UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERIZATION OF HETEROGENEOUS MAGNETIC BIFUNCTIONAL CATALYST DERIVED FROM RICE HUSK FOR BIODIESEL PRODUCTION FROM USED COOKING OIL

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By

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SYNTHESIS AND CHARACTERIZATION OF HETEROGENEOUS MAGNETIC BIFUNCTIONAL CATALYST DERIVED FROM RICE HUSK FOR BIODIESEL PRODUCTION FROM USED COOKING OIL

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Chairman : Umer Rashid, PhD
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The heterogeneous magnetic bifunctional catalysts supported on rice husk biochar were successfully synthesized by impregnated K_2O and magnetic compound Fe_2O_3 and NiO. The synthesized catalysts were characterized by using XRD, BET, TPD-CO_2, TPD-NH_3, TGA-DTA, FTIR, FESEM-EDX and VSM. The prepared catalysts consisted with high surface area more than 20 m^2g^{-1} with highly porous structure that increased the number of active sites, overcame diffusion problem between solid catalyst-oil-methanol and enhanced the catalytic transesterification.

The utilization of magnetic catalyst is known to ease separation process from reaction medium by introduce magnetic field. The VSM analysis revealed that RHC/K_2O-20 wt.%/Fe-5 wt.% and RHC/K_2O-20 wt.%/Ni-5 wt.% exhibited sufficient magnetism of 7.88 $emug^{-1}$ and 2.31 $emug^{-1}$ respectively for separation of catalyst. Furthermore, the recovery percentage of magnetic catalysts were recorded approximately more than 80 % after reaction was catalyzed 6 times.

In this study, the catalysts were used for transesterification of used cooking oil (UCO) to biodiesel by using a conventional reflux. The catalytic activities of RHC/K_2O-20 wt.%/Fe-5 wt.% and RHC/K_2O-20 wt.%/Ni-5 wt.% had demonstrated the highest biodiesel yield at 98.6% and 98.2% under following moderate optimum condition: catalyst loading 4wt.%, methanol-to-molar ratio of 12:1, reaction temperature of 75 ^oC and 65 ^oC within reaction time of 4 h and 2 h respectively. Both catalysts were reused for 5 transesterification consecutive cycles with a biodiesel yield more than 70.0 %.

The fuel properties of the biodiesel reviewed by the ASTMD 6751 method, and it was found to be within allowable limits and has properties almost similar to diesel fuel. To conclude, both of heterogeneous magnetic bifunctional catalysts RHC/K_2O-20 wt.%/Fe-5 wt.% and RHC/K_2O-20 wt.%/Ni-5 wt.% have the potential in the transesterification of low-grade feedstocks due to high catalytic performance, recovery, reusability, and stability.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
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SINTESIS DAN PENCIRIAN MANGKIN HETEROGEN BERMAGNETIK DWI-FUNGSI TERBITAN SEKAM PADI UNTUK PENGHASILAN BIODIESEL DARIPADA MINYAK TERPAKAI

Oleh

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Pemangkin dwi-fungsi heterogen berjaya disintesis dengan impregnasi K_2O dan sebatian magnet seperti Fe_2O_3 dan NiO ke atas arang sekam padi. Pemangkin yang disintesis diciri menggunakan XRD, BET, TPD-CO_2, TPD-NH_3, TGA-DTA, FTIR, FESEM-EDX and VSM. Luas permukaan pemangkin yang disediakan melebihi 20 m^2g^{-1} dan mempunyai tinggi struktur berliang yang mampu meningkatkan bilangan tapak aktif, mengatasi masalah resapan antara pemangkin-minyak-metanol dan meningkatkan pemangkinan transesterifikasi.

Penggunaan pemangkin bermagnet dikenali boleh memudahkan proses pemisahan pemangkin daripada medium tindak balas dengan menggunakan medan magnet. Analisis VSM menunjukkan pemangkin RHC/K_2O-20 wt.%/Fe-5 wt.% dan RHC/K_2O-20 wt.%/Ni-5 wt.% masing-masing mempunyai sifat magnet 7.88 $emug^{-1}$ dan 2.31 $emug^{-1}$ memadai untuk pemisahan pemangkin. Tambahan pula, peratusan pemulihan pemangkin bermagnet dianggarkan melebihi 80 % selepas 6 kali tindak balas pemangkinan.

Dalam kajian ini, pemangkin digunakan untuk transesterifikasi minyak masak terpakai kepada biodiesel dengan menggunakan radas refluks konvensional. Aktiviti pemangkinan oleh RHC/K_2O-20 wt.%/Fe-5 wt.% and RHC/K_2O-20 wt.%/Ni-5 wt.% menunjukkan penghasilan biodiesel tertinggi sebanyak 98.6 % dan 98.2 % pada keadaan separa optimum seperti berikut; muatan pemangkin 4 wt.% nisbah pemangkin kepada kepekatan methanol iaitu UCO 12:1 suhu tindak balas masing-masing pada 75 °C dan 65 °C serta masa tindak balas iaitu 4 jam dan 2 jam. Pemangkin boleh diguna semula sehingga lima kitaran dan menghasilkan biodiesel melebihi 70.0 %.

Sifat bahan api biodiesel dikenalpasti dengan menggunakan ASTMD 6751 dan hasil mendapati biodiesel berada pada had yang dibenarkan serta bersifat yang hampir sama dengan bahan api diesel. Kesimpulannya, kedua-dua pemangkin dwi-fungsi heterogen bermagnet RHC/K_2O-20 wt.%/Fe-5 wt.% and RHC/K_2O-20 wt.-%/Ni-5 wt.% menujukkan keupayaan dalam transesterifikasi sumber minyak gred rendah kerana tinggi kecekapan pemangkinan, pemulihan, kebolehgunaan semula dan stabil.

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LIST OF ABBREVIATIONS

^{1}H-NMR	Proton Nuclear Magnetic Resonance
AOCS	American Oil Chemist's Society
ASTM	American Standard Testing Method
ATR-FTIR	Attenuated Total Reflection- Fourier Transform Infrared
AV	Acid Value
BET	Brunauer-Emmett-Teller
EDX	Energy Dispersive X-ray
FAME	Fatty Acid Methyl Ester
FESEM	Field Emission Scanning Electron Microscope
FFA	Free Fatty Acid
GC-FID	Gas Chromatography-Flame Ionisation Detector
MW	Molecular Weight
RH	Rice Husk
RHC	Rice Husk Char
RHA	Rice Husk Ash
SV	Saponification Value
TGA-DTA	Thermogrametric Analyser- Differential Thermal Analyser
TPD-CO_2	Temperature Programmed Desorption-Carbon Dioxide
TPD-NH_3	Temperature Programmed Desorption- Ammonia
UCO	Used Cooking Oil
VSM	Vibrating- Sample Magnetometer
XRD	X-ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Industrialization, metropolitan growth, power production and transportation are dependent on the non-renewable sources such as petrol fuel, coal and natural gases whose reserves of the source continue to decline yearly (Balajii and Niju, 2020). As reported, global oil reserves can only sustain and provide energy and chemicals for four decades (Deng et al., 2016). Furthermore, the burning of fossil fuels contributes to the emission of greenhouse gases that has led to serious environmental issues, mainly climate change (Sahar et al., 2018). Thus, the development of a renewable, sustainable and environmental friendly energy source to replace fossil fuels with green natural resources has drawn the attention of researchers over the past twenty years (Ramli et al., 2017).

Biofuels such as biodiesel, biogas and bioethanol had been recognized as a new alternative to overcome the energy crisis, due to the availability of feedstocks (non-edible oils, animal fats and biomass wastes) for the conversion of biofuels through different chemical process and technologies. These types of biofuels are biodegradable, emit non-toxic gases and release less carbon dioxide than fossil fuels (Ahmad et al., 2011). Among of the different type of biofuels, biodiesel is increasingly appealing given the physiochemical properties which can be used directly or blend with diesel and utilized in the compression ignition engine without modification (Shivakumar and Dinesha, 2019).

Biodiesel can be produced through three well-established methods such as blending, dilution, micro-emulsion, thermal cracking, and transesterification (Stephen and Periyasamy, 2018). However, the most common method employed is the transesterification of vegetable oils and animal fats with alcohol in the presence of the suitable catalyst (Aransiola et al., 2014). So far, the biggest barrier to commercializing biodiesel is the cost production, thus the exploitation of waste material such as used cooking oil (UCO) for biodiesel production could be useful to reduce raw material cost and making the process more economical also reducing pollution that wasted into the environment (Mansir, et al., 2018). Nevertheless, UCO contains a high level of free fatty acids (FFAs) which lead to saponification. Therefore, to overcome this problem heterogeneous catalysts fabrication and design has been intensively studied to replace the utilization of homogeneous catalysts for large scale biodiesel production (Li et al., 2019).

Lately, bifunctional heterogenous catalysts have gained considerable recognition for biodiesel synthesizing from low-cost feedstocks because these catalysts can carry out esterification of FFAs and transesterification of triglycerides simultaneously without producing soap and corroding the reactor also the by-product purification steps can be omitted (Helwani et al., 2009). Moreover, the bifunctional heterogeneous catalyst can be regenerated and reused for multiple cycles. This type of catalyst can be classified into several groups, for example, carbon-based catalyst (Kumar et al., 2019), metal-based catalyst (Prabhakaran et al., 2017) and polymer-based catalyst (Kumar et al., 2020). Unfortunately, each catalyst has its own disadvantages for instance, mass transfer limitation, low surface area and high materials cost. Therefore, a more efficient, easier to prepare, cheaper and more environmentally friendly bifunctional heterogeneous catalyst should be used to produce biodiesel on a large scale.

To date, low-cost catalyst derived from carbon biomass waste materials had received tremendous attention due to several distinct properties such as high surface area and porosity, high stability, also can be modified and functionalized with active group metals (Bhoi et al., 2020). Basically, the porous carbon-based catalyst can be synthesized from agricultural waste (rice husk, palm kernel shell, corn cob, coconut peats and shells and etc.) and animal waste (manure) by pyrolysis (Deng et al., 2016). Unfortunately, the recovery process of solid carbon-based catalyst remains as the main disadvantages because it is conventionally performed by filtration and centrifugation techniques by which would reduce the catalyst amount. Hence, the fabrication of solid catalyst with magnetic materials could ease and fasten the separation process of catalyst from the reaction medium by applied external magnetic field with high rate of catalyst recovery than non-magnetic carbon-based catalyst (Ullah et al., 2014).

1.2 Problem Statement

The heterogeneous catalyst is slightly less reactive due to the different phase in the reaction medium which results in a diffusion problem and limited mass transfer between solid catalyst-oil-methanol. In addition, a low number of active sites available, less porosity and derived from expensive materials are common defects found from solid catalyst. Therefore, it can be replaced by utilizing a cheap solid catalyst derived from agricultural waste such as rice husk due to the fact that the catalyst supported with biomass waste char possesses with more specific surface area for catalytic active sites, high porosity, and high stability to well perform catalytic transesterification.

Conversion of biodiesel form UCO by using highly basic heterogeneous carbon-based catalyst is unfavourable due to emulsion and soap formation. Thus, in this study, the catalysts were modified by introducing both of acidic and basic functional groups on the rice huck char which simultaneously can perform esterification and transesterification.

Normally, the removal process of heterogeneous catalyst from the reaction medium, involves filtration and centrifugation, which are impractical due to longer time and energy consuming and ineffective separation techniques with low catalyst recovery. As a result, magnetic metals such as Fe and Ni were developed into the biochar support to simplify the catalyst removal process by magnetic separating technique. These metals provided paramagnetic property that attracted to applied magnetic field and able to recover catalysts from reaction medium more than 80% in comparison to other conventional catalyst recovery methods.

1.3 Objectives of the Research

The aim of this research to synthesize magnetic heterogeneous bifunctional catalyst supported on rice husk char. There are four objectives have been highlighted and addressed as follows:

I. To synthesize and characterize the magnetic heterogenous bifunctional catalysts supported by rice husk char.
II. To optimize the parameter of catalytic transesterification reaction of UCO.
III. To study reusability and deactivation of synthesized heterogeneous magnetic catalyst.
IV. To determine and evaluate the properties of biodiesel derived from UCO.

1.4 Scope of Research

This research consisted of the synthesis of magnetic bifunctional heterogeneous catalysts supported on rice husk char for biodiesel production derived from UCO. The physical and chemical characterization of prepared magnetic RHC catalysts was studied and performed by using BET, TPD, TGA, XRD, FTIR, VSM and FESEM-EDX. The behaviour of prepared catalysts towards transesterification via catalytic parameter optimization was investigated and discussed thoroughly in this thesis. In addition, the catalyst reusability tests were carried out and the deactivation of used catalysts were studied and characterized. The quality assessment of synthesized biodiesel was determined by using ASTM D6751 standard.

1.5 Organization of the Thesis

This thesis is divided into six chapters. Chapter One presents the research background on the benefits of biodiesel; the types of catalysts involved in biodiesel production and emphasizes the purposes of the study. Chapter Two describes a comprehensive literature review of the benefits of biodiesel as a new energy resource, biodiesel synthesis methods, the generations of biodiesel feedstocks, types of catalysts involved in the transesterification of triglycerides as well as the reports on utilizing bifunctional magnetic solid catalysts supported on biomass char. Chapter Three discusses the materials and methods of synthesis and characterization of bifunctional magnetic catalysts, transesterification process and FAME analysis. Chapter Four and Chapter Five provide the experimental results and the explanation of the analysis results. Chapter six presents summarization and highlights of the finding of this research as well as the recommendations for future research work.

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