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Transesterification of Palm Oil with Methanol in a Millichannel Reactor: Effects of Methanol to Oil Molar Ratio and Reaction Temperature

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ABSTRACT

Biodiesel, a biomass-derived fuel, is becoming increasingly important due to the increase in the price of petroleum and the environmental concerns about the pollution that release to the environment by vehicles and factories. Recently, millichannel reactor has become one of the methods to produce biodiesel because it can overcome several issues in the traditional approached of biodiesel production. In this paper, transesterification of palm oil using millichannel reactor was conducted to study the effect of methanol to oil molar ratio (6:1 to 23:1) and reaction temperature (40-65 °C), for two different internal tube diameters; 1.6 and 1.0 mm on the FAME yield. Results showed that the highest FAME yield was at 21:1 methanol to oil molar ratio and the temperature was 60 °C with internal diameter 1.0 mm.

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INTRODUCTION

Biodiesel is becoming essential due to the environmental concerns of exhaust gases from petroleum diesel fuels. Biodiesel is biodegradable and non-toxic product. It is made from renewable sources such as palm oil, rapeseed oil, sunflower oil, and canola oil, consists of the simple alkyl esters of fatty acids (Chongkhong *et al*., 2009). Biodiesel can be produced through the transesterification process by reacting vegetable oils with methanol in the presence of a catalyst such as sodium hydroxide (NAOH) or potassium hydroxide (KOH) (Maa *et al*., 1999).

Transesterification process could be done using batch stirred reactor and continuous millichannel reactor. The latter reactor (continuous millichannel reactor) produces greater FAME yield at the same residence time (Gerpen, 2005 and Tanawannapong *et al*., 2013). This is due to millichannel reactor have many advantages over conventional reactors (batch stirred reactor), including increase the reaction rate and product yield, as well as energy efficiency. One of the previous paper reported by Guan *et al*. (2010) stated millichannel reactor produced around 100 % FAME yield at shorter residence time (100 s) as compared to batch stirred reactor which takes longer time (600 s) to achieve 100 % FAME yield. High mass transfer rate due to high interfacial surface area and internal circulation flow in millichannel reactor resulting to high FAME yield (Guan *et al*., 2009 and Rashid *et al*., 2014). Besides, the reaction between immiscible liquids can be enhanced using a slug-flow millichannel reactor (Chongkhong *et al*., 2009). However, there are a few comprehensive studies on condition parameters for the production of biodiesel in a millichannel reactor.

In this study, transesterification was carried out to elucidate the influence of methanol to oil molar ratio and temperature towards biodiesel yield for two different internal tube diameters; 1.6 and 1.0 mm at 6:1-23:1 methanol to oil molar ratio and temperature from 40 - 65 °C using millichannel reactor.

Experimental:

The feedstock was refined palm oil which was purchased from FFM Berhad, Malaysia. Methanol and acetic acid were obtained from Merck Co. Ltd. and potassium hydroxide was supplied by R&M Chemicals. Transparent polytetrafluoroethylene (PTFE) tubes (inner diameter = 1.6 and 1.0 mm, length = 1000 mm) were used for millichannel reactor.

Fig. 1 shows a schematic diagram of the millichannel reactor system. Two syringe pumps were used to inject the liquids (methanol containing KOH and oil). The methanol to oil molar ratio were varying from 6:1 to 23:1 by changing the flow rates. Both oil and

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methanol containing 5 wt% of KOH concentration were mixed at a T-shape joint prior to introduction into the milichannel reactor. The residence time for reaction is 180 s. The millichannel reactor was placed on a hot plate to control the temperature (40-65 °C). The acetic acid was used to terminate the reaction of the product and it was rinsed with deionized water for three times using centrifuge to remove all the residues. Then, it was analyzed using a HPLC from Shimadzu Prominence equipped with silica-gel column (Shimpack CLC-SIL) and refractive index detector.

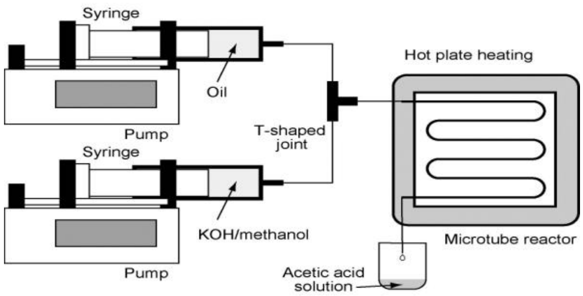

Fig. 1: Millichannel reactor system.

RESULT AND DISCUSSION

The FAME yield for the two internal tube diameters (1.6 and 1.0 mm) increases with the increasing of the methanol to oil molar ratio. This result shows that the highest FAME yield was at 21:1 methanol to oil molar ratio and lowest at 6:1 molar ratio. Guan *et al*. (2009) stated that the FAME yield increases with increase in methanol to oil molar ratio. However, higher molar ratio of alcohol to vegetable oil interferes in the separation of glycerol. From Fig. 2, it was observed that methanol to oil molar ratio at 21:1 gives the highest yield of the biodiesel and it slightly decrease when increase the methanol to oil molar ratio to 23:1. Between the two internal diameters investigated, the FAME yield increased with decreasing internal tube diameter.

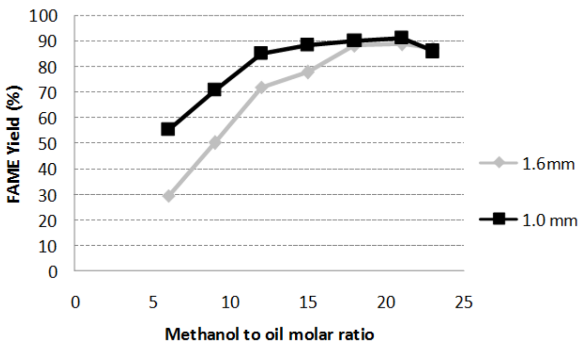

Fig. 2: Effect of methanol to oil molar ratio on FAME yield (inner diameter;1.6 and 1.0 mm, length;1000 mm, temperature;60 °C, KOH conc.; 5.0 wt%, residence time; 180 s).

Increase of FAME yield in methanol to oil molar ratio is due to higher contact surface area, thus enhancing the reaction rate (Guan *et al*., 2009). In addition, the behaviours of segmented flow will be affected by the size of millichannel reactor. So, it will increase the mass transfer between methanol and oil as the average liquid velocity is faster in a smaller internal tube diameter. Therefore, while using smaller internal tube diameter of millichannel reactor, higher FAME yield can be achieved. Fig. 3 shows the interfacial area between oil and methanol phases per unit methanol volume (S/v) throughout the entire reaction tube for the effect of methanol to oil molar ratio. This trend of the interfacial surface area may be attributed to the FAME yield difference for different internal tube diameters.

Fig. 4 shows the effect of temperature towards the FAME yield. Based on report by Sun *et al*. (2007), further increase in temperature (70 °C) resulted in negative effect on the FAME yield because the boiling point of methanol is 64.4 °C. In this study, the experiment was conducted at 4 different temperatures (40, 50, 60 and 65 °C). Based on Fig. 4, as the reaction temperature increases, the FAME yield will increase. Highest FAME yield was observed at 60 °C. This is because the solubility between the methanol and triglycerides will increase when the reaction temperature increase (Guan *et al*., 2010). Thus, the yield of biodiesel increases.

However, if the temperature is too high it will favour the saponification process (Atadashi *et al*., 2012). Therefore, at 65 °C the FAME yield decreases slightly. Besides, FAME yield also decreases because vaporization of methanol. In addition, FAME yield produced in 1.0 mm result in highest FAME yield for all temperature in comparison to 1.6 mm. This shows that smaller internal diameter produce higher FAME yield.

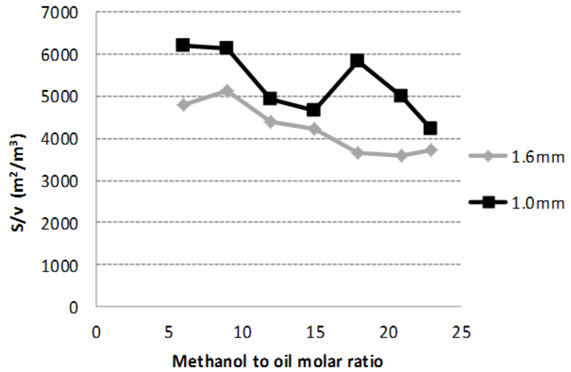

Fig. 3: Effect of methanol to oil molar ratio on interfacial area between oil and methanol phases per unit methanol volume (S/v).

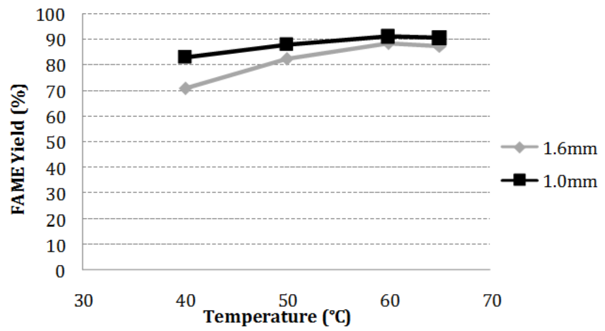

Fig. 4: Effect of temperature on FAME yield (inner diameter;1.6 and 1.0 mm, length;1000mm, methanol to oil molar ratio;21:1, KOH conc.; 5.0 wt%, residence time; 180 s).

Fig. 5 shows the interfacial area between oil and methanol phases per unit methanol volume (S/v) for the effect of reaction temperature. Based on graph in Fig. 5, 1.0 mm shows higher interfacial area compare to 1.6 mm internal diameter throughout the entire reaction tube.

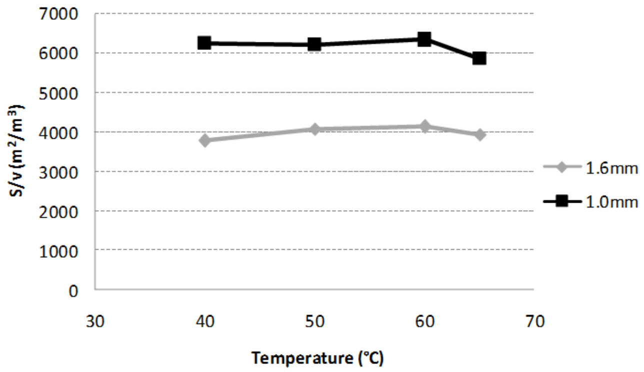

Fig. 5: Effect of temperature on interfacial area between oil and methanol phases per unit methanol volume (S/v).

Conclusion:

This study presented an experimental work of transesterification process using a millichannel reactor. The FAME yield was influenced by the methanol to oil molar ratio, temperature and internal tube diameter. The effect of methanol to oil molar ratio may be attributed to the enhancing effect from methanol concentration. The effect of the temperature can be explained by the positive effect of temperature on the reaction rate constant. These two facts and interpretation have been reported by many researchers for batch reactors. The third effect that influenced the FAME yield: the effect of internal tube diameter is our main concern. The smaller internal tube diameter resulted in high FAME yield. This may be explained by higher interfacial area between oil and methanol phases per unit methanol volume for smaller internal diameter.

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