Intensification of Biodiesel Production from Palm Oil Using Rotating Tube Reactor

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Abstract

Biodiesel is an attractive renewable fuel to replace petroleum diesel. It is mainly produced via transesterification of triglyceride in oil and methanol to biodiesel or fatty acid methyl ester (FAME) and glycerol in presence of a catalyst. The reaction suffers from the immiscibility of oil and methanol, and therefore, the enhancement of mixing efficiency for the reaction mixture is required to achieve higher biodiesel yield. This work focuses on the development of a rotating tube reactor for intensifying the continuous biodiesel production from palm oil and methanol. The effects of various operating parameters (methanol to oil molar ratio and concentration of catalyst) on biodiesel yield were investigated. The suitable condition to achieve a biodiesel yield target of 96.5% was at a methanol to oil molar ratio of 6:1, 1 wt.% of sodium hydroxide (as a catalyst) and a rotational speed of 1,000 rpm at room temperature. The production rate and yield efficiency of the optimum condition were 23.5 mL/min and 22.9 x 10^{-4} g/J, respectively.

Keywords: Biodiesel; Transesterification; Process intensification; Rotating tube reactor; Yield efficiency

1. Introduction

Currently, the usage of renewable energy instead of the conventional energy becomes more reasonable because renewable energy can produce from a renewable resource such as biomass, solar and wind, etc. Biodiesel is one of a renewable energy resource which is produced from triglyceride and methanol in presence of an acid, base or enzyme catalyst. The main product and byproduct of biodiesel production via transesterification are fatty acid methyl ester (FAME) and glycerol, respectively (Gerpen 2005). The several reactions can produce biodiesel including, esterification of free fatty acid (FFA), transesterification of triglyceride (TG) or 2 steps of hydrolysis of TG-esterification-FFA. However, alkali catalyzed transesterification is a major reaction which is used to produce biodiesel in commercial because it is the highest efficiency process.

The limit of biodiesel production is the presence of mass transfer resistance derived from the immiscibility of the alcohol and vegetable oil resulting in the low reaction rate. To enhance heat and mass transfer, several researches study suggested the application of intensification technology to enhance the biodiesel production efficiency, including ultrasonic irradiation (US) (Stavarache, Vinatoru et al. 2005), microwave irradiation (MW) (Lertsathapornsuk, Pairintra et al. 2008), spinning disc reactor (SDR) (Qiu, Petera et al. 2012), and rotating tube reactor (RTR) (Holl 2010).

Rotating tube reactor (RTR) is one of the process intensification technology which can enhance the mixing performance and generate sufficient heat for biodiesel production. This RTR mainly composes of rotor and stator. The rotor operates at high rotational speed generating thin film of substances and leading to expand interfacial area of substances. Heat is also generated by shear force between rotor and stator (Qiu, Zhao et al. 2010).

The objective of this work is to study the performance and the energy consumption of RTR for biodiesel production through alkali-catalyzed transesterification under mild reaction conditions with various operational parameters such as methanol to oil molar ratio and concentration of catalyst.

2. Materials and methods

2.1 Materials

The main reactant in this study was refined palm oil and methanol. Refined palm oil in brand of Morakot industry co. Ltd. was purchased from local store in Thailand. Analytical grade methanol (99.8% purity) was provided by Loba Chemie Pvt. Ltd. Sodium hydroxide (NaOH) pellet (99.0% purity) was used as a homogeneous catalyst in this study and purchased from Merck company Ltd. For biodiesel yield analysis, heptane (99.5% purity) was used as solvent for GC analysis and purchased from Ajax Finechem Pty Ltd. Moreover, Methyl heptadecanoate (95% purity) used as internal standard for GC analysis following EN14103 was provided by Sigma-Aldrich.

2.2 Equipment

RTR consisted of 3 tubes: (1) an inner tube acted as the rotor that rotates rapidly inside a concentric stationary; (2) a middle tube which acted as the stator; and (3) an outer tube containing some heating/cooling fluid to control reaction temperature. The small gap between an inner tube and a middle tube was the reaction zone while the large gap between a middle tube and an outer tube was the heating/cooling zone. Rotor rotation generated thin film in the reaction zone, resulting in increase of the surface area of substances to enhance mass transfer. Furthermore, the rapid rotation of rotor in the small gap of reaction zone can generate shear stress leading to provide sufficient heat for biodiesel production.

The RTR prototype for continuous biodiesel production system is shown in Fig.1. The RTR connected to the 3-phase motor and used inverter to control the rotational speed. Two peristaltic pumps were used for feeding substances while three vessels were used to control oil, methanol/catalyst, mixture and products.

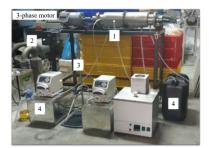


Fig.1 RTR prototype for continuous biodiesel production system including: (1) RTR, (2) AC motor and inverter, (3) Peristaltic pumps, and (4) Storage tank.

2.3 Biodiesel production in a continuous-flow rotating tube reactor

The experiments are carried out by using the different amount of NaOH catalysts (1 and 1.5 wt.%). NaOH was homogeneously mixed with methanol at room temperature (RT). Then, the container of methanol/catalyst mixture and refined palm oil was connected to the inlet port of RTR. The substances were pumped into RTR using peristaltic pumps. Methanol to oil molar ratio of 3:1, 4.5:1, 6:1, and 9:1 were examined. Total feed flow rate was fixed at 30 mL/min. The rotational speed of 1,000 and 1,200 rpm of RTR were also used and controlled by inverter. The reaction operated at atmospheric pressure and room temperature for 180 min and sample was collected at 15, 30, 45, 60, 90, 120, 150, and 180 min to confirm the steady state condition. The container of collecting sample was submerged in cool water to stop the reaction. Then the resulting reaction mixture was separated using gravity technique providing the different layer of unreacted methanol, fatty acid methyl ester from the reaction mixture and glycerol before being analyzed by gas chromatography (GC).

2.4 Biodiesel yield analysis

The biodiesel yield was analyzed following EN14103 using Shimadzu GC-2010 Plus, with DB-WAX capillary column and detected by flame ionization detector (FID).

2.4.1 Biodiesel yield calculation

Biodiesel yield which was defined as the methyl ester purity of biodiesel is calculated by Equation (1).

Biodiesel yield(%) =
$$\frac{(\Sigma A) - A_{IS}}{A_{IS}} \times \frac{C_{IS} \times V_{IS}}{m_S} \times 100$$
 (1)

Where (ΣA) , A_{IS} , C_{IS} , V_{IS} , m_S refer to total area of peak, area of methyl heptadecanoate (internal standard), concentration of methyl heptadecanoate (mg/mL), volume of methyl heptadecanoate (mL), and mass of biodiesel sample (mg), respectively.

2.4.2 Yield efficiency calculation

The yield efficiency was defined in Equation (2).

$$Yield_{eff} = \frac{m_{prod}}{P \times t}$$
(2)

Where $Y_{ield_{eff}}$, m_{prod} , P, t refer to yield efficiency, amount of product produced (g), power supplied (J/s), and reaction time (s), respectively.

3. Results and discussion

3.1 Effect of the molar ratio of methanol to oil

The effect of methanol to oil molar ratio on biodiesel yield of palm oil via transesterification using RTR is illustrated in Fig.2(a). The reaction was carried out at room temperature, catalyst loading of 1 wt.% NaOH, rotational speed of 1,000 rpm, and total flowrate of 30 mL/min. The results showed that NaOH catalyzed transesterification in RTR were reached steady stead at 60 min for methanol to oil molar ration of 3:1 to 6:1, while the conditions of methanol to oil molar ration of 9:1 was faster reached steady state at 30 min. The biodiesel yield increased from 86.75% to 94.41% when increased methanol to oil molar ratio from 3:1 to 6:1 in 60 min of reaction time. The stoichiometry of reversible transesterification, one mole of triglyceride required three moles of methanol to produce three moles fatty acid methyl ester (FAME) or biodiesel and one mole of glycerol. The excess methanol is required to shift the forward transesterification to produce more biodiesel. Actually, a molar ratio of methanol less than 6 might be insufficient to complete reaction because some of methanol dissolved in glycerol phase which results in only the remaining methanol in upper phase can react with palm oil to produce biodiesel (Choedkiatsakul, Ngaosuwan et al. 2014).

On the contrary, the excess of a molar ratio of methanol to oil from 6:1 to 9:1 was found to decrease the biodiesel yield from 94.41% to 58.76%. It is observed that the high molar ratio of methanol more than 6 leads to decrease biodiesel yield. This is more like due to the separation of biodiesel from glycerol became more difficult since hydroxyl group in methanol could contribute to the production of water, monoglyceride, and diglyceride resulting in emulsion formation (Eevera, Rajendran et al. 2009). Moreover, the increase of methanol molar ratio resulted to decrease the concentration of palm oil (triglyceride) in the reaction mixture resulting to lower transesterification rate (Chuah, Yusup et al. 2015).

Yield efficiency is an indicator to produce economical biodiesel which usually use to evaluate the performance of the reactor in the production process. Yield efficiency is defined as the amount of biodiesel product produced per energy consumed and time required. Fig.2(b) shows the yield efficiency based on 80% biodiesel yield using rotating tube reactor for palm oil. The condition was obtained catalyst concentration of 1 wt.% of NaOH, rotational speed of 1,000 rpm and total flowrate of 30 mL/min at room temperature. It has been observed that the yield efficiency increased from 20.0 x 10^{-4} g/J to 22.5 x 10^{-4} g/J when increasing the methanol to oil molar ratio from 4.5:1 to 6:1. These results were consistent with the biodiesel yield because the increase of methanol to oil molar ratio resulted in the increase of biodiesel yield which is the major parameter to enhance the yield efficiency. The previous study focused on the continuous process to produce methyl ester from palm fatty acid distillate (PFAD) using helical static mixers as reactors (Somnuk, Soysuwan et al. 2019). The results showed that the average energy consumption was 0.0473 kWh/L for producing 22.4 L of biodiesel. Therefore, the yield efficiency of biodiesel was approximately 21.69 x 10⁻⁴ g/J which is slightly lower than the yield efficiency of biodiesel production using RTR in this study. This could be related to the reaction temperature of our work was lower than that of previous work (Somnuk, Soysuwan et al. 2019).

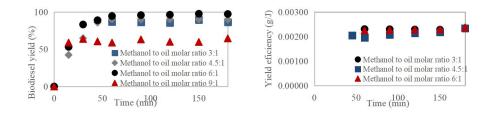


Fig.2 (a) Effect of the molar ratio of methanol to oil on biodiesel yields and (b) yield efficiency based on 80% biodiesel yield using RTR for palm oil. Catalyst concentration, 1 wt.% of NaOH; rotational speed, 1,000 rpm; total flowrate, 30 mL/min at RT.

3.2 Effect of the NaOH concentration

Using catalyst in the reaction is to enhance the reaction rate by generating the active species which conducts as the active site to obtain easily reaction (Mohod, Gogate et al. 2017). Fig.3(a) demonstrates that the effect of the NaOH concentration on biodiesel yield of palm oil via transesterification using RTR. The reaction condition was obtained methanol to oil molar ratio of 6:1, rotational speed of 1,200 rpm, and total flowrate of 30 mL/min at room temperature. The results founded that using different NaOH concentration provided the different steady state as 45 and 90 min for NaOH of 1 and 1.5 wt.%, respectively. Moreover, the biodiesel yield was increased from 18.79 to 91.22% using1 wt.% and 1.5 wt.% NaOH, respectively at 90 min of reaction time. It was observed that lower NaOH concentration was insufficient active sites to catalyze transesterification resulting to obtain lower biodiesel yield. The biodiesel yield was increased when NaOH concentration increases dealing with the enhancement in the rate of reaction. Generally, NaOH concentration between 0.4 and 2 wt.% is an appropriate condition for transesterification of palm oil. However, the biodiesel yield was decreased using the excess NaOH concentration due to the presence of side reaction (saponification) or increase the viscosity of reaction mixture (Meher, Vidyasagar et al. 2006, Gnanaprakasam, Sivakumar et al. 2013, Talebian-Kiakalaieh, Amin et al. 2013). This should be noted that biodiesel yield obtained from different rotational speed of 1,000 and 1,200 were also different. Therefore, the effect of RTR on biodiesel yield and yield efficiency should be further investigated.

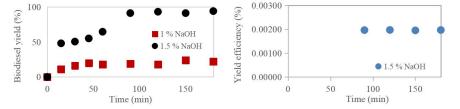


Fig.3 (a) Effect of the NaOH concentration on biodiesel yields and (b) yield efficiency based on 80% biodiesel yield using RTR for palm oil. The molar ratio of methanol to oil, 6:1; rotational speed, 1,200 rpm; total flowrate, 30 mL/min at RT.

Fig.3(b) illustrated the effect of the NaOH concentration on yield efficiency based on 80% biodiesel yield. The results showed that yield efficiency was $19.7 \times 10^{-4} \text{ g/J}$ using 1.5 wt.% of NaOH. It was also noted that the yield efficiency using 1 wt.% of NaOH

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could not determine because the biodiesel yield obtained from this condition was lower than 80%.

4. Conclusions

The presence work revealed the invention and operating condition of using RTR to produce biodiesel from palm oil at room temperature. RTR is one of the process intensification technology which is appropriate for transesterification because it can enhance the mixing efficiency of the insoluble reaction mixture (oil and methanol) by reducing heat and mass transfer resistance. Methanol to oil molar ratio and NaOH concentration were significantly effect on the biodiesel yield and yield efficiency. To achieve a biodiesel yield target of 96.5%, the methanol to oil molar ratio of 6:1 and 1 wt.% of NaOH (as a catalyst) and a rotational speed of 1,000 rpm at room temperature was carried out. This condition provided the high yield efficiency was 22.9 x 10^{-4} g/J. However, the higher biodiesel yield and yield efficiency could be produced in this RTR by varying the rotational speed, total feed flowrate and reaction temperature which should be further studied.

5. Acknowledgement

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