UNIVERSITI PUTRA MALAYSIA

SYNTHESIS OF POLYPYRROLE/TANNIN/CETYLTRIMETHYLAMMONIUM BROMIDE NANOCOMPOSITES FOR DOPAMINE DETECTION

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FS 2019 15

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

November 2018

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

SYNTHESIS OF POLYPYRROLE/TANNIN/CETYLTRIMETHYLAMMONIUM BROMIDE NANOCOMPOSITES FOR DOPAMINE DETECTION

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November 2018

Chair : Mahnaz M.Abdi, PhD
Faculty : Science

Polypyrrole-Tannin (PPy/TA) nanocomposite was prepared in the presence of cationic surfactant, cetyltrimethylammonium bromide (CTAB) using the chemical polymerization method. The resulting PPy/TA/CTAB nanocomposite was used to modify screen printed carbon electrode (SPCE) for developing a dopamine (DA) biosensor. The presence of TA was able to repel the interference molecules (AA and UA) in DA detection, while CTAB act as a soft-template in tailoring the nanostructure of PPy. The electrochemical properties of the modified electrode were studied via cyclic voltammetry (CV) and SPCE/PPy/TA/CTAB-modified electrode revealed a higher current response compared to modified electrode of SPCE/PPy and SPCE/PPy/TA. The results obtained from Electrochemical Impedance Spectroscopy (EIS) exhibited a lower value of charge transfer resistance (Rct) for the SPCE/PPy/TA/CTAB-modified electrode indicating an enhancement in the electron transfer rate.

The Fourier-Transform Infrared (FTIR) spectra of nanocomposite displayed the typical characteristic peaks of TA and CTAB which are evidences of the incorporation of these components into PPy. Electron Microscopy (TEM) revealed a rod-like structure with a lumpy surface for PPy/TA/CTAB nanocomposite, justifying the highest current response for the modified electrode. Brunauer-Emmet-Teller (BET) measurement displayed a surface area of 23.5 m^2/g for PPy/TA/CTAB nanocomposite.

Response Surface Methodology (RSM) has been applied to optimize the selected parameters and conditions in order to maximize the biosensor's performance and its sensitivity. Amperometry and Differential Pulse Voltammetry (DPV) analysis were employed for all electrochemical measurements and dopamine detection in two different ranges of 0.1–2 μM and 2–50 μM. The good adhesion of nanocomposite on the electrode surface, as well as the high surface area and porosity of the modified

electrode, enhanced the diffusion of DA molecules inside the matrix. Amperometry analysis of the SPCE/PPy/TA/CTAB modified electrode displayed a good sensitivity of 0.039 μA $(\mu M)^{-1}$ towards dopamine with the limit of detection (LOD) of 2.9×10^{-7} M. The modified biosensor also excludes the interfering species of ascorbic acid (AA) and uric acid (UA) which makes the sensor suitable for DA determination. This biosensor showed an acceptable reproducibility and repeatability with low relative standard deviations (RSD) of 4.8% and 4.4%, respectively. The current response remained about 81.5% of the original value after 21 days indicating an accepted stability over time.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

SINTESIS POLIPIROL/TANIN/SETILTRIMETILAMMONIUM BROMIDA KOMPOSIT NANO BAGI PENGESANAN DOPAMIN

Oleh

NUR FARHANA WAHEEDA BINTI MOHD AZLI

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Komposit nano polimer konduktor Polipirol-Tanin (PPy/TA) telah disediakan dengan kehadiran surfaktan kationik, setiltrimetilammonium bromida (CTAB) dengan menggunakan kaedah pempolimeran kimia. PPy/TA/CTAB komposit nano yang dihasilkan telah digunakan untuk mengubah suai karbon elektrod skrin bercetak (SPCE) untuk membangunkan biosensor dopamin (DA). Kehadiran TA mampu menangkis molekul gangguan (AA dan UA) dalam pengesanan DA, sementara CTAB bertindak sebagai templat-lembut dalam melaraskan struktur nano PPy. Ciri-ciri elektrokimia elektrod yang diubahsuai telah dikaji melalui Voltammetri Berkitar (CV) dan elektrod yang diubahsuai oleh SPCE/PPy/TA/CTAB mendedahkan tindak balas arus yang lebih tinggi berbanding elektrod yang diubahsuai oleh SPCE/PPy dan SPCE/PPy/TA. Keputusan yang diperolehi daripada EIS mempamerkan nilai rintangan pindah cas (Rct) yang rendah bagi elektrod yang diubahsuai oleh SPCE/PPy/TA/CTAB, yang menunjukkan bahawa penggabungan CTAB ke dalam komposit dapat meningkatkan kadar pemindahan elektron.

Spektrum Inframerah Transformasi Fourier (FTIR) komposit nano masing-masing memaparkan ciri tipikal puncak yang merupakan bukti penggabungan komponen-komponen ini ke dalam PPy. Mikroskopi Elektron Transmisi (TEM) mededahkan struktur rod dengan permukaan yang berketul-ketul bagi komposit nano PPy/TA/CTAB, mewajarkan tindak balas arus tertinggi bagi elektrod yang diubahsuai. Keputusan yang diperolehi daripada analisis termal menunjukkan bahawa interaksi TA dengan PPy dapat meningkatkan kestabilan termal PPy sebanyak 307 °C. Pengukuran Brunauer-Emmet-Teller (BET) memaparkan luas permukaan sebanyak 23.5 m^2/g bagi komposit nano PPy/TA/CTAB.

Kaedah Permukaan Penyelidikan (RSM) telah digunakan untuk mengoptimumkan parameter dan keadaan yang dipilih bagi memaksimumkan prestasi dan sensitiviti biosensor. Analisis Amperometri dan Voltammetri Denyut Pembeza (DPV) telah digunakan untuk semua pengukuran elektrokimia dan pengesanan dopamin dalam dua julat berbeza iaitu 0.1–2 μM dan 2–50 μM. Lekatan komposit nano yang baik pada permukaan elektrod, serta kawasan permukaan dan keliangan yang tinggi elektrod yang diubahsuai, mempertingkatkan resapan molekul DA di dalam matriks. Analisis Amperometri elektrod diubahsuai SPCE/PPy/TA/CTAB memapar sensitiviti yang baik pada nilai 0.039 μA $(\mu M)^{-1}$ terhadap dopamin dengan had pengesanan (LOD) 2.9×10^{-7} M. Biosensor yang diubahsuai juga menyingkirkan spesis gangguan asid askorbik (AA) dan asid urik (UA) yang membuatkan sensor ini sesuai untuk penentuan DA. Biosensor ini menunjukkan kebolehpenghasilan semula dan kebolehulangan yang boleh diterima dengan sisihan standard relatif rendah (RSD) masing-masing 4.8% dan 4.4%. Tindak balas arus kekal kira-kira 81.5% daripada nilai asal selepas 21 hari yang menunjukkan kestabilan di atas masa yang diterima.

ACKNOWLEDGEMENTS

Alhamdulillah, thank you, Allah, for giving me the opportunity, strength, and good health throughout my master's study.

Special thanks to my beloved parents, Mohd Azli Hassan and Noorain Yussuf, my aunt, Ramlah Yussuf, and my siblings (Najwa, Mohammad, Azim and Izzati), for always providing me with encouragement and unfailing support in spiritually throughout my studies (researching and writing this thesis) and my life in general.

I would like to dedicate this thesis to all my respective supervisory committee, especially my research supervisor, Dr. Mahnaz M. Abdi for the continuous support of my master's study and related research, for her patience, motivation, and immense knowledge. Her guidance helped me all time in research and writing of the thesis since the beginning until the end of my studies. I could not have imagined having a better supervisor and advisor for my master study. Appreciation also goes to my co-supervisors too, Prof. Paridah Mat Tahir and Assoc. Prof. Dr. Janet Lim Hong Ngee, who's provided me with the opportunity to be part of their team, and gave me an access to the laboratory, research facilities, suggestions and opinions regarding my research. Without their precious support, it would not be possible to conduct this research.

I want to thank Prof. Zulkarnain Zainal Abidin and his fellow postgraduate students who provided good laboratory facilities, stimulating discussions, sharing knowledge and problems solving. Also, thank you to my fellow lab mate, Rawaida Liyana Razalli for always be there and help me during my hard time especially regarding my research. Thank you for all the fun that we had and the memories we created.

I would also want to express my gratitude to the all the staff involved from the Faculty of Science, Institute of Tropical Forestry and Forest Products (INTROP), and Institute of Bioscience (IBS) for their kindness in solving problems and expert in conducting analysis of my samples.

Not to forget to all my UPM friends, for their encouragement and to be part of my studies life in teaching me the meaning of friendship and to be someone with a strong will to success in life. This accomplishment would not have been possible without them.

Lastly, I would like to extent my gratitude to Ministry of Education Malaysia for providing support in my study fees through MyMaster.

Thank you so much for everything.

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

µM	Micromolar
^0C	Degree Celsius
AA	Ascorbic Acid
APS	Ammonium Persulfate
AsF_5	Arsenic Pentafluoride
BET	Brunauer-Emmet-Teller
C	Capacitance
CCRD	Central composite rotatable design
CE	Counter Electrode
cm	Centimeter
CMC	Critical Micelle Concentration
CP	Conducting Polymer
CPE	Constant Phase Element
CTAB	Cetylltrimethylammonium Bromide
CV	Cyclic Voltammetry
Da	Dalton
DA	Dopamine
DIW	De-ionized Water
DNA	Deoxyribonucleic Acid
DPV	Differential Pulse Voltammetry
E	Potential
EIS	Electrochemical Impedance Spectroscopy
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier-Transform Infrared Spectroscopy
g	gram
I	Current
I_2	Iodine
K	Kelvin
kv	Kilovolt
LOD	Limit of Detection
M	Molar
mg	Milligram
ml	Milliliter
mM	Millimolar
mVs^{-1}	Millivolt per second
N	Nitrogen
N_2	Nitrogen gas
Na	Sodium
nm	Nanometer
PBS	Phosphate Buffer Solution
PPy	Polypyrrole
Py	Pyrrole
R^2	Regression
Rct/Q	Charge Transfer Resistance
RE	Reference Electrode
rpm	revolutions per minute

R_s	Solution Resistance
RSE	Relative Standard Error
RSM	Response Surface Methodology
S	Second
SN	Polysulfur Nitride
SPCE	Screen Printed Carbon Electrode
TA	Tannin
TEM	Transmission Electron Microscopy
TGA	Thermogravimetry Analysis
UA	Uric Acid
V	Volt
Vs^{-1}	Volt per second
W	Warburg
WE	Working Electrode
π	Pie
Ω	Ohm

CHAPTER 1

INTRODUCTION

1.1 Background

Research in conducting polymers has been established for years with many applications in nanoscience and nanotechnology, such as electric energy storage systems, chemical-to-electric or vice-versa energy conversion, sensors and biosensors, and corrosion protection system. To date, even though there is various literature on conducting polymers, studies to improve the physical and chemical properties of these materials are still ongoing (Naveen *et al*., 2017). There are a few developments involving composites of conducting polymer-based biomolecules, and surfactant using screen-printed carbon electrodes (SPCEs) in the biosensor application.

Polypyrrole is one of the most extensively used as a conducting polymer in the design of bioanalytical sensors as well as for other purposes. However, polypyrrole had drawbacks such as low solubility, dispensability, and response time, limit their application in sensors. Thus, many attempts have been made to overcome these shortcomings and develop new composites of conducting polymers with biopolymers, surfactants, and nanoparticles for biomolecule detection. Ghanbari and Hajheidari, 2015, had modified the glassy carbon electrode with nanocomposite of hybrid nanostructured materials of zinc oxide nanosheets and copper oxide ($ZnO\text{-}Cu_xO$) coupled with conducting polymer of PPy ($ZnO\text{-}Cu_xO\text{-}PPy$) for simultaneous detection of dopamine, ascorbic acid, and uric acid. Selvarajan *et al*., 2018, used the copper oxide (Cu_2O) with silver-polypyrrole (Ag/PPy) to enhance the electrocatalytic activity of the biosensor, where the modified glassy carbon electrode was able to exhibit higher electrocatalytic activity and good sensitivity for the detection of serotonin (5HT).

As a consequence, the aim of this study is to enhance the physical and chemical properties of polypyrrole by means of incorporation of tannin (biopolymer) and a cationic surfactant to form polypyrrole nanocomposite. The presence of TA enhances the physical and mechanical properties of polypyrrole. In addition, hydroxyl groups with negative charges in TA structure encourage accumulating positive charges of DA and repel AA and UA on the modified electrode via electrostatic interaction.

Surfactants are one of the most widely used materials for modification CPs, leading to many applications in sensing devices. Surfactants act as a template for a formation of nano- and mesostructured particle during synthesis, which leads to the increase in the penetration rate of the target molecules in the sensing area of the biosensor and thus decreases its response time (Viana *et al.,* 2012). Therefore, the ability of hard or soft templates of the surfactants to produce a high surface material for the sensing

applications, attract the researcher interest to utilize it through the synthesis of the conducting polymer nanostructure (Xia *et al.,* 2010).

In the current research, a cationic surfactant of cetyltrimethylammonium bromide (CTAB) was utilized as a soft template to prepare a nanostructured compound in the developing of a highly porous substrate with a high rate of electron transfer. The composites of polypyrrole/tannin (PPy-TA) were prepared in the presence of different concentration of CTAB. The nanocomposite of PPy/TA/CTAB was made via chemical polymerization and then was used for the fabrication of a biosensor for biomolecule detection (such as dopamine and ascorbic acid).

Among biomolecules, dopamine (DA) is a neurotransmitter that carries signals between brain cells and helps control the brain's reward and pleasure centers. The abnormal amount of DA in body systems may frequently cause varied neurological disorders, including Parkinson's disease and schizophrenia (Stopper *et al.,* 2009). However, the electrochemical determination of dopamine is mainly affected by the oxidation of ascorbic acid (AA) and uric acid (UA), which often coexist with the DA in the biological samples and exhibit a similar oxidation potential (Naseri & Majidi, 2011). Therefore, an electrode modification is necessary to detect DA in the presence of other biomolecules (Wang *et al.,* 2014).

In order to optimize parameters and conditions for biosensor fabrication and to achieve the best performance, all experiments were designed using response surface methodology (RSM), which is one of the well-known design of experimental technique.

1.2 Problem Statement

Conducting polymer sensors have received growing attention due to their favorable properties. Polypyrrole (PPy) is an example of an electronically conducting polymer that has widely been used in biosensors. It is identified to possess outstandingly high electrical conductivity, simply deposits from aqueous and non-aqueous media, highly adherent to many types of substrates, which sums up the advantages it offers (Singh *et al*., 2011). However, the problem arises due to the disadvantages of PPy such as having a lack of mechanical properties (Wang *et al.,* 2001), a low level of dispersion ability, and is structurally bulky, which limit its application in sensors, especially in the purposes of obtaining the high sensitivity in the detection of the target analyte.

Thus, in this research, CTAB as a surfactant has been utilized in order to provide a nanostructure composite, and the main reason for using TA was to enhance the sensor performance for dopamine detection in the presence of other biomolecules, especially AA and UA, as TA molecules could help to repel those interferences. In addition, TA also was used to enhance the physical and electrochemical properties of PPy.

1.3 Research Objectives

The main aim of this research is to prepare PPy/TA/CTAB nanocomposite and study its physical and electrochemical properties for sensor application. The objectives are as follows:

1. To prepare and characterize the electrochemical and physical properties of the PPy/TA/CTAB nanocomposites.

2. To evaluate the application of the modified electrode using PPy/TA/CTAB nanocomposites for biomolecules (dopamine and/or ascorbic acid and/or uric acid) detection using DPV and CV.

3. To find the optimal conditions of the target analyte by using the fabricated biosensor with the aid of Research Surface Methodology (RSM) and evaluate the biosensor in terms of its performance in detection of the analyte.

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In 2009, she continues her study in Penang Matriculation College, Pulau Pinang, as a biological science student and undergoes her fourth education in the field of Environmental and Analytical Chemistry in Universiti Malaysia Terengganu (UMT), Kuala Terengganu, Terengganu in 2011. She graduated three years later in Bachelor of Science (Honours)-Environmental and Analytical Chemistry.

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PUBLICATIONS

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Abdi, M. M., Azli, N. F. W. M., Lim, H. N., Tahir, P. M., Razalli, R. L., & Hoong, Y. B. (2017). Chemical polymerization and characterization of surfactant directed of polypyrrole-tannin-CTAB nanocomposites. In *AIP Conference Proceedings* (Vol. 1901, pp. 1–5). http://doi.org/10.1063/1.5010442

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