ELECTROCHEMICAL SENSOR USING CHITOSAN-BASED NANOMATERIALS MODIFIED SCREEN PRINTED CARBON ELECTRODE FOR BISPHENOL A DETECTION

By

NOR FAEZAH BINTI MOHD DAUD

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

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DEDICATION

This thesis is dedicated to my beloved family, lovely husband, my daughters, and all Muslim.

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

ELECTROCHEMICAL SENSOR USING CHITOSAN-BASED NANOMATERIALS MODIFIED SCREEN PRINTED CARBON ELECTRODE FOR THE DETECTION OF BISPHENOL A

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ABSTRACT

Bisphenol A (BPA) is an organic compound found in plastic products that used every day. It is mostly transferred into foods and drinks including water bottles, lining of food cans, and dental fillings. It is known that BPA is considered as an endocrine-disrupting compound which related to various kinds of health concerns such as the development of cancers and reproduction problems. Thus, the development of a reliable analytical approach for the detection of BPA is an urgent issue. In this study, electrochemical sensor based on modification of screen-printed carbon electrode (SPCE) with two fabricated sensor which are carbon dots-chitosan (CDs-CS) and gold nanoparticles/carbon dots-chitosan (AuNPs/CDs-CS) have been explored for the detection of BPA. Each sensor was prepared using drop casting technique. The detection of BPA using these sensors was studied employing differential pulse voltammetry (DPV). Characterization was done by Field emission scanning electron Microscopy (FESEM) and Energy Dispersive X-Ray Spectroscopy (EDX), High-Resolution Transmission Electron Microscopy (HRTEM) and Fourier Transform Infrared Spectroscopy (FTIR). FTIR spectra for CDs-CS shows peak at 1648 cm^{-1}, 1411 cm^{-1} and 1038 cm^{-1} for vibration of C=O hydroxyl group on CDs. New peak at 1100 cm^{-1} are related to the strong C-O stretching of AuNPs/CDs-CS. Optimum parameter for the sensor development such as CS concentration of 1% (m/v) and CDs : CS ratio of 1/1 (v/v), and 7 µL of CDs-CS volume drop casted on SPCE for first sensor, the volume ratio of the composite CDs-CS and AuNPs was 1 : 1.5 (v/v) and 3 µL of AuNPs/CDs-CS drop casted on SPCE for the second sensor was evaluated to improve the performance of the developed sensors. The results showed that SPCE modified with CDs-CS composite and AuNPs/CDs-CS has a great electrocatalytic features for detecting BPA in the concentration range of 0.4 µM to 10 µM with sensitivity of 0.46 µA/µM (R^2 = 0.9911) and limit of detection (LOD) of 0.37 µM for the CDs-CS/SPCE sensor while concentration range of 0.4 µM to 10 µM of BPA with sensitivity of 0.28 µA/µM (R^2 = 0.9937) and LOD of 0.1 µM for the AuNPs/CDs-CS/SPCE sensor.

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Good reproducibility of the sensors with relative standard deviation (RSD) 3.66 % (n = 15) for CDs-CS/SPCE while 1.35 % (n = 15) for AuNPs/CDs-CS/SPCE, respectively. For storage stability, both sensors retained their performances to 79.1% and 90.3% for CDs-CS/SPCE and AuNPs/CDs-CS/SPCE of their original response after a month of storage. The interferences studies of these sensors showed that there is no significant interfering effect towards phenol, 2,4-dinitrophenol, 4-chlorophenol, K^+,Cu^{2+}, Cl^-, and SO_4^{2-} even with 10-fold higher concentration compared to BPA. The fabricated sensors were successful applied for the determination of BPA in tap water of disposable plastic cup and PVC mineral water bottle with satisfactory recovery results from 96.21% to 101.40% for CDs-CS/SPCE while 98.89% to 100.37% for AuNPs/CDs-CS/SPCE. These findings suggest that the developed sensors have a promising potential for the detection of BPA in real sample for environmental monitoring and industry quality control.

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

SENSOR ELEKTROKIMIA MENGGUNAKAN KITOSAN-BERASASKAN NANOMATERIAL PADA ELEKTROD KARBON BERCETAK SKRIN TERUBAHSUAI UNTUK PENGESANAN BISFENOL A

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ABSTRAK

Bisfenol A (BPA) adalah sebatian organik yang terdapat dalam produk plastik yang digunakan setiap hari. Ia kebanyakannya dipindahkan ke dalam makanan dan minuman termasuk botol air, lapisan tin makanan, dan tampalan gigi. Adalah diketahui bahawa BPA dianggap sebagai sebatian gangguan endokrin yang berkaitan dengan pelbagai jenis masalah kesihatan seperti perkembangan kanser dan masalah pembiakan. Oleh itu, pembangunan pendekatan analitikal yang boleh dipercayai untuk pengesanan BPA adalah isu yang mendesak. Dalam kajian ini, penderia elektrokimia berdasarkan pengubahsuaian elektrod karbon cetakan skrin (SPCE) dengan dua penderia yang telah difabrikasi iaitu titik karbon-kitosan (CDs-CS) dan zarah nano emas/titik karbon-kitosan (AuNPs/CDs-CS) telah diterokai untuk pengesanan BPA. Kedua-dua penderia telah disediakan menggunakan teknik penuangan titis. Pengesanan BPA pada penderia ini dikaji menggunakan voltammetri nadi pembezaan (DPV). Pencirian dilakukan oleh Mikroskopi elektron pengimbasan pelepasan medan (FESEM) dan Spektroskopi X-Ray Penyebaran Tenaga (EDX), Mikroskop Elektron Transmisi Resolusi Tinggi (HRTEM) dan Spektroskopi Inframerah Transformasi Fourier (FTIR). Spektrum FTIR untuk CDs-CS menunjukkan puncak pada 1648 cm^{-1}, 1411 cm^{-1} and 1038 cm^{-1} untuk getaran kumpulan hidroksil C=O pada CDs. Puncak baharu pada 1100 cm^{-1}berkaitan dengan regangan C-O AuNPs/CDs-CS yang kuat. Parameter optimum untuk pembangunan penderia seperti kepekatan CS 1% (m/v), nisbah CDs : CS 1/1 (v/v), dan 7 µL isipadu CDs-CS yang dituang titis pada SPCE untuk penderia pertama, nisbah isipadu bagi komposit CDs-CS dan AuNPs 1 : 1.5 (v/v) dan 3 µL AuNPs/CDs-CS yang dituang titis pada SPCE untuk penderia kedua telah dinilai untuk meningkatkan prestasi penderia yang dibangunkan. Keputusan menunjukkan bahawa SPCE terubah suai dengan komposit CDs-CS dan AuNPs/CDs-CS mempunyai ciri elektrokatalitik yang hebat untuk mengesan BPA dalam julat kepekatan 0.4 µM hingga 10 µM dengan sensitiviti 0.46 µA/µM (R^2 = 0.9911) dan had pengesanan

(LOD) sebanyak 0.37 µM untuk penderia CDs-CS/SPCE manakala julat kepekatan 0.4 µM hingga 10 µM BPA dengan kepekaan 0.28 µA/µM (R^2 = 0.9937) dan LOD 0.1 µM/CDs untuk AuNPs /CDs-CS/SPCE. Kebolehasilan yang baik bagi penderia ini dengan sisihan piawai relatif (RSD) 3.66 % (n = 15) untuk CDs-CS/SPCE manakala 1.35 % (n = 15) untuk AuNPs/CDs-CS/SPCE, masing-masing. Untuk kajian penyimpanan, kedua-dua penderia mengekalkan prestasinya pada 79.1% dan 90.3% untuk CDs-CS/SPCE dan AuNPs/CDs-CS/SPCE daripada respons asalnya selepas sebulan penyimpanan. Kajian gangguan penderia ini menunjukkan bahawa tiada isyarat ketara terhadap fenol, 2,4-dinitrofenol, 4-klorofenol, K^+,Cu^{2+}, Cl^-, and SO_4^{2-} walaupun dengan kepekatan 10 kali ganda lebih tinggi berbanding BPA. Penderia yang direka telah berjaya digunakan untuk penentuan BPA untuk air paip dalam cawan plastik pakai buang dan botol air mineral PVC dengan hasil pemulihan yang memuaskan daripada 96.21% kepada 101.40% untuk CDs-CS/SPCE manakala 98.89% kepada 100.37% untuk AuNPs/CDs-CS/SPCE. Penemuan ini menunjukkan bahawa sensor yang dibangunkan mempunyai potensi yang membberangsangkan untuk pengesanan BPA dalam sampel sebenar untuk pemantauan alam sekitar dan kawalan kualiti industri.

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LIST OF ABBREVIATIONS

BPA	Bisphenol A
PC	Polycarbonate
EFSA	European Food Safety Authority
t-TDI	Temporary Tolerable Daily Intake
HPLC	High-Performance Liquid Chromatography
LC-MS	Liquid Chromatography– Mass Spectrometry
GC-MS	Gas Chromatography Coupled- Mass Spectrometry
CDs	Carbon Dots
CS	Chitosan
AuNPs	Gold Nanoparticles
SPCE	Screen Printed Carbon Electrode
LSPR	Local Surface Plasmon Resonance
DPV	Differential Pulse Voltammetry
CV	Cyclic Voltammetry
EIS	Electrochemical Impedance Spectroscopy
FESEM	Field Emission Scanning Electron Microscopy
EDX	Energy Dispersive X-Ray Spectroscopy
HRTEM	High-Resolution Transmission Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
LOD	Limit Of Detection
RE	Reference Electrode
CE	Counter Electrode
WE	Working Electrode

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LDR	Linear Dynamic Range
HCl-KCl	Hydrochloric Acid-Potassium Chloride Buffer Solution
PBS	Phosphate Buffer Solution
CBBS	Carbonate-Bicarbonate Buffer Solution
SD	Standard Deviation
RSD	Relative Standard Deviation

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CHAPTER 1

INTRODUCTION

1.1 Background Study

Bisphenol A (BPA 2,2-bis(4-hydroxyphenyl)propane) is a compound that usually used as a monomer in the synthesis of epoxy resins and polycarbonate (PC). In combination with other materials used for production of plastic. Packaging for food and beverage, also for engineering plastics, drinking water and wastewater samples is one of the products which contain BPA (Santana et al., 2017). In 2015, the European Food Safety Authority (EFSA) has proposed the minimum amount of temporary tolerable daily intake (t-TDI) of BPA should not exceed $4\ \mu g\ kg^{-1}$ of bodyweight day^{-1} (Shi et al., 2018). It may harm human and worst lead to cancer of prostate, testicular or breast besides reproductive disorders including lower down sperm quality, defects of birth and impaired humans brain development even with a very small amount (Watabe et al., 2004). Thus, a technique for detection of BPA with rapid, simple, sensitive and selective is immediately needed.

There are many research focusing on determining of BPA including conventional technique such as high-performance liquid chromatography (HPLC), liquid chromatography–tandem mass spectrometry(LC–MS-MS) (Halle et al., 2015) and gas chromatography coupled with mass spectrometry (GC–MS) (Cunha et al., 2015). Although these techniques offer the remarkable sensitivity and low detection limit, but they involve several operational steps and tedious procedure, which hindered for rapid processing of samples. On top of that, these instrumentations are expensive, complicated operational and are hardly employed for on-site measurement (Yin, Zhou, et al., 2010). Hence, there is a demand for determination of BPA by using simple and rapid analytical technique.

Latest technology has introduced commercially available screen printed carbon electrode (SPCE) which offer inherent advantages including low cost, simple fabrication, miniaturization, disposability, and portability (Fartas et al., 2017). Besides, screen-printed technology for production of electrochemical sensor have been widely used in various fields, including environmental, pharmaceutical, clinical, and food quality control(Fartas et al., 2017). Moreover, the best part of this technology is their potential to be use for on-site measurement.

BPA (Figure 1.1) is an electroactive species because it is contains phenolic hydroxyl group that could be oxidized at the electrode surface. The direct uses of bare electrode for the detection of BPA are rare because the response of BPA

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is weak without any helps of other materials and the oxidation of BPA always requires a relatively high over potential (Farajzadeh & Mogaddam, 2012). Thus, to enhance the sensitivity and selectivity of the electrode for BPA determination, many types of materials have been suggested for electrode modification such as ionic liquids (Nikahd & Khalilzadeh, 2016), enzymes, molecularly imprinted polymers (Wang et al., 2016), ferroferric oxide nanoparticles (Fe_3O_4NPs) (Zhang et al., 2013), carbon materials, such as carbon nanotubes (Goulart et al., 2016), multiple-walled carbon nanotube (MWCNT) (Li et al., 2012), single-walled (SWCNT) (Moraes et al., 2013), graphene (Fan et al., 2012) and metal nanoparticle-based composites, such as gold nanoparticles (AuNPs) (Niu et al., 2013).

Over the past few years, carbon nanomaterials have attracted attention of many researchers due to its great properties including low toxicity (Zou et al., 2015), remarkable conductivity (Zuo et al., 2016), large surface area and high stability (Baruah et al., 2015). So far, CDs have been well-known in wide-spread of applications, inclusive of biosensing, bioimaging and also electrochemical. Gold nanoparticles (AuNPs) have attracted special attention recently due to its local surface plasmon resonance (LSPR) which provide a special physical and chemical properties that lead application in diverse fields, such as catalysis and sensing. Since it provides massive specific surface area, their conductivity became one of the most exceptional properties which can be used for enhancing electric signal (Liu & Wulff, 2004). These combination of multiple nanomaterials offer great performance towards detection of BPA compared to single nanomaterial use for modification of electrode.

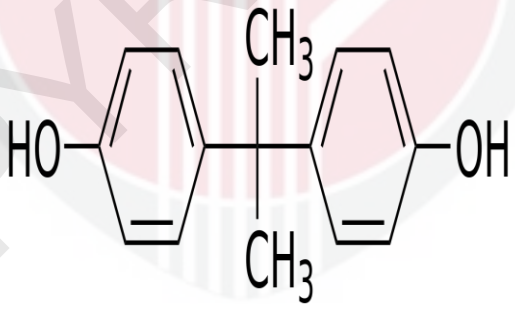

Figure 1.1: Bisphenol A (BPA) structure

1.2 Problem Statement

As people are aware that even a small amount of BPA may harm to human, in fact its amount cannot be more than $4\ \mu g\ kg^{-1}$ of bodyweight day^{-1} which have been proposed the European Food Safety Authority (EFSA) in 2015 (Shi et al., 2018). Researchers are desperate to conduct research for determining BPA to that concentration extend. Although there are many conventional techniques

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have been developed for that purpose such as high-performance liquid chromatography (HPLC) liquid chromatography–tandem mass spectrometry (LC–MS-MS) (Halle et al., 2015) and gas chromatography coupled with mass spectrometry (GC–MS) (Cunha et al., 2015), but there were limitations involve by these techniques.

These techniques involve several operational steps and tedious procedure, also prohibited for rapid processing of samples, expensive, complicated operational and are hardly employed for on-site measurement (Yin, Zhou, et al., 2010) even though these techniques offer the remarkable sensitivity and low detection limit. Hence, there is a demand for determination of BPA by using simple and rapid analytical technique.

In this research, carbon dots-chitosan (CDs-CS) composite and gold nanoparticle/carbon dots-chitosan (AuNPs/CDs-CS) deposited on screen printed carbon electrode (SPCE) for prominent catalytic performance and conductivity of BPA determination has been exploited. Additionally, it is expected that the designed fabricated sensors display excellent analytical performance for the detection of BPA with high sensitivity, wide linear range and lower detection limit.

1.3 Objective of the study

The purpose of this study is to develop a simple and sensitive electrochemical sensor for the detection of BPA in aqueous sample. This can be performed by immobilizing nanomaterials (CDs and AuNPs) on SPCE for the development of 1) CDs-CS sensor and 2) AuNPs/CDs-CS sensor for the detection of BPA. These following objectives comes out to achieve this research study goal:

i. To synthesize and characterize nanomaterial of carbon dots and gold nanoparticles.
ii. To immobilize carbon dots-chitosan (CDs-CS) and Au nanoparticles/ carbon dots-chitosan (AuNPs/CDs-CS) onto SPCE for the detection of BPA.
iii. To evaluate the performance of the developed sensors by using differential pulse voltammetry (DPV) technique for the detection of BPA.

1.4 Scope of study

The scope of this study is divided into two type of sensors which are carbon dots-chitosan (CDs-CS) and Au nanoparticles/carbon dots-chitosan (AuNPs/CDs-CS) composite deposited on screen printed carbon electrode (SPCE). They have been uses for the determination of BPA as they provide excellent catalytic

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performance and conductivity. These composites offer several advantages such as remarkable conductivity, large surface area, good film forming capability and high stability. These sensor-based electrode provide great electrocatalytic activity toward oxidation of BPA, which representing a new electrochemical sensor development platform.

However, there is a limitation in these sensors study. The most noticeable one is the performance of the electrode. The fluctuation signal reading leads to unstable outcomes and extra work must be done such as pre-treatment of the electrode to ensure the good result and hence reduce the error. Furthermore, most of the solution and chemical must be freshly prepare which longer time required before proceeding to the analysis of the sensors.

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