UNIVERSITI TEKNOLOGI MARA

SURFACE MODIFICATION OF SCREEN-PRINTED CARBON ELECTRODE BY ELECTROSPUN POLYACRYLIC ACID/ POLYACRYLONITRILE AND GOLD NANOPARTICLES FOR NON-ENZYMATIC GLUCOSE SENSOR

ILYANI BINTI ISMAIL

MSc

January 2021

AUTHOR'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 results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

Name of Student	:	Ilyani Binti Ismail
Student I.D. No.	:	2016723631
Programme	:	Master of Science (Chemical Engineering) – EH750
Faculty	:	Chemical Engineering
Thesis Title	:	Surface Modification of Screen-Printed Carbon Electrode by Electrospun Polyacrylic Acid/Polyacrylonitrile and Gold Nanoparticles for Non-enzymatic Glucose Sensor
Signature of Student	:	………………………………………………..
Date	:	January 2021

ABSTRACT

The extensive knowledge and understanding regarding nanofiber through the electrospinning method has empower revolutionary of polymeric composites to be utilized for glucose sensor application. As compared to customary drop-casting method used to modify electrode surface, electrospun fibers score more points. This is the starting point of this research, modifying electrode structure using electrospinning. Nanofibers enable porous structure which lead to high surface to volume ratio. Apart from that, nanofibers allow diffusion of analytes with minimal resistance and improve electron transfer by having great capacity for electron to transport. However, little attempt is made to study the correlation between the effect of electrospinning solution parameter on fiber morphology and how the fiber morphology difference affect the performance of sensor. From this perspective, the first part of this thesis focused on the manipulation of electrospinning solution concentration and resulted electrospun fiber morphology. The polymer used for the first part of this research was polyacrylic acid (PAA). Understanding the electrospun fiber morphology effect on its conductivity enables the correct PAA concentration selection to ensure the best sensor performance. PAA electrospun fiber was not viable to be directly applied for sensor application as the fiber structure cannot be retained if immersed in water. Thus, PAN solution at 9.5 wt% is introduced to optimal 6% (wt) PAA solution prior to electrospinning to render the fiber from insoluble in water. Three volume ratios of PAA/PAN were manipulated to select appropriate formulation for the best conductivity. The results showed that PAA/PAN at ratio 6:4 has the best conductivity. The noble metal gold in nano-sized (AuNPs) was employed in this research to increase the sensing element. The performance of sensor is related to surface area for analyte to adhere at active site. The electrochemically active surface area (ECSA) of an electrode is highly dependent on the nano-structured particles to generate high currents. Thus, adoption of AuNPs increased the sensitivity of sensor as it creates better electron transfer media and add more active sites. An electrochemical sensor consisting of a screen-printed carbon electrode (SPCE) modified with electrospun polyacrylic acid/ polyacrylonitrile and AuNPs (PAA/PAN/AuNPs) was developed for the non-enzymatic determination of glucose. To achieve the optimal electrocatalytic oxidation of glucose, the pH, deposition potential and deposition time were optimized using differential pulse voltammetry (DPV). Under the optimal conditions; pH:10, deposition potential: -0.35 V and deposition potential: 210 s calibration curves were made at which the sensor had a detection limit of 1.52 µM, sensitivity of 1071.1 $\mu A\ cm^{-2}\ mM^{-1}$. Two linear ranges which are 2 to 10 Mµ and 2 mM to 16 mM were constructed. The modification made the device compact, low cost and reliable non-enzymatic glucose sensor. The modified SPCE was successfully used for the sensing of glucose.

ACKNOWLEDGEMENT

Firstly, I am deeply thankful and grateful to Allah for everything. Then, not to forget my supervisors who had trained me to act and think like a researcher. Especially Assoc. Prof. Ir. Dr. Noor Fitrah Abu Bakar for always trusting me, being gentle and gave me thousands of second chances. The art from her brain had turned my rubbish into a masterpiece. My other supervisors; Dr Tan Huey Ling and Dr Norhidayah Ideris helped me to point the direction of my research. Also, thanks to my labmates a.k.a labrats A, S, H, Q, PGS for being a good friend, helper, rival and most of the time mentoring me to finish this journey.

Last but never the least, I would like to thank my family especially my mom for consistently asking me when I will finish my masters (in a very supportive way) and being my backbone to strengthen me; physically, emotionally, mentally, spiritually and financially.

TABLE OF CONTENTS