

UNIVERSITI TEKNOLOGI MARA

**STUDIES ON
ELECTROPOLYMERIZATION OF
ORTHO-PHENYLENEDIAMINE ON
PLATINUM MICROELECTRODE
AND ITS DETECTION OF
HYDROGEN PEROXIDE AND
ASCORBIC ACID USING
ELECTROCHEMICAL IMPEDANCE
SPECTROSCOPY**

AZIAH BT AHMAD ARIFFIN

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of the requirements for the degree of
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AUTHORS'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the result of my own work, unless otherwise indicated and acknowledged as references work. This thesis has not been submitted to any other academic institutions or non-academic institution for any degree or qualification.

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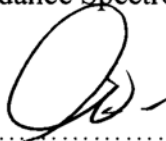
Name of Student : Aziah Bt Ahmad Ariffin

Student I.D. No : 2010204712

Programme : Master of Science (AS780)

Faculty : Applied Science

Thesis Title : Studies on electropolymerization of *Ortho*-Phenylenediamine on Platinum Microelectrode and Its Detection of Hydrogen Peroxide and Ascorbic Acid Using Electrochemical Impedance Spectroscopy

Signature of Student : 

Date : February 2015

ABSTRACT

Poly-*ortho*-phenylenediamine (PoPD) has been successfully electropolymerized and characterized on Platinum disk (125 μm internal diameters) using Electrochemical Impedance Spectroscopy (EIS) and Cyclic Voltammetry (CV). *o*PD was first prepared using CV by dissolving *o*PD monomer into Phosphate Buffer Saline (PBS) using various monomer concentration, pH of PBS and scan rate to determine the optimum condition. The optimum condition (300 mM *o*PD in pH 7.2 PBS with scan rate 100 mVs^{-1}) recorded oxidation potential $E = 500 \text{ mV}$ at the highest anodic peak current, $9.16 \times 10^5 \text{ mA cm}^{-2}$. Electropolymerization of *o*PD was carried out by EIS with applying the optimum condition and oxidation potential to determine the redox-kinetic parameters and electrical behavior of the system. Up to our knowledge, *o*PD electropolymerization on Pt disk microelectrode is firstly reported in this work. The redox-kinetic parameters of PoPD obtained from impedance data are charge transfer resistance, R_{ct} , ($1.80 \times 10^2 \text{ k}\Omega$), diffusion coefficient, D , ($1.23 \times 10^5 \text{ cms}^{-1}$) and double-layer capacitance, C_{dl} , ($0.14 \text{ }\mu\text{F}$). These parameters were obtained as the impedance spectra were fitted to three proposed equivalent circuit models beginning with simple Randles Model, $(R(Q[RW]))$ to a more complex models such as $([R(RQ)([RW]Q))$ and $[R([RW]C)(RC)]$. They showed chi square values, χ^2 , less than zero indicating the models fits to the impedance spectra. The PoPD coated electrode was used as analytical probes towards the detection of Hydrogen Peroxide (H_2O_2) and Ascorbic Acid (AA). EIS used to investigate the blocking ability of PoPD to the ascorbic acid and hydrogen peroxide is also firstly reported in this work. In various analytes concentrations, the impedance value of AA higher ($4.4 \text{ k}\Omega$ to $6.6 \text{ k}\Omega$) than H_2O_2 ($2.8 \text{ k}\Omega$ to $3.7 \text{ k}\Omega$) indicated the larger size of AA species impeded by PoPD layer to the electrode surface compared with smaller size of H_2O_2 species allowed to permeate. Meanwhile, dielectric constant values were also determined from the impedance data and exhibited higher values for ascorbic acid compared hydrogen peroxide as well. PoPD formation onto the electrode surface was confirmed by FESEM by exhibited a rough and crater-like surface formed.

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