



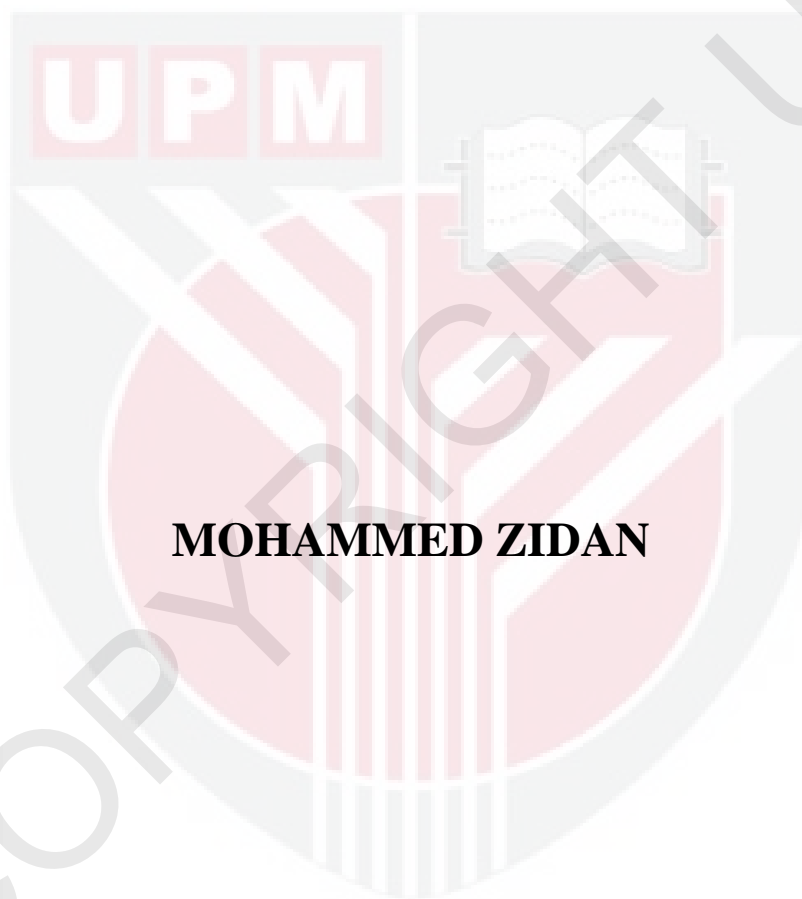
**UNIVERSITI PUTRA MALAYSIA**

**ELECTROCHEMICAL CHARACTERIZATION AND APPLICATION OF  
CARBON NANOTUBES AND METAL OXIDE NANOPARTICLE MODIFIED  
ELECTRODES**

**MOHAMMED ZIDAN**

**FS 2011 93**

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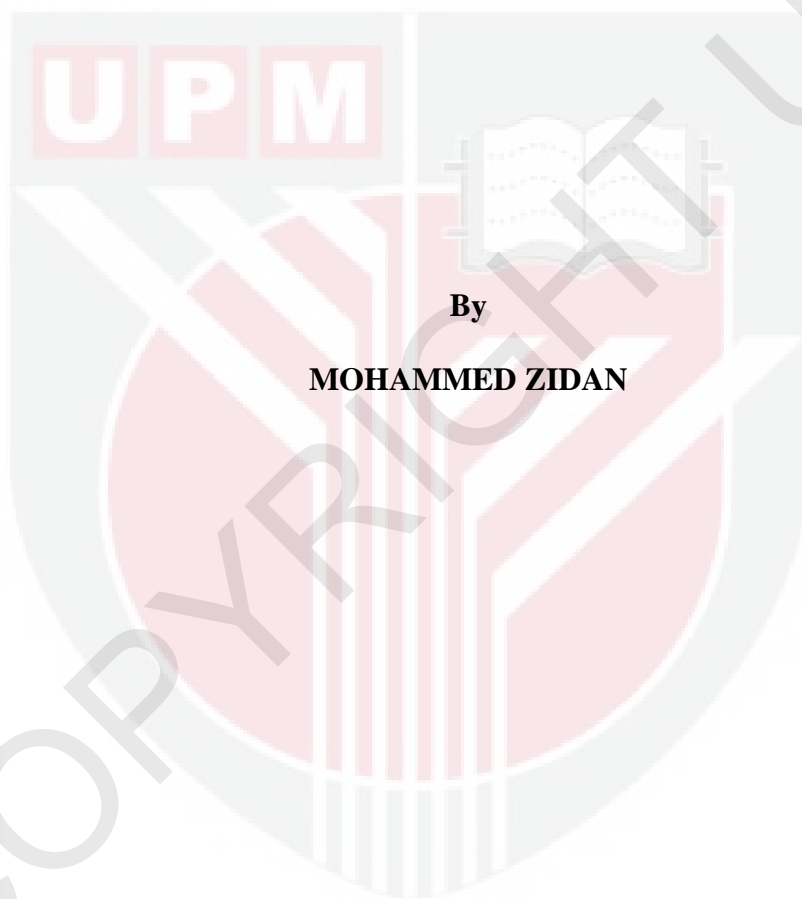


**MOHAMMED ZIDAN**

**DOCTOR OF PHILOSOPHY  
UNIVERSITI PUTRA MALAYSIA**

**2011**

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ELECTRODES**



By

**MOHAMMED ZIDAN**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia in  
Fulfilment of the Requirements for the  
Degree of Doctor of Philosophy**

**June 2011**

## DEDICATION

To my beloved parents, wife and daughter for their support and patience.



Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in  
fulfilment of the requirement for the degree of Doctor of Philosophy

**ELECTROCHEMICAL CHARACTERIZATION AND APPLICATION OF  
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**MOHAMMED ZIDAN**

**June 2011**

**Chairman : Associate Professor Tan Wee Tee, PhD**

**Faculty : Science**

The modified glassy carbon electrodes by adhered microcrystals of bismuth oxide ( $\text{Bi}_2\text{O}_3$ ) and zinc oxide ( $\text{ZnO}$ ) has been studied extensively in characterization and application of these modified electrodes in mediating some selected compounds such as ascorbic acid and paracetamol in the presence of aqueous electrolytes.

It is evident that the use of a lithium doped Bismuth oxide and multi-walled carbon nanotubes (MWCNT) modified glassy carbon electrode ( $\text{Bi}_2\text{O}_3/\text{Li}^+/\text{CNT}/\text{GC}$ ) tends to enhance the oxidation current of ascorbic acid during cyclic voltammetry compared to bare GC and ( $\text{Bi}_2\text{O}_3/\text{Li}^+/\text{CNT}$ ) modified electrode. Peak potential was observed to shift slightly to less positive value by about 220mV and current was significantly hanced by about two folds. Under conditions of cyclic voltammetry, the sensitivity of the current is significantly dependent on pH, temperature, and electrolyte and scan rate. The result of

scanning electron micrograph shows that the size increased slightly from 1 – 4  $\mu\text{m}$  after electrolysis using  $\text{Bi}_2\text{O}_3/\text{Li}^+/\text{CNT}$  modified electrode. The detection limit of this modified electrode was found to be 50  $\mu\text{M}$ . The oxidation current of ascorbic acid decreased sharply after the first cycle and became stable with minor decreases after second cycle. It is therefore apparent that the  $\text{Bi}_2\text{O}_3/\text{Li}^+/\text{CNT}$  modified GC electrode possesses some degree of stability.

During cyclic voltammetry, an oxidation peak of paracetamol appearing at +6.11 V vs. Ag/AgCl was observed. Enhance the oxidation current of paracetamol compared to bare GC and ( $\text{Bi}_2\text{O}_3/\text{Li}^+/\text{CNT}$ ) modified electrode. Peak potential was observed to shift slightly to less positive value by about 220 mV and current was significantly enhanced by about 3.2 folds. The sensitivity under conditions of cyclic voltammetry is significantly dependent on pH, temperature, and scan rate. Calibration plot reveals linearity from the range  $5.0 \times 10^{-7}$  to  $2 \times 10^{-3}$  M with a correlation coefficient of 0.998. The detection limit was estimated to be  $7.4 \times 10^{-7}$  M.

The usefulness of zinc oxide (ZnO) modified of a glassy carbon (GC) electrode was extended in mediating the oxidation of ascorbic acid in 0.1 M  $\text{KH}_2\text{PO}_4$  electrolyte solution by cyclic voltammetry (CV). ZnO/GC electrode exhibited obvious enhancing and electrocatalyzing effect as it causes the oxidation current of ascorbic acid to increase by 1.5 times as compared to bare GC electrode. The variation of scan rate study shows that the system undergoes diffusion-controlled process. Diffusion coefficient and rate constant of ascorbic acid were determined using hydrodynamic method (rotation disk electrode) with values of  $5.4 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$  and  $2.5 \times 10^{-3} \text{ cm s}^{-1}$  respectively for

unmodified electrode, while the values of diffusion coefficient and rate constant of ascorbic acid using ZnO/GC electrode were  $5.7 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$  and  $2.1 \times 10^{-3} \text{ cm s}^{-1}$  respectively.

Zinc oxide (ZnO) microparticles have been mechanically attached on the surface of a glassy carbon (GC) electrode and the effect on oxidation of paracetamol in 0.1 M  $\text{KH}_2\text{PO}_4$  electrolyte solution by cyclic voltammetry (CV). Peak potential was observed to shift slightly to less positive value by about 150 mV and current was significantly enhanced by about 1.1 folds as compared to bare GC electrode. In addition, calibration plot reveals linearity from the range  $2.0 \times 10^{-5}$  to  $5.0 \times 10^{-3}$  M with a correlation coefficient of 0.997.

Also evident that use of  $\text{MgB}_2/\text{Li}^+/\text{CNT}/\text{GC}$  modified electrode tends to enhance the oxidation of paracetamol in 0.1 M  $\text{KH}_2\text{PO}_4$  supporting electrolyte at pH 4.2 at bare (unmodified) GC electrode,  $\text{MgB}_2/\text{GC}$  modified electrode,  $\text{MgB}_2/\text{Li}^+/\text{GC}$  modified electrode and  $\text{MgB}_2/\text{Li}^+/\text{CNT}/\text{GC}$  modified electrode. While at the modified GC electrode, peak shift of 130 mV and 150 mV towards less positive region was observed for  $\text{MgB}_2/\text{Li}^+/\text{CNT}/\text{GC}$  and  $\text{MgB}_2/\text{Li}^+/\text{GC}$  respectively with a slight current increase as compared to those of an unmodified electrode. However, at  $\text{MgB}_2/\text{Li}^+/\text{CNT}/\text{GC}$  current increased by 3 times with a similar peak shift as those of  $\text{MgB}_2/\text{Li}^+/\text{GCE}$ , indicating presence of a electrocatalysis process effected by the  $\text{MgB}_2/\text{Li}^+$ , and  $\text{MgB}_2/\text{Li}^+/\text{CNT}$  coating.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia  
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

**PENCIRIAN DAN APLIKASI ELEKTROKIMIA TERHADAP KARBON NANO  
TIUB DAN OKSIDA LOGAM NANOPARTIKEL  
MODIFIKASI ELEKTROD**

Oleh

**MOHAMMED ZIDAN**

**June 2011**

**Pengerusi : Profesor Madya Tan Wee Tee, PhD**

**Fakulti : Sains**

Modifikasi elektrod karbon kaca melalui pelekatan mikrokrystal bismut oksida ( $\text{Bi}_2\text{O}_3$ ) dan zink oksida ( $\text{ZnO}$ ) telah dikaji dengan terperinci dalam pencirian dan aplikasi elektrod-elektrod ini sebagai perantaraan beberapa sebatian-sebatian terpilih seperti asid askorbit, parasetamol dalam kehadiran elektrolit berair.

Ianya jelas menunjukkan yang penggunaan litium bismuth oksida terdop dan MWCNT menggunakan elektrod karbon berkaca ( $\text{Bi}_2\text{O}_3/\text{Li}^+/\text{CNT}/\text{GC}$ ) cenderung untuk meningkatkan arus pengoksidaan asid askorbik semasa siklik voltametri berbanding dengan GC kosong dan ( $\text{Bi}_2\text{O}_3 / \text{Li}^+/\text{CNT}$ ) modifikasi elektrod. Keupayaan puncak telah diperhatikan berubah sedikit kearah nilai kurang positif iaitu pada lebih kurang 220 mV dan arus meningkat dua kali ganda. Peningkatan arus ini adalah bergantung kepada pH, suhu, elektrolit dan kadar imbasan. Hasil pengimbas mikroskop elektron menunjukkan saiz bertambah sedikit dari 1 – 4  $\mu\text{m}$  selepas elektrolisis menggunakan modifikasi  $\text{Bi}_2\text{O}_3 / \text{Li}^+/\text{CNT}$ . Had pegasan modifikasi elektrod ini adalah 50  $\mu\text{M}$ . Arus pengoksidaan



asid askorbik meningkat selepas pusingan pertama dan menjadi stabil dengan sedikit pengurangan selepas kitaran kedua. Ia adalah terbukti yang  $\text{Bi}_2\text{O}_3 / \text{Li}^+/\text{CNT}$  modifikasi elektrod GC memiliki beberapa tahap kestabilan.

Semasa siklik voltammetri, puncak pengoksidaan parasetamol diperhatikan muncul pada +6.11 V (lawan. Ag/AgCl). Arus pengoksidaan parasetamol ditingkatkan berbanding dengan GC tanpa modifikasi dan elektrod ( $\text{Bi}_2\text{O}_3 / \text{Li}^+/\text{CNT}$ ) yang dimodifikasi. Potensi puncak diperhatikan berubah sedikit kepada kurang nilai positif iaitu pada lebih kurang 220 mV dan dengan nyata sekali ditingkatkan pada lebih kurang 3.2 kali ganda. Kepekaan pada keadaan siklik voltammetri nyata sekali bergantung kepada pH, suhu, dan kadar imbasan. Plot penentukuran menunjukkan kelinearan dari julat  $5.0 \times 10^{-7}$  hingga  $2 \times 10^{-3}\text{M}$  dengan satu pekali korelasi iaitu 0.998. Had pengesanan adalah dianggarkan  $7.4 \times 10^{-7}$  M.

Penggunaan zink oksida ( $\text{ZnO}$ ) modifikasi elektrod karbon berkaca (GC) telah dilanjutkan dalam perantaraan pengoksidaan asid askorbik dalam 0.1M larutan elektrolit  $\text{KH}_2\text{PO}_4$  menggunakan siklik voltammetri (CV). Elektrod  $\text{ZnO} / \text{GC}$  menunjukkan dengan jelas peningkatan dan kesan electropemangkinan kerana menyebabkan arus pengoksidaan asid askorbik meningkat sebanyak 1.5 kali ganda berbanding dengan elektrod GC tanpa modifikasi. Perbezaan kajian kadar imbasan menunjukkan yang sistem ini mengalami proses pengawalan pembauran. Pekali pembauran dan kadar pemalar asid askorbik ditentukan menggunakan kaedah hidrodinamik (elektrod cakera putaran) dengan nilai masing-masing sebanyak  $5.4 \times 10^{-6} \text{cm}^2\text{s}^{-1}$  and  $2.5 \times 10^{-3} \text{cm}^2\text{s}^{-1}$  untuk elektrod tanpa modifikasi, sementara nilai-nilai pekali pembauran dan kadar

pemalar asid askorbik menggunakan elektrod ZnO / GC masing-masing ialah  $5.7 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$  and  $2.1 \times 10^{-3} \text{ cm}^2 \text{ s}^{-1}$ .

Zink oksida (ZnO) microparticles telah dilekatkan secara mekanik di atas permukaan elektrod karbon kaca (GC). Tujuan kajian ini ialah untuk mengkaji secara kritis pengubahsuaian elektrod GC dengan zinc oksida microparticles dan kesan pada pengoksidaan parasetamol dalam  $0.1 \text{ M KH}_2\text{PO}_4$  larutan elektrolit oleh siklik voltametri (CV). Aktiviti elektropemangkin yang baik ke arah pengoksidaan parasetamol telah diperhatikan. Keupayaan puncak telah diperhatikan bergerak ke nilai kurang positif iaitu pada  $150 \text{ mV}$  dan arus nyata sekali meningkatkan pada lebih kurang 1.1 kali ganda berbanding dengan elektrod GC tanpa modifikasi. Perbezaan kajian kadar imbasan menunjukkan yang sistem ini mengalami proses pengawalan pembauran. Sebagai tambahan, plot penentuan menunjukkan kelinearan dari julat  $2.0 \times 10^{-5}$  hingga  $5.0 \times 10^{-3} \text{ M}$  dengan 0.997 pekali korelasi.

Juga terbukti dengan penggunaan modifikasi  $\text{MgB}_2/\text{Li}^+/\text{CNT}/\text{GC}$  elektrod ianya cenderung untuk meningkatkan pengoksidaan parasetamol dalam  $0.1 \text{ M KH}_2\text{PO}_4$  larutan elektrolit pada pH 4.2 di elektrod tanpa modifikasi GC, modifikasi  $\text{MgB}_2 / \text{GC}$  elektrod, modifikasi  $\text{MgB}_2/\text{Li}^+/\text{GC}$  elektrod dan modifikasi  $\text{MgB}_2/\text{Li}^+/\text{CNT} / \text{GC}$  elektrod. Manakala pada elektrod GC yang dimodifikasi, anjakan puncak  $130 \text{ mV}$  dan  $150 \text{ mV}$  ke arah kurang positif telah diperhatikan untuk  $\text{MgB}_2 / \text{Li}^+/\text{CNT} / \text{GC}$  dan  $\text{MgB}_2/\text{Li}^+/\text{GC}$  masing-masing dengan sedikit peningkatan arus berbanding dengan elektrod tanpa modifikasi. Bagaimanapun , di  $\text{MgB}_2/\text{Li}^+/\text{CNT} / \text{GC}$  arus ditingkatkan sebanyak 3 kali

dengan anjakan puncak yang sama seperti  $\text{MgB}_2 / \text{Li}^+ / \text{GCE}$ , menunjukkan kehadiran proses elektromangkikan kesan daripada  $\text{MgB}_2 / \text{Li}^+$  , dan salutan  $\text{MgB}_2 / \text{Li}^+ / \text{CNT}$ .



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In The Name of ALLAH, the Most Merciful and Most Beneficent

All praises be to Allah, Lord of the universe. Only by His grace and mercy this thesis could be completed.

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## APPROVAL SHEETS

I certify that an Examination Committee met on    to conduct the final examination of MOHAMMED ZIDAN on the Doctor of Philosophy thesis entitled **ELECTROCHEMICAL CHARACTERIZATION AND APPLICATION OF CARBON NANOTUBES AND BISMUTH OXIDE, ZINC OXIDE AND MAGNESIUM DIBORIDE MODIFIED GLASSY CARBON ELECTRODES** ” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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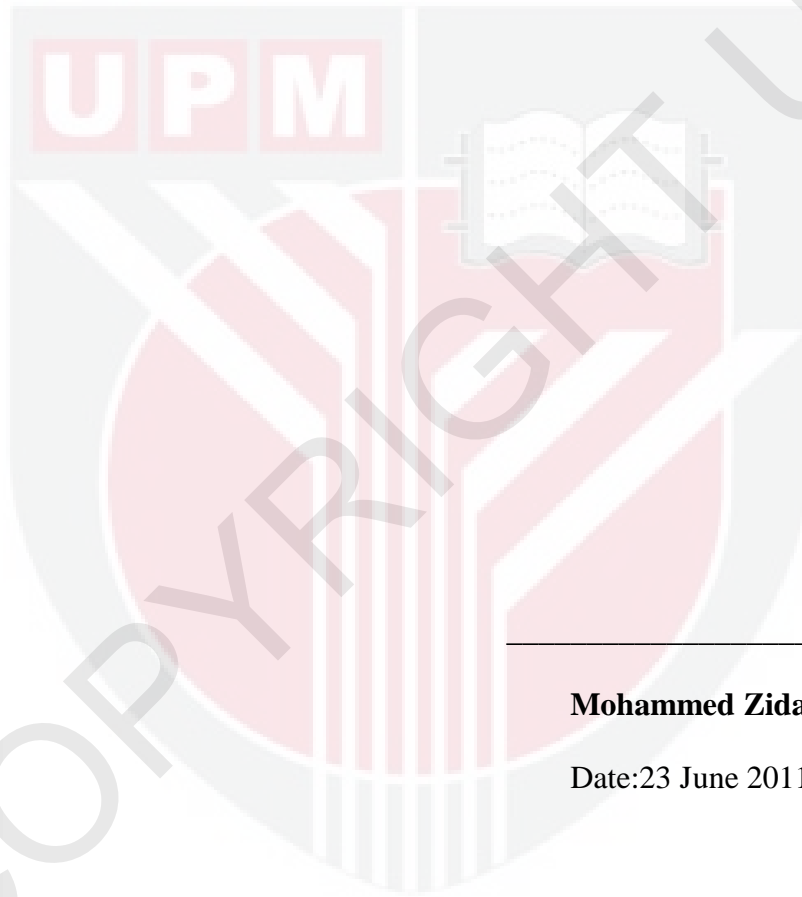
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## DECLARATION

I declare that the thesis is my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or other institutions.



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**Mohammed Zidan**

Date:23 June 2011



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