DEVELOPMENT OF PHOSPHORUS-DOPED NOBLE METAL (Pt, Pd) ELECTROCATALYSTS FOR SELECTIVE OXIDATION OF GLYCEROL TO VALUE ADDED CHEMICALS

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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 Doctor of Philosophy.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations 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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ABSTRAK

Gliserol adalah produk sampingan yang terhasil dari penghasilan biodiesel melalui proses transesterifikasi minyak sayuran. Boleh dikatakan 10Kg gliserol akan terhasil daripada penghasilan 100Kg biodiesel. Pelbagai kajian telah dilakukan untuk mengubah gliserol kepada pelbagai proses pemangkin termasuk pembaharuan, oksidasi, hidrogenolisis, eterifikasi, esterifikasi, dan sebagainya. Ia dapat diubah kepada pelbagai sebatian kimia seperti dihydroxyacetone (DHA), gliseraldehyde (GALD), asid mesoxalic (MOXA), asid glyseric (GLY), asid tartronic (TAT), dan asid oksalat (OXA). Sasaran utama semua teknik ini adalah untuk mencapai penukaran gliserol yang cekap dan ekonomik kepada produk yang diinginkan dengan selektiviti tertinggi. Elektro-pengoksidasi gliserol (GOR) merupakan kaedah yang selamat, tidak merbahaya, dan kaedah mampan untuk sintesis produk daripada gliserol. Untuk kerja masa ini, tindak balas telah dikatalisis menggunakan nanopartikel diubahsuai logam (Pt, Pd) dan '(P-doped Pt, P-doped Pd)' yang disokong oleh 'multiwalled carbon nanotube' dalam tiga sistem sel elektrod telah dilaporkan untuk elektro-pengoksidaan gliserol. Pemangkin akan dianalisis menggunakan BET, XRD, XPS, FESEM-EDX dan TEM untuk dapatkan pandangan yang lebih baik mengenai kesan luas permukaan, saiz kristal, ukuran zarah, dan keadaan pengoksidaan logam yang didoping untuk prestasi elektrokatalis GOR. Sifat-sifat elektrokimia pemangkin diukur melalui 'cyclic voltammetry (CV)' dan 'chronoampermetry CA' menggunakan potentiostat dan HPLC digunakan untuk mnganalisa dan mengira kuantiti produk GOR. Elektrokatalis Pt / CNT dan Pd / CNT dengan size partikel 4.2 nm dan 5.13 nm masing-masing telah disediakan dengan menggunakan kaedah 'hydrazine reduction method'. Pemangkin menunjukkan ketahanan yang tinggi, aktiviti elektrokimia selektiviti produk 90-95% ke arah sintesis terpilih asid tartronik dalam media alkali. Pt-Pd/ CNT telah dihasilkan dan menunjukkan permukaan luas elektrokimia yang lebih tinggi (322.0 m2 / g), dengan selektiviti asid mesoxalic (87%). Walaupun logam terbukti sebagai pemangkin yang menjanjikan selektiviti yang tinggi tetapi disebabkan oleh kekurangan komersil disebabkan harga yang mahal, penggabungan bukan logam (P) dipilih sebagai kaedah terbaik untuk menyelesaikan masalah ini. Oleh kerana homogenitasi non-logam masih menjadi masalah, maka dalam kerja ini, 'P-doped Pt/ CNT' dan 'P-doped Pd/ CNT' dihasilkan menggunakan kaedah hidrotermal menghasilkan diameter 6.23 nm dan 3.7 nm. Menghasilkan keputusan yang bagus ECSA (335.95 m2 / g & 392.22 m2 / g) iaitu terbaik dari pemangkin logam. Tambahan, didapati selektiviti DHA dan TAT adalah 90.82% dan 47%. Telah didapati bahawa semua pemangkin Pd '(Pd/ CNT & P-doped Pd)' menunjukkan ketumpatan arus yang lebih tinggi, potensi permulaan yang lebih rendah, ECSA yang tinggi, saiz zarah yang kecil, selektiviti yang tinggi untuk produk yang dikehendaki dan rintangan yang lebih baik '(If/Ic =2.37)' berbanding dengan pemangkin Pt. Keputusan 'P-doping' Pt dan Pd mencadangkan penambahan P tidak hanya menyumbang untuk aktiviti tinggi tetapi juga pendekatan ekonomi untuk pengkomersialan elektrokatalisis GOR. Selektiviti produk juga dapat ditingkatkan untuk pemangkin yang didoping paling rendah; 'onset potential', potensi terapan bersama dengan tinggi; ECSA dan kepadatan arus.

ABSTRACT

Glycerol is a by-product of biodiesel obtained during transesterification of the vegetable oil (one kg in every 10 kg of biodiesel). Its improper handling can create environmental issues. Much work has been devoted to the transformation of glycerol by various catalytic processes involving reforming, oxidation, hydrogenolysis, etherification, esterification and so on. It can be converted into various valuable chemicals, such as dihydroxyacetone (DHA), glyceraldehyde (GALD), mesoxalic acid (MOXA), glyceric acid (GLY), tartronic acid (TAT), and oxalic acid (OXA) by using different chemo-catalytic and electrocatalytic techniques. Main target of all these techniques is to achieve efficient and economical conversion of glycerol to desired products with highest selectivity. Glycerol electro-oxidation reaction (GOR) could offer potentially safe, non-hazardous and sustainable method to produce value-added products from glycerol. In this work, the effect of noble metals (Pt & Pd) and non-metals modified nanoparticles (P-doped Pt, P-doped Pd) supported over multiwalled carbon nanotubes in three electrode cell system is reported for electro-oxidation of glycerol. As prepared catalysts were physicochemically characterized by Brunauer-Emmett-Teller (BET), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), Field emission electron spectroscopy (FESEM), energy-dispersive X-ray spectroscopy (EDX) and Transmission electron microscopy (TEM) to get better insight about the effect of specific surface area, crystallite size, particle size, and oxidation states of doped non-noble metal on performance of electrocatalysts for GOR. The electrochemical properties of the catalysts were measured through cyclic voltammetry and chronoampermetry using a potentiostat and HPLC was used to analyze and quantify the GOR products. Initial studies found that the Pt/CNT and Pd/CNT electrocatalysts with a particle size of about 4.2 nm and 5.13 nm respectively was prepared by using hydrazine reduction method. It was demonstrated a high durability, electrochemical activity can achieve product selectivity of 90-95% for selective synthesis of tartronic acid in alkaline media. Bi-metallic (Pt-Pd/CNT) electrocatalysts showed higher electrochemical surface area (322.0 m^2/g), and selectivity of mesoxalic acid (87%). Although noble metals proved to be promising catalyst with high selectivity but due to commercial constraints of costly noble metals, incorporation of non-metal (P) was chosen as best method to solve this problem. Since, homogeneity of non-metals remains a problem, so in this work, P-doped Pt/CNT and P-doped Pd/CNT were prepared using hydrothermal method with a diameter of 6.23 nm and 3.7 nm were obtained. The results showed superior ECSA (335.95 m^2/g & 392.22 m^2/g, respectively) than previously mentioned noble metal catalysts. In addition, DHA and tartronic acid selectivity of 90.82% and 47% respectively was obtained. It was found that all the Pd catalyst (Pd/CNT & P-doped Pd) showed higher current density, lower onset potential, high ECSA, narrow particle size, high selectivity for desired products and better resistance (I_f/I_b =2.37) as compared to Pt catalysts confirming the higher catalytic activity of these catalysts. The results of P-doping into Pt and Pd suggested that addition of phosphorous (P) not only contributed for high activity but also an economical approach for future commercialization of GOR electrocatalysts. It was also found that the product selectivity remarkably enhanced for doped catalysts showing lowest; onset potential, applied potential along with high; ECSA and current density.

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

θ	Diffraction angle
∑	Summation
Å	Angstrom
µ	Micro
I_f	Anodic current
I_b	Cathodic current
E_f	Anodic potential
E_b	Cathodic potential
n	No. of moles
ρ	Density
M	Molarity
m	Mass

LIST OF ABBREVIATIONS

GALD	Glyceraldehyde
MOXA	Mesoxalic acid
DHA	Dihydroxyacetone
GLY	Glyceric acid
TAT	Tartronic acid
OXA	Oxalic acid
FA	Formic acid
GLYC	Glycerol
CV	Cyclic voltammetry
CA	Chronoamperometry
LSV	Linear sweep voltammetry
BET	Brunauer, Emmet, and Teller
ECSA	Electrochemical surface area
SA	Specific activity
SSA	Specific electrochemical active surface area
MA	Mass activity
HPLC	High-performance liquid chromatography
MWCNTs	Multi walled carbon nanotubes
FE-SEM	Field electron-scanning electron microscopy
TEM	Transmission electron microscopy
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction spectroscopy
EDX	Energy-dispersive X-ray spectroscopy
ICP-OES	Inductively coupled plasma atomic emission spectroscopy
CHNOS	Carbon, hydrogen, nitrogen, oxygen and sulfur analyser

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