UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF BIFUNCTIONAL CATALYSTS SYNTHESIZED FROM PYROLYZED AND HYDROTHERMALIZED PALM WASTE FOR BIODIESEL PRODUCTION USING WASTE COOKING OIL

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By

ROSE FADZILAH ABDULLAH

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Dedication

They said, "Exalted are You, we have no knowledge except what You have taught us. Indeed, it is You who is the Knowing, the wise". Quran (Surah Al-Baqarah 2:32)

Mohd Zahid

Nur Aisyah Zulaikha

Abdullah & Zaleha

Shamsudin & Hamimah

Ramlah, Norazlinda & late Normarina

Family & friends

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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The abundance of waste biomass from palm oil industries creates a new potential for producing activated carbon as catalyst support. The hydrothermal carbonization (HTC) process provides a brilliant alternative to produce activated carbon from biomass samples with higher water content than conventional pyrolysis techniques. Therefore, this study aims to produce activated carbon from palm kernel shell (PKS), mesocarp fiber (MF) and empty fruit bunch (EFB) using pyrolysis and HTC techniques. The HTC process favours the breakage of large polymeric molecules in the lignocellulosic material into lower molecular weight organic compounds through hydrolysis, dehydration, decarboxylation, aromatization and recondensation process. Subsequently, the performance of the prepared activated carbon was determined through the application as catalyst support of a bifunctional catalyst. Therefore, the activated carbon was impregnated with K_2CO_3 and CuO via wet impregnation provided bifunctional characteristics. The state-of-the-art characterization of the synthesized bifunctional catalysts was conducted including N_2 adsorption-desorption analysis, functional group determination, surface morphology observation, crystallography study, electron dispersive X-ray mapping, elemental distribution analysis, concentration of basicity and acidity measurement and thermal degradation behaviour analysis. The results show that the HTC technique successfully produced a highly mesoporous activated carbon derived from PKS that had better textural properties than pyrolyzed with a BET surface area of 1411.16 $m^2 g^{-1}$ and 3368.60 $m^2 g^{-1}$, respectively. The produced bifunctional catalyst was applied in the simultaneous esterification and transesterification reaction from the waste cooking oil (WCO) by the conventional reflux system. The high BET surface area provides extra space for active sites impregnation which is very important for producing maximum biodiesel yield from WCO. The catalytic performance results show that the hydrothermalized-activated carbon from PKS (PKSHAC) derived catalyst produced a higher biodiesel yield of 95.3% against 95.0% over the pyrolyzed-activated carbon from PKS (PKSAC) derived catalyst. The activated carbon preparation was continued using MF and EFB, namely hydrothermalized-activated carbon from MF (MFHAC) and EFB (EFBHAC), which exhibited a BET surface area of 3909.33 $m^2 g^{-1}$ and 4056.17 $m^2 g^{-1}$,

and produced a maximum biodiesel yield of 96.4% and 97.1%, respectively. The synthesized catalysts sustained up to 5 reaction cycles with more than 80% of biodiesel yield. The carbon structure collapsed due to multiple calcination during the reactivation procedure, and the catalyst poisoning by glyceroxide on the catalyst surface was identified as a deactivation factor. On the other hand, the transformation of biodiesel from WCO was confirmed via proton nuclei magnetic resonance (^{1}H NMR), Fourier transform Infrared (FTIR) and thermal gravimetric analysis (TGA). Fuel properties revealed kinematic viscosity of 3.3 mm^2 s^{-1}, the cetane number of 51, the flashpoint of 160.5 °C, cloud and pour point of 11 °C and -3 °C, respectively. Overall, the finding shows the excellent potential of waste materials, especially PKS, MF, and EFB in producing high quality activated carbon as catalyst support via pyrolysis and HTC techniques. The functionalization with K_2CO_3 and CuO via wet impregnation provides bifunctional that opens the opportunity in utilizing WCO to produce high-quality biodiesel.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMBANGUNAN PEMANGKIN DWI-FUNGSI DARIPADA SISA KELAPA SAWIT SECARA PIROLISIS DAN HIDROTERMAL UNTUK PENGHASILAN BIODIESEL DARIPADA SISA MINYAK MASAK

Oleh

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Lambakan sisa biomass daripada industri kelapa sawit dilihat mempunyai potensi dalam penghasilan karbon teraktif sebagai bahan asas pemangkin. Proses karbonisasi hidrotermal (HTC) adalah alternatif yang terbaik dalam penghasilan karbon teraktif daripada sampel biomas yang mempunyai kandungan air yang tinggi berbanding teknik pirolisis konvensional. Oleh itu, kajian ini bertujuan untuk menghasilkan karbon teraktif daripada tempurung kelapa sawit (PKS), serat mesokap (MF) dan tandan buah yang telah dikosongkan (EFB) dengan menggunakan teknik pirolisis dan HTC. Proses HTC merangsang pemecahan molekul polimerik besar dalam bahan lignoselulosa menjadi sebatian organik yang lebih kecil melalui proses hidrolisis, dehidrasi, dekarboksilasi, aromatisasi dan rekondensasi. Selepas itu, prestasi karbon teraktif diaplikasikan sebagai bahan asas untuk pemangkin dwifungsi. Oleh itu, karbon teraktif diimpregnasikan dengan K_2CO_3 dan CuO melalui proses impregnasi basah yang menghasilkan pemangkin dwifungsi. Pencirian telah dilakukan terhadap pemangkin dwifungsi termasuk analisis penjerapan-penyahjerapan N_2, penentuan kumpulan fungsi, pemerhatian morfologi, kajian kristalografi, pemetaan sinar-X, analisis taburan unsur, kepekatan bes dan asid dan analisis degradasi terma. Hasil kajian menunjukkan bahawa teknik HTC berjaya menghasilkan karbon aktif daripada PKS yang sangat mesopori dan mempunyai sifat tekstur yang lebih baik daripada pirolisis dengan masing-masing mempunyai luas permukaan BET sebanyak 1411.16 m^2 g^{-1} dan 3368.60 m^2 g^{-1}. Pemangkin dwifungsi telah digunakan dalam reaksi esterifikasi dan transesterifikasi serentak daripada sisa minyak masak (WCO) dengan menggunakan sistem refluks konvensional. Luas permukaan BET yang tinggi memberikan ruang tambahan untuk impregnasi bahan aktif yang sangat penting untuk menghasilkan jumlah biodiesel yang maksimum daripada WCO. Hasil prestasi pemangkin menunjukkan bahawa pemangkin yang menggunakan karbon teraktif hidroterma berasaskan PKS (PKSHAC) menghasilkan biodiesel yang lebih tinggi iaitu sebanyak 95.3% berbanding 95.0% daripada pemangkin yang menggunakan karbon teraktif pirolisis (PKSAC). Penyediaan karbon teraktif kemudiannya dilanjutkan dengan menggunakan MF dan EFB, menghasilkan MFHAC dan EFBHAC, yang menunjukkan luas permukaan BET 3909.33 m^2 g^{-1} dan 4056.17 m^2

g^{-1} dan masing-masing menghasilkan jumlah biodiesel yang maksimum iaitu sebanyak 96.4% dan 97.1%. Semua pemangkin yang telah disintesis dapat bertahan sehingga 5 kitaran tindak balas dengan lebih daripada 80% hasil biodiesel. Keruntuhan struktur karbon akibat daripada karbonisasi berulangan untuk pengaktifan semula pemangkin, dan keracunan oleh gliseroksida pada permukaan pemangkin telah dikenal pasti sebagai faktor penyahaktifan. Selain daripada itu, transformasi biodiesel daripada WCO telah disahkan melalui analisis ^{1}H NMR, FTIR dan TGA. Sifat bahan api menunjukkan kelikatan kinematik pada 3.3 $mm^2\ s^{-1}$, bilangan cetane 51, titik kilat 160.5 °C, dan titik awan dan titik tuang masing-masing 11 °C dan -3 °C. Secara keseluruhan, penemuan menunjukkan potensi yang sangat baik daripada bahan buangan terutama PKS, MF dan EFB dalam menghasilkan karbon teraktif berkualiti tinggi sebagai bahan asas pemangkin melalui teknik pirolisis dan HTC. Pengungsian dengan K_2CO_3 dan Cu_O melalui impregnasi basah memberikan sifat dwifungsi yang membuka peluang untuk memanfaatkan WCO dalam menghasilkan biodiesel yang berkualiti tinggi.

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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 Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

^{1}H-NMR	Hydrogen-1 nuclei magnetic resonance
BET	Brunauer-Emmett-Teller
BJH	Barret-Joyner-Halenda
EDX	Energy dispersive X-ray
EFB	Empty fruit bunch
EFBH	EFB-hydrochar
EFBHAC	Empty fruit bunch hydro-activated carbon
FAME	Fatty acid methyl ester
FESEM	Field emission scanning electron microscopy
FFA	Free fatty acid
FTIR	Fourier transform infrared spectroscopy
GC-FID	Gas chromatography - flame ionization detector
HTC	Hydrothermal carbonization
MF	Mesocarp fiber
MFHAC	Mesocarp fiber hydro activated carbon
PKS	Palm kernel shell
PKSHAC	Palm kernel shell hydro activated carbon
PKSHC	Palm kernel shell hydro carbon
TGA/DTG	Thermogravimetric analysis/ differential thermogravimetric
TPD-CO_2	Temperature programmed desorption carbon dioxide
TPD-NH_3	Temperature programmed desorption ammonia
WCO	Waste cooking oil
XRD	X-ray diffraction

CHAPTER 1

INTRODUCTION

1.1 Background of study

Biodiesel is one of the best alternatives for conventional fossil fuels and its use is growing all over the world. In conjunction with global economic evaluation and the expansion of the automobile industry sectors, researchers consistently provide the most remarkable discoveries and inventions to produce alternatives to fossil fuel (F. Martins et al., 2019). Biodiesel has gained popularity worldwide because it is renewable and energy efficient as it is primarily made from crop waste and recycled resources. Biodiesel was found a promising new source of energy as it is highly biodegradable and could reduce global warming gas emissions as it emits lesser hydrocarbon (HC), carbon monoxide (CO), 10 diesel engines with no or only minor modifications. The potential of waste cooking oil (WCO) was discovered as the best candidate for biodiesel production in this study. The non-value by-product from daily cooking and frying activities reported that 15 Mt had been disposed of annually worldwide (A. F. Lee et al., 2014a). Most of the time, the WCO is drained off into the drainage system which will cause water pollution. This situation has caused difficulties for authorities in waste management, especially when dealing with WCO. Thus, utilizing WCO for biodiesel production in extent will help to reduce the cost of waste management.

Theoretically, biodiesel was produced via esterification or transesterification reaction. Each reaction requires a catalyst to accelerate the reaction. The acidic catalyst was involved in the esterification reaction of feedstock with a high content of free fatty acid (FFA), while the basic catalyst facilitates the transesterification reaction of feedstock with high content percentages of triglycerides. The development of a catalyst for biodiesel production shows an excellent opportunity for activated carbon as catalyst support. The use of biomass-derived activated carbon for heterogeneous catalysts seems to be a promising option as it eliminates the tediousness and problems faced by homogeneous reactions. In addition, biomass is ubiquitous in nature, wide distribution, CO_2 neutrality and economic feasibility (Ma et al., 2012). Therefore, the synthesizing activated carbon originating from palm oil biomass such as palm kernel shell (PKS), empty fruit bunch (EFB) and mesocarp fiber (MF) provides a promising opportunity in the utilization of waste material into valuable activated carbon as catalyst support for biodiesel production.

The introduction of the hydrothermal carbonization (HTC) process provides a brilliant alternative in producing activated carbon from high moisture biomass. Instead of drying the materials, the HTC operated in the presence of subcritical water conditions with autogenous pressure in the range of 2-10 Mpa in the sealed reactor, thus promoting dissolution of lignocellulosic components into the hot water (He et al., 2013). The HTC technique involves hydrolysis, dehydration, decarboxylation, aromatization, re-condensation, and other complicated unknown processes to produce biochar, namely hydrochar, bio-oil, and gases at the end of the process (Fang et al., 2018; Kalderis et al., 2014). The subcritical water accelerates the large polymeric molecules contained in the lignocellulosic materials into lower molecular weight organic compounds (Khan et al., 2019). Other than that, the HTC process has an advantage in producing 70-80% of yield and the produced hydrochar exhibited high aromaticity structure with a higher content of oxygen-containing functional groups (Yihunu et al., 2019).

1.2 Problem Statement

Nowadays, the biodiesel production process suffers from technological challenges in producing highly effective catalysts to accommodate feasibility and sustainable development. Homogeneous catalyst is widely used by industry nowadays cause several technical problems such as reaction corrosion due to high acidity/basicity, generate large amounts of waste water via product washing and acid neutralization step, separation problems caused by unfavourable aqueous emulsion; which thereby increase the overall cost of biodiesel production (Farooq, Ramli, Naeem, & Saleem khan, 2016; Mardhiah et al., 2017). In contrast, a heterogeneous catalyst has the ability to be recovered and re-used provides excellent economic benefits to the product liability (Meher et al., 2013). Other than that, different heterogeneous catalysts such as zirconia, ferric alginate, titanium dioxide, silica nanoparticles, and others require expensive materials, have complicated synthesis routes, and record low catalytic activity. To overcome this problem, an activated carbon was prepared using waste biomass as a catalyst support.

Conventionally, the activated carbon was produced by the pyrolysis technique. The pyrolysis techniques require high temperature up to 700 °C in an inert atmosphere to produce activated carbon. As a result, the produced activated carbon, namely biochar, will have high BET surface area and porosity. However, the pyrolysis technique requires an energy-intensive drying process and is inconvenient for biomass-based material as it contains high moisture content. The reaction condition is also critical as the oxygen should be avoided throughout the process by providing inert gas such as nitrogen; otherwise, it will produce ash instead of activated carbon. Besides that, pyrolysis emits toxic gasses during carbonization, requiring specific safety precautions to handle the emission. In this work, the environmentally friendly of HTC process was introduced to skip the drying process of the biomass material by using water as solvent to produce hydrochar that has been used as catalyst support to produce biodiesel.

Biodiesel was formerly produced from edible oils creates unhealthy competition between food and biodiesel crops. The used of inedible oil of WCO become a challenge as the it contains of high triglycerides (85%), FFA (15%), moisture and solid residue. Previously, the biodiesel production from WCO was catalyzed with a homogeneous catalyst by two-step esterification–transesterification, where esterification is to reduce the FFA before transesterification reaction. Thus, the two-step reaction has been double the production cost and time and it needs a neutralization step to the treatment system before it is safe for the environment (Baroutian et al., 2010). Therefore, the acid-base bifunctional catalyst has been synthesized to catalyze both transesterification and esterification reactions simultaneously to fully utilize the oil under mild conditions exhibits great potential for industrial application.

1.3 Objectives

This study aims to produce biomass based bifunctional catalyst for biodiesel production from WCO. The aim will be supported with the following objectives:

i. to discover potential of palm waste in producing activated carbon for catalyst support using pyrolysis technique,
ii. to access capability of HTC technique in producing activated carbon for catalyst support,
iii. to determine the physical and chemical properties of the bifunctional catalyst for simultaneous esterification-transesterification reaction, and
iv. to identify the deactivation factor that caused the deterioration of biodiesel yield that effect the reusability of the bifunctional catalyst.

1.4 Scope

This study was focused on the utilization of PKS, EFB and MF in producing activated carbon via pyrolysis and HTC techniques. Initially, the activated carbon was produced from PKS using pyrolysis and HTC techniques. A comparison of its physical and chemical properties was done to choose the best technique to produce high-quality activated carbon. Later, both of the produced activated carbon were doped with potassium carbonate (K_2CO_3) and copper nitrate ($Cu(NO_3)_2$) via wet impregnation followed by calcination to produced bifunctional catalyst and tested its catalytic performance. Subsequently, after considering best results, the catalysts derived from EFB, and MF were prepared using the HTC technique. All catalysts were subjected to detailed characterizations to assess their surface characteristics, morphological and elemental analysis, amount of basicity and acidity, crystallinity, surface functional group and thermal degradation behaviour. The optimum loading of K_2CO_3 and $Cu(NO_3)_2$ were determined by various percentages loading onto the activated carbon to find the best combination as a bifunctional catalyst for biodiesel production using a conventional reflux system for simultaneous esterification and transesterification reaction of WCO. The reaction condition of catalyst loading, methanol to oil molar ratio, temperature and reaction duration were optimized through one-factor-at-a-time techniques. Afterwards, the detailed reusability study was carried out using the best catalyst and the properties of the spent catalyst were characterized to discover the deactivation factor. This study also includes a quality assessment of the produced biodiesel to confirm the conversion of WCO to biodiesel and was further compared with ASTM D-6751.

1.5 Thesis Outline

This thesis contains nine chapters that include four chapters based on published work. Chapter 1 provides the background of this study and the deliberation of problem statements that initiate the study. This chapter also highlights the aim and objectives to solve the problems and scope of the study. Chapter 2 discusses palm oil-based waste's potential as a source of activated carbon with its availability in Malaysia and technologies in producing the activated carbon. Afterward, the application of the homogeneous and heterogeneous catalysts in biodiesel production was reviewed and quality standards associated with biodiesel were listed. Chapter 3 started with a list of chemicals and materials used in this study. It also delivers a methodology covering the preparation of activated carbon, synthesizing bifunctional catalyst, and illustrates the experiment set up for the esterification-transesterification reaction. Other than that, the details of catalysts characterization were included. Additionally, analyses relating to biodiesel assessment and properties were inserted. Chapter 4, 5, 6 and 7 present the potential of PKS, MF and EFB as catalyst support of bifunctional catalysts in biodiesel production. These chapters include the characterizations of the synthesized catalysts, the experiment set up for application in producing biodiesel from WCO, the optimization of the reaction condition, the reusability study, and its characterization. Chapter 8 focuses on the biodiesel evaluation and deliberation of its properties. Chapter 9 summarizes by highlighting the main contribution and significant findings of this study and recommendations for future investigation.

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