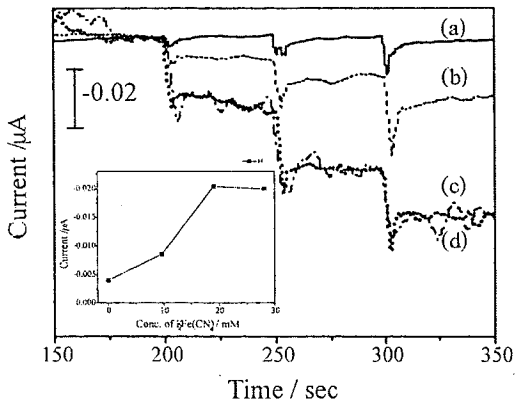


Figure 2 Amperograms of the each addition 10 μL of 15 mM KNO_2 in 0.05 M phosphate buffer (pH 7.5) with different concentration of (a) 0.0 mM, (b) 9.68 mM, (c) 19.06 mM, and (d) 28.15 mM $K_4Fe(CN)_6$ at a pencil electrode and fixed potential 0.0 V. The inset was relative to concentration of $K_4Fe(CN)_6$ and average cathodic current KNO_2 (n=3).

The current was increased from -0.004 μA, -0.0086 μA, -0.0204 μA, and -0.02 μA while increased the concentration of $K_4Fe(CN)_6$ 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 $K_4Fe(CN)_6$ (Figure 2c), that higher than without $K_4Fe(CN)_6$ about 5 times and the reaction was responded time less than 2 seconds. This phenomena proved that the $K_4Fe(CN)_6$ have been potent to catalyte 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].

$$NO_2^- + 4H_2O + 4Fe^{2+} \rightarrow NH_2OH + 5OH^- + 4Fe^{3+} \quad (1)$$

$$Fe^{3+} + e^- \rightarrow Fe^{2+} \quad (2)$$

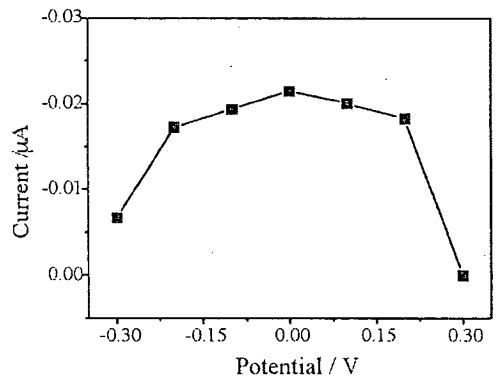


Figure 3 Effect of potential on determination 5 mM KNO_2 in 0.05 M phosphate buffer (pH 7.5) + 20 mM $K_4Fe(CN)_6$ solution at a pencil electrode using amperometric determination.

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 $K_4Fe(CN)_6$. Under the various applied potentials of -0.3–0.3 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.

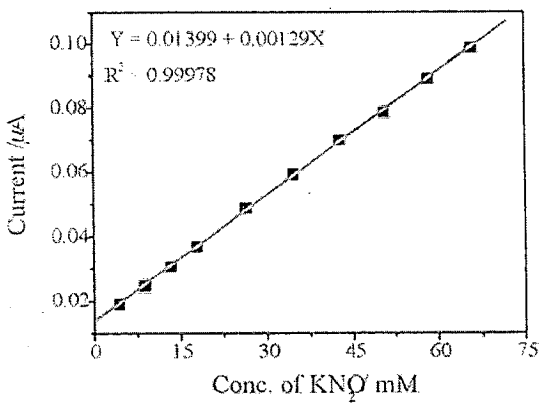


Figure 4 The linear correlation curve of cathodic current at 0.0 V *vs.* the concentration of KNO_2 (4.5 - 65 mM).

The KNO_2 could be determinates in the concentration of 4.5-65 mM by the amperometric method. Sensitivity was 0.00129 μA/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 μA/mM and the detection limit 1 mM.

Acknowledgments

Anchana Preechaworapun acknowledges The Research and Development Institute, Faculty of Science and Technology of Pibulsongkram Rajabhat University.

References

[1] M.N. Abbas; & G.A. Mostafa. (2000). *Anal. Chim. Acta.* 410: 185-192.

[2] N. Cabaleiro; et al. (2010). *Talanta.* 83: 386-390.

[3] B. Haghighi; & A. Tavassoli. (2002). *Talanta*, 56: 137-144.

[4] C.E. López Pasquali; P. Fernández Hernando; & J.S. Durand Alegía. (2007). *Anal. Chim. Acta.* 600:177-182.

[5] S. Senra-Ferreiro; et al. (2010). *Anal. Chim. Acta.* 668: 195-200.

[6] N. Bosch Bosch; et al. (1995). *J. of Chromatog. A.* 706: 221-228.

[7] S.B. Butt; M. Riaz; & M.Z. Iqbal. (2001). *Talanta.* 55: 789-797.

[8] S.A. Everett; et al. (1995). *J. Chromatogr. A.* 706: 437-442.

[9] M.I.H. Helaleh; & T. Korenaga. (2000). *J. Chromatogr. B: Biomedical Sciences and Applications.* 744: 433-437.

[10] K. Ito, et al. (2005). *J. Chromatogr. A.* 1083: 63-67.

[11] E. Salhi; & U. von Gunten. (1999). *Water Res.* 33: 3239-3244.

[12] C.D. Stalikas; C.N. Konidari; & C.G. Nanos. (2003). *J. Chromatogr. A.* 1002: 237-241.

[13] M. Akyüz; & S. Ata. (2009). *Talanta.* 79: 900-904.

[14] J.W. Dallinga; et al. (1998). *Cancer Lett.* 124: 119-125.

[15] D. Tsikas; et al. (1997). *Anal. Biochem.* 244: 208-220.

[16] D. Tsikas; et al. (1999). *J. Chromatogr. B.* 731: 285-291.

[17] D. Tsikas; et al. (2010). *Anal. Biochem.* 397: 126-128.

[18] D. Tsikas; J. Sandmann; & J.C. Frölich. (2002). *J. Chromatogr. B.* 772: 335-346.

[19] T. Aokil; & M. Wakabayashi. (1995). *Anal. Chim. Acta.* 308: 308-312.

[20] D. He; et al. (2007). *Food Chem.* 101: 667-672.

[21] H. Kodamatani; et al. (2009). *J. Chromatogr. A.* 1216: 3163-3167.

[22] P. Mikuska; Z. Vecera; & Z. Zdrahal. (1995). *Anal. Chim. Acta.* 316: 261-268.

[23] E. Nagababu; & J.M. Rifkind. (2007) *Free Radical Biol. Med.* 42: 1146-1154.

[24] M.M. Pelletier; et al. (2006). *Free Radical Biol. Med.* 41: 541-548.

[25] Q. Yue; & Z. Song. (2006). Microchem. J. 84: 10-13.

[26] M.C. Barciela Alonso; & R. Prego. (2000). *Anal. Chim. Acta.* 416: 21-27.

[27] C. Merusi; et al. (2010). *Food Chem.* 120: 615-620.

[28] A.A. Okemgbo; et al. (1999). *J. Chromatogr. A.* 844: 387-394.

[29] N. Öztekin; M.S. Nutku; & F.B. Erim. (2002). *Food Chem.* 76: 103-106.

[30] I. Martins; et al. (2010). *Toxicology Lett.* 196: S292-S292.

[31] S. Nouroozi; & R. Mirshafian. (2009). *Talanta.* 79: 1149-1153.

[32] A.S. Adekunle; J. Pillay; & K.I. Ozoemena. (2010). *Electrochim. Acta.* 55: 4319-4327.

[33] B.O. Agboola; K.I. Ozoemena; & T. Nyokong. (2006). *Electrochim. Acta.* 51: 6470-6478.

[34] L.H. Chen; et al. (2008). *Electrochim. Acta.* 53: 3442-3445.

[35] H.J. Choi; G. Kwag; & S. Kim. (2001). *J. Electroanal. Chem.* 508: 105-114.

[36] V. Di Matteo; & E. Esposito. (1997). *J. Chromatogr. A.* 78: 213-219.

[37] R. Geng; et al. (2008). *Biomaterials.* 29: 2794-2801.

[38] X. Huang; et al., (2008). *Sens. Actuats B: Chemical.* 134: 780-786.

[39] L. Jiang; et al. (2005). *Electrochem. Commun.* 7: 597-601.

[40] S. Kerkeni; E. Lamy-Pitara; & J. Barbier. (2002) *Catalysis Today.* 75: 35-42.

[41] T.-S. Liu; et al. (2009). *J. Electro. Chem.* 632: 197-200.

[42] F. Manea, et al. (2010). *Talanta.* 83: 66-71.

[43] E.A. Moschou; et al. (2000). *Aquacultural Engineering.* 22: 255-268.

[44] D. Quan; et al. (2006). *Bioelectrochem.* 69: 267-275.

[45] F. Vicente; et al. (1995). *Electrochim. Acta.* 40: 1121-1126.

[46] D. Zheng; et al. (2009). *Electrochim. Acta.* 54: 4910-4915.

[47] N. Zhu; et al. (2009). *Electrochem. Com.* 11: 2308-2311.

[48] S.Q. Liu; & H.X. Ju. (2003). *Analyst.* 128: 1420-1424.

[49] M. Li; et al. (2009). *J. Hazard. Mater.* 171: 724-730.

การเผยแพร่ 2

THE 6th PURE AND APPLIED CHEMISTRY INTERNATIONAL CONFERENCE 2012

PACCON 2012-
CHEMISTRY BEYOND BOUNDARIES

11 - 13 JANUARY 2012, THE EMPRESS CONVENTION CENTER
CHIANG MAI, THAILAND

PROCEEDINGS

DEPARTMENT OF CHEMISTRY, FACULTY OF SCIENCE, CHIANG MAI UNIVERSITY
& THE CHEMICAL SOCIETY OF THAILAND UNDER THE PATRONAGE OF
HER ROYAL HIGHNESS PRINCESS CHULABHORN MAHIDOL

PACCON
PURE AND APPLIED CHEMISTRY INTERNATIONAL CONFERENCE 2012

"Chemistry Beyond Boundaries"

Pure and Applied Chemistry International Conference (PACCON2012)

January 11-13, 2012

Organized by:

Department of Chemistry, Faculty of Science, Chiang Mai University
and Chemical Society of Thailand

List of Reviewers

Achara Kleawkla
Akarin Intaniwet
Anansawake Haewsungjarern
Angkana Saovapakhiran
Annop Suriyasomboon
Anucha Watcharapasorn
Anurak Prasatkhetragarn
Aphichart Limpichaipanit
Apinpus Rujiwatra
Apirat Laobuthee
Apisit Songsasen
Arinthip Thamchaipenet
Arnon Chaipanich
Atitaya Siripinyanond
Banchob Wonno
Boon-ek Yingyongnarongkul
Boonsom Liawruangrath
Boosayarat Tomapatanaget
Bunjerd Jongsomjit
Burapat Inceesungvorn
Bussarin Ksapabutr
Chaikarn Liewhiran
Chaiyasit Banjongprasert
Chalermporn Thongpoon
Chamnan Randorn
Chanatip Samart
Chanchai Thonpin
Chanchira Jubsilp
Chantana Boonyarat
Charoen Nakasan

Chatchai Nimmol
Chidchanok Meechaisue
Choladda Srisuwannaket
Chor. Wayakron Phetphaisit
Chumni Sangpakdee
Datchanee Pattavarakorn
Dau Hung Anh
David J. Harding
Dheerawan Boonyawan
Duangjai Nacapricha
Dusit Ngamrungroi
Ekasith Somsook
Gobwute Rujijanagul
Hathaikarn Manuspiya
Jakrapong Kaewkhao
Jantrawan Pumchusak
Jaroon Jakmunee
Jatuphorn Wootthikanokkhan
Jidapha Tinoi
Jintawat Chaichanawong
Jirasak Kongkiatkajorn
Jitladda T.Sakdapipanich
Joongjai Panpranot
Kamonpan Pengpat
Kanarat Nalampang
Kanchana Dumri
Kanchana Uraisin
Kanchana Watla-iad
Kanchaya Honglertkongsakul
Kasinee Hemvichian
Kawee Srikulkit
Khemika Lomthaisong
Kiattikhun Manokruang
Kongkiat Trisuwan
Konlayutt Punyawudho
Kontad Ounnunkad
Kraisri Pisithkul
Kritapas Laohhasurayotin
Kunakorn Poochinda
Lalida Shank
Laongnuan Srisombat
Luckhana Lawtrakul
Lucksagoon Ganranoo
Malee Santikunaporn
Maliwan Amatatongchai
Metha Ruttanakornpitak
Metta Chareonpanich
Mongkon Rayanakorn
Monthon Nakpathom
Mookda Pattarawarapan
Moontree Sawangphruk
Munetsuku Kawashima
Nadtanet nunthaboot
Nantakan Muensit
Nantanit Wanichacheva
Nantiya Hansupalak
Napachat Tareelap
Naratip Vittayakorn
Nason Phonpoke
Natda Wetchakun
Nattakan Soykeabkaew
Natthawut Choengchan
Nawadon Petchwattana
Nawee Kungwan
Neti Waranuch
Nirun Witit-anun
Nithima Khaoropong
Nithinart Supakarn
Nongnuch Ruangjit
Nopakarn Chandet
Nopparat Plucktaveesak
Nuanlaor Ratanawimarnwong
Nuanphan Chantarasiri
Nuchjira Dejang
Nurak Grisadanurak
Nuttee Suree
Nutthita Chuankrerkkul
Orapin Chienthavorn
Orawan Kritsunankul
Orawon Chailapakul
Orn-Anong Arquero
Padchanee Sangthong
Paisan Suwannoi
Pakawadee Sutthivaiyakit
Pakawan Puangsombat
Pamornrat Kueseng
Panchika Prangkio
Panya Wongpanit
Pariya Na Nakorn
Patchara Punyamoonwongsa
Patnarin Worajittiphon
Paul Topham
Paweena Sureeyatanapas
Pawinee Deetae

Phanphen Wattanaarskit
Phimphaka Harding
Phiriyatorn Suwanmala
Phumon Sookwong
Phunuphai Phunnawadee
Piamsook Pongsawasdi
Pitchaya mungkornasawakul
Piyarat Nimmanpipug
Pongdhorn Sae-Oui
Ponusa Songtipya Jitphuthi
Poonsakdi Ploypradith
Pornchai Rachtanapun
Pornsak Sriamonsak
Pornsri Pakeyangkoon
Pornsuda Bomlai
Potjaman Poolmee
Pramoch Rangsunvijit
Pranee Chumsamrong
Prapairat Seephonkai
Prapaporn Boonme
Praput Thavornyutikarn
Prasat Kittakoop
Prasert Reubroycharoen
Pratarn Wong sarivej
Pusit Pookmanee
Puttinan Meepowpan
Rangrong Yoksan
Rapeephun Dangtungee
Rasamee Chaisuksant
Rattanaphol Mongkholrattanasit
Rattikorn Yimnirun
Rewadee Wongmaneerung
Robert Molloys
Rodjana Burakham
Ruangsri Watanesk
Rukkiat Jitkati
Runglawan Somsunan
Saengrawee Sriwichai
Sairoong Saowsupa
Sakda Jongkaewwattana
Saowanit Tongpim
Saranagon Hemavibool
Sarunyoo Songkro
Sasiprapha Rattanadilok Na Phuket
Satit Phiyanalinmat
Sila Kittiwachana
Siriluk Chiarakorn
Siripat Suteerapataranon
Siriporn Jungsuttiwong
Sirirat Chancharunee
Siriwan Kittinaovarat
Siriwan Srisorrachatr
Sittichai Wirojanupatu
Sombat Thanawan
Somchai Lapanantnoppakhun
Somchai Thongtem
Somkiat Ngamprasertsith
Somkiat Srijaranai
Sommai Pivsa-Art
Somporn Chantara
Sorapong Janhom
Srung Smanmoo
Suchart Siengchin
Sugunya Wongpornchai
Sujittra Youngme
Sukanda Jiansirisomboon
Sukjit Kungwankunakorn
Sukon Phanichphant
Sulawan Kaowpong
Sumrit Mopoung
Sumrit Wacharasindhu
Sunan Tiptipakorn
Supa Hannongbua
Supab Choopan
Supakorn Boonyuen
Supalux Srijaranai
Supaporn Kradtap Hartwell
Suparin Chaiklangmuang
Supicha Rungrodnimichai
Supin Tangwiwat
Supon Ananta
Surat Laphookhieo
suwaporn luangkamin
Tawan Sooknoi
Taweechai Amornsakchai
Teerapol Srichana
Teinthong Thongpanchang
Terdthai Vatanatham
Thammarat Panyathanmaporn
Thapanee Sarakonsri
Theeraphan Machan
Thidarat Eksittikul
Thirawudh Pongprayoon
Thunwadee Ritthiwigrom

Tinakorn Kanyanee
Tirayut Vilaivan
Titipun Thongtem
Ubonrat Siripatrawan
Ungkana Wongsiriwan
Urai Tengjaroenkul
Vannajan Sanghiran Lee
Varawut Tangpasuthadol
Varit Srilaong
Verasak Sahachaisaree
Vorapot Kanokkantapong
Voravee P. Hoven
Wancheng Sittikijyothin
Wandee Thamjaree
Wanida Janvikul
Wannee Chinsirikul
Wanwilai Vittayakorn
Warakorn Limbut
Waraporn Tanakulrungsank
Waret Veerasai
Wasinee Channuan
Weena Siangproh
Wim Nhuapeng
Wimolpun Rungprom
Winita Punyodom
Wirach Taweepreda
Wittaya Ngeontae
Woraluk Angsuwaranggoon
Woraratana Pattaraprakorn
Worraanong Leewattanapasuk
Wundee Onriebroi
Yongsak Sritana-Anant
Yupaporn Ruksakulpiwat

PACCON
PURE AND APPLIED CHEMISTRY INTERNATIONAL CONFERENCE 2012

"Chemistry Beyond Boundaries"

Content

PACCON 2012 proceedings are published in electronic form only. There is no index page but reader can search for interested keywords in this content by using pdf program searching tool. The full articles in the proceedings are grouped into various sessions and have been in separated files in order to reduce file size for convenient downloading. The sessions and associated files are listed below:

Session: Advances in Flow Analysis and Sensors

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

Nootjarin Inngam[1], Kulwadee Pinwattana[1], Tanin Tangkuaram[2], Anchana Preechaworapun[1,*]

1 Program of Chemistry, Faculty of Science and Technology, Pibulsongkram Rajabhat University, Phitsanulok, Thailand 65000
[2] Department of Chemistry, Faculty of Science, Maejo University, Chiang Mai, Thailand 50290

*Author for correspondence ; E-Mail: anchanaph@yahoo.com, Tel./Fax. +66 55 267106

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 NO_2^- 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 elctrocatalyst 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, $R \geq 18.2$ MΩ 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 mm^2.

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 $HAuCl_4$ 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.

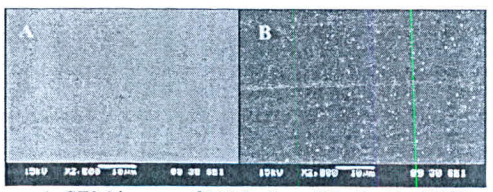


Figure 1. SEM image of (A) Indium-Tin Oxide (ITO) and (B) ITO after AuNPs electrodeposition by cyclic voltammetry.

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.

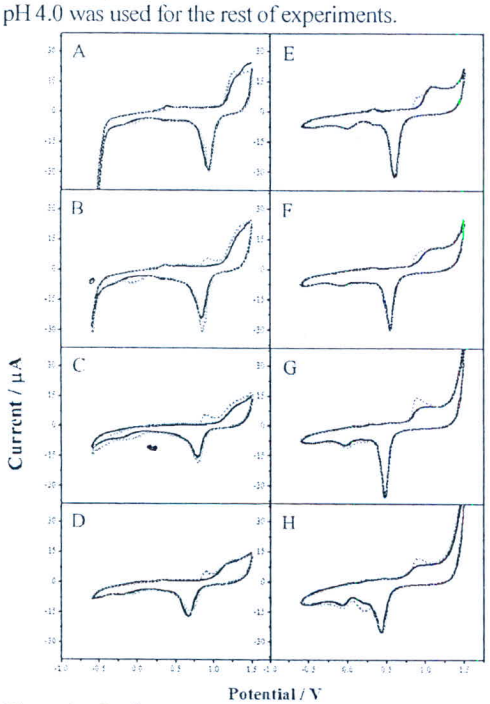


Figure 2. Cyclic voltammograms of 0 mM (solid lines) and 1 mM (dotted lines) Nitrite in pH 2(A), pH 3(B), pH 4(C), pH 5(D), pH 6(E), pH 7(F), pH 8(G), and pH 9(H) 0.05 M PBS buffer using the AuNPs modified pencil carbon electrodes at 50 mV / s

AuNPs were electrodeposited on pencil carbon electrod 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.

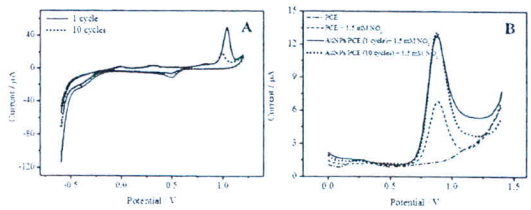


Figure 3 (A) Cyclic voltammograms of 10 ppm Au deposited on pencil carbon electrodes at 1 cycle and 10 cycles using scan rate of 0.05 V/s. (B) Square wave voltammograms of 1.5 mM Nitrite in 0.05 PBS buffer pH 4.0

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

$$NO_2^- + H_2O \xrightarrow{AuNPs} NO_3^- + 2H^+ + 2e^-$$

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].

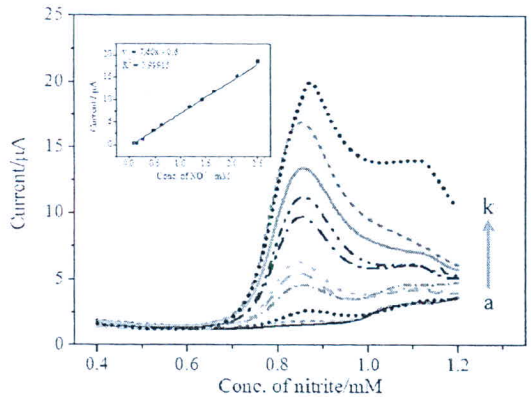


Figure 4. Square wave voltammograms of various nitrite concentrations (0 to 2.5 mM (a to k)). The inset shows a calibration plot of oxidation peak current versus nitrite concentration using square wave voltammetry.

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 μA/mM and the detection limit of 30 μM were obtained by the proposed electrode.

Acknowledgements

Anchana Preechaworapun acknowledges The Research and Development Institute, Faculty of Science and Technology of Pibulsongkram Rajabhat University.

References

[1] D. Demetriades, A. Economou, A. Voulgaropoulos, *Anal. Chim. Acta* **519** (2004) 167-172.
[2] E. Mirmomtaz, A.A. Ensafi, S. Soleimanian-Zad, *Electrochim. Acta* **54** (2009) 1141-1146.
[3] A.A. Ensafi, E. Heydari-Bafrooei, M. Amini, *Biosen. Bioelectron.* **31** (2012) 376-381.