TAILORING MANGANESE DIOXIDE ELECTROCATALYST BY PLATINUM AND CARBON NANOTUBE FOR AIR-CATHODE MICROBIAL FUEL CELL

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Master of Science

UNIVERSITI MALAYSIA PAHANG

SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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I hereby declare that the work in this thesis is based on my original work except for quotations and citation which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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LIST OF SYMBOLS

E^0	Standard cell potential (V)
R	Ideal gas constant (J/(mol K))
T	Temperature (K)
n_e	Number of electrons transferred in the reaction
F	Faraday's constant (C/mol e^{-1})
π	Chemical activity of products divided by those of reactants
P	Power (W)
V	Potential (V)
I	Current (A)
i	measured current (A)
i_k	kinetic current (A)
i_d	diffusion-limiting current (A)
P_v	Volumetric power density (W/m^3)
P_A	Area power density (W/m^2)
I_A	Current density (A)
Vol.	Total volume of anolyte (mL)
S.Area	Total surface area of the air-cathode (m^2)
COD_i	Initial COD (mg/L)
COD_f	COD of the effluent in the anode chamber at any time (mg/L)
t	Grain size (nm)
β	Angular width (nm)
λ	Wavelength of the radiation used (nm)
C	Capacitance (F)
R	Parallel resistance (Ω)
n	Empirical constant
Q	Pseudo-capacitance (F)
W_o	Process of diffusion of ions from the electrolyte to the interface
W_R	Ohmic resistance (Ω)
W_T	Capacitive part (F)
W_P	Exponent
R_{ct}	Charge transfer resistance (Ω)
R_{total}	Total resistance (Ω)
C_{SC}	Space-charge capacities (F)
ε_o	Permittivity of free space
e	Charge of electron
ε	Dielectric constant
E_{fb}	Flat-band potential (V)
E	Applied potential (V)
k	Boltzmann constant ($J.K^{-1}$)
N_D	Donor density
M_O	Molecular weight of oxygen (kg/mol)

Δ_{COD}	Change of COD concentration (mg/L)
n	Overall number of electron transferred

LIST OF ABBREVIATION

AEM	Anion exchange membrane
BET	Brunauer-Emmett-Teller
BOD	Biological oxygen demand
CE	Coulombic efficiency
CEM	Cation exchange membrane
CNT	Carbon nanotube
COD	Chemical oxygen demand
CPE	Constant phase element
CV	Cyclic Voltammetry
CV-TF-RRDE	Cyclic Voltammetry using a thin-film rotating ring-disk electrode
DI	Deionized
EDX	Energy-Dispersive X-ray Spectroscopy
EIS	Electrochemical Impedance Spectroscopy
FESEM	Field Emission Scanning Electron Microscopy
FT-IR	Fourier Transform Infrared
GC	Glassy electrode
LSV	Linear Sweep Voltammetry
MEA	Membrane-electrode-assembly
MFC	Microbial Fuel Cell
NN	Nitrate nitrogen
OCP	Open circuit potential
ORR	Oxygen reduction reaction
PACF	Polyacrylonitrile carbon felt
PEM	Proton exchange membrane
POME	Palm oil mill effluent
Pt-sol	Platinum solution
RDE	Rotating disk electrode
TDS	Total dissolved solids
TS	Total solids
TSS	Total suspended solids
XPS	X-ray Photoelectron Spectroscopy
XRD	X-ray Diffraction analysis

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ABSTRAK

Sel-sel bahan api mikrob katod-udara (MFCs) merupakan teknologi hijau yang berpotensi untuk menjana bio-elektrik dan merawat air sisa secara serentak. Namun, reaksi pengurangan oksigen (ORR) merupakan salah satu faktor yang mempengaruhi penjanaan tenaga bagi sel tersebut. Oleh itu, pemangkin adalah diperlukan bagi mempercepatkan kadar ORR dan meningkatkan prestasi MFC katod-udara. Platinum (Pt) adalah pemangkin konvensional yang telah digunakan untuk pelbagai applikasi dan mempunyai activiti pemangkinan yang amat baik dengan stabilan yang tinggi. Namun begitu, kos Pt yang amat tinggi merupakan satu halangan bagi penghasilan dan aplikasi Pt sebagai pemangkin dalam MFCs. Oleh itu, pemangkin ORR alternatif telah dihasilkan bagi menggantikan Pt sebagai pemangkin. Sejak kebelakangan ini, mangan dioksida (MnO_2) telah dikaji secara meluas dan dilaporkan bahawa ia mempunyai potensi yang tinggi sebagai pemangkin ORR kerana ia mempunyai sifat-sifat yang unik, kos rendah, mudah penghasilan, pemangkinan ORR aktiviti yang baik serta kurang memudaratkan alam sekitar. Namun, pemangkinan ORR aktiviti bagi MnO_2 masih rendah berbanding dengan pemangkin Pt kerana MnO_2 mempunyai kekonduksian elektrik yang rendah. Oleh itu, modifikasi pada MnO_2 diperlukan bagi meningkatkan aktiviti ORR supaya boleh menggantikan applikasi pemangkin Pt dalam MFC katod-udara. Dalam karya ini, MnO_2 telah dihasilkan melalui kaedah hidroterma dan diubahsuai dengan gabungan Pt dalam amaun yang rendah, carbon nanotube (CNT) dan kedua-dua Pt dan CNT untuk menghasilkan Pt/MnO_2, MnO_2/CNT and Pt-MnO_2/CNT. Maklamat karya ini adalah menghasilkan pemangkin ORR yang effektif untuk menaikkan prestasi MFC untuk penjanaan tenaga serta merawat sisa buangan kilang minyak sawit (POME). Pemangkin tersebut dicirikan secara komprehensif dengan menggunakan kaedah Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-ray analisis (EDX), Fourier Transform Infrared Spectroscopy (FT-IR), X-ray Diffraction analisis (XRD), X-ray Photoelectron Spectroscopy (XPS), Brunauer-Emmett-Teller analisis (BET), Cyclic Voltammetry (CV), Linear Sweep Voltammetry (LSV), Electrochemical Impedance Spectroscopy (EIS) and Mott-Schottky analisis untuk mengkaji morfologi permukaan, penghabluran, pengoksidaan dan aktiviti elektrokimia pemangkin-pemangkin tersebut. Untuk mengkaji keberkesanan ORR aktiviti pemangkin, pemangkin tersebut digunakan dalam MFC katod-udara dimana POME dan enapcemar anaerobik digunakan sebagai substrat anod dan inokulum. Prestasi MFC yang mengunakan pemangkin-pemangkin tersebut ditentukan melalui ujian polarisasi. Kestabilan, kecekapan coulombic (CE) dan keberkesanan penyingkiran COD menggunakan MFC dengan pemangkin-pemangkin tersebut juga ditentukan. Hasilnya, Pt-MnO_2/CNT merupakan antara pemangkin ORR yang terbaik dimana ia mempunyai ORR aktiviti yang tinggi serta jumlah caj dan caj pemindahan yang rendah dengan penghasilan maksima ketumpatan kuasa, volt litar terbuka dan penyingkiran COD yang paling tinggi and berkesan iaitu 100.63 mW/m^2, 629.30 mV, 34.17% dan 75.55%. Mengikut kajian di atas, didapati bahawa kehadiran CNT telah meningkatkan luas permukaan BET dan konduktiviti pemangkin elektro. Pada masa yang sama, kehadiran PT meningkatkan aktiviti pemangkin ORR, konduktiviti dan kestabilan pemangkin elektro. Melalui kombinasi kedua-dua Pt dan CNT dalam pemangkin elektro MnO_2, aktiviti pemangkin ORR yang tinggi berserta dengan konduktiviti tinggi, kestabilan dan luas permukaan BET pemangkin elektro (Pt-MnO_2/CNT) terbentuk dan ini menunjukkan prestasi MFC meningkat, kestabilan operasi dan keberkesanan penyingkiran COD.

ABSTRACT

Air cathode microbial fuel cell (MFC) is a high potential green technology which could simultaneously generate bio-electricity and conducting wastewater treatment. However, the slow oxygen reduction reaction (ORR) is one of the limiting factors that bounds the power generation of the cell. Hence, ORR catalytic electrocatalysts are required to enhance the performance of the air-cathode MFC. Platinum (Pt) is the conventional electrocatalysts which have been used for various applications as it has the preeminent ORR catalytic activity with high stability. However, the precious metal electrocatalyst creates a big obstacle in the development and application of Pt electrocatalysts in MFCs. Therefore, alternative ORR electrocatalysts were developed to replace the Pt electrocatalyst. In recent years, manganese dioxide (MnO_2) has been studied extensively and found that it has a great potential as an effective ORR electrocatalyst due to its unique properties, low cost, easy preparation and possesses ORR catalytic activity. However, the ORR activity of MnO_2 is still low compared to that Pt electrocatalyst due to the intrinsic low electrical conductivity of MnO_2. Therefore, modifications are needed to enhance the ORR activity of MnO_2 to substitute the Pt electrocatalyst in air-cathode MFC application. In present work, MnO_2 was developed via hydrothermal method and modified by incorporating trace amount of Pt nanoparticles, carbon nanotube (CNT) and both Pt and CNT to develop Pt/MnO_2, MnO_2/CNT and Pt-MnO_2/CNT, respectively. The goal of this work is to develop an effective ORR electrocatalyst for improving the performance of the MFC for power generation and simultaneously treating palm oil mill effluent (POME). The as-prepared electrocatalysts were characterized comprehensively through Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-ray analysis (EDX), Fourier Transform Infrared Spectroscopy (FT-IR), X-ray Diffraction analysis (XRD), X-ray Photoelectron Spectroscopy (XPS), Brunauer-Emmett-Teller analysis (BET), Cyclic Voltammetry (CV), Linear Sweep Voltammetry (LSV), Electrochemical Impedance Spectroscopy (EIS) and Mott-Schottky analysis where the surface morphology, crystallinity, oxidation state and electrochemical activity of the as-prepared electrocatalysts were examined, respectively. The effectiveness of the electrocatalysts were tested in the air-cathode MFC with POME and anaerobic sludge as the anolyte and inoculum, respectively. The performance of the MFC was determined via polarization test. The stability, coulombic efficiency (CE) and chemical oxygen demand (COD) removal efficiency of the MFC with the respectively electrocatalysts were investigated. As the result, Pt-MnO_2/CNT was found to be the best ORR electrocatalysts among the modified electrocatalysts which has the highest ORR activity with lowest total and charge transfer resistances which showed high stability and the highest maximum power density, open circuit potential (OCP), CE and COD removal efficiency of 100.63 mW/m^2, 629.30 mV, 34.17% and 75.55%, respectively. From the study, it was found that the presence of the CNT increases the BET surface area and the conductivity of the electrocatalyst meanwhile the presence of Pt increases the ORR catalytic activity, conductivity and the stability of the electrocatalysts. By the combination of both Pt and CNT in the MnO_2 electrocatalyst, a high ORR catalytic activity with high conductivity, stability and BET surface area electrocatalyst (Pt-MnO_2/CNT) was developed which showed an improved MFC performance, operational stability and COD removal efficiency.

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