Research Article

Effects of Weight Hourly Space Velocity and Catalyst Diameter on Performance of Hybrid Catalytic-Plasma Reactor for Biodiesel Synthesis over Sulphated Zinc Oxide Acid Catalyst

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Abstract

Biodiesel synthesis through transesterification of soybean oil with methanol on hybrid catalytic-plasma reactor over sulphated zinc oxide (SO_4^{2-}/ZnO) active acid catalyst was investigated. This research was aimed to study effects of Weight Hourly Space Velocity (WHSV) and the catalyst diameter on performance of the hybrid catalytic-plasma reactor for biodiesel synthesis. The amount (20.2 g) of active sulphated zinc oxide solid acid catalysts was loaded into discharge zone of the reactor. The WHSV and the catalyst diameter were varied between 1.55 to 1.546 min^{-1} and 3, 5, and 7 mm, respectively. The molar ratio of methanol to oil as reactants of 15:1 is fed to the reactor, while operating condition of the reactor was kept at reaction temperature of 65 °C and ambient pressure. The fatty acid methyl ester (FAME) component in biodiesel product was identified by Gas Chromatography - Mass Spectrometry (GC-MS). The results showed that the FAME yield decreases with increasing WHSV. It was found that the optimum FAME yield was achieved of 56.91 % at WHSV of 0.895 min^{-1} and catalyst diameter of 5 mm and reaction time of 1.25 min. It can be concluded that the biodiesel synthesis using the hybrid catalytic-plasma reactor system exhibited promising the FAME yield. Copyright © 2017 BCREC Group. All rights reserved

Keywords: Biodiesel; Sulphated zinc oxide; Hybrid catalytic-plasma reactor; Transesterification

1. Introduction

Biodiesel is a promising renewable energy replacing fossil without requiring modifications to the engines and produce environmental benign emissions. Biodiesel has higher cetane number than petroleum diesel, biodegradable, contains no sulphur and aromatic compounds and resulting in lower emissions than petroleum diesel [1-4]. Various methods have been employed for biodiesel production, both conventional [5-8], enzymatic [9-11], or even advanced technologies [12-16] methods. Conventionally, biodiesel was produced by homogeneous and heterogeneous catalysts, either acid or base catalysts as previously found [5-8]. However, transesterification reaction using homogeneous

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catalysts are constrained by the complexity of the purification and separation steps [17,18]. Those problems on homogeneous catalysts can be solved by heterogeneous catalysts. In the heterogeneous catalysts, purification processes would be simpler due to catalysts and product phase difference. Therefore, the catalysts can be easily regenerated and recycled and reused several times tailoring minimum material and process cost. Also, this process is environmentally friendly and could be applied to a batch or continuous process [19-21].

Many researchers developed batch and continuous reactor systems over heterogeneous catalysts for the biodiesel production [21-28]. Endalew *et al.* [21] studied solid base and acid catalysts for transesterification in a batch reactor. However, soap formation was still found to be the main problem during the reaction. Transesterification in a batch reactor using supercritical methanol was investigated by Olivares-Carrillo and Quesada-Medina [23]. Unfortunately, use of this supercritical batch reactor was less economical due to requiring very high temperature (350 ^{o}C) and pressure (36-41 MPa). For the case of continuous reactor system, a fixed bed tubular continuous reactor to produce biodiesel has been developed by Hsieh *et al.* [24]. Other researchers, Feng *et al.* [25] and Ren *et al.* [26], proposed continuous reactor using fixed bed reactor for biodiesel synthesis which focused on continuous esterification and continuous transesterification, respectively. However, the continuous fixed bed catalytic reactor systems have some weaknesses, such as: water content in oil hindered the ester formation with the esterification, deactivating catalyst active sites, and hydrolyzing FAME. These problems would decrease the yield of FAME and slow down the reaction rate [29]. Also, the transesterification reaction using heterogeneous catalyst over fixed bed tubular reactor still needs longer residence time.

Recently, use of plasma technology is promising and advanced on production of biodiesel due to shorter reaction time significantly. Lawson *et al.* [30] and Istadi *et al.* [15] developed electro-catalysis system in biodiesel production. They reported that high energetic electrons would collide with the reaction mixtures and interfere electrons pairs in covalent bonding within reactant molecules which in turn excitation or even dissociation would be occurred. However, difficulty in controlling the reaction mechanism within the plasma electro-catalysis system was existed due to continuing uncontrolled reactions due the action of high energetic electron and difficulty in controlling

which covalent bonds would be excited or dissociated. Regarding improving the problems, Buchori *et al.* [16] developed hybrid catalytic-plasma reactor system to improve the selectivity of biodiesel, especially fatty acid methyl esters. They reported that the high energetic electrons collided with reactants molecules on the catalyst surface of catalyst. The results showed that biodiesel yield reached 92.39 % and 73.91 % by using active carbon and modified active carbon catalysts, respectively.

This research aims to study effects of weight hourly space velocity (WHSV) and catalyst diameter on performance of hybrid catalytic-plasma reactor for biodiesel synthesis over sulphated zinc oxide acid catalyst. Yield of FAME product over the hybrid catalytic-plasma reactor is compared to that of fixed bed catalytic reactor without plasma.

2. Materials and Methods

2.1. Materials

In this research, soybean oil (Happy Soya Oil) and methanol (Merck, 99.9 %) were used as raw materials. Commercial soybean oil was purchased from local market. Sulfuric acid (H_2SO_4, Merck, 95-97 %) was used as catalyst pretreatment of commercial zinc oxide (Merck, 99 %).

2.2. Catalyst preparation

The sulphated zinc oxide was prepared by impregnation of zinc oxide within H_2SO_4 solution 2 M for 3 h at room temperature (28 ^{o}C). The mixed solid was dried at 110 ^{o}C in a Memmert oven for overnight. The resulted product was calcined at 300 ^{o}C for 3 h in a box furnace. The calcined catalyst was crushed, milled and then made in the form of pellets with diameter of 3, 5, and 7 mm, respectively.

2.3. Hybrid catalytic-plasma reactor system

Biodiesel synthesis was performed in a hybrid catalytic-plasma reactor using Dielectric-Barrier Discharge (DBD) type. The plasma reactor was consisted of high voltage and ground electrodes as well as a glass plate as dielectric barrier. The reactor was equipped with electric furnace, temperature control, DC type high voltage power supply (power up to 330 Watt), high voltage probe (SEW PD-28), and Sanwa multimeter. The temperature control was used to adjust the reactor temperature, while the high voltage power supply is used to provide

high voltage on the reactor electrodes giving energetic electrons flow between the electrodes. A Sanwa multimeter was used to measure voltage during the biodiesel synthesis process. The glass barrier was purposed to act as a dielectric and to distribute the electrons from high voltage to ground electrodes. Catalyst was placed into tubular reactor within discharge zone. Distance between high voltage electrode and ground electrode was 1.27 cm.

2.4. Procedures of transesterification reaction over hybrid catalytic-plasma reactor

Transesterification reaction in biodiesel synthesis was conducted by mixing the reactants, i.e. soybean oil and methanol with methanol/oil molar ratio of 15:1 [16,31]. Then, the mixture of methanol and oil was flown over sulphated zinc oxide catalyst (SO_4^{2-}/ZnO) within the plasma reactor. The reactor was heated up to 65 °C at atmospheric pressure which in turn a 7 kV DC high voltage was applied on the discharge zone. The high voltage electrode was connected to high voltage probe, while the ground electrode was connected to earth ground. The high voltage electrode flew high energetic electrons toward ground electrode through the glass dielectric barrier. The energetic electrons collided to the liquid reactants molecules that flow over the surface of catalyst pellet. Effect of the reactant flowrate on reactor performance was studied by varying Weight Hourly Space Velocity (WHSV), i.e. 0.895, 0.974, 1.069, 1.264, and 1.546 min^{-1}, while the methanol to oil molar ratio, catalyst diameter, and voltage were kept constant at 15:1, 5 mm, and 7 kV, respectively. Effect of catalyst diameter on reactor performance was investigated by varying diameter as 3, 5, and 7 mm, while the methanol to oil molar ratio, WHSV, and voltage were kept constant at 15:1, 0.895 min^{-1}, and 7 kV, respectively.

2.5. Characterizations of biodiesel product and catalyst

To determine content of fatty acid methyl esters from resulted product, the reactor products were analyzed by Gas Chromatography-Mass Spectrometry (GC-MS) (QP2010S SHIMADZU, DB-1 column). Within the GC-MS, the samples were analyzed with an oven temperature of 50 °C (5 minutes) and raised 10 °C min^{-1} up to a temperature of 260 °C and held for 33 minutes.

Yield of FAME can be calculated using Equation (1), while yield of biodiesel can be calculated in Equation 2).

$$Yield_{FAME} = \frac{\%FAME\,GC\,area\,x\,\rho_{biodiesel}\,xV_{biodiesel}}{weight\,of\,soybean\,oil\,feed}\,x100\% \quad (1)$$

$$Yield_{FAME} = \frac{\rho_{biodiesel}\,xV_{biodiesel}}{weight\,of\,soybean\,oil\,feed}\,x100\% \quad (2)$$

Surface area of the catalysts (powder, diameter of 3, 5, and 7 mm, respectively) were analyzed by BET (Brunauer–Emmett–Teller) method. The surface area measurements were conducted using Nitrogen adsorption-desorption at -196 °C (77 K) in a Quantachrome. The samples were degassed for 5 h at 300 °C (573 K) under vacuum pressure (6.58 × 10^{-5} Torr) before the sorption measurements.

3. Results and Discussion

3.1. Effects of weight hourly space velocity on reactor performance

Weight Hourly Space Velocity (WHSV) is the mass flow rate of the reactants divided by the mass of the catalyst in the reactor. Effect of WHSV on performance of hybrid catalytic-plasma reactor is shown in Figure 1. Dependent variables of reaction performance of biodiesel synthesis is presented as yields of FAME and biodiesel. From the Figure 1, FAME yield decreases with increasing WHSV. The higher WHSV the shorter contact time between energetic electrons and reactant molecules. The contact time is the length of time required by high-energetic electrons to contact with reactant molecules. The principle of plasma system is collision between high energetic electrons, flows from high voltage electrode, and electron pairs from covalent chemical bonding in reactants. The high energetic electrons would interfere the electrons pairs from covalent bonding of the reactant to become excited or dissociated so that the activation energy required by catalyst surface reaction could be reduced [15,16,30]. Shorter contact time leads to shorter the collision process so that only a few electron pairs of covalent bonding in reactant molecules are excited. This lower contact time between the energetic electrons and the reactant molecules affects on lowering yield of FAME or biodiesel.

Figure 2 presented effect of reaction time on yield of FAME. The reaction time is the amount of time required by the reactants (fluid) to react along the catalyst bed. In this

research, the highest yield of FAME achieved up to 56.91 % within 1.25 minutes reaction time. This results showed that the hybrid catalytic-plasma reactor system is promising due to producing high yield of FAME or biodiesel in a very short time. In comparison, the reaction time required to produce yield of FAME or biodiesel in a catalytic fixed bed reactor (without plasma) is very long (1-4 hours). De Moura *et al.* [32] need 3 hour reaction time to produce biodiesel yield of 98.54 % on methanolysis of babassu oil used strontium oxide (SrO) as catalyst at room temperature. Hsieh *et al.* [24] achieved 95% yield of FAME over $Ca(C_3H_7O_3)_2/CaCO_3$ catalyst with the reaction temperature of 60 °C using conventional reactor. On transesterification of soybean oil over fixed bed reactor found by Ren *et al.* [26], the

conversion of biodiesel achieved 95.2 % within a residence time of 56 min at temperature reaction of 50 °C and feed flow rate of 1.2 mL/min. Shibasaki-Kitakawa *et al.* [33] investigated a continuous transesterification of crude triolein with ethanol in fixed bed reactor with conversion achieved 80 % at residence time of 60 min. Ketchong *et al.* [28] studied transesterification process to produce FAME using limestone as a catalyst in a fixed bed reactor with FAME yield of 94.1 wt.% and kept constant over 1500 min of reaction time. In continuous esterification processes by Feng *et al.* [25], conversion reached over 98 % within 500 h running time in a fixed bed. Comparison of FAME yield obtained and the reaction times by researchers is exhibited in Table 1.
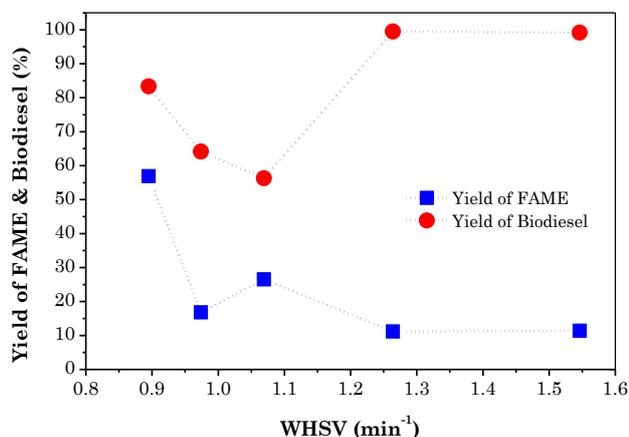

Figure 1. Effect of weight hourly space velocity on the FAME yield in hybrid catalytic-plasma reactor using SO_4^{2-}/ZnO catalyst at 65 °C, atmospheric pressure, alcohol:oil molar ratio 15:1, catalyst diameter of 5 mm, and 7 kV DC high voltage

Figure 2. Effect of reaction time on the FAME yield in hybrid catalytic-plasma reactor using SO_4^{2-}/ZnO catalyst at 65 °C, atmospheric pressure, alcohol:oil molar ratio 15:1, catalyst diameter of 5 mm, and 7 kV DC high voltage
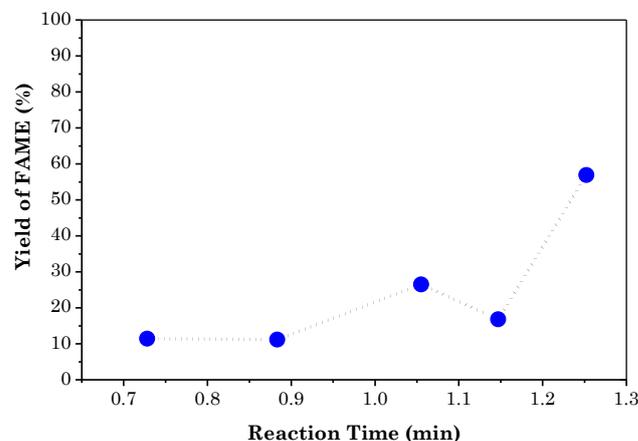
Table 1. Yield of FAME (i.e. biodiesel) obtained with the reaction time in fixed bed catalytic reactor

Yield of FAME (i.e. biodiesel) (%)	Reaction time (min)	References
93	600	Ketcong *et al.* [28]
95.2	56	Ren *et al.* [26]
80	60	Shibasaki-Kitakawa *et al.* [33]
98	30000 (stably)	Feng *et al.* [25]
98.54	180	De Moura *et al.* [32]
95	168	Hsieh *et al.* [24]
75	300	Da Silva *et al.* [27]
95	1200	Furuta *et al.* [22]
56.91	1.25	This study (plasma reactor)

Istadi *et al.* [34] investigated transesterification process of soybean oil to fatty acid methyl ester (FAME) using sulphated zinc oxide as catalyst conducted in a batch reactor system. Yield of FAME obtained in this study was 75.5 % within 4 h reaction time. This reaction time when using conventional reactor system was longer compared to this research when using hybrid catalytic-plasma reactor that only required 1.25 min reaction time and resulting 56.91 % yield of FAME.

3.2. Effects of catalyst diameter on reactor performance

Catalyst diameter has a significant effect on the yield of FAME as presented in Figure 3. In the Figure 3, yield of FAME over catalyst diameter of 5 mm is higher than that of 3 mm and 7 mm. Surface area of the 5 mm diameter of catalyst (9.889 m^2g^{-1}) larger than that of 3 mm diameter (8.536 m^2g^{-1}) and 7 mm diameter (8.920 m^2g^{-1}). Surface area of the catalyst is shown in Table 2. The larger surface area of catalyst would produce higher adsorption capacity of reactant molecules providing more ad-
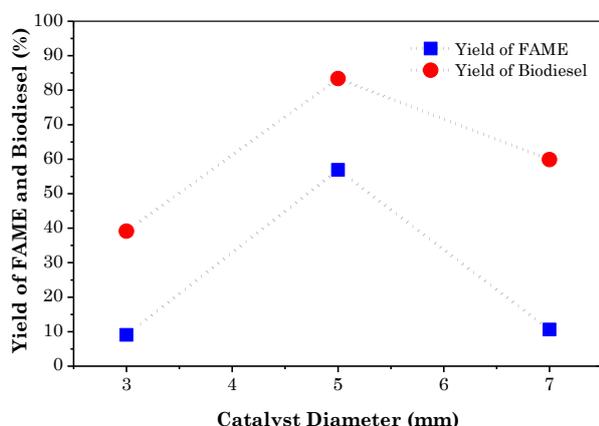

Figure 3. Effect of catalyst diameter on FAME yield over hybrid catalytic-plasma reactor using SO_4^{2-}/ZnO catalyst at 65 °C, atmospheric pressure, alcohol:oil molar ratio 15:1, WHSV of 0.895 min^{-1}, and 7 kV DC high voltage.

Table 2. Surface area of the catalyst

Catalyst diameter (mm)	Surface area (m^2g^{-1})
powder	10.677
3	8.536
5	9.889
7	8.920

sorption sites. In hybrid catalytic-plasma reactor system, the catalyst was directly in contact with energetic electrons within discharge zone generating reactive species (free radicals, excited atoms, molecules and ions) within plasma discharge zone and pores of the catalyst [16,35,36]. The formation of reactive species in the discharge zone and the pores of the catalyst would increase reaction activity between the adsorbed reactants on the catalyst surface and the active species which in turn forms FAME product.

The small surface area of catalyst would reduce contact surface area and decreases yield of FAME product. In the hybrid catalytic-plasma process, there is a synergetic effect between plasma systems with heterogeneous surface catalysis roles. The plasma has roles on providing high enough energy to interfere the electron pairs of covalent bonding of the reactant molecules that would be vibrated, excited, dissociated, and even ionized on the catalyst surface. The catalysts play a role on directing the selectivity of formation of fatty acid methyl ester products and controlling reaction mechanism within plasma discharge zone. Small contact surface area between electrons and the catalyst surface would decrease the role of catalyst which in turn reducing the synergistic effect of plasma and catalysis. This decrease of the catalyst role caused the uncontrollable reaction mechanisms and unavoidable continuing reaction, so that cracking the reactant bond chain (methanol and oil) becomes undesirable [15,37].

In the 3 and 7 mm diameter of catalyst, yield of FAME product decreases. This study has the same trend as the study carried out by Marlinda *et al.* [38] and Chen *et al.* [39]. Biodiesel conversion increased with the size of the catalyst to a peak before decreasing on a similar pattern. Optimum diameter catalyst found 5 mm that gives maximum yield of FAME.

The use of larger catalyst diameter for the same catalyst loading (weight) results in larger void fraction in the reactor. As the result, the larger discharge gap in a plasma reactor and eventually discharge volume increase. At that condition, the yield of FAME reaches the optimum point. The yield of FAME declines when the catalyst diameter is larger than the optimum diameter ($d > d_{opt}$) which is caused by a decrease in discharge power [40].

4. Conclusions

The hybrid catalytic-plasma reactor system can produce fatty acid methyl esters or biodiesel in a very short reaction time from soybean oil and methanol. The FAME yield achieved up to 56.91 % within 1.25 min reaction time, 5 mm catalyst diameter, and WHSV of 0.895 min^{-1}. As comparison, the transesterification process within batch reactor system (without plasma) with the same catalyst needed longer reaction time (up to 4 h). Also, the continuous fixed bed catalytic process also required longer reaction time to produce FAME or biodiesel product. This fact proves that the plasma activity plays important role in the transesterification process. Surface area of catalyst influences interactions between the plasma and the catalyst roles. Larger surface area of the catalyst would enhance the adsorbed reactants on the catalyst surface leading to increased reaction activity between adsorbed reactant molecules on the surface of catalyst and reactive species (free radicals, excited atoms, molecules and ions). The synergistic effect between plasma and catalyst roles would drive the transesterification reactions to produce FAME or biodiesel.

Acknowledgement

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