



Steady-State and Dynamic Simulation Study of Reactive Distillation for FFA Esterification in Biodiesel Synthesis

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Abstract

Reactive distillation (RD) holds promise for process intensification in biodiesel production since it integrates reaction and separation. It simplifies the process and enhances the conversion of the equilibrium limited reactions. To ensure stability in RD operation, sensitivity study and process control simulation are necessary. In this work, RD was employed for free fatty acid (FFA) esterification of mixed non-edible oils in biodiesel synthesis. Non-edible oils used were waste cooking oil, crude jatropha oil, and crude nyamplung oil (*Calophyllum inophyllum* L). The simulation was conducted using ASPEN Plus V8.8. The sensitivity study was carried out to determine the effects of the operating condition alteration. A dynamic simulation was performed as a Proportional-Integral-Derivative (PID) controller tuning. It was revealed that the highest FFA conversion was 85%, achieved at the feed stage of 7, distillate rate of 0.22 kmol/hr, and oil to methanol molar ratio of 1:5. Level, pressure, and temperature controls were installed in RD. Then, a dynamic simulation was applied as a PID controller tuning. Three different controller tuning methods, viz. Ziegler-Nichols, Cohen-Coon, and Internal Model Control were studied. The best PID parameter was obtained by using Cohen-Coon method which provided the fastest rise time, lowest settling time, and lowest overshoot.

Keywords: Biodiesel; Reactive Distillation; Dynamic Simulation; Free Fatty Acid; Proportional-Integral-Derivative

1. Introduction

Nowadays, fuel demand has increased following the increase of public need for transportation, industries, power plant, household, and many other activities. The increasing fuel demand is not in line with the available energy sources. Generally, fuel oil consumption in the world has exceeded its domestic production, and petroleum resources are expected to be available only 10 – 15 years from now (Daya and Day, 2017). In addition, the problem of fossil fuel reserves depletion and energy security, the issue of climate change caused by the utilization of fossil fuel has promoted the necessity of a worldwide transition to renewable energy resources which are more environmentally friendly. Biodiesel is attractive alternative energy to conventional fuel since it is renewable, sustainable, biodegradable, and non-toxic (Supaporn and Yeom, 2017). Biodiesel also reveals good

properties and performance when it is applied in diesel engine (Widjanarko et al., 2020).

Generally, biodiesel (fatty acid methyl esters) can be produced through alkaline catalyzed transesterification of vegetable oils. However, for vegetable oils containing high free fatty acid (FFA), the FFA should be first converted to methyl ester through esterification reaction before the main transesterification reaction (Kusuaningtyas et al., 2016). In this study, the mixture of three different oil containing high FFA was used as a feedstock in biodiesel production. The three oil sources were waste cooking oil (WCO), crude jatropha oil (CJO), and crude nyamplung oil (CNO) or *Calophyllum inophyllum* L oil. WCO is one of the common biodiesel sources since it is economical and plentiful. WCO might harm the environment if it is not processed properly. One of the solutions is by processing it into biodiesel (Pugazhendhi et al., 2020; Suriaini et al., 2019). CJO and CNO are also suitable as biodiesel feedstocks since they are

non-edible oils. These vegetable oils have high FFA which is not desirable for food production (Fadhullah et al., 2015; Kusumaningtya et al., 2017; Jain et al., 2018; Arumugam and Ponnusami, 2019). *Jatropha* plant (*Jatropha curcas L.*) is easily found in tropical and subtropical countries. It is well known as a bioenergy plant since this plant can live in the marginal and arid land. Meanwhile, its seed contains 40%-60% oil, which is considered abundant as biodiesel feedstock (Cañadas-López et al., 2018). Nyamplung (*Calophyllum inophyllum L.*) plant is also famous with other names such as honne, tamanu, and polanga. This plant is advantageous as a source of biodiesel feedstock as it bears fruits all over the year, starting from the age of 7 years to 58 years (Ong et al., 2019). It is easy to cultivate and its seeds comprise 40-73% oils (Atabani et al., 2018).

Due to this reason, the mixture of these three different oil sources was used in this research as biodiesel feedstock. Since the mixed non-edible oils contain high FFA, this oil mixture underwent an esterification reaction to reduce the FFA content. The FFA esterification experiment has been previously conducted in a reactive distillation column (Kusumaningtyas et al., 2018; Li et al., 2018). This work focuses on the dynamic simulation for process control of reactive distillation column for FFA esterification reaction in mixed non-edible oils (WCO, CJO, and CNO) in biodiesel production.

Reactive distillation (RD) is a combination of reaction and distillation in a single column simultaneously. RD has the same function as the reactor strung with separator. However, RD has some advantages when compared to reactor-separator, viz. an increase in selectivity and conversion, better heat control, reaching for hard separation, reducing the catalyst needed, avoid azeotrope, and more effective heat integration for energy conservation (Kiss, 2011). RD is more efficient in capital cost because it reduces the distillation column as a separation unit. Moreover, RD is useful and applicable for carrying out reversible reactions such as esterification, transesterification, and etherification processes since continuous product separation on RD will shift the reaction equilibrium towards product formation (Banchero et al., 2014). Thus, it can be affirmed that RD is such a great intensification process in the chemical industry.

Reactive distillation (RD) is a complex process influenced by several operating parameters such as reflux ratio, reacting zone, feed stage of reactants, the ratio of reactants, and distillate rate (Kiss et al., 2011). Thus, computer-aided process simulation and control are essential to ensure the stability of the operating condition. Process simulation was initially performed to simulate the process at the steady-state condition. Steady-state simulation is conducted based on the mass and energy balance at the condition which does not count on time. Based on the steady-state simulation, the performance of the RD column in terms of the FFA conversion on the esterification process at certain operating conditions can be predicted. The effect of each process variable on the reaction conversion can also be estimated using sensitivity analysis. This work presents simulation and a sensitivity study of an RD process for the esterification reaction of FFA in mixed non-edible oil in biodiesel synthesis in the presence of tin (II) chloride catalyst using ASPEN PLUS V8.8 process simulator.

Furthermore, to study the feasibility of this process as a real plant, dynamic simulation with an installed level, pressure, and temperature control has been performed. In the dynamic simulation, time-reliance is developed into the model through derivative terms. By using dynamic simulation, time-dependent description, prediction, and control of real processes in real-time have turned out to be viable (Huang et al., 2017). Thus, dynamic modeling and simulation have become more important in the industrial process nowadays.

In this study, steady-state simulations were conducted. Furthermore, the optimum operating condition was obtained from the sensitivity study analysis. Continuously, level, pressure, and temperature controls were installed in RD simulation to ensure the operation condition kept stable. Proportional-Integral-Derivative (PID) controllers were applied in this simulation. A PID controller continuously computes an error value as the dissimilarity between the desired set-point (SP) and a measured process variable (PV) and composes a correction based on proportional, integral, and derivative terms, which are symbolized by P, I, and D respectively. The best operating condition in the sensitivity study was employed as a basic dynamic simulation to obtain the best PID parameter for a stable process. Control stability was tested through two different tests which were servo and regulatory tests.

2. Materials and Methods

2.1. Steady-State Simulation

Steady-state simulation is needed before the dynamic simulation as the basic operation condition. The simulation of FFA esterification of mixed non-edible oil using RD column in the presence of tin (II) chloride catalyst was carried out using ASPEN PLUS V8.8. The parameters for steady-state simulation which were exhibited in Table I and Table II were established based on the previous experiment performed by Kusumaningtyas et al. (2014). Feed compositions for inlet feed are shown in Table 1.

Table 1. Column Feed Entry Components

Component	Flowrate (kmol/hr)
Oleic acid	0.0422
Linoleic acid	0.0201
Palmitic acid	0.0291
Stearic acid	0.0090
Methanol	6.0249
Total	6.1253

The initial operating condition for the reactive distillation is presented in Table 2.

Table 2. Reactive Distillation Operating Condition

Parameter	Value
Number of stages	20
Reacting Zone	13
Feed Stage	7
Feed Temperature	50°C
Total Feed	6.12534 kmol/hr
Pressure	1 Atm.
Condenser Type	Total
Reboiler Type	Kettle

The reaction rate of the esterification process follows the pseudo homogeneous reversible second order reaction as shown in Eq. (1).

$$-\frac{dC_A}{dt} = k_1 C_A C_B - k_2 C_C C_D \quad (1)$$

Where, C_A , C_B , C_C , and C_D denote the concentrations of oleic acid, methanol, methyl oleate, and water respectively. Meanwhile, k_1 and k_2 are kinetic rate constants for forward and backward reactions respectively. k_{0i} and E_{ai} is the pre-exponential factor and activation energy for the kinetic reaction. The reaction rate constants and activation energy values are presented in Table 3, referring to the data obtained in our previous work (Kusumaningtyas et al., 2014).

Table 3. Kinetic and Thermodynamic Parameters of the Reacting System

Parameter	Value
k_{01}	2.33×10^{10}
E_{a1}	64.5 kJ/mol
k_{02}	4.29×10^8
E_{a2}	40.7 kJ/mol

2.2. Sensitivity Study

The sensitivity study of RD was performed to understand the effects of the main parameter of the process on the reaction conversion of FFA esterification. Besides, it can also be conducted to determine the optimum operating condition of the reaction. The parameters studied in this work were reflux ratio, distillate rate, feed stage, and oil to methanol molar ratio. The sensitivity study parameter for this RD is shown in Table 4.

Table 4. Sensitivity Study of RD Operating Condition

Parameter	Min.	Max.	Unit
Reflux ratio	10	500000	
Feed stage	2	19	
Distillate rate	0.00001	6.1253	kmol/hr
Oil: Methanol	0.00001	60	

The optimum operating condition from the sensitivity study was then employed as the base condition in dynamic simulation.

2.3. Dynamic Simulation

In this dynamic simulation, 2 level control (LC), 1 pressure control (PC), and 1 temperature control (TC) were installed as shown in Figure 1.

LC-1 and LC-2 were operated to control the liquid levels in the condenser and reboiler, respectively. PC-1 was functioned to control the pressure inside the condenser, meanwhile, TC-1 was applied to control the temperature of the 14th stage of reactive distillation. Based on the steady-state simulation, additional sizing of the RD column was calculated and the results are shown in Table 5.

Table 5. Sizing of RD Column

Equipment	Height (m)	Diameter (m)
RD Column	12.44	2.39
Boiler	2.32	1.16
Condenser	1.86	0.93

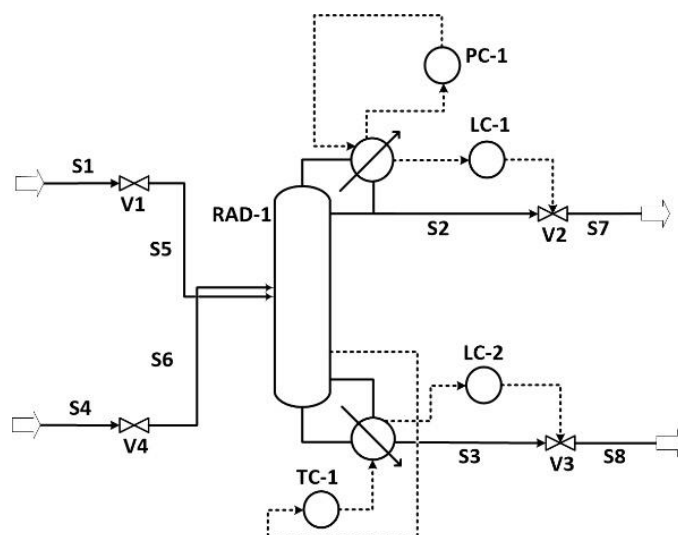


Figure 1. Control Instruments in Reactive Distillation Column

2.4. Proportional-Integral-Derivative Tuning (PID)

In this study, PID tuning for each controller is performed by using three different methods i.e. Ziegler-Nichols (Z-N), Cohen-Coon (C-C), and Internal Model Control (IMC). PID parameter from each tuning method was then tested using servo and regulatory tests. The best PID parameter was determined based on its performance (rise time, overshoot, and settling time).

3. Results and Discussion

Steady-state simulation of RD was performed by using operating conditions as shown in Table 1 and 2. The result revealed that the FFA conversion at this condition was 75.8%. To increase the FFA conversion, sensitivity analysis was done using the parameter as shown in Table 4.

3.1. Sensitivity Analysis

Effect of Reflux Ratio on the FFA Conversion. The effect of the reflux ratio was studied by maintaining the value of distillate rate, feed stage, and methanol to oil molar ratio while reflux ratio was varied from 10 to 500,000. In this simulation, a reflux ratio of 500,000 indicates that the process uses total reflux without distillate as a product. Figure 2 shows that a higher reflux ratio also provided higher FFA conversion.

Meanwhile, it will only have a little amount of distillate product. A higher amount of liquid

returned from the top of reactive distillation

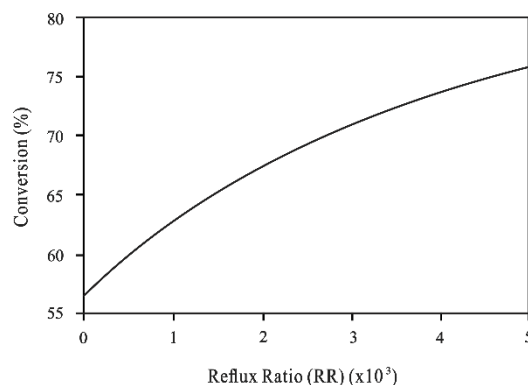


Figure 2. Effect of reflux ratio to FFA conversion

will increase the contact time between vapor and liquid along RD column (Mallaiyah and Reddy, 2016). On the other hand, a higher reflux ratio also increased reboiler duty to vaporize the liquid into vapor. Reboiler duty for 10 and 500,000 reflux ratio were 1,658.58 cal/s and 60,195.76 cal/s, respectively. It can be concluded that achieving higher FFA conversion requires higher energy consumption (Tavan and Hosseini, 2013). Based on the sensitivity study at a lower increment, a reflux ratio of 5000 provided a significant increase in the FFA conversion.

Effect of Reflux Ratio on the FFA Conversion. Figure 3 shows the influence of distillate rate on the FFA conversion. Lower FFA conversions were obtained at the lowest and highest distillate rate (Kumar, 2010). The increase of distillate rate will reduce the

volatile amount at the bottom of RD, the lesser amount of volatile at the bottom will increase its purity. In this study, the best condition was found at a distillate rate of 0.22 kmol/hr with 99.2% FFA conversion.

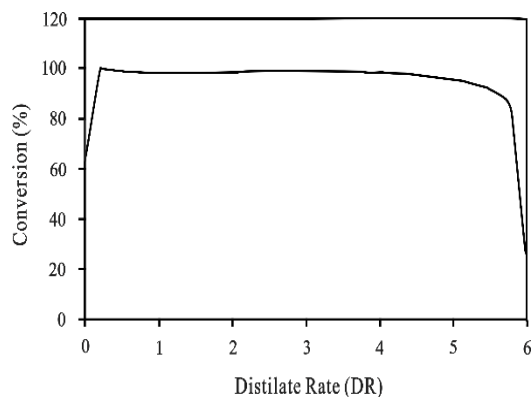


Figure 3 Effect of distillate rate on FFA conversion

Effect of Feed Stage on the FFA Conversion. The feed stage was varied from 2 to 19, while the other parameters were kept the same. Fig. 4 illustrates the influence of the feed stage on the FFA conversion. It can be seen that there was no significant contribution of the feed stage to the FFA conversion. As stated by Kumar (Kumar, 2010) that changes on the feed stage without changing the reactive stage will not change material composition at the bottom and distillate. Reboiler duty at different feed stages also showed a slight difference, where reboiler duty for the feed stage at 1st and 7th stage were 2243.44 cal/s and 2243.11 cal/s, respectively. Feed stage at 7th stage was selected based on the consideration of column height which will affect pump total head to transport the fluid into the feed stage.

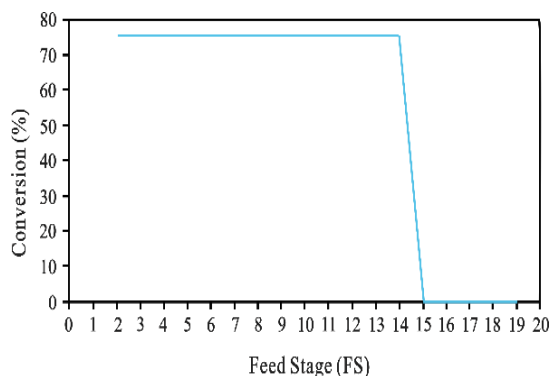


Figure 4. Effect of feed stage to FFA conversion

Effect of Methanol to Oil Molar Ratio. The effect of methanol to oil molar ratio on the FFA conversion was studied by changing the ratio from 0.00001 to 60. Theoretically, a larger amount of methanol will reduce the

purity of the product at the bottom (Mallaiah and Reddy, 2016). Based on the base case simulation, the oil to methanol molar ratio at 1:60 only resulted in 1.2% of product purity. The lower purity of the product requires higher energy for product separation. The effect of oil to methanol ratio on the FFA conversion is presented in Fig. 5. In this study, the oil to methanol ratio 1:5 was chosen to increase the FFA conversion and also to increase the purity of the product. This ratio is also suggested from previous experiments conducted by Boonauwat et al. (2015) and Huang (2018).

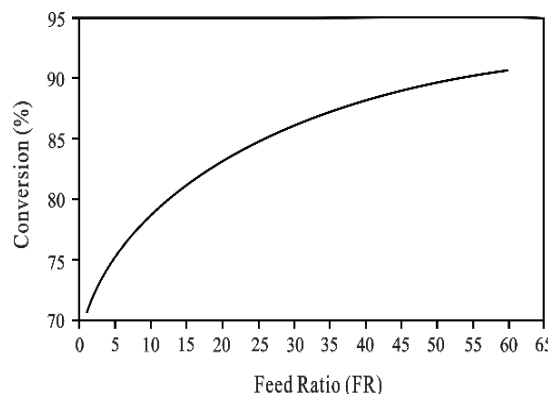


Figure 5 Effect of oil to methanol molar ratio on FFA conversion.

Based on this sensitivity study, a new parameter to obtain higher FFA conversion was determined. The details of the operating parameter are presented in Table 6.

FFA conversion from base case simulation which used operating condition presented in Table 2 was 75.7%. Meanwhile, the optimized operating condition in Table 6 increased FFA conversion to 85.3%. It shows that the FFA conversion still can be improved by changing the feed stage, feed ratio, distillate rate, and reflux ratio. The purity of the product also increased from 1.2% to 22.12%. Subsequently, the operating condition in Table 6 was used for the dynamic simulation.

Table 6. Optimized RD Operating Condition

Parameter	Value
Feed stage	7
Total feed	0.60248 kmol/hr
Pressure	1 atm
Feed ratio	1: 5
Distillate rate	0.22 kmol/hr
Reflux ratio	5000

3.2. Dynamic Simulation

Performance of Level Control (LC-1 and LC-2). LC-1 and LC-2 were used to control

the liquid level inside the condenser and reboiler respectively. The dimension of each equipment is presented in Table 5. Control performance was observed based on their rise time (RT), overshoot (OS), and settling time (ST) during servo and regulatory tests. PID tuning and control performance of LC-1 are shown in Table 7. While Table 8 shows PID tuning and control performance of LC-2.

Table 7 demonstrated that the PID tuning result from the Z-N method provided the best control performance. It has the fastest rise time, settling time, and lowest overshoot on both regulatory and servo tests. While in Table 2, PID tuning from the C-C method was chosen since it has the best control performance. PID tuning from Z-N and C-C method almost has control performance in the servo test. Based on the regulatory test, the Z-N method showed a fast RT but its OS was 50% and the settling time required was 1.85. It means that even it had a fast rise time, but PID tuning from the Z-N method needed a longer time to reach steady-state condition. This was in agreement with the result of Giwa (2016), in which Cohen-Coon was the best tuning technique for PID controller in reactive distillation process.

Performance of Pressure Control (PC-1). Pressure control is used to control pressure changes in the RD column. This pressure control is employed to ensure that the

pressure inside the condenser does not exceed the allowed design pressure limit and ensures condensation of the top product returned to the main column is in the accordance with the reflux ratio. PID tuning and PC-1 control performances are shown in Table 9.

Based on Table 9, it can be seen that PID tuning using the Cohen Coon method gave the best response for regulatory and servo tests. The settling time required for regulatory and servo tests was the fastest among Z-N and IMC methods. Fast settling time indicates that the process quickly reaches the setpoint when there are changes in the process and also able to reach a new setpoint when there is a change in setpoint rapidly.

Performance of Temperature Control (TC-1). Temperature control is used to control the temperature changes in the reactive distillation column which can be caused by changes in operating pressure and feed temperature. Temperature control in this process is very important to determine the FFA conversion in the RD column. In this process, the temperature was kept at 70°C. This temperature was subsequently used as an initial setpoint for this process. Table 10 shows the PID tuning parameter and control performance of TC-1.

Table 7. PID Tuning and Control Performance of LC-1

Tuning Method	Parameter			Regulatory Test			Servo Test		
	P	I	D	RT	ST	OS	RT	ST	OS
Z-N	259.7	2.49	0.62	0.01	0.02	0	0.61	0.66	0
C-C	313.5	0.37	0.11	0.42	0.58	0	0.63	0.69	2
IMC	3.33	0.37	0.38	0.45	5.2	20	0.57	2.43	15

Table 8. PID Tuning and Control Performance of LC-2

Tuning Method	Parameter			Regulatory Test			Servo Test		
	P	I	D	RT	ST	OS	RT	ST	OS
Z-N	142	3.1	0.77	0.05	1.85	50	2.35	2.6	2
C-C	177	2.9	0.51	0.12	0.38	20	2.43	2.63	2
IMC	3.16	0.4	0.43	1.15	2.85	80	2.33	5.98	15

Table 9. PID Tuning and Control Performance of PC-1

Tuning Method	Parameter			Regulatory Test			Servo Test		
	P	I	D	RT	ST	OS	RT	ST	OS
Z-N	20.0	1.2	0.3	0.5	3	30	0.065	0.29	0
C-C	22.3	1.4	0.2	0.21	1.22	40	0.018	0.045	5
IMC	0.53	16	0.29	0.36	1.98	50	0.036	0.261	20

Table 10. PID Tuning and Control Performance of TC-1

Tuning Method	Parameter			Regulatory Test			Servo Test		
	P	I	D	RT	ST	OS	RT	ST	OS
Z-N	49	1.1	0.2	0.3	2.12	20	0.45	0.97	1
C-C	57	1.3	0.2	0.1	0.8	35	0.032	0.222	25
IMC	11	2.3	0.2	0.2	1.17	30	0.085	0.625	20

PID parameter that was obtained using Cohen-Coon method has the best control performance compared to the other methods. Even it had an overshoot of 25 and 35% for servo and regulatory tests, but it reaches the set point 200% faster than Z-N and IMC method. This finding was in line with the report of Joseph and Olaiya (2018). It was found that ZN-PID tuning method revealed lower effectiveness in the control of non linear and complex system. It revealed huge inertia and lag since it generated vastly higher overshoot, rise time and settling time in the system operation. Meanwhile, IMC often shows lower performance in overwhelming load disturbance (Mokhatab and Poe, 2012).

4. Conclusion

This simulation was performed to represent the biodiesel synthesis from a non-edible oil mixture. The best operating condition was obtained from the sensitivity study where the feed stage was in the 7th stage, using oil to methanol molar ratio of 1:5, at distillate rate of 0.22 and reflux ratio of 5000. Three different tuning methods were used to find the PID parameters of LC-1, LC-2, PC-1, and TC-1. The C-C method exhibited the best PID tuning result for LC-2, PC-1, and TC-1, while the PID parameter for LC-1, optimized using the Z-N method, demonstrated the best control performance.

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