



Production of methyl esters from waste cooking oil using a heterogeneous biomass-based catalyst



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ABSTRACT

Fatty acid methyl esters (FAME) production from waste cooking oil was successfully carried out using a newly developed heterogeneous biomass-based catalyst. Activated carbon produced from oil palm biomass was calcined with potassium phosphate tri-basics (K_3PO_4) in order to synthesize a high catalytic heterogeneous catalyst. As it is characterized with substantial surface area of $680 \text{ m}^2/\text{g}$ and basicity amount of 11.21 mmol/g , 98% of FAME yield was achieved under optimum reaction parameters of 5 wt% catalyst loading, 12:1 methanol to oil molar ratio at $60 \text{ }^\circ\text{C}$ for 4 h. The catalyst was shown to be reusable, with more than 76% FAME yield after 5 consecutive cycles.

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1. Introduction

Besides global warming, population growth and global industrialization have caused increased energy demand and inevitable depletion of fossil fuels [1]. Increasing concern on environmental conservation and energy security issue have encouraged research efforts for alternative fuels, such as biodiesel [2]. Biodiesel is a renewable, green and clean-burning fuel that consists of long-chain methyl esters, which are normally produced from agricultural oils [3].

The lack of policies and inefficient waste management of waste cooking oil (WCO) have led to its indiscriminate disposal [4]. Since

it was abundant and cheap, some unregulated industries have made huge profits out of recycling this waste. Therefore, the exploitation of waste cooking oil (WCO) in biodiesel production is a potential alternative that is beneficial towards the environment [5].

Recently, heterogeneous catalyst has received attention for biodiesel production. Apart from its advantages on reusability and ease of separation, it could be prepared from cheap biomass [6]. It has been reported that oil palm empty fruit bunch (OPEFB) can be used as a catalyst for biodiesel production, after being functionalized with acids or bases [6,7]. In a previous report, biochar supported calcium oxide (CaO) was used in transesterification of Mesua ferrea seed oil resulting > 90% of fatty acid methyl esters (FAME) yield produced under the optimal conditions [8]. In other study, activated carbon supported heteropoly acid catalyst was used in transesterification of crude Jatropa oil in an ultrasound-assisted reactor system. The catalyst with 20% catalyst loading resulted in 87.3% of FAME yield in 40 min [7]. Recently, transesterification of waste cooking oil was found to be efficiently

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catalysed by potassium phosphate tri-basics (K_3PO_4), producing 97.3% of FAME yield [9]. Previously, K_3PO_4 catalyst is generally used as an anti-microbial agent for poultry processing and food additives [4,5]. Therefore, since it is safe and basic in characteristic, it should be explored for heterogeneous based transesterification reaction.

The aim of the present work was to synthesize and characterize a biomass-based catalyst for FAME production using WCO as feedstock. The catalyst was characterized with scanning electron microscope (SEM), energy-dispersive X-ray (EDX) spectroscopy, Brunauer–Emmett–Teller (BET), Barrett–Joyner–Halenda (BJH), Fourier transform infrared (FT-IR), X-ray diffraction (XRD) and temperature-programmed desorption of carbon dioxide (TPD- CO_2). The influence of methanol to oil molar ratio, catalyst loading and reaction temperature on FAME production was studied. The catalyst reusability and leaching properties were also being carried out for several consecutive reaction cycles.

2. Materials and methods

2.1. Materials

Samples of OPEFB were collected from FELDA Seriting Hilir Palm Oil Mill in Negeri Sembilan, Malaysia. For oil feedstock, WCO was collected from a nearby residential area located around Taman Sri Serdang, Seri Kembangan Selangor, Malaysia. KOH (95.5%) and analytical grade *n*-hexane (99.9%) were supplied by Merck, USA. Methanol (95%) was purchased from Friendemann Schmidt Chemical, Australia. K_3PO_4 (98%) was purchased from Sigma-Aldrich, USA. N_2 (99%) was purchased from Malaysian Oxygen Berhad (MOX). Supelco FAME mix standard was purchased from Sigma-Aldrich, Germany. Methyl heptadecanoate (99%) was purchased from Sigma-Aldrich, Germany.

2.2. Catalyst preparation

The collected press-shredded OPEFB was ground using cyclone grinder (Sima, Malaysia) to produce samples of 29 ± 1 mm in size. The sample was then washed several times with distilled water to remove dirt and residues, followed by drying at $105^\circ C$ for 16 h.

Activated carbon was produced by a two-step process, beginning with carbonization in a furnace (Densply Creamco, USA) under continuous flow of N_2 at $700^\circ C$ for 2 h. The biochar was ground using Waring blender (Hung Chuan Machinery, Taiwan) and sieved to achieve biochar size of $250 \mu m$, followed with KOH impregnation for 2 h at 1:0.5 biochar to KOH weight ratio. The mixture was then dried at $105^\circ C$ for 16 h and activated at $700^\circ C$ for 2 h under continuous flow of N_2 . The OPEFB-derived activated carbon obtained was neutralized with 0.1 M HCl, followed with hot water repeatedly until the pH of the washing solution reached 6 to 7, and dried at $105^\circ C$ for 16 h.

Biomass-based catalyst was prepared via wet-impregnation and calcination process. At 1:1 OPEFB-derived activated carbon to K_3PO_4 weight ratio, both were mixed in 100 mL of distilled water, which was continuously stirred at 80 rpm for 2 h. The impregnation mixture was then dried at $105^\circ C$ for 16 h and subsequently calcined for 3 h under continuous flow of N_2 at $500^\circ C$. The catalyst produced was stored in the desiccator at $25^\circ C$. The produced potassium phosphate tri-basics supported activated carbon catalyst (K_3PO_4/AC) was later used in the biodiesel reaction from WCO.

2.3. Catalyst characterization

Catalyst surface morphology and composition were examined using scanning electron microscopy (SEM) (JEOL, JSM- 6290LV instrument) equipped with energy-dispersive X-ray (EDX)

(Shimadzu, EDX-720). In order to evaluate the surface area, pore volume and pore size, surface area measurement analyser (Micromeritics ASAP 2000, USA) was used according to Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) method. Catalyst crystallinity and functional groups were examined via X-ray diffraction (XRD) using a Shimadzu Diffractometer Model XRD 6000 and Fourier transform infrared (FT-IR) spectroscopy, respectively. Temperature-programmed desorption of carbon dioxide (TPD- CO_2) was performed to determine basicity of the developed catalyst using AutoChem II 2920 chemisorption analyser.

2.4. Methyl esters production

At first, the WCO was filtered through vacuum filtration to remove the food impurities and dried at $105^\circ C$ for 1 h to remove the residual moisture. The reaction was performed by using a 1 L three-neck round-bottom flask equipped with magnetic stirrer bar and reflux condenser on a digital heating mantle (Misung Scientific Co., Korea). In the presence of 50 g of pre-treated WCO, the methanol and K_3PO_4/AC were added into the flask. After the reaction was completed, the product mixture was separated via centrifugation at 10,000 rpm for 10 min. The effect of the reaction variables such as methanol to oil molar ratio (3:1–15:1), catalyst loading (1–6 wt%), and reaction temperature (30 – $70^\circ C$) was investigated by sampling at an hourly interval during the 6 h reaction time.

The quantitative analysis of the produced FAME was analysed using gas chromatography (Shimadzu GC-14C) equipped with flame ionization detector (GC-FID) and polar RTX65 capillary column ($30 m \times 0.5 mm \times 0.25 \mu m$). For sample preparation, 100 μL of sample was added with 100 ppm of methyl heptadecanoate (internal standard) and dissolved in *n*-hexane (solvent) at the desired dilution factor. The injector and detector ports were set to $230^\circ C$ and $270^\circ C$, respectively. One μL of sample was injected into the oven at $140^\circ C$ and heated up to $250^\circ C$ at $5^\circ C/min$. The FAME yield was determined by following Eq. (1).

$$FAME \text{ yield}(\%) = \frac{\text{Total mole of methyl esters}}{\left(\frac{\text{Weight of WCO}}{\text{Molecular Weight of WCO}} \right) \times 3} \times 100\% \quad (1)$$

2.5. Catalyst reusability and leaching

The reusability of the catalyst was studied by conducting 4 successive reaction cycles. The spent catalyst was recovered from the reaction mixture and re-calcined for 3 h under continuous flow of N_2 . The leaching of potassium (K) and phosphorus (P) was determined by inductively coupled plasma-atomic emission spectrometric (ICP–AES) using Perkin Elmer Emission Spectrometer Model Plasma 1000.

3. Result and discussions

3.1. Catalyst characterization

The reason behind carbonization is to increase the carbon content and generate porosity, while activation helps in pores enlargement. In this work, potassium hydroxide (KOH) is used as activating agent in order to enhance surface area and pore structure of the mesoporous carbon [10]. After activation is completed, neutralization is applied in order to remove the excess KOH from the surface of OPEFB-derived activated carbon and to allow immobilization by K_3PO_4 catalyst during the calcination process.

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