UNIVERSITI PUTRA MALAYSIA

ALUMINIUM-BASED METAL-ORGANIC FRAMEWORK-FABRICATED ELECTROCHEMICAL SENSOR FOR ULTRASENSITIVE HYDROQUINONE HAZARD DETECTION IN WATER SAMPLES

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By

SIM SIEW MING

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

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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

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October 2020

Chair : Janet Lim Hong Ngee, PhD
Institute : Advanced Technology

A sensitive electrochemical sensor based on aluminium-based metal-organic framework (CAU-1) deposited glassy carbon electrode (GCE) was utilized to detect hydroquinone (HQ), an environmental pollutant in the water sample. The synthesized CAU-1 was characterized by Fourier transform infrared (FT-IR) spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDS). The FT-IR results revealed the C=O vibrations presented in the as-synthesized CAU-1 sample which proved the incorporation of the amino-terephthalic acid in CAU-1. Besides, since the XRD pattern obtained for the as-synthesized CAU-1 sample was in good agreement with the simulated CAU-1 (CCDC: 723320), this strongly proved the successful synthesized of the CAU-1. Furthermore, CAU-1 showed rod-shaped morphology with an average length of 280 $\pm$ 33 nm through SEM analysis. Besides, CAU-1 had a Langmuir surface area of 1349 m^2g^{-1} and a micropore volume of 0.41 cm^3 g^{-1}. The CAU-1 was successfully fabricated on GCE using nafion as a immobilization matrix. The electrochemical behavior of the CAU-1/GCE was studied by cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) analysis. The EIS result proved that the modified CAU-1/GCE had lower resistance, which is 683 Ω compared to bare GCE. This indicated that CAU-1 with high porosity and high surface area had contributed to the fast electron transfer between electrode-electrolyte interface. The electrochemical behavior of HQ was investigated at CAU-1/GCE surface and studied the kinetics of HQ oxidation. The CAU-1 fabricated electrode highly catalyzed the oxidation of HQ than the bare GCE. The CAU-1/GCE was proven to have excellent repeatability with relative standard deviation (RSD) of 0.69% for the same sensor that runs 20 cycles consecutively and RSD of 3.96% for four sensors reproduced by using the same procedure. Besides, this sensor also shows good selectivity and anti-interference properties in HQ detection with the detection limit of 0.015 μM with an excellent sensitivity of 1555.7

μA/mM/cm^2. Furthermore, the recovery of HQ was 99.34 % to 103.93 % which proved the practical applicability and reliability of the modified CAU-1/GCE in the real-life application.

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

FABRIKASI SENSOR ELEKTROKIMIA KERANGKA ORGANIK-LOGAM BERASASKAN ALUMINIUM UNTUK PENGESANAN ULTRASENSITIF HIDROKUINON DALAM SAMPEL AIR

Oleh

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Sensor elektrokimia sensitif berdasarkan kerangka organik logam berasaskan aluminium (CAU-1) elektrod karbon berkaca (GCE) digunakan untuk mengesan hidrokuinon (HQ), komponen pencemaran alam sekitar dalam sampel air. CAU-1 yang disintesis dicirikan dengan spektroskopi inframerah transformasi Fourier (FT-IR), difraksi sinar-X (XRD), mikroskop elektron imbasan (SEM) dan analysis sinar-X penyebaran tenaga (EDS). Hasil FT-IR menunjukkan getaran C = O dalam CAU-1 yang disintesis. Ini membuktikan penggabungan asid amino-terephthalic dalam CAU-1. Selain itu, disebabkan spektrum XRD yang diperolehi daripada sampel CAU-1 sama dengan spekrtum CAU-1 yang disimulasikan (CCDC: 723320), ini membuktikan CAU-1 telah berjaya disintesis. Selanjutnya, CAU-1 menunjukkan morfologi berbentuk batang dengan ukuran purata 280 nm $\pm$ 33 nm melalui analisis SEM. Selain itu, CAU-1 memiliki luas permukaan Langmuir 1349 m^2g^{-1} dan isipadu mikroliang 0.41 cm^3 g^{-1}. CAU-1 berjaya difabrikasi pada GCE dengan menggunakan nafion sebagai matrik pemegun. Tingkah laku elektrokimia CAU-1/GCE dikaji dengan analisis melalui kitaran voltammetri (CV) dan spektroskopi impedans elektrokimia (EIS). Hasil EIS membuktikan bahawa CAU-1/GCE yang diubahsuai mempunyai rintangan yang lebih rendah, iaitu 683 Ω berbanding dengan GCE yang belum diubahsuai. Ini menunjukkan bahawa CAU-1 dengan luas permukaan yang tinggi telah menyumbang kepada pemindahan elektron yang cepat berlaku antara permukaan elektrod-elektrolit. Tingkah laku elektrokimia HQ disiasat di permukaan CAU-1/GCE dan kinetik pengoksidaan HQ juga dikaji. Elektrod fabrikasi CAU-1 memangkinkan pengoksidaan HQ daripada GCE yang tidak diubahsuai. CAU-1/GCE terbukti mempunyai kebolehulangan yang sangat baik dengan sisihan piawai relatif (RSD) 0.69% untuk sensor yang sama yang menjalankan 20 kitaran berturut-turut dan RSD 3.96% untuk empat sensor yang dihasilkan semula dengan menggunakan prosedur yang sama. Selain itu, sensor ini juga menunjukkan sifat selektif dan anti-gangguan yang baik dalam pengesanan HQ dengan had

pengesanan 0.015 µM dan sensitiviti yang sangat baik, iaitu 1555.7 µA/mM/cm^2. Selanjutnya, perolehan semula HQ adalah 99.34% hingga 103.93% yang membuktikan kebolehlaksanaan praktikal dan kebolehpercayaan CAU-1/GCE dalam aplikasi kehidupan sebenar.

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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

3DFG	Three-Dimensional Functionalized Graphene
P1,5-DAN	Poly(1,5-Diaminonaphthalene)
AA	Ascorbic Acid
AB	Acetylene Black
BET	Brunauer Emmett And Teller
BMITFB	1-Butyl-3-Methylimidazolium Tetrafluoroborate
BQ	Benzoquinone
CAU	Christian-Albrechts University
CC	Catechol
CE	Capillary Electrophoresis
Chi/CS	Chitosan
CNTs	Carbon Nanotubes
CP	Conducting Polymer
CPE	Carbon Paste Electrode
CV	Cyclic Voltammetry
DHB	Dihydroxybenzene
DP	Dopamine
DPV	Differential Pulse Voltammetry
EDS	Energy Dispersive X-Ray Spectroscopy
EIS	Electrochemical Impedance Spectroscopy
EPA	Environmental Protection Agency
EU	European Union
FT-IR	Fourier Transform Infrared Spectroscopy

GCE	Glassy Carbon Electrode
GC-MS	Gas Chromatography-Mass Spectrometry
GR	Graphene
HPLC	High Performance Liquid Chromatography
HQ	Hydroquinone
Hz	Hertz
IBU	Inorganic Building Unit
IL	Ionic Liquid
M	Molarity
MCPE	Modified Carbon Paste Electrode
MIL	Matériaux De l’Institut Lavoisier
MOF	Metal-Organic Framework
MWCNTs	Multi-Walled Carbon Nanotubes
ND	Nanodiamond
Nf	Nafion
OMIM-PF_6 IL	1-Octyl-3-Methylimidazolium Hexafluorophosphate Ionic Liquid
PBS	Phosphate Buffer Solution
PCH	2-(Phenylazo) Chromotropic Acid
PTH	Poly(Thionine)
RS	Resorcinol
RSD	Relative Standard Deviation
RTIL	Room Temperature Ionic Liquid
SEM	Scanning Electron Microscopy
SWCNTs	Single-Walled Carbon Nanotubes

SWV	Square-wave Voltammetry
TMO	Ternary Metal Oxide
UA	Uric Acid
UV-Vis	Ultraviolet-Visible Spectrophotometry
XRD	X-Ray Diffraction
ZIF-8	Zeolitic Imidazolate Framework-8

LIST OF SYMBOLS

°	Degree
Å	Angstrom
˚C	Degree Celcius
ΔE_p	Separations of Peak Potential
E_{pa}	Anodic Peak Potential
E_{pc}	Cathodic Peak Potential
I_p	Peak Current
R^2	Correlation Coefficient
R_{et}	Electron Transfer Resistance
$v^{\frac{1}{2}}$	Square Root of Scan Rate
Ω	Ohm
λ	Wavelength

CHAPTER 1

INTRODUCTION

1.1 Background

Hydroquinone (HQ) is a well-known phenolic environmental pollutant produced by the industries that involved photographic processes and from coal gasification condensate water (Hu et al., 2012; Xie et al., 2006). Some of the company will discharge the waste containing HQ into the river or air without monitoring the concentration of HQ that contained in the waste discharged. This action will seriously pollute the environment due to the high toxicity of hydroquinone. According to Environmental Quality (Industrial Effluent) Regulations 2009, the discharge of industrial effluent should not greater than 0.001 ppm into any inland waters within the catchment area as specified in Sixth Schedule and less than 1.0 ppm into any other Malaysian waters (Malaysia, 2009). The unauthorized person who discharged HQ-containing waste might bring huge impact to our environment and living things.

In addition, hydroquinone is non-biodegradable and it will affect our ecosystem where it can harm aquatic life and directly bring bad health effects to human beings (Hu et al., 2012; Zhang et al., 2019). A person in contact with even low concentration of hydroquinone may suffer from skin irritation or inflammation because hydroquinone can be absorbed through skin easily (Ahmed et al., 2018; Zhang et al., 2019). According to the Ministry of Health, the drinking water quality standard is less than 0.002 mg/l for phenolic compound (Ministry of Health Malaysia, 2006). A person may suffer from sore throat or vomiting if ingested hydroquinone. Moreover, in long term effect, hydroquinone might cause central nervous system, respiratory tract, kidney and blood disorder (Young, 2008). In worst case humans may suffer from cancer or even fatality (Ahmed et al., 2018). Hence, it is highly essential to determine the level of HQ in environmental water samples.

There were various analytical methods such as HPLC, UV-VIS, GC-MS and CE being introduced to test the amount of HQ that presence in specific sample. These analytical methods can be used to detect HQ at trace levels. However, the expensive instrument that required a qualified operator to operate had limit the application of these analytical methods for daily HQ detection. Besides, detecting HQ by using these instruments usually takes a long time. In some cases, the sample pre-treatment was needed before HQ detection (Dursunoğlu et al., 2018). Hence, a simple but sensitive HQ sensor is preferable.

Up to date, graphene, carbon nanotubes, nanoparticles such as nanodiamond (ND) and nanocomposites had been widely introduced for the modification on the bare GCE. This modified GCE was proven to enhance the current response of the GCE towards HQ and at the same time provides an effective but simple way for the HQ determination. Each modified electrode possesses its own sensitivity, stability, repeatability, reproducibility and selectivity towards the HQ determination. Hence, development in this modification of GCE is still on-going in fabricating a simple, sensitive and selective electrochemical sensor in detecting HQ.

1.2 Problem Statement

Due to the rapid development in industries in various fields such as cosmetics, medicines, paints and dyes, pesticides and so on, the environmental problems that arose from the illegal discharge of waste into the drain or river had gain attention from many peoples. Especially, the waste that contains hydroquinone even in extremely low concentrations also can lead to a huge impact on our ecological system. The aquatic life will be first affected by the HQ discharge and then it will directly affect the animals or humans that consume the fish and water.

Consequently, many analytical methods such as HPLC, UV-VIS, GC-MS and CE were introduced to detect the concentrations of HQ presented in the samples. However, these methods have drawbacks where they required expensive instruments and an expert hand to operate the instruments. In addition, in some cases, pre-treatment of the samples is required for the detection of HQ in the samples and sometimes is time-consuming to even test a sample. Also, some instruments required solvent such as methanol that is not environmentally friendly too. All these limitations had caused the determination of HQ in samples cannot be run easily and routinely.

Hence, the electrochemical method had been introduced to overcome the limitations of conventional analytical methods. Electrochemical sensors are well-known for their simple and facile analytical apparatus that can show fast response towards the detection of various chemical components. Besides, their amendable properties had attracted many researchers to modify the electrodes to enhance the electrode sensitivity for ultra-sensitive detection of trace amounts of chemical compounds (Yao et al., 2016; Zhou et al., 2015). Various modifiers such as graphene, nanoparticles, nanocomposites, CNTs was introduced as a modifier for the bare GCE or CPE in order to enhance the sensitive detection of HQ. However, researchers are still discovering the electrochemical method that is simple yet cost-effective and time-effective for the sensitive determination of HQ.

In conjunction with the development of electrochemical sensors, metal-organic frameworks (MOFs) also gained attention from many researchers due to the porous structure, high surface area and tunable properties. MOFs have a porous structure that is suitable as a modifier on the electrode, where the large effective surface area could enhance the adsorption or diffusion of the ions and hence promote the electrochemical reaction to occur.

At the best of the author's knowledge, the application of aluminium-based MOF: CAU-1 as the modifier at the GCE surface has not been reported yet. CAU-1/GCE with the high effective surface area was expected to show improved current response towards HQ than the bare GCE. Besides, the CAU-1/GCE was expected to improve the electron transfer at the GCE surface during the electrochemical reaction. Furthermore, the modified CAU-1/GCE is expected to have good sensitivity, repeatability, reproducibility, anti-interference properties and reliability to detect the HQ in real-life water samples.

1.3 Thesis Objectives

This research is to fabricate a modified CAU-1/GCE sensor for the sensitive detection of hydroquinone. The key objectives of the study are outlined below:

i. To synthesis CAU-1 as an electrode material for hydroquinone detection.
ii. To study the electrochemical behaviors of hydroquinone at the modified CAU-1/GCE.
iii. To evaluate the practical application of the CAU-1 modified sensor on hydroquinone detection in the real application.

1.4 Scope and Relevance

Chapter 1 shows the overview of this thesis where it includes the background of the study, problem statement, objectives of this research as well as the scope of the research. **Chapter 2** of this thesis included the introduction of hydroquinone and its effect on livings things and the environment. Besides, various analytical methods of hydroquinone detection with their pro and cons are presented here. Different methods of modification of carbon paste electrode and glassy carbon electrode on the hydroquinone detection are also discussed. In addition, the properties of CAU-1 are discussed here. **Chapter 3** shows the methodology for the synthesis of CAU-1, the fabrication of modified CAU-1/GCE and the instruments used for the CAU-1 characterizations. Besides, the electrochemical measurements of the modified CAU-1/GCE on the detection of hydroquinone are discussed.

In **Chapter 4**, FT-IR, XRD, SEM, EDS, and BET were used to characterize the as-synthesized CAU-1 in order to identify the functional group, crystalline quality, surface morphology and the surface area of CAU-1. Besides, electrochemical impedance spectroscopy (EIS) was conducted to qualitatively verify the kinetic control process of the CAU-1/GCE in the presence of HQ.

Moreover, the electrochemical behavior of HQ at the modified CAU-1/GCE was identified through cyclic voltammetry (CV), differential pulse voltammetry (DPV) and amperometry. The optimized conditions for HQ detection were also been identified. Furthermore, the repeatability, reproducibility, sensitivity and the recovery study of the modified CAU-1/GCE towards HQ were also been investigated. **Chapter 5** concludes all the results obtained throughout the entire research. Furthermore, the future work to be done is presented at the end of this chapter.

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