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นางสาวนันดา พิกรานนท์

วิทยานิพนธ์เป็นส่วนหนึ่งของการศึกษาทางด้านอุตสาหกรรมปูนซูตรปริญญาโทในสาขาเคมีที่
มหาวิทยาลัยมหิดล ภาควิชาเคมี ภาควิชาเคมี
ผู้วิทยานิพนธ์ ดร. วุฒิกร มนัสวนิช
ปีการศึกษา 2552
จัดตีพิมพ์โดยสถาบันเทคโนโลยีไทย-ญี่ปุ่น



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อุปกรณ์

นางสาว นันทนา พิเคราะห์

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สาขาวิชาเคมี ภาควิชาเคมี
คณะวิทยาศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย
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ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย



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**DETERMINATION OF HEAVY METALS BY BISMUTH-CARBON
NANOTUBE MODIFIED ELECTRODES**

Miss Nantana Pikroh

A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Science Program in Chemistry

Department of Chemistry

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By Miss Nantana Pikroh
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Thesis Advisor Parichatr Vanalabhpatana, Ph.D.

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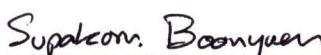
 Dean of the Faculty of Science
(Professor Supot Hannongbua, Dr. rer. nat.)

THESIS COMMITTEE

 Chairman
(Assistant Professor Warinthorn Chavasiri, Ph.D.)

 Thesis Advisor
(Parichatr Vanalabhpatana, Ph.D.)

 Examiner
(Associate Professor Orawon Chailapakul, Ph.D.)

 External Examiner
(Assistant Professor Supakorn Boonyuen, Ph.D.)

นันทนา พิเคราะห์ : การตรวจวัดโลหะหนักโดยข้าไฟฟ้าบิสมัท-ท่อนาในคาร์บอนดัดแปลง (DETERMINATION OF HEAVY METALS BY BISMUTH-CARBON NANOTUBE MODIFIED ELECTRODES) อ.ที่ปรึกษาวิทยานิพนธ์หลัก : อ.ดร. ปาริชัตร วนลาภพัฒนา , 80 หน้า.

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งานวิจัยนี้ได้สังเคราะห์บิสมัท-ท่อนาในคาร์บอนคอมโพสิตด้วยกระบวนการพอลิออลเพื่อพัฒนาเป็นข้าไฟฟ้าใช้งานสำหรับการวิเคราะห์หาปริมาณของแคนเดเมียมและตะกั่วด้วยเทคนิคแควร์เวฟแอโนดิกสทริปปิงโกลแทมเมตري ได้ศึกษาลักษณะทางโครงสร้างของบิสมัท-ท่อนาในคาร์บอนคอมโพสิตที่เตรียมขึ้นด้วยเทคนิคเอ็กซ์เรย์ดิฟแฟรากชัน ทราบสมิชชันอิเล็กตรอน-ไมโครสโคป และเอนเออวีจิดิสเพอร์ซีฟเอ็กซ์เรย์ฟลูออเรสเซนต์スペกโตรสโคป ข้าไฟฟ้ากลาสซีคาร์บอนที่ดัดแปลงด้วยบิสมัท-ท่อนาในคาร์บอนคอมโพสิตแสดงสัญญาณสทริปปิงที่ชัดเจนและแยกออกจากกันได้สำหรับไอออนของโลหะแคนเดเมียมและตะกั่ว อีกทั้งค่าเบี่ยงเบนมาตรฐานสัมพัทธ์สำหรับไอออนของแคนเดเมียมและตะกั่วเท่ากับ 2.44 และ 3.19 เปอร์เซ็นต์ (ทำซ้ำ 8 ครั้ง) ตามลำดับ สำหรับสารละลายน้ำ ไมโครกรัมต่อลิตรของไอออนโลหะทั้งสอง นอกจากนี้ข้าไฟฟ้าดัดแปลงให้ช่วงความเป็นเส้นตรงสองช่วงความเข้มข้น คือ 5 ถึง $150 \text{ } \mu\text{g}\cdot\text{L}^{-1}$ และ 150 to 240 $\text{mg}\cdot\text{L}^{-1}$ ที่ช่วงความเข้มข้นต่ำ ค่าสัมประสิทธิ์สัมพันธ์เป็น 0.9986 สำหรับไอออนแคนเดเมียมและ 0.9990 สำหรับไอออนของตะกั่ว ส่วนกราฟมาตรฐานที่ความเข้มข้นของไอออนโลหะ 150–240 ไมโครกรัมต่อลิตร ให้ค่าสัมประสิทธิ์สัมพันธ์เป็น 0.9983 และ 0.9970 สำหรับไอออนของโลหะแคนเดเมียมและตะกั่ว ตามลำดับ ภายใต้ภาวะการทดลองที่เหมาะสมที่สุดพบว่าข้าไฟฟ้ากลาสซีคาร์บอนที่ดัดแปลงด้วยบิสมัทแบบ *in situ* บนข้าไฟฟ้านาในคาร์บอนดัดแปลงบนกลาสซีคาร์บอน สุดท้ายได้นำข้าไฟฟ้าพิล์มบิสมัทแบบ *in situ* บนข้าไฟฟ้านาในคาร์บอนดัดแปลงบนกลาสซีคาร์บอนไปใช้สำหรับวิเคราะห์หาปริมาณไอออนของโลหะแคนเดเมียมและตะกั่วในตัวอย่างน้ำประปาและน้ำทิ้งจากโรงงานอุตสาหกรรมพบว่าผลการทดลองที่ได้เป็นที่น่าพอใจ

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NANTANA PIKROH : DETERMINATION OF HEAVY METALS BY BISMUTH-CARBON NANOTUBE MODIFIED ELECTRODES. THESIS

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In this research, the bismuth–carbon nanotube (Bi–CNT) composites were synthesized by polyol process and have been developed as working electrodes for the determination of cadmium (II) and lead (II) ions by means of square wave anodic stripping voltammetry. Prepared Bi–CNT composites were characterized by X-ray diffraction (XRD) technique, transmission electron microscopy (TEM), and energy dispersive X-ray fluorescence (EDXRF) spectroscopy. Bi–CNT/GC electrode exhibited well-defined and sharp stripping signals for both metal ions and a reproducibility of 2.44% and 3.19% ($n = 8$) for $25 \mu\text{g}\cdot\text{L}^{-1}$ cadmium (II) and lead (II) ions, respectively. In addition, the modified electrode displayed excellent linear behavior in two concentration ranges: 5 to $150 \mu\text{g}\cdot\text{L}^{-1}$ and 150 to $240 \mu\text{g}\cdot\text{L}^{-1}$. At the lower concentration range, the correlation coefficients (R^2) were 0.9986 for cadmium (II) ion and 0.9990 for lead (II) ion. For the calibration curve from 150 to $240 \mu\text{g}\cdot\text{L}^{-1}$, the R^2 of 0.9983 and 0.9970 was obtained for cadmium (II) and lead (II) ions. Under optimal conditions, the Bi–CNT/GC electrode displayed more attractive voltammetric responses than *in situ* bismuth film on CNT modified glassy carbon (*in situ* BiF/CNT/GC) electrode. Finally, the developed electrode was applied to tap water and waste water samples for the analysis of cadmium (II) and lead (II) ions with satisfactory results.

Department :.....Chemistry.....Student's Signature.....*Nantana Pikroh*.....

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LIST OF ABBREVIATIONS

A	Ampere
ASV	Anodic Stripping Voltammetry
AAS	Atomic Absorption Spectroscopy
Bi	Bismuth
BiF	Bismuth Film
BiFE	Bismuth Film Electrode
Bi ₂ O ₃	Bismuth Oxide
Cd	Cadmium
CNT	Carbon Nanotube
CE	Counter Electrode
Co	Cobalt
Cl	Chloride
CPE	Carbon Paste Electrode
CPmE	Carbon Paste mini-Electrode
°C	Degree Celsius
O ₂	Dioxygen
DME	Dropping Mercury Electrode
XRF-EDX	Energy Dispersive X-ray Fluorescence Spectroscopy
FTIR	Fourier Transform Infrared
FAAS	Flame Atomic Absorption Spectroscopy
GC	Glassy Carbon
GCE	Glassy Carbon Electrode
GFAAS	Graphite Furnace Atomic Absorption Spectroscopy
Au	Gold
HMDE	Hanging Mercury Drop Electrode
IUPAC	International Union for Pure and Applied Chemistry
ICP-OES	Inductively Coupled Plasma-Optical Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
Pb	Lead

LGC	Levitational Gas Condensation
Li	Lithium
Hg	Mercury
MFE	Mercury Film Electrode
M	Metal
MT	Methallothionein
Mo	Molybdenum
MWCNT	Multi-Walled Carbon Nanotube
MCA	Multichannel Analyzer
nm	Nanometer
NCBFE	Nafion Coated Bismuth Film Electrode
NEAs	Nanoelectrode Arrays
Ni	Nickel
Pd	Palladium
Pt	Platinum
ppb	Part Per Billion
ppm	Part Per Million
E	Potential
RE	Reference Electrode
NaBH ₄	Sodium Borohydride
Si	Silicon
SPCE	Screen-Printed Carbon Electrode
SPGCE	Screen-Printed Glassy Carbon Electrode
Ag/AgCl	Silver/Silver Chloride
SWCNT	Single-Walled Carbon Nanotube
Ag	Silver
SWASV	Square-Wave Anodic Stripping Analysis
SIA	Sequential-Injection Analysis
TEM	Transmission Electron Microscopy
V	Volt
WE	Working Electrode
XRD	X-ray Diffraction
ZDCPE	Zeolite Doped Carbon Paste Electrode