

Facile preparation of Heterogeneous Silica-Based Catalyst for Transesterification of Palm Fatty Acid Distillate

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Abstract

In this study, heterogeneous silica-based catalyst was prepared from rice husk using a facile method for transesterification of palm fatty acid distillate (PFAD) into biodiesel. Huskcatacid and Huskcatbase were characterized using scanning electron microscopy (SEM), Energy Dispersive X-ray (EDX), Brunauer-Emmett-Teller (BET) surface area and pore size analysis. Results showed that Huskcatacid was efficiently employed in esterification of PFAD to afford 91.6% ester in 1:1 (PFAD:MeOH) and 5 wt% catalyst, followed by transesterification in 1:1 (oil: MeOH) using Huskcatbase (1 wt%) to produce 97.5% biodiesel in 30 min, with high percentage of methyl palmitate (34.43%) and methyl oleate (57.86%). Both Huskcatacid and Huskcatbase have high porosity (2.726×10^{-3} cm³/g and 4.985×10^{-3} cm³/g) and surface area (7.362 m²/g and 14.493 m²/g), respectively, which offer efficient esterification, easy glycerol separation and reusability up to three times to afford biodiesel ranging from 90-97% yield. The biodiesel obtained was applied on Megatech®-Mark III engine and demonstrated a proportional increased of torque (r) with the B100 biodiesel loading. Rice husk demonstrated a good potential as solid support heterogeneous catalysts and feedstock for value-added products, which also assists to overcome agricultural waste management issues.

Keywords: *rice husk; heterogeneous; catalysts; esterification; acid, base; biodiesel*

1. INTRODUCTION

The demand for sustainable alternative to petroleum diesel fuel is increasing due to the depletion of fossil resources (Huang et al., 2012). Biodiesel produced from vegetable oil was traditionally used as an alternative biodiesel feedstock. However, converting vegetable oil to biodiesel has caused great concerns due to food crisis issues. Consequently, non-edible biodiesel feedstock from agricultural waste are in high demand for biodiesel production (Chongkhong et al., 2007; Atadashi et al., 2010). For example, palm fatty acid distillate (PFAD), an agricultural waste massively generated from palm oil industry is a potential biodiesel feedstock attributed to its high free fatty acids (FFAs) content (72.7 – 92.6%) with triglycerides residues (Zahan and Kano 2018).

Catalyst plays a vital role in the transesterification reaction of free fatty acids (FFA) to form biodiesel. Sulfuric acid (H₂SO₄) and hydrochloric acid (HCl) are common acid catalysts used to accelerate the transesterification process for a higher yield of biodiesel (Chongkhong et al., 2007; Canakci and Van Gerpen, 1999; Fadhil et al., 2012; Zuo et al., 2013). Major issues of acid catalysts are the corrosive effect on the reactor and high amount of alcohol required, which incur high production cost (Leung et al., (2010). Homogeneous base-catalyst such as sodium hydroxide (NaOH) and potassium hydroxide (KOH) are employed as an alternative to acid catalyst in transesterification process to exhibit faster and non-corrosive reaction (Ma and

Hanna (1999). Employing homogeneous base catalyst, however, suffers disadvantage due to saponification effect which reduce the yield of biodiesel (Leung et al., 2010; Romero et al., 2011).

Heterogeneous base-catalyst such as calcium oxide and silica has been reported to produce a higher yield of biodiesel compared to homogeneous base catalyst (Leung et al., 2010). Nevertheless, the catalyst has a difficulty to diffuse into the product mixture to form oil, alcohol and catalyst (Mbaraka and Shanks, 2006). This limitation has led to the introduction of catalyst support to overcome the weaknesses of heterogeneous catalyst (Mbaraka and Shanks, 2006; Zabeti et al., 2010). Catalyst support is a solid carrier impregnated with an active species (Mabena et al., (2011). NaOH/Al₂O₃, NaOH/SiO₂ and ZS/Si are examples of catalyst supports used in the production of biodiesel (Taufiq-Yap et al., 2011; Jacobson et al., 2008; Arzamendi et al., 2007). The incorporation of active species into catalyst support can increase the reaction with triacylglycerol (Zabeti et al., 2010).

We have reported on using *Imperataacid* and *Imperatabase* as silica-based heterogeneous catalysts from *Imperata cylindrica* (*I. cylindrica*) for a facile transesterification reaction of palm oil mill sludge (POMS) (Ngaini et al., 2016). The high porosity of silica in *I. cylindrica* offers convenient esterification and transesterification in higher percentage yield. A very low amount of silica content (1-8%) in *I. cylindrica* has led our study to search for agricultural waste with higher silica content for preparation of catalyst support. Rice husk has been reported to have high silica content (90-97%) (Della et al., 2002) and is abundantly available as agricultural waste in South East Asia, especially Malaysia.

Herein we report on the production of biodiesel from PFAD employing silica-based catalyst support from rice husk impregnated with H₂SO₄ and NaOH as solid acid catalyst (SiA) and solid base catalyst (SiB), respectively. The formation of biodiesel from PFAD was performed *via* esterification using SiA followed by transesterification using SiB. The biodiesel produced was evaluated based on several reaction parameters such as oil to methanol ratio, amount of catalyst, reaction times and temperature and the optimization of parameters was carried out.

2. EXPERIMENTAL

Materials

PFAD was obtained from Bau Palm Oil Mill, Sarawak. Rice husk was obtained from Selangor, Malaysia, washed and air dried prior to use. Sodium hydroxide pellets (NaOH), sulfuric acid (H₂SO₄) 99% and methanol were purchased from Merck and used as received.

Preparation of *Huskatacid* and *Huskatbase*

Dried rice husks (1000g) was heated at 800 °C to form calcined rice husk (RHA), and further treated with NaOH (20%) to produce activated ash ARHA (200g). The activated ash was impregnated with concentrated H₂SO₄ *vi* a sulfonation for 24 h. The excess acid was decanted, and the residual wet solids were heated at 120 °C for 18 h (Kaster et al., 2012) to form heterogeneous *Huskatacid*.

Solid base catalyst *Huskatbase* was prepared by soaking the activated ash (RHA) in NaOH solution (5M) for 2 h, followed by filtration. Sample was then heated at 700 °C for 10 min (Perrichon and. Durupty, 1988; Refaat, 2011) to afford *Huskatbase*.

Characterization of *Huskatacid* and *Huskatbase*

The X-Ray Diffraction (XRD) patterns of calcined ash was obtained on a Rigaku Miniflex X-ray diffractometer equipped with a scintillation counter to generate pattern at ambient temperature. Surface morphology of the catalyst was observed under Scanning Electron Microscope (SEM) (JEOL 7500F-1) with magnification

10000x. Quantitative element composition of the catalysts was characterized using Energy Dispersive X-ray (EDX) (JEOL JSM-6390 LA). Specific surface area and total pore volume of the catalysts were determined using Brunauer-Emmett-Teller (BET) (Quantachrome ASIQC0000-3) with liquid nitrogen adsorption at 77 K.

Esterification and transesterification reaction of PFAD

Due to high content of FFAs, the production of biodiesel from PFAD was performed *via* esterification followed by transesterification process. During esterification, *Huskcatacid* (0.5 g, 5%) was added into methanol (10.0 mL) and heated at 65 °C for 1 h in a round bottom flask. Preheated PFAD (10.0 g) was added into the flask and heating was continued for 24 h at 120 °C. The mixture was cooled to room temperature, filtered, and washed with methanol. The filtrate was evaporated to eliminate water and excess methanol to obtain esterified PFAD as a brown oil.

In transesterification process, *Huskcatbase* (1%, 0.1 g) was added into a 10.0 mL methanol and heated at 65°C for 1 h in a 3-neck round bottom flask. The esterified oil (10.0 g) was added into the flask and continued heating for 1 h. The mixture was cooled to room temperature, filtered, and washed with methanol. The filtrate was evaporated to remove excess methanol and formed layers of glycerol and biodiesel [23], whereby the upper layer was collected as biodiesel. The effect of reaction times (15 min, 30 min, 45 min, 60 min) employing ratio 1:1 (methanol: oil) and 1% of catalyst was investigated. The acid value and FFA (%) of the esterified oil was calculated using Equation 1 and Equation 2 (Banani et al., 2015).

$$\text{Acid value (mgKOH/g)} = (N_{\text{KOH}} \times \text{MW}_{\text{KOH}} \times V_t) / W_s \text{ Eq. 1}$$

where, N_{KOH} is the normality of KOH used, MW_{KOH} is the molecular weight of KOH (56.11 g/mol), V_t is the volume of titrant used, and W_s is the mass of sample, g.

$$\text{FFA \%} = \text{AV}_s / 2 \text{ Eq. 2}$$

where, AV_s = Acid value of PFAD, mgKOH/g

Analysis of Biodiesel

The esterified oil and biodiesel analysis were examined using Fourier Transform Infrared (FTIR) (Thermo Scientific Nicolet iS10) with zinc-selenide (Zn-Se) disc as the transmitter. The composition of the biodiesel was analyzed using Gas Chromatography Mass Spectroscopy (GCMS) (Shimadzu GC-MS model QP2010 Plus) with helium as gas carrier with constant flow rate at 0.94 mL/min and linear velocity at 36.6 cm/sec. Chromatographic separation was operated on a nonpolar capillary column, BPX-5 (30.0 m x 0.25 µm x 0.25mm). MS was operated using electronic impact (70 eV) in selected ion monitoring (SIM) mode. The injector port was operated in splitless mode. The temperature of both injector and interface were at 250 °C. The oven temperature was maintained at 10 °C/ min, which initiated at 110 °C and held for 1 min. The temperature was increased to 220 °C at 20 °C/min and held for 8 min.

3. RESULTS AND DISCUSSION

Physicochemical Characterization of Silica-Based Heterogeneous Catalyst

The physicochemical characterization of the calcined ash (RHA), activated ash (ARHA), *Huskcatacid* and *Huskcatbase* was performed using XRD analysis as shown in Fig. 1. The calcination of rice husk at 800 °C afforded calcined ash as a white silica powder (Arzamendi, et al., 2007). All samples showed similar crystallinity pattern with the decrease in intensity of certain peaks, which indicate good dispersion of H_2SO_4 and NaOH on silica (Taufiq-Yap, et al., 2011). The calcined ash (Fig. 1a) showed a strong and sharp peak of cristobalite silica (SiO_2) at $2\theta=21.68$ (Taufiq-Yap, et al., 2011). A series of small peaks were detected at $2\theta=28.11$,

31.21 and 35.87 in the calcined ash which corresponded to traces of metal elements (Kumar et al., 2013). These peaks were reduced in the activated ash (Fig. 1b) ascribed the effects of NaOH to the activated ash. A sharp and well-defined peaks at $2\theta=25.05, 29.38, 34.93, 37.21, 48.08, 52.02$ and 65.68 were observed in *Huskcatabase* (Fig. 1c) due to cement effect of sodium loading on the activated ash during mineralization and crystallization of larger crystallites (Taufiq-Yap, et al., 2012; Kumar et al., 2013). Cement effect of sodium and H_2SO_4 acted by filling up the intergranular porosity of the silica and sticking the particles together, resulting of larger crystallites or crystalline structure of the catalysts (Zabeti, et al., 2009; Taufiq-Yap, et al., 2011). The XRD analysis shows a broad diffraction peak in *Huskcatacid* (Fig. 1d), which indicated that no substantial difference in the XRD pattern before and after sulfonation by H_2SO_4 on the activated ash. Similar observation has also been reported during impregnation of H_3PO_3 and H_2SO_4 onto carbon-based materials (Fu, et al., 2011; Fu, et al., 2012)

Figure 1

SEM analysis was performed on the calcined ash, activated ash, *Huskcatacid* and *Huskcatabase* to examine the surface morphology of the samples before and after treatment. SEM micrograph of activated ash (Fig. 2b) showed agglomerated particles and increased pores network and surface area after underwent activation process. SEM micrograph of *Huskcatabase* after impregnation with NaOH is shown in Fig. 2c with some assembled needle-like structures (Fu, et al., 2011; Arzamendi, et al., 2007). The morphology observed could be due to repeated washing of the sample which caused exfoliation to the outer surface and thus, exposing the internal structure. SEM micrograph of *Huskcatacid* (Fig. 2d) after sulfonation showed a disintegration of the surface caused by the partial destruction of the porous structure during impregnation, which resulted in larger pores (Janaun and Ellis, 2011). Large pore structure has been reported to indirectly improve catalytic activity (Shu, et al., 2009) during esterification.

Figure 2

The EDX analysis on the elemental composition of the calcined ash, activated ash, *Huskcatacid* and *Huskcatabase* showed the presence of silica (SiO_2) as the main element detected in all the samples (Table 1). The successful impregnation showed by the presence of sulfur (3.25%) and sodium (0.40%) in *Huskcatacid* and *Huskcatabase*, respectively.

Table 1

BET analysis showed that the surface area and total pore volume of calcined ash was increased from $1.914 m^2/g$ to $18.947 m^2/g$ after activation process (Table 2). The activation process has apparently caused the formation and enlargement of pores, as observed in SEM, which increase the surface area of the activated ash. The surface area of *Huskcatabase* ($14.493 m^2/g$) and *Huskcatacid* ($7.362 m^2/g$) was found to be lower than the activated ash. The introduction of active species such as alkali metals and sulfonation onto a support material has reduced the surface area of the catalyst (Perrichon and Durupt, 1988; Janaun and Ellis, 2011).

Table 2

Esterification of PFAD with Methanol

Effect of Catalyst Loading

The nature of PFAD with higher FFA content has undergone a two-step process which are esterification of PFAD, followed by transesterification of the oil with methanol (MeOH) (Shu, et al., 2009). In the initial stage, the esterification of PFAD was performed in 1:5 wt% (PFAD:MeOH) using *Huskcatacid* (1 wt%) under reflux for 24 h (Gole and Gogate, 2012). The effect of *Huskcatacid* loading during esterification of PFAD is illustrated in Fig. 3. The FTIR spectra showed two peaks at $1706 cm^{-1}$ and $1743 cm^{-1}$ attributed to $-COOH$ and $-COOR$, respectively, which indicated an incomplete esterification of the carboxyl group (Fig. 3b) (Yong-Ming, et al., 2014). An increased amount of *Huskcatacid* to 5 wt% showed a complete formation of ester by the appearance of a sharp peak at $1742 cm^{-1}$ and disappearance of peak at $1706 cm^{-1}$ (Fig. 3c).

Figure 3

Effect of PFAD to Methanol

A minimum amount of MeOH was investigated for the esterification of PFAD using 5 wt% *Huskcatacid* under reflux for 24 h. Incomplete esterification was observed in 1:0.5 (PFAD:MeOH) by the appearance of -COOH peak at 1709 cm^{-1} . A complete formation of ester (Fig. 4b) occurred in 1:1 (PFAD:MeOH) in the presence of 5 wt% *Huskcatacid*. The influence of PFAD to MeOH ratio is illustrated in Fig. 4.

Figure 4

Effect of reaction time

Reaction time is crucial and incur the production cost of biodiesel. The esterification was optimized hourly with constant ratio 1:1 wt% (PFAD:MeOH) and 5 wt% *Huskcatacid* (Fig. 5). FTIR spectra showed a mixture of COOH and COOR (ester) peaks presence after 1-1.5 h (Fig. 5a). Further increased of the reaction time to 2 h (Fig. 5b) afforded esterified oil at 1741 cm^{-1} with yield 91.6%. A reversible reaction is believed to occur during 24 h esterification (Moser, 2009; Chongkhong, et al., 2007; Kumar, et al., 2013).

Figure 5

The esterified oil was characterized using GCMS and shown in Table 3. No significance carboxyl group was detected in the esterified oil as compared to PFAD. The palmitic acid, for instance, was completely converted into methyl palmitate (Table 4).

- Table 3
- Table 4

The acid value of the esterified oil was 6 mgKOH/g with 3% of FFA. FFA value in the range of 3% indicates that the sample is suitable to undergo base catalysed-transesterification reaction (Meher, et al., 2006).

Transesterification of PFAD

Effect of catalyst loading

Huskcatbase was employed in the transesterification of the esterified oil at various catalyst loading (0.5 -5%) with constant reaction temperature and oil to methanol ratio (Table 5). Further increase of *Huskcatbase* (5 wt%) was observed to produce undesirable viscous, jelly-like oil, and turned cloudy upon addition of water during separation of biodiesel (Fig.6a). This could be due to saponification phenomenon (Moser, 2009) owing to the possible sodium leaching from the catalyst during reaction (Arzamendi, et al., 2007). The optimum amount of *Huskcatbase* (1 wt%) for 1 h contributed to the formation of biodiesel and glycerol as by-product (Fig. 6b).

- Table 5
- Figure 6

Effect of reaction time

Reaction time is another crucial factor in biodiesel production process. Different reaction times were monitored for the transesterification of esterified oil with methanol (1:1) and *Huskcatbase* (1 wt%) as a catalyst (Gole and Gogate, 2012). The conversion of esterified oil to biodiesel was monitored with the gradual formation of glycerol, which formed after 15 min and constant at 30-60 min. The formation of biodiesel was increased from 96.7% (15 min) to 97.5% (30 min) and decreased after 60 min (Fig. 7). Reusability of the catalyst up to three times afforded biodiesel ranging from 90-97% w/w. The percentage conversion (%) of biodiesel from esterified oil was determined based on Equation 4 (Banani, et al., 2015).

$$\text{Conversion, \%} = \frac{\text{Weight of biodiesel produced, g}}{\text{Weight of feedstock, g}} \times 100\% \text{ Eq.4}$$

Figure 7

Calorific Value of Biodiesel

The efficiency of the PFAD biodiesel was characterized based on the calorific values (CV) of the neat biodiesel (B100), 5% biodiesel to 95% commercial diesel (B5), commercial B5 (CB5) and commercial diesel fuel (CD) using Oxygen Bomb Calorimeter (Parr 6400) (Sivaramakrishnan and Ravikumar, 2012; Lang, et al., 2001). The CV of B100 was 39.35 MJ/kg while CV of B5 was increased to 45.07 MJ/kg. Based on the CV obtained for CB5 (45.53 MJ/kg) and CD (45.67 MJ/kg), it can be deduced that B5 showed a comparable CV and efficiency which is acceptable as a diesel substitution.

Table 6

GCMS analysis

The composition of the biodiesel produced via two step reactions (esterification and transesterification) was analyzed using GCMS. Table 7 shows higher percentage of methyl palmitate (34.43%) and methyl oleate (57.86%) from the PFAD biodiesel. This phenomenon is not surprising as PFAD has higher amount of palmitic acid (73.99%) and oleic acid content (13.25%) in as compared to other types of fatty acids (Sugiarto, et al., 2015). No significant carboxyl group was detected in PFAD biodiesel. In other words, initial esterification of PFAD improved the ability to form complete transesterification of PFAD for a high biodiesel yield (Ngaini et al, 2026).

Table 7

Combustion capability test

The combustion capability test of PFAD biodiesel (B100) was demonstrated on the alternative diesel engine (Megatech- Mark III) (Fig. 8). Ethanol was initially introduced into the combustion chamber to achieve 400-500°C before B100 was introduced. A bright orange flame with less smoke was released upon combustion of B100 in the chamber in 10-15 mins as compared to CD, which normally emitted black smoke due to incomplete combustion (Akihama, et al., 2002). A slight, odourless smoke was produced during combustion of B100 indicated a sufficient and complete combustion of the engine.

Figure 8

The performance of fuel using diesel combustion engine was demonstrated based on torque, r, load and revolution per minute (rpm) (Fig. 9), where torque was proportionally increased with the B100 loading. This observation indicated that the engine was able to operate up to the maximum load of 100 lbs using B100 PFAD. The rpm was decreased when the load of B100 increased. This is because high force (torque) is needed to endure the load and cause the rpm to decrease (Ngaini et al, 2016).

Figure 9

4. CONCLUSION

Huskcatacid and *Huskcatbase* have been successfully developed from rice husk ash and employed as heterogeneous solid support for the production of biodiesel from PFAD. The catalysts were conveniently prepared and employed in the esterification and transesterification reaction to afford high yield of biodiesel (>97% yield) with reusability up to three times. The biodiesel performance was demonstrated on alternative diesel engine with only small amount of smoke emission compared to the commercial diesel. This study indicated that rice husk demonstrated a good potential as solid support heterogeneous catalysts and feedstock for value-added products, which also assists to overcome agricultural waste management issues.

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