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SEMARAK ILMU PUBLISHING

Journal of Advanced Research in Fluid Mechanics and Thermal Sciences

Journal homepage:
https://semarakilmu.com.my/journals/index.php/fluid_mechanics_thermal_sciences/index
ISSN: 2289-7879

Biodiesel Production from Used Cooking Oil Using Integrated Double Column Reactive Distillation: Simulation Study

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ARTICLE INFO

Article history:
Received 10 November 2021
Received in revised form 6 March 2022
Accepted 9 March 2022
Available online 9 April 2022

Keywords:
Reactive distillation; biodiesel; simulation; Aspen; UNIQUAC

ABSTRACT

Dependency on fossil – based energy has become a global problem. Indonesia has started a movement in changing energy sources from fossil to new and renewable energy. Biodiesel is one of the renewable energy sources. Used cooking oil is one of the prospective feed-stock in biodiesel production. Used cooking oil contains 21.84% free fatty acid and 78.16% triglycerides. This study simulated the production of biodiesel from used cooking oil using the Aspen Plus v10 software. The production system used an Integrated Double Column Reactive Distillation (IDC-RD) system. The thermodynamic method employed in the simulation system was UNIQUAC. This study used the sensitivity analysis function in the Aspen Plus v10 application to analyze the effect of reflux ratio, the bottom to feed ratio, and the methanol to oil ratio on the reaction conversion. The reflux ratio applied in the simulation was 0.5 – 200, the bottom to feed ratio was 0.25 - 0.9, and the methanol to oil ratio was 1: 3 - 9:8. It was found that the esterification column with a reflux ratio of 0.5, a bottom to feed ratio of 0.5, and a methanol-oil ratio of 4: 1 resulted in a conversion of 96.59%. It was also demonstrated that the transesterification column with a reflux ratio of 0.5, a bottom to feed ratio of 0.5, and a methanol-oil ratio of 3: 1 resulted in a conversion of 99.98%.

1. Introduction

The availability of fossil energy, especially crude oil in Indonesia, has become increasingly scarce, causing Indonesia to become an importer of crude oil and its derivative products [1]. Based on data from the Indonesian Central Statistics Agency [2], it was stated that in 1996 Indonesia's oil imports amounted to 10.6893 million tons and in 2018 Indonesia's oil imports increased to 26.7371 million tons. Meanwhile, oil exports in Indonesia in 1996 amounted to 10.1338 million tons and in 2018 decreased to 3.1221 million tons. This shows that Indonesia experienced an oil balance surplus in 1996 of 555.5 thousand tons, while it experienced an oil balance deficit in 2018 of 23.615 million tons.

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https://doi.org/10.37934/arfmts.94.1.152162

The increase in energy demand caused by the high rate of economic growth and population growth coupled with the depletion of world oil reserves and the problem of gas emissions caused by excess fossil fuels are strong reasons for every country to immediately develop and utilize renewable alternative energy This alternative energy has advantages when compared to fossil energy currently in use, namely that it will not run out if it is managed properly and is environmentally friendly, one of which is biodiesel [3]. Based on the environmental perspective, biodiesel is an interesting renewable energy to be widely applied since it is non-toxic, has a low aromatic content, and release a lower greenhouse gases such as unburned hydrocarbon, carbon monoxide, and particulate matters [4].

Indonesia has created a new policy to address the problem of fossil energy and its increasing energy needs. The new national energy policy is regulated in Government Regulation No. 79/2014. The new regulation set a target for Indonesia's new and renewable energy target in 2025 which is 23% of the total national energy. One of the ways for Indonesian government to achieve the target of new and renewable energy is by issuing a mandatory policy of B30 (Biodiesel 30%) [5]. In addition to B30, the Government is starting to develop in achieving the B100 (Biodiesel 100%) program.

Biodiesel is an alternative diesel engine fuel which consists of a mixture of fatty acid alkyl esters. Biodiesel can be produced through transesterification reactions of triglycerides contained in animal fats and plant oils with light alcohol and the presence of alkaline or acid catalysts. The reaction results in biodiesel as main product and glycerine as the by-product [6]. The direct transesterification process cannot be applied when the oil raw materials used contain high *free fatty acids* (FFA) (> 2%). In the transesterification reaction, the FFA content in the raw material must be lower than 1-2% to avoid lathering reaction between FFA and alcohol which can result in increased catalyst consumption, reduce biodiesel yield, and inhibit product separation [7]. Esterification reaction must be done to reduce FFA levels by reacting FFA with alcohol.

The materials for producing biodiesel from vegetable oil consist of used cooking oil, Calophyllum inophyllum oil, castor oil, and many more. Used cooking oil has FFA levels of 21.84% [8], Calophyllum inophyllumoil and castor oil have 39% and 19.8%, respectively [9]. Biodiesel cannot have high viscosity because it will damage and interfere with engine performance so that in the selection of raw materials, viscosity is a major consideration. The viscosity of used cooking oil, Calophyllum inophyllum oil, and castor oil is 30 cst [10], 38.17 cst [11], and 162.8 cst [12], respectively. Used cooking oil compared to *Calophyllum inophyllum* oil and castor oil has more advantages in viscosity and FFA in used cooking oil is lower than that of *Calophyllum inophyllum* oil so that used cooking oil is suitable as a raw material for making biodiesel.

Used cooking oil is an alternative material that can be used as an ingredient for making biodiesel which can minimize environmental pollution due to the free and unmanaged disposal of used cooking oil. If left unchecked and prevented immediately, there will be a pile of carcinogenic waste oil which is detrimental for health and can also cause poisoning in the body [13]. Therefore, used cooking oil should be recycled and used as raw material for producing biodiesel. Used cooking oil production is related to the total consumption of palm oil in Indonesia. In 2018, there were 12.2 million tonnes of oil consumed domestically.

There are several advanced biodiesel manufacturing processes such as reactive distillation (RD) and ionic liquid (IL) application. The use of Ionic Liquid has disadvantages for its expensive price of Ionic Liquid and the long reaction time. On the other hand, reactive distillation integrates reaction and separation in one unit column [14]. It has advantages since it needs a small ratio of oil alcohol, low operating temperature, lower costs, less waste products, low reactant costs, increased conversion through product removal, and useful to limit the azeotropic conditions in the column [15].

There are some parameters that affect the performance of the reactive distillation for biodiesel production. Modeling and simulation is often used for determining the effect of main parameters on the biodiesel conversion to reduce the number of experimental investigation which is usually costly. Modeling, simulation and numerical method are widely applied in engineering area. Many work related to the simulation using various tools have been reported in literature [16,17,18,19].

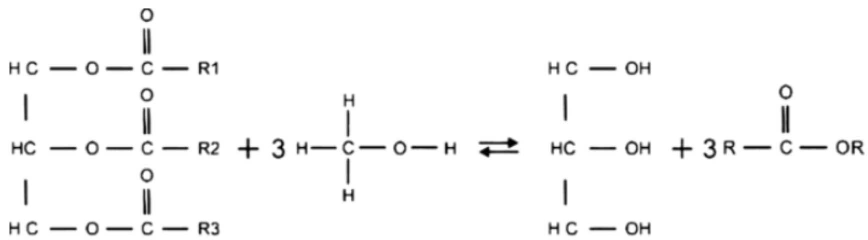
In this work, reactive distillation simulation was conducted using Aspen Plus software version 10.0 in a steady state condition. Souza *et al.*, [20] have optimized the process of making biodiesel using HYSYS v7.2 resulting in an increase in conversion of 18% and an error of 3.2%. Smejkal *et al.,* [21] has made a comparison between simulation using Aspen Plus and Hysys in the reactive distillation system. It was found that the error produced by Aspen Plus and Hysis were 1.2% and 2.8%, respectively. Based on the error parameter and the description above, Aspen Plus has more advantages in simulating the reactive distillation process. Therefore, in this work, Aspen Plus was employed for the simulation of reactive distillation to produce biodiesel. Process simulation is urgent for designing processing equipment to scale up and predict tool performance on an industrial scale. In this simulation, process optimization was carried out using the sensitivity analysis feature on the variable reflux ratio (RR), feed ratio (FR), feed stage (FS), and distillate rate (DR).

The thermodynamic model used in this study was the UNIQUAC model which was rarely used in other literature. The UNIQUAC model is more suitable for calculating the non-ideal characteristic of the liquid mixture such as the system in biodiesel synthesis [22]. Çağatay and Karacan [23] cagatay have performed a simulation using reactive distillation and heterogeneous catalysts for biodiesel production using used cooking oil as raw material. However, the system used in the research was a single column which only involved a transesterification reaction while the esterification as pre-treatment step was carried out separately. This study offered a novelty in biodiesel manufacturing process technology which was Integrated Double Column Reactive Distillation (IDC-RD). IDC-RD used a two column reactive distillation system which integrated the esterification and transesterification processes in one set of equipment to produce biodiesel with a more optimal yield. This work revealed the effect of the main parameters on biodiesel manufacturing process using IDC-RD which will be useful for process design and scale-up.

2. Methodology

In this work, an integrated double column reactive distillation (IDR-RC) was applied to produce biodiesel. Two reactions, namely esterification and transesterification reactions, were performed consecutively in the two reactive distillation column. The esterification reaction was conducted as the pre-treatment step to reduce the free fatty acid (FFA) content in used cooking oil in the first column. Subsequently, transesterification reaction as the main reaction to produce biodiesel was carried out in the second reactive distillation column. The simulation process of biodiesel production from used cooking oil using IDC-RD was performed with ASPEN Plus V10.0.

The formula for the transesterification reaction is exhibited in Eq. (1).



Triglyceride 3 (Methanol) Glycerol 3 (Alkyl Esters) (1)

The feed-stock for biodiesel synthesis in this work was used cooking oil which contained 21.84% FFA and 78.16% triglycerides. Fatty acid composition of the raw material was presented in Table 1 [8].

Table 1
Fatty Acid Composition of the Used Cooking Oil

Component	% mole
Palmitic Acid	1.4196
Linoleic Acid	8.3429
Oleic Acid	8.8452
Stearic Acid	0.3058
Linolenic Acid	2.9266
Tripalmitate	5.0804
Trilinoleate	29.8571
Trioleate	31.6548
Tristearat	1.0942
Trilinolenic	10.4734

The kinetics data of the esterification reaction taken from the literature can be seen on Table 2 [24].

Table 2
Esterification reaction data of used cooking oil

Parameter	Collision Factor (A)	Activation Energy (Ea)
	A^{-}_{1} = 128.8 /minute	Ea^{-}_{1} = 4.239 kJ/mole
	A^{+}_{1} = 1.307 x 10^3 /minute	Ea^{+}_{1} = 18.59 kJ/mole
T boiling point methanol	64.7°C	

The kinetics data of the transesterification reaction kinetics taken from the literature is demonstrated in Table 3 [25].

Table 3
Data of used cooking oil transesterification reactions

Parameter	Collision Factor	Activation Energy
	A_2 = 2.9 x 10^7 / minute	Ea_2 = 53.99 kJ/mole
T boiling point methanol	64.7°C	

3. Results

In this study, an Integrated Double Column Reactive Distillation (IDC-RD) simulation has been performed. IDC-RD applied two column reactive distillations which integrated in one set of equipment to accomplish the esterification and transesterification processes consecutively. Esterification reaction of used cooking oil with methanol is a crucial step to reduce the high FFA content prior to the main transesterification reaction to produce biodiesel. The process flow simulation is shown in Figure 1. The Basic simulation and sensitivity analysis was performed to examine the effect of the reflux ratio, the methanol to oil ratio, and the bottom to feed ratio on the reaction conversion.

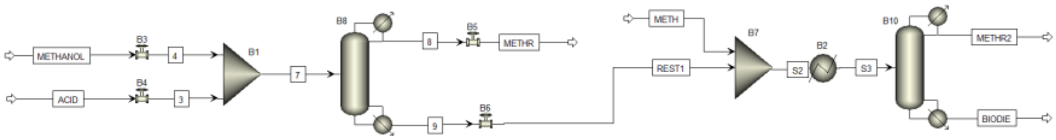


Fig. 1. Steady State Aspen Plus v10.0 Simulation Flowsheet of the IDC-RC of Biodiesel Production

3.1 Basic Simulation of Steady State Reactive Distillation (RD) Esterification Column

Esterification is a crucial step for reducing the FFA content prior to transesterification reaction in order to avoid the undesired saponification side reaction [26]. The main parameters of the IDC-RD for the esterification process were inputted into the Aspen Plus V10.0 using the radfrac type device. The basic simulation parameters were as follows: the number of stages was 5, the reaction zone was at stage 3, the input feed zone was at stage 2, the feed temperature was 60 °C, the pressure was set at the atmospheric pressure, the feed (oil to methanol) ratio was 1: 4, the bottom to feed ratio was 0.5, and the reflux ratio was 0.5. The results showed that the FFA, which was composed of oleic acid, linoleic acid, palmitic acid, stearic acid, and linolenic acid, decreased up to 96.59 % due to the reaction process with methanol. Those fatty acids were converted into methyl oleate, methyl linoleate, methyl palmitate, methyl stearic, and methyl linolenic acid, correspondingly. This result was considerably higher than the similar reaction conducted using the other types of biodiesel synthesis technology. Agustian *et al.,* [27] reported that the esterification of used cooking oil using ultrasonic reactor resulted in a conversion of 42.08%. Another study conducted by Mengyu *et al.,* [24] conveyed that the esterification of used cooking oil using conventional methods in the presence of Fe_2SO_4/C catalyst resulted in 96% conversion.

3.1.1 Effect of the reflux ratio on the FFA conversion

Figure 2 displays the effect of the reflux ratio on the FFA conversion. The higher reflux ratio led to the higher FFA conversion. This was due to the fact that the higher reflux denoted the longer reaction time. It implied that more intensive contact between the reactant molecules, yielding the higher conversion. Based on sensitivity analysis, the optimum result was obtained at the reflux ratio of 25 with a FFA conversion of 98.95%.

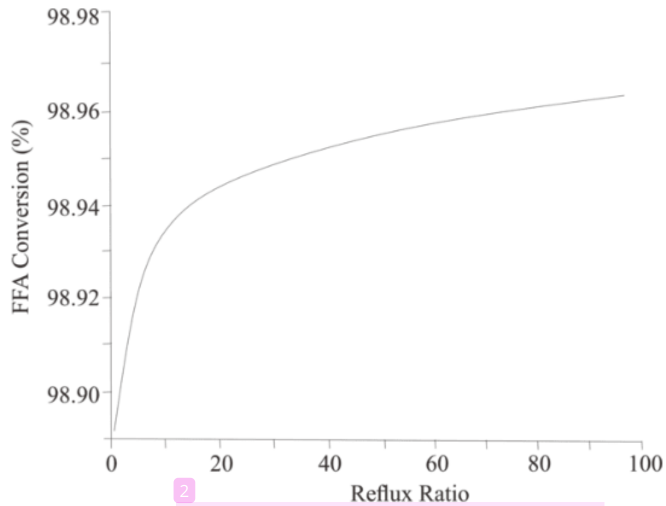


Fig. 2. The Effect of Reflux Ratio on the FFA Conversion

3.1.2 The effect of the feed ratio on the FFA conversion

Figure 3 reveals the effect of feed (methanol to oil) ratio on the FFA conversion. It was revealed that the higher methanol to oil ratio brought about the higher FFA conversion. This phenomenon occurred since the addition of the methanol will caused the excess of the reactant, which improved the reaction conversion. The result of the sensitivity analysis disclosed that the highest conversion was achieved at the methanol to oil ratio of 13, resulting in the FFA conversion of 99.591. This result was higher to a great extent compared to the conversion provided by the conventional reactor for the identical value of the reactant molar ratio.

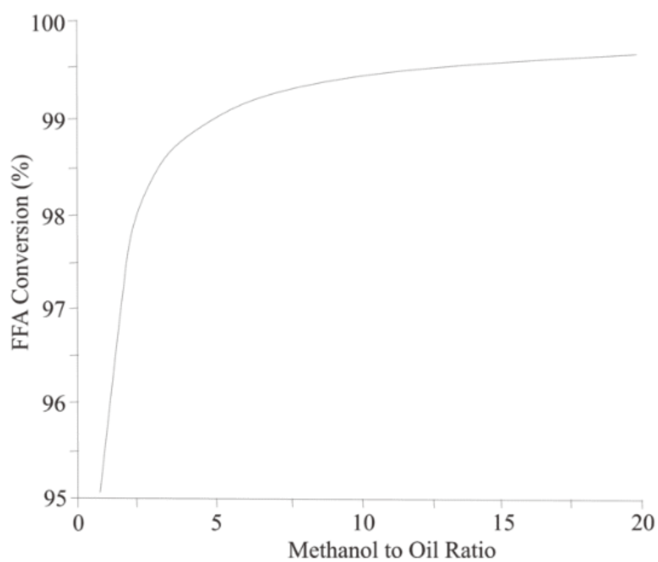


Fig. 3. The Effect of the Methanol to Oil Ratio on the FFA Conversion

3.1.3 The Effect of Bottom to Feed Ratio on FFA Conversion

Figure 4 indicates that the higher bottom to feed ratio led to the higher FFA conversion. This arose since the higher methanol concentration at the bottom will cause a high excess methanol to FFA. On the other hand, esterification is an equilibrium limited reaction, where the far excess reactant will shift the reaction towards the product formation and precede a higher conversion. Based on sensitivity analysis, the optimum result was obtained at a bottom to feed ratio of 0.75 with a conversion of 98.828%.

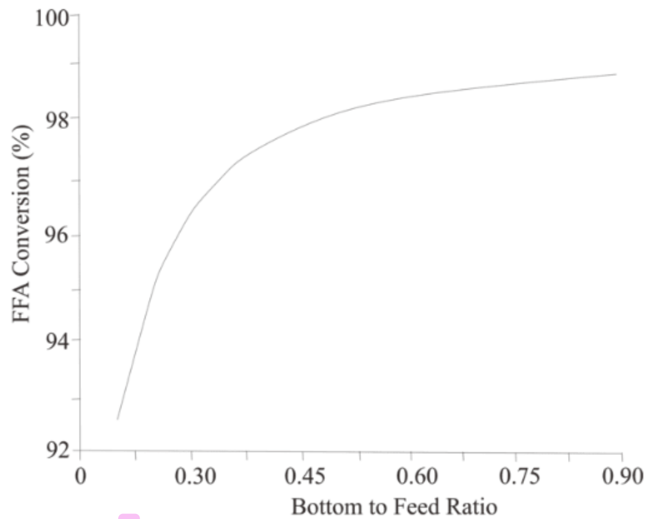


Fig. 4. The Effect of Bottom to Feed Ratio on the FFA Conversion

3.2 Basic Simulation of Steady State Reactive Distillation (RD) Transesterification Column

The main parameters were inputted into the Aspen Plus V10.0 using the radfrac type device. The basic simulation parameters set up were as follows: the number of stages was 5, the reaction zone was at stage 3, feed point location was at stage 2, the feed temperature was 65°C, the operation was conducted at the atmospheric pressure, the feed ratio of oil to methanol was 1: 3, the bottom to feed ratio was 0.5, and the reflux ratio was 0.5. The results exhibited that triglycerides in the form of tripalmitin, tristearin, triolein, trilinolein, and trilinolenin reacted with methanol almost completely. The conversion attained in the transesterification reaction using IDC-RD was 99.98%. It was notably superior compared to those resulted using conventional method and ultrasonic reactor which were 53.6% [28] and 92% [29].

3.2.1 The effect of reflux ratio on triglyceride conversion

Figure 5 discloses the effect of reflux ratio on triglyceride conversion. It can be observed that the higher reflux ratio enhanced the triglyceride conversion. This fact occurred since more reflux generated a longer contact between the reactants, leading to the higher reaction conversion.

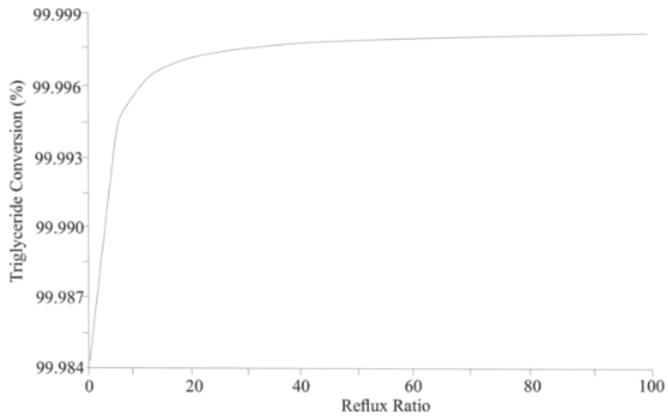


Fig. 5. The Effect of Reflux Ratio on Triglyceride Conversion

3.2.2 The effect of methanol-oil ratio on triglyceride conversion

The effect of feed (methanol to oil) ratio on the triglyceride conversion was disclosed in Figure 6. The simulation shows that the higher methanol to oil ratio promoted the triglyceride conversion. This was attributable to the fact that the higher excess of reactant will drive the equilibrium-limited reaction to right, affording a higher reaction conversion.

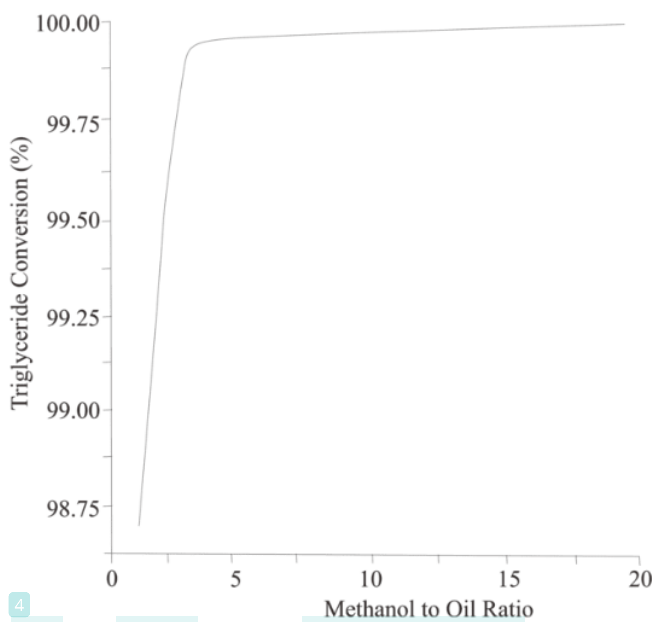


Fig. 6. The Effect of the Feed (Methanol-Oil Ratio) on Triglyceride Conversion

3.2.3 Effect of bottom to feed ratio on triglyceride conversion

Figure 7 shows that the higher bottom to feed ratio leads to the lower triglyceride conversion in the transesterification. This was because the higher bottom to feed ratio initiated the less reflux ratio in the distillate, thus reducing the conversion.

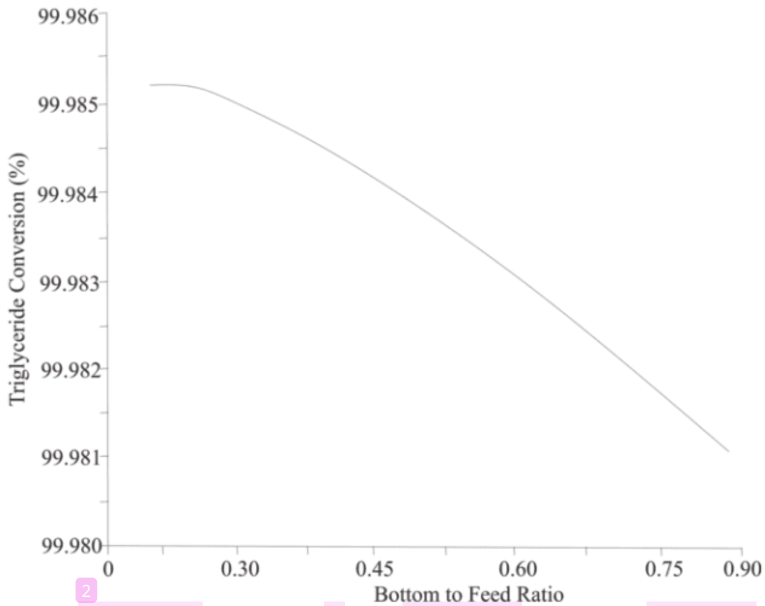


Fig. 7. The Effect of the Bottom to Feed Ratio on the Triglyceride Conversion

4. Conclusions

Based on the results of the steady state simulation study of Integrated Double Column Reactive Distillation (IDC-RC) esterification and transesterification of used cooking oil into biodiesel using Aspen Plus V10 software, it can be inferred as follows. The basic simulation of FFA esterification process of used cooking oil using IDC-RD provided a conversion of 96.59 % with a reflux ratio of 0.5, a bottom to feed ratio of 0.5, and a feed methanol-oil ratio of 4: 1. The effect of each parameter was evaluated independently using sensitivity analysis. The sensitivity analysis revealed that the higher reflux ratio, methanol to oil ratio, and bottom to feed ratio increased the FFA conversion. It was also discovered that esterification using a reflux ratio of 25 resulted in a conversion of 98.95%, a bottom to feed ratio of 0.75 gave a conversion of 98.828%, and a feed (methanol to oil) ratio of 13: 1 contributed to the conversion of 99.591%. The second column was assigned for the transesterification reaction. The basic simulation on the transesterification process of used cooking oil on IDC-RD denoted a conversion of 99.98% with a reflux ratio of 0.5, a bottom to feed ratio of 0.5, and a feed methanol-oil ratio of 3: 1. However, the results of sensitivity analysis on the IDC-RD transesterification demonstrated that changing of the process parameter slightly influenced the reaction conversion since a high conversion has been reach using basic parameter condition.

Acknowledgement
The funding resource from the Directorate of Research and Community Service – Ministry of Research, Technology and Higher Education (*DRPM* – Kemenristekdikti) through the Hibah Pasca

Doktor Grant with the contract number of 078/SP2H/LT/DRPM/2019 (internal contract number of 15.18.3/UN37/ PPK.3.1/2019).

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