

# **UNIVERSITI PUTRA MALAYSIA**

# ELECTROCHEMICAL STUDY OF MAGNESIUM DIBORIDE MODIFIED ELECTRODE

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## ELECTROCHEMICAL STUDY OF MAGNESIUM DIBORIDE MODIFIED ELECTRODE



By

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#### March 2011

Chair : Assoc. Prof. Tan Wee Tee, PhD

Faculty : Faculty of Science

Use of a glassy carbon (GC) modified by adhered microparticles of MgB<sub>2</sub> mediates the reduction process of  $Fe(CN)_6^{3-}$  during cyclic voltammetry. Potential at reduction peak was observed to shift slightly from 0.20 to 0.23 V and current is significantly enhanced by about two folds. The sensitivity under conditions of cyclic voltammetry is significantly dependent on pH, electrolyte and scan rate. The result of scanning electron micrograph of MgB<sub>2</sub> obtained before and after electrolysis show the size of the MgB<sub>2</sub> microparticles increased slightly to the size ranging from 2 - 5.5  $\mu$ m attributing to the hydration effect and/or incorporation of some ionic species into the crystal lattices of MgB<sub>2</sub>. Interestingly, redox reaction of Fe(III) solution using modified GC electrode remain constant even after 15 cycle reflecting the usability of the MgB<sub>2</sub> film attached to the GC electrode surface.

Use of a lithium doped magnesium diboride modified glassy carbon electrode enhance the oxidation current of ascorbic acid during cyclic voltammetry compare to bare GC and MgB<sub>2</sub> modified electrode. Peak potential was observed to shift slightly from around 0.40 to 0.25 V and current is significantly enhanced by about two folds. The sensitivity under conditions of cyclic voltammetry is significantly dependent on pH, temperature, electrolyte and scan rate. The result of scanning electron micrograph of MgB<sub>2</sub> with Li<sup>+</sup> doped obtained before and after electrolysis show the size increased slightly to the size ranging from  $0.5 - 1.3 \mu m$  to  $3 - 7 \mu m$  attributing to the hydration effect and/or incorporation of some ionic species into the crystal lattices of MgB<sub>2</sub>.

The oxidation current of ascorbic acid decreased sharply after the first cycle and become stable with minor decreases after second cycle. The recovery values of  $99.0\pm0.4\%$  was obtained after the addition of 0.5 mM ascorbic acid into rose flavour syrup while recovery of  $99.2\pm0.1\%$  was obtained after the addition of 0.05 mM ascorbic acid into rose syrup. Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan ijazah Master Sains

## KAJIAN ELEKTROKIMIA MODIFIKASI ELEKTRODE MAGNESIUM DIBORIDA

Oleh

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Elektrod karbon kaca (KK) diubahsuai dengan perantara mikropartikel  $MgB_2$  proses penurunan  $Fe(CN)_6^{3-}$  dalam kitaran voltammetri. Puncak potensi yang diperoleh mengalami sedikit pergeseran daripada 0.20 ke 0.23 V dan arus secara signifikan meningkat kira-kira dua kali ganda. Kepekaan dalam keadaan kitaran voltammetri secara signifikan bergantung pada pH, elektrolit dan kadar kitaran. Keputusan imbasan mikrograf elektron  $MgB_2$  diperolehi sebelum dan selepas elektrolisis menunjukkan saiz sedikit peningkatan dengan saiz berkisar 2-5.5 µm kesan penghidratan dan/atau penggabungan beberapa spesies ion ke dalam kekisi kristal  $MgB_2$ . Menariknya, reaksi redoks larutan Fe(III) menggunakan elektrod ini tetap konsisten setelah 15 kitaran mencerminkan kegunaan dari filem  $MgB_2$  melekat pada permukaan elektrod KK.

Lekatan lithium pada modifikasi KK yang diubahsuai dengan MgB<sub>2</sub> meningkatkan arus pengoksidaan paling tinggi pada kitaran voltammetri asid askorbik berbanding dengan elektrod KK tanpa modifikasi dan elektrod karbon kaca (KK) diubahsuai dengan mikropartikel MgB<sub>2</sub>. Puncak potensi mengalami sedikit pergeseran dari 0.40 ke 0.25 V dan arus secara signifikan meningkat kira-kira dua kali ganda. Kepekaan dalam keadaan kitaran voltammetri secara signifikan bergantung pada pH, suhu, elektrolit dan kadar kitaran. Keputusan imbasan mikrograf elektron MgB<sub>2</sub> dengan lekatan lithium diperolehi sebelum dan selepas elektrolisis menunjukkan saiz sedikit peningkatan dengan saiz berkisar antara 0.5 - 1.3 µm ke 3 - 7 µm kesan penghidratan dan/atau penggabungan beberapa spesies ion ke dalam kekisi kristal MgB<sub>2</sub>.

Puncak pengoksidaan asid askorbik turun mendadak selepas kitaran pertama dan menjadi stabil selepas kitaran kedua. Penentuan kandungan asid askorbik ditentukan dengan nilai 99.0±0.4% diperoleh untuk sampel air sirap yang ditambah kepekatan asid askorbik 0.5 mM manakala 99.2±0.1% diperoleh daripada penambahan kepekatan asid askorbik 0.05 mM ke dalam sampel sirap.

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I certify that a Thesis Examination Committee has met on **2011** to conduct the final examination of **Mohd Farhan bin Yusri** on his thesis entitled **Electrochemical Study of Magnesium Diboride Modified Electrode** in accordance with the Universities and University Colleges Act 1971 and the Constitution of the University Putra Malaysia [P.U. (A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science

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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 at any other institution.



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