UNIVERSITI PUTRA MALAYSIA

UTILIZATION OF BIOMASS-DERIVED ACTIVATED CARBON AS CATALYST SUPPORT AND BIOADSORBENT IN BIODIESEL PRODUCTION USING WASTE COOKING OIL AS FEEDSTOCK

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By

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The depletion of non-renewable fossil fuels and the growing environmental awareness, biodiesel is seen as a promising substitute for the conventional diesel. Its eco-friendly properties such as being renewable, biodegradable and less carbon emission have brought new hope for a greener future. Presently, waste cooking oil and oil palm empty fruit bunch were extensively used as the raw materials for a low-cost feedstock and catalyst for biodiesel production. Apart from its economic objective, exploitation of these abundant waste sources for biodiesel production is a step ahead in saving the environment from pollution, as it is typically being disposed indiscriminately.

In this study, improved production of biodiesel from waste cooking oil was achieved by using a newly developed potassium phosphate tri-basic supported activated carbon catalyst. In order to produce high surface area activated carbon, press-shredded oil palm empty fruit bunch was subjected to carbonization at 700^{O}C for 2 h followed by activation with potassium hydroxide at 700^{O}C for 2 h. To produce the catalyst, calcination was performed at different potassium phosphate tri-basic impregnation concentrations (1:0.25 to 1:1 activated carbon to potassium phosphate tri-basic weight ratio) and temperatures (400^{O}C to 700^{O}C). Prior to transesterification, waste cooking oil was analysed for its physicochemical properties and pre-treated to remove moisture and residues. Under the optimum condition of 5 wt% catalyst loading, 12:1 methanol to oil molar ratio at 60^{O}C for 4 h, 98% of biodiesel yield was achieved, which surpassed the European Biodiesel Standard (EN 14214). The catalyst was reusable for 5 successive reaction cycles, achieving almost 80% of biodiesel yield.

In addition, the activated carbon produced from the press-shredded oil palm empty fruit bunch was also utilized as bioadsorbent to remove impurities from the crude biodiesel. The purification process was performed using different adsorbent loadings (1 to 5 wt%) under continuous stirring condition at 500 rpm for 1 h. Approximately 89.71% of methanol, 81.74% of water, 36.67% of FFA and 98.61% of potassium (K) were successfully removed after purification at 5 wt% of bioadsorbent loading, which met the European Biodiesel Standards (EN 14214). In comparison to other commercial adsorbents and conventional water washing method, purification using the biomass-derived bioadsorbent resulted in better removal of methanol, water and triglyceride impurities with only a small loss of biodiesel yield.

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

PENGGUNAAN KARBON TERAKTIF DIPEROLEH DARIPADA BIOJISIM SEBAGAI PENYOKONG PEMANGKIN DAN BIO-PENJERAP DIDALAM PENGHASILAN BIODIESEL MENGGUNAKAN SISA MINYAK MASAK SEBAGAI BAHAN MENTAH

Oleh

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Dengan pengurangan bahan api fosil yang tidak boleh diperbaharui dan kesedaran alam sekitar yang semakin meningkat, biodiesel dilihat sebagai pengganti yang memberangsangkan untuk diesel konvensional. Ciri-ciri mesra alam seperti boleh diperbaharui, biodegradasi dan kurang pelepasan karbon telah membawa harapan baru untuk masa depan yang lebih hijau. Kini, sisa minyak masak telah digunakan dengan meluas sebagai bahan mentah dan pemangkin bagi pengeluaran biodiesel kos rendah. Selain daripada objektif ekonomi, eksploitasi sumber sisa yang banyak ini bagi pengeluaran biodiesel adalah satu langkah ke hadapan dalam menyelamatkan alam sekitar daripada pencemaran dan penyumbatan sistem kumbahan sanitari, kerana ia biasanya dilupuskan secara sewenang-wenangnya.

Dalam kajian ini, peningkatan pengeluaran biodiesel daripada sisa minyak masak telah dicapai dengan menggunakan pemangkin potassium fosfat tri-asas disokong karbon teraktif. Dalam usaha untuk menghasilkan luas permukaan karbon teraktif yang tinggi, tandan kelapa sawit kosong ditekan-cincang telah dikarbonisasi pada suhu 700^{O}C selama 2 jam diikuti oleh pengaktifan dengan kalium hidroksida pada 700^{O}C selama 2 jam. Pengkalsinan dilakukan pada kepekatan impregnasi kalium fosfat tri-asas (1: 0.25 kepada 1: 1 diaktifkan karbon kalium fosfat nisbah berat tri-asas kepada) dan suhu (400^{O}C untuk 700^{O}C) yang berbeza. Sebelum transesterifikasi, sifat fizikokimia sisa minyak masak telah dianalisis dan dipra-rawat untuk membuang kelembapan dan residu. Di bawah keadaan optimum 5% berat unit muatan pemangkin, 12:1 nisbah molar metanol kepada sisa minyak masak pada 60^{O}C untuk bagi 4 jam, 98% hasil biodiesel telah dicapai, yang melepasi Piawaian Biodiesel Eropah (EN 14214). Pemangkin ini boleh diguna semula untuk 5 kitaran reaksi berturut-turut, mencapai hampir 80% hasil biodiesel.

Di samping itu, karbon teraktif yang dihasilkan daripada tandan kelapa sawit kosong yang ditekan-cincang juga digunakan sebagai bio-penjerap untuk membuang kekotoran daripada biodiesel mentah seperti air, asid lemak bebas (FFA), metanol, gliserin bebas, trigliserida dan kalium. Proses penulenan biodiesel mentah yang dihasilkan daripada sisa minyak masak telah dilakukan dengan menggunakan muatan penjerap yang berbeza (1 hingga 5% berat unit) di bawah keadaan kacau berterusan pada 500 rpm selama 1 jam. Kira-kira 89.71% metanol, 81.74% air, 36.67% FFA and 98.6% kalium telah berjaya dibuang selepas penulenan pada 5% berat unit muatan bio-penjerap, yang mana telah melepasi Piawaian Biodiesel Eropah (EN 14214). Berbanding dengan penjerap komersial yang lain dan kaedah konventional basuhan air, penulenan menggunakan bio-penjerap yang diperolehi daripada biojisim telah mengakibatkan penyingkiran metanol, air dan trigliserida yang lebih baik dengan kehilangan hasil biodiesel yang kecil.

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I certify that a Thesis Examination Committee has met on 18 April 2017 to conduct the final examination of Mohammed Abdillah bin Ahmad Farid on his thesis entitled "Utilization of Biomass-Derived Activated Carbon as Catalyst Support and Bioadsorbent in Biodiesel Production using Waste Cooking Oil as Feedstock" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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LIST OF ABBREVIATIONS

ASTM D6751	American biodiesel standard
BET	Brunauer–Emmett–Teller
BJH	Barrett-Joyner-Halenda
CH_3OH	Methanol
CO_2	Carbon dioxide
Cu	Copper
EN14214	European biodiesel standard
EDX	Energy-dispersive X-ray
FAME	Fatty acids methyl esters
FELDA	Federal Land Development Authority
FTIR	Fourier transform infrared spectroscopy
FFA	Free fatty acids
GC	Gas Chromatography
He	Helium gas
h	Hour
H_2	Hydrogen gas
HCl	Hydrochloride acid
K	Potassium
KBr	Potassium bromide
KOH	Potassium hydroxide
K_3PO_4	Potassium phosphate tri-basic
MB	Methylene blue
min	Minute
M	Molar

N_2	Nitrogen gas
OPEFB	Oil palm empty fruit bunch
P	Phosphorus
rpm	Rotation per minute
SEM	Scanning electron microscope
TPD	Temperature Programmed Desorption
XRD	X-ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 General introduction

It has been two centuries since petroleum was found and now almost 90 % of world energy supplies relied on fossil fuels (Chew & Bhatia, 2008). Nevertheless, due to its growing demand and uncontrollable consumption, the earth has begun to bear the negative consequences such as depletion of fossil fuel reserves and climate change (Adam *et al.*, 2011). Based on the current estimate, it was expected that world fossil fuel reserves could only last for another 53 years (Xu *et al.*, 2016). Undisputedly, the upsurge in fossil fuels utilization has increased the amount of CO_2 emission which leads to global warming (Yusuf *et al.*, 2011). Therefore, the scientific community has been intensively conducting studies to seek solutions for these issues.

Biodiesel is one of the potential biofuels that could reduce our energy dependency on fossil fuels. It consists of long-chain fatty acid alkyl esters and are normally produced from agricultural oils which are renewable (Berrios *et al.*, 2010). It has been regarded as an alternative towards the petroleum diesel due to its better features such as low carbon emission, greater lubricity, biodegradable and less toxic (Dehkhoda *et al.*, 2010).

1.2 Problem statement

With current technologies implemented, biodiesel production process is cost-ineffective. High price of feedstocks, non-reusable catalysts and poor downstream purification method have inevitably caused an expensive final product and costly wastewater treatment (Shu *et al.*, 2010).

The economics of biodiesel production is strongly linked to feedstock cost, catalyst cost and wastewater treatment (Kastner *et al.*, 2012). It has been estimated that 95% of global biofuel production uses edible oil as feedstocks (Sanjid *et al.*, 2016). As a consequence, competition between energy markets with food sectors has caused the increase of edible oil prices. In biodiesel production itself, approximately 70 to 90% of overall biodiesel production cost was accounted solely for feedstock expenditure (Farooq *et al.*, 2013). Therefore, employing cheap raw material in biodiesel production such as waste cooking oil (WCO) is a better option. Utilization of WCO in biodiesel production reduced the cost of production down to 60% in comparison to high-grade vegetable oil consumption (Talebian-Kiakalaieh *et al.*, 2013). Due to lack of disposal policy and inefficient waste management, WCO has been directly dumped into sewer and the environment, which led to pipeline blockage and pollution (Mara & Alam, 2008). On top of that, due to its cheap price, some unregulated industries have made profit out of recycling this waste by blending into a new cooking oil product. Thus, based on these issues, it is beneficial to employ WCO as feedstock for biodiesel production.

Transesterification is a process that is involved in biodiesel conversion by detaching 3 free fatty acids (FFA) from a molecule of triglyceride and combining them with alkyl group to form alkyl ester molecules (Sharma *et al.*, 2011). In this process, a homogenous base catalyst, such as sodium hydroxide (NaOH) and potassium hydroxide (KOH), was commonly being used due to its cheap price and high in reactivity (Tariq *et al.*, 2012). Recently, carbon based catalyst has gained attention due to its reusability and high catalytic activity (Konwar *et al.*, 2014). Previously, the development was focused on solid acid catalyst. Nevertheless, since implementation of acid-catalysed reaction was usually associated with impractical procedures such as long reaction time, high amount of alcohol usage, and high temperature condition, solid base catalyst is used due to its benefits such as higher reaction yield, short reaction period, less alcohol usage and conducted at mild reaction condition (Chew & Bhatia, 2008). However, these solid catalysts are expensive that leads to high price of biodiesel. Therefore, development of carbon based catalyst from agricultural waste is gaining interest among researchers due to its low-cost of production (Konwar *et al.*, 2012).

Biodiesel purification step is important in order to improve the quality of the final fuel product in fulfilling the quality specifications standard (EN 14214). The presence of unwanted impurities in biodiesel such as free FFA, free glycerol, catalyst trace, moisture, remaining alcohol and unreacted triglycerides reduced the quality of the fuel (Ngamlerdpokin *et al.*, 2011). Usually, water washing method was applied to purify the crude biodiesel. However, this conventional method produced wastewater at the end of the process, which has to be treated before being discharged (Atadashi *et al.*, 2011; Enweremadu & Mbarawa, 2009). In contrast to purification method using adsorbents, no wastewater are produced and less product loss (Berrios *et al.*, 2011). Activated carbon was recognized as the material with excellent adsorbent properties for purification (Konwar *et al.*, 2014). According to Fadhil *et al.* (2012), utilization of activated carbon in biodiesel purification produced a better yield and quality compared to conventional water washing method.

Palm oil industry is one of the fast growing economic sectors in Malaysia (Alam, 2008). Increased demand on oil palm led to accumulation of huge amount of biomass waste, such as oil palm empty fruit bunch (OPEFB). As of now, OPEFB was either applied at the plantation as compost fertilizer, burned illegally or act as fuel to generate steam at the mill. Since it was fibrous in structure, this material was considered good for high surface area activated carbon production (Tan *et al.*, 2009).

The overall objective of this research is to produce heterogeneous catalyst and adsorbent from oil palm biomass for biodiesel production from waste cooking oil. This research also targeted to improve the biodiesel production and purification process in comparison to conventional methods.

1.3 Specific Objectives

1) To produce activated carbon from empty fruit bunch (OPEFB) via two-steps carbonization and chemical activation method.
2) To develop and optimize carbon-supported potassium phosphate tri-basic (K_3PO_4/AC) catalyst for biodiesel production from WCO.
3) To purify and compare the quality of WCO-derived biodiesel using biomass-derived adsorbent with other purification methods.

1.4 Experimental overview

The overall experimental overview is shown in **Figure 1.1**. The first objective is on the development of K_3PO_4/AC catalyst for biodiesel production from waste cooking oil. By using OPEFB as raw material, the catalyst was developed through the process of carbonization, KOH-activation and K_3PO_4 calcination, consecutively. Subsequently, the prepared K_3PO_4/AC catalyst was characterized in order to study its physicochemical properties. In transesterification of WCO, the variables affecting the process were optimized in order to determine the highest biodiesel yield. The efficiency of the developed catalyst was also evaluated by conducting reusability and leaching test.

The second objective is to exploit the feasibility of the activated carbon produced from OPEFB biomass in purifying the biodiesel produced from WCO. The quality of the purified biodiesel using the activated carbon was analysed and compared with the conventional water washing method and other commercial adsorbents such as bentonite, silica gel and talc.

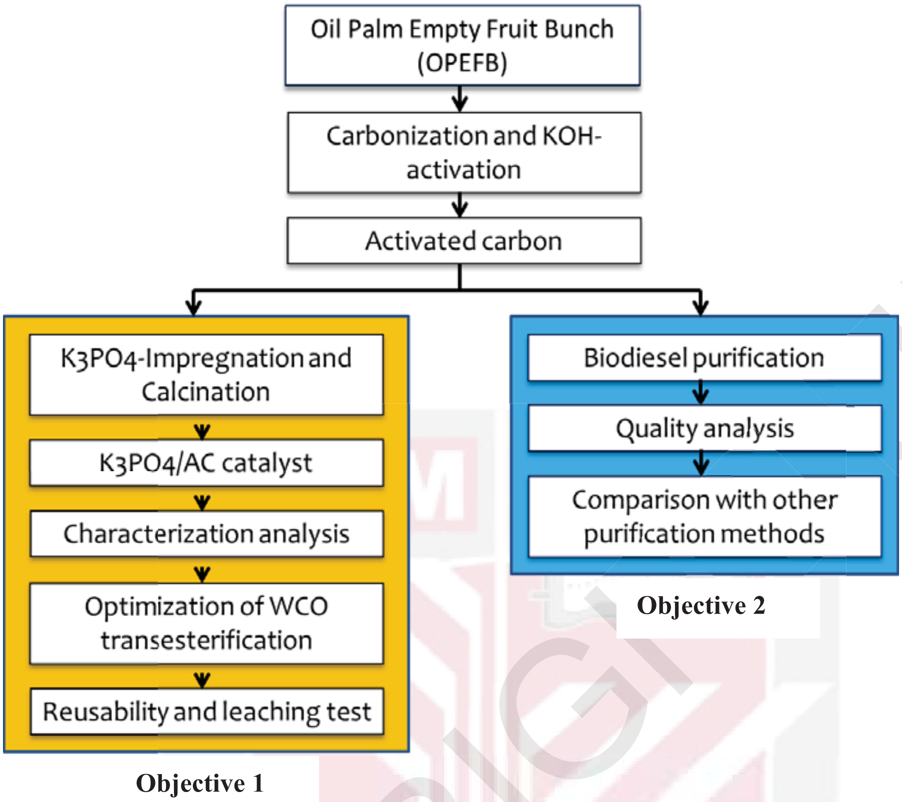

Figure 1.1: Research work flow

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