

Simulation and Control of Reactive Distillation of Biodiesel Production

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Abstract: The control of the transesterification process was conducted using MATLAB® codes as well as its Simulink environment. To realize the aim of the study, the dynamics data of methyl-oleate (BIODIESEL), decanter duty (manipulated variable) and reflux ratio (selected disturbance variable) were elicited from the Aspen Plus®. dynamic simulation of the formulated process model and this was used to get the first-order-plus-dead-time transfer function relation between methyl-oleate, decanter duty and reflux ratio with the help of MATLAB®. Open loop simulation was achieved by introducing steps to the input variables (reboiler duty and reflux ratio). The feed oil (Trolein) (98.3%) was converted into methyl-oleate and the final composition of the exit streams was 72.9% methyl-oleate, 1.7% triolein, 24.5% glycerol, 1.3% purge methanol. It was observed that a net duty of 5kW is required to achieve this production after 6000 mins at 100 °C. The controller was successfully tuned by Zeigler-Nichols (ZN) and Cohen-Coon (CC) techniques to conduct the disturbance rejection of the process. The performance of the CC tuning and ZN adjusting techniques in the disturbance rejection control simulation had Integral Square Error (ISE) and Integral Absolute Error (IAE) values of 1.269/ 4.09 and 1.126/3.909, respectively. It was noticed that the performance of the CC tuning technique was better than that of the ZN tuning technique in the disturbance rejection control simulation due to its lower ISE and IAE values. This study suggested that the reactive distillation process could be effectively operated to act as required using PID control to produce clean methyl-oleate.

Keywords: Aspen Plus®, Biodiesel, glycerol, MATLAB, PID control, Reactive distillation

I. Introduction

The increase in energy demand worldwide along with the non-renewability of crude oil has led to the quest for an alternate source of energy, particularly renewable energy [1]. One of these

renewable energies is biodiesel. Biodiesel has become more attractive since it is made from renewable sources and combines high performance with safer environmental impact. Biodiesel is non-toxic, biodegradable, free of sulphur and aromatic compounds and contributes less to greenhouse emission since the CO₂ emitted are absorbed in the production process [2-4].

The transesterification process of wholesome oil, uneatable oil and animal fat is commonly used to produce biodiesel through short-chained aliphatic alcohols though its commercial-scale production is still challenging. Batch and continuous processes use excessive alcohol more than the required molar stoichiometry in biodiesel production (3:1) to obtain an optimum yield of biodiesel per unit oil used. At the end of

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the production procedure, the unreacted alcohol has to be retrieved in a different distillation column. The residual alcohol recovery helps to increase the purity of the product. This increases both the capital and operating costs of the process invariably. Therefore, biodiesel production via reactive distillation process proffers a solution to the high cost of biodiesel production without affecting the product quantity and quality of the biodiesel [2].

The reactive distillation process integrates the reaction operations and separation process into one reacting vessel to minimize both the operational and equipment costs [5]. The products are withdrawn immediately after they are generated, this leads to suppression of the thermodynamic equilibrium of the result, thereby leading to high conversion rate and selectivity. Thus, the reactive distillation process is productive for reversible reactions such as the transesterification process of edible oil, non-edible oil and animal fats to methyl oleate. The blending of the reaction and separation process into one entity leads to various difficulties of the procedure, this has made the dynamics and control study of this procedure a major challenge to Process Engineers [5].

The residual alcohol recovery helps to increase the purity of the product, but the cost of production through standard means to establish the maximum purity is quite high. This exorbitant cost is a major challenge which must be tackled by developing an alternative means that incorporates the development of a dependable control method. This will allow the process to behave efficiently and also minimizes the cost of commercial production of biodiesel. Therefore, it is essential to examine the dynamics of the methyl oleate production procedure via simulation of the reactive distillation process. Since the need to maximize the mass and energy

of the raw materials makes the biodiesel reactive distillation process difficult, therefore, it is imperative to develop an appropriate control system for the procedure [5]. Hence, it is important to establish another cheap and efficient method for mass production of biodiesel which will not reduce the quantity and quality of biodiesel produced. One of such alternative method is the reactive distillation process oil [1].

Dynamics in engineering means the process by which variables change with time in response to input types. In chemical engineering, process variables vary with time and one of the examples is the transesterification process. The external intervention is the process control needed to ascertain the satisfaction of operational requirements such as safety, production specifications, environmental regulations, operational constraints, and economics of a process [6]. Since the structure of the biodiesel reactive distillation process is difficult, it is important to maximize mass and energy of the raw materials, hence, an appropriate control system for the process must be developed.

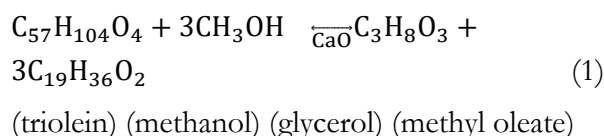
Several research works have been reported on modelling, simulation and control of biodiesel production. The production of biodiesel from transesterification reaction between soybean oil and methanol in a reactive distillation column was reported by [7-10]. Various works on modelling, simulation and control of biodiesel production have also been reported by [2]. However, research on the dynamics and control of biodiesel production via transesterification reactive distillation process using ASPEN PLUS AND MATLAB has not been considered and reported to the best of authors' knowledge. Therefore, the main drive behind this research was to model, simulate, and develop a control system for the transesterification process. Also,

to provide an outlook on the dynamics of biodiesel production by reactive distillation process by employing Aspen Plus® and MATLAB® process modelling and simulation software. This goal would be established by realizing the following objectives; developing a steady-state model of the process and convert it into a dynamics model within the Aspen Plus® simulation environment, develop the process transfer functions of the generated data required for the PID controller and obtain the tuning parameters for Zeigler-Nichols and Cohen-Coon methods with the help of MATLAB®, apply the PID controller to ensure the biodiesel conversion attain the desired set-point value within the Simulink environment of MATLAB and investigate the performance of the PID controller using open-loop and closed-loop models.

II. Materials and Methods

A. Model Development

This transesterification process was modelled in Aspen plus® to know the optimal conditions that will reduce the conversion of wholesome oil to biodiesel. The wholesome oil and methyl oleate are represented as TRIOLEIN and methyl oleate in the simulation. Triolein reacted with an alcohol to generate methyl oleate and glycerol, according to Eq (1), which is a general representation, where the CaO acts as a heterogeneous catalyst.



The two RGibbs reactors operated in series (Figure 1) were used for the process model. The wholesome oil and alcohol (MeOH) were measured using their stoichiometric value, mixed

and then poured into the RGibbs reactor 1(R1) thereafter catalyst was added into the R1. The product formed in R1 was transferred into a flash drum separator (R1-SEP) to remove unreacted alcohol (MeOH-R1) which was transferred to the RGibbs reactor 2 (R2). The product leaving the R1-SEP (PROD-01) was then transferred into a centrifuge to have two distinct layers such as glycerol (GLYC-1) and a mixture of unreacted wholesome oil and biodiesel (R2-FEED). The wholesome oil -biodiesel mixture was pump to the R2 with recycled alcohol which was condensed before feeding into the condenser (COND). The R2 product surge (R2-OUTFLOW) was flashed to eliminate and recycle alcohol (MeOH-R2). This stream was diverged (RECDIVERGE) later to facilitate a strip surge (STRIP). The base liquid (PROD-02) was centrifuge (CENTRIFUGE2) to separate the biodiesel (BIODIESEL) and glycerol (GLYC-2). The glycerol surges were combined (GLYC-MIX) to generate the process output known as the final glycerol product [11].

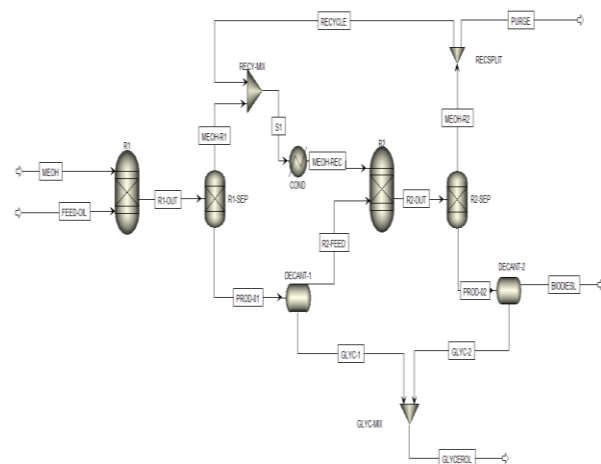


Figure 1: Aspen Plus® Model for the Reactive Distillation Process (Modified from [11])

B. Process Simulation Description

The simulation of the biodiesel production process was carried out using Aspen Plus®

software based on the Steps of Procedures (SOP).

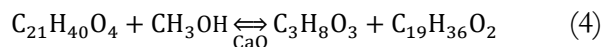
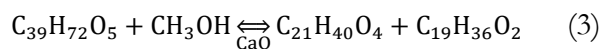
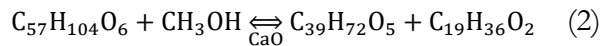
- i. The basic chemical components (Wholesome (Triolein), Alcohol (Methanol), Methyl oleate, calcium oxide, and Glycerol) used in the process were selected from the Aspen Plus database:
- ii. The conditions of the feed streams were stated as displayed in Table 1[12].

Table 1: Stream Operating Parameters

Stream Name	Methanol	Triolein
Temperature (°C)	60	60
Pressure (bar)	1	1
Total Flow (kmol/hr)	3	1
Feed (%wt)	100	100

C. Process Modelling and Reaction Kinetics

The forward and backward reactions are given in eqns (2-4) and the transesterification reaction that takes place within the column was modelled with the aid of kinetic rate expressions eqns (5-10),



The reaction rates are as follows:

$$r_1 = k_1 C_{TO} C_M \quad (5)$$

$$r_2 = k_2 C_{DO} C_{MOE} \quad (6)$$

$$r_3 = k_3 C_{DO} C_{MOH} \quad (7)$$

$$r_4 = k_4 C_{MO} C_{MOE} \quad (8)$$

$$r_5 = k_5 C_{MO} C_M \quad (9)$$

$$r_6 = k_6 C_G C_{MOE} \quad (10)$$

where k_i and C_i are the rate constant and concentration of each specie, TO , DO , MO , M ,

MOE , and G denote tri-olein, di-olein, mono-olein, alcohol(methanol), biodiesel and glycerol respectively.

Equation 11 represent the rate constant (k)

$$k = k^0 \times e^{-\frac{E}{RT}} \quad (11)$$

where k^0 is the frequency factor in $\text{mol}^{-1}\text{litre min}^{-1}$,

E = activation energy in cal/kmol,

R = universal gas constant and

T = reaction temperature in Kelvin.

The kinetic data (Table 2) adopted in this work was gotten from the study of [1].

Table 2 : Kinetic Data for the Transesterification Process [1]

i	Frequency Factor (k_i^0 , $\text{mol}^{-1}\text{litre min}^{-1}$)	Activation Energy (E_i , cal/kmol)
1	1.469×10^8	14040
2	105100	10739
3	1.19×10^{10}	16049
4	1.725×10^8	13907
5	24940	7173
6	627700	10997

D. Process Dynamics Description

The dynamic simulation of the reactive distillation process of methyl oleate production was conducted by using the developed converged steady-state reactive distillation model, from the 'Convergence Parameters' sub-menu, the dynamics option was chosen from the 'Steady-state/Dynamics' dropdown menu. The dynamics simulation were experimented using two run-time steps. The 'pick surges from flowsheet' option were checked from the 'Record surges' sub-menu. In the first run step, 18 mins with a 0.1 mins gap was used while for the second run step, 120 h with a 500 min gap was used. The dynamics data obtained from step 2 was used as the mole fraction of biodiesel in

the column base product surge, and these data were computed on an excel worksheet and MATLAB workspace for analysis [12].

E. Process Control

i. Transfer Function Generation

The process model was derived from the transfer function relationship between biodiesel mole fraction (output variable), decanter duty and reflux ratio (input variables) using the developed Aspen Plus® model data. The model type selected was a First-Order-Dead-Time Transfer (FOPDT) function model eq (12). The process model formulation was done within the MATLAB® environment.

$$x_{MOE}(s) = \frac{K_{pp}e^{-T_{dp}s}}{\tau_{pp}s+1}D(s) + \frac{K_{pd}e^{-T_{dd}s}}{\tau_{pp}s+1}R(s) \quad (12)$$

where x_{MOE} = Mole fraction of the methyl oleate,

D = Decanter duty column

R = Ratio of the column reflux.

ii. Simulink Modelling and Open-Loop Simulation

The equation (12), was modeled in MATLAB® Simulink environment after transferring and obtaining the function of the process by joining the various appropriate required blocks. The proposed open-loop case of the system for Simulink model (Figure 2), were examined by applying a unit step change using user-defined codes to the disturbance and manipulated variable and running the open-loop model of the Simulink.

iii. Proportional-integral-derivative (PID) Controller Tuning

A Proportional-Integral-Derivative (PID) control method was used for the control of the process to achieve a maximum fraction of the output variable (methyl oleate mole fraction).

Zeigler-Nichols and Cohen-Coon tuning techniques were applied using the expression in Table 3, with the transfer function of the controller given eq (13). The performance criteria values, Integral Absolute Error (IAE) and Integral Square Error (ISE) for both the Zeigler-Nichols and Cohen-Coon-tuned PID control systems were calculated to determine the controller parameters that will be more efficient in controlling the process.

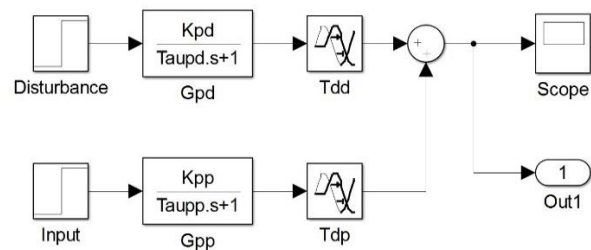


Figure 2: Proposed Open Loop Model of the Process [10]

Table 3: PID Tuning Parameters [6, 10]

Type of control	Zeigler-Nichols	Cohen-Coon
K_c	$\frac{K_u}{1.7}$	$\frac{1}{K} \frac{\tau_{pp}}{\tau_{pd}} \left(\frac{4}{3} + \frac{T_{dp}}{4\tau_{pp}} \right)$
τ_I	$\frac{P_u}{2}$	$T_{dp} \left(\frac{32 + \frac{6T_{dp}}{\tau_{pp}}}{13 + \frac{8T_{dp}}{\tau_{pp}}} \right)$
τ_D	$\frac{P_u}{8}$	$T_{dp} \left(\frac{4}{11 + \frac{2T_{dp}}{\tau_{pp}}} \right)$

iv. Simulink Modelling and Closed-loop Simulation

The developed closed-loop Simulink model (Figure 3) was used to carry out the closed-loop simulation using the value of the tuning parameters obtained by applying the expressions given in Table 3 after developing the Simulink model of the process and examining its open-loop behaviours. The closed-loop dynamics of

the process for both set-point tracking and disturbance rejection were also examine [13].

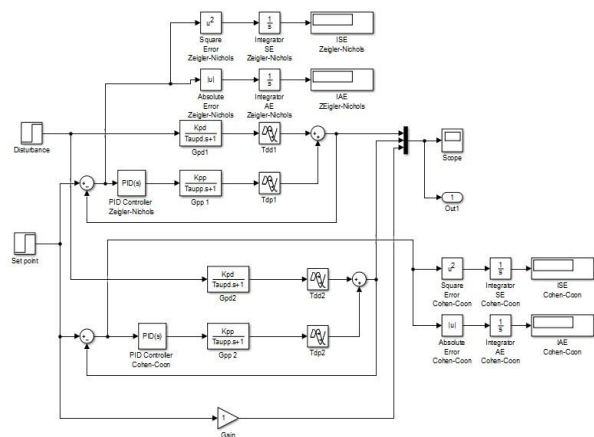


Figure 3 :Proposed Closed Loop Simulink Model of the Process with PID Controllers

III. Results and Discussion

A. Aspen Plus® simulation Result

The results gotten from the Aspen Plus® simulation for the methyl oleate production process, based on the process flowsheet output (Figure 4) are presented in Tables 4. The exit conditions for the three main streams are 101 °C, 104 °C and, 198 °C for the BIODIESEL, GLYCEROL and PURGE streams, respectively, at 1 bar. The BIODIESEL, GLYCEROL, and

PURGE streams have an output flow rate of 2.97 kmol/hr, 0.98 kmol/hr and 0.05 kmol/hr which represents 74%, 24.5% and 1.3% of the overall output stream, respectively.

The total mass (889.688 kg/hr) and mole flow (2.96879 kmol/hr) was greater than the BIODIESEL stream (1.65835 kg/hr) and lowest at the PURGE stream (0.051755 kmol/hr). The total volume flow (2.46622 m³/hr) and temperature (198 °C) are greater than the PURGE stream. The ratio of methanol to feed oil (triolein) in the two reactors, indicated that 85.9% of the oil was used up in the first reactor (R1), while the recycled methanol (MeOH-REC), in the second reactor (R2), combined with the remaining triolein from R1 in the ratio of 6.3:1 with a conversion rate of 96.7% (Table 5).

Table 5: Feed Oil Conversion

	Feed Ratio (Methanol: Feed Oil)	Feed Oil Concentration (%)	Feed Oil Conversion (%)
Reactor 1	3:1	25	85.9
Reactor 2	6.3:1	13.7	96.7

Table 6: Product Purity

Key Products	Stream	Total Flow (kmol/hr)	Total Flow (%)	Composition (wt.%)			
				Methyl-Oleate	Glycerol	Methanol	Triolein
Methyl Oleate	BIODIESEL	2.9688	(74.2%)	98.25	3.5x10 ⁴	0	0.0172
Glycerol	GLYCEROL	0.9795	(24.5%)	4.38x10 ⁴	99.96	0	1.98x10 ¹¹
Methanol	PURGE	0.0518	(1.3%)	0	0	100	0
Total		4.0000					

The properties of the biodiesel in the bottom stream along with the biodiesel are the remaining triolein and little unseparated glycerol (Table 6). The overall feed oil conversion was 98.3% which indicated that only 1.7% of unreacted triolein was left in the biodiesel produced. A maximum

of 73% (i.e., 98.3%*74.2) of biodiesel was obtained when the system is considered as a whole and hence, the need to vary important process parameters. The first reactor (R1) had the most duty with 84 kW as a result of the exothermic reactivity of the production process.

The net duty balance was computed and approximately 5 kW was needed for the whole biodiesel production process (Table 7).

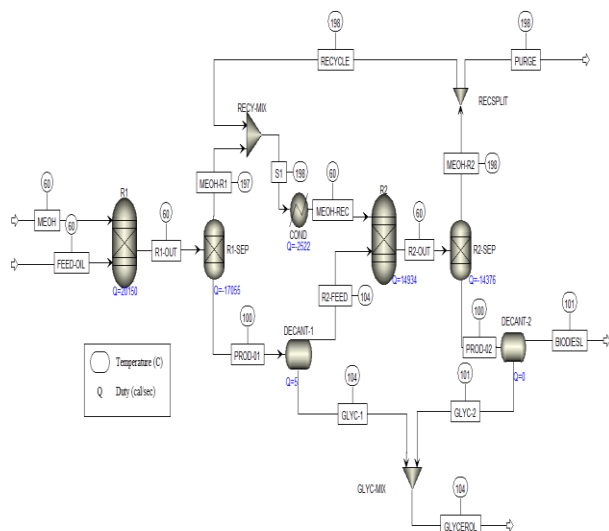


Figure 4: Aspen Plus® Simulation Result Flowsheet for the Production Process

B. Process Dynamics

The dynamic response of biodiesel and glycerol production obtained from the Aspen Plus process dynamics window is shown in Figures 5a and b, respectively. There was a step-wise increase in production after 6000 mins of simulation and maximum production of 98.3% biodiesel with a mass flow of 889.7 kg/hr at 101 °C and 1 bar, was attained. The sudden increase was a result of the combination of the effects caused by the reboiler duty (REBOILER-2 duty) and reflux ratio (R2 Methanol-Triolein ratio) as suggested by [12]. The glycerol production simulation dynamics window indicated that 90.30 kg/hr of glycerol was produced at a temperature of 111.1 °C and pressure of 1 bar. There is a continuous production even at 7200 mins and this could be attributed to the ongoing reaction between triolein and methanol left in the exit stream. However, the continuation of glycerol production may not be cost-effective since the focus is on the production of biodiesel.

Table 7: Process Duty

Process	Block	Duty (kcal/sec)	Duty (kW)
Reactor 1	R1	20.150	84.21
Reactor 1	R1-SEP	-17.051	-
Flash Drum			71.36
Reactor 1	DECANT-1	1.194	5
Decanter			
Condenser	COND	-2.521	-
			10.56
Reactor 2	R2	13.841	57.89
Reactor 2	R2-SEP	-14.383	-
Flash Drum			60.16
Reactor 2	DECANT-2	0	0
Decanter			
Net Balance		1.230	5.02

C. Process Control Results

i. Open-loop Simulation

The biodiesel mole fraction in the overall transfer function of the final BIODIESEL stream (controlled/output variable) to the reboiler (REBOILER-1) duty (manipulated variable) and the reflux ratio (R2 Methanol-Triolein ratio) was obtained as given in eq (14), using MATLAB developed codes,

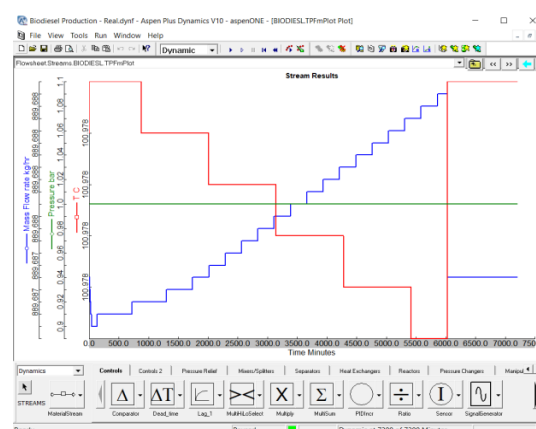


Figure 5a: Dynamic Response of Biodiesel (Methyl Oleate) Production

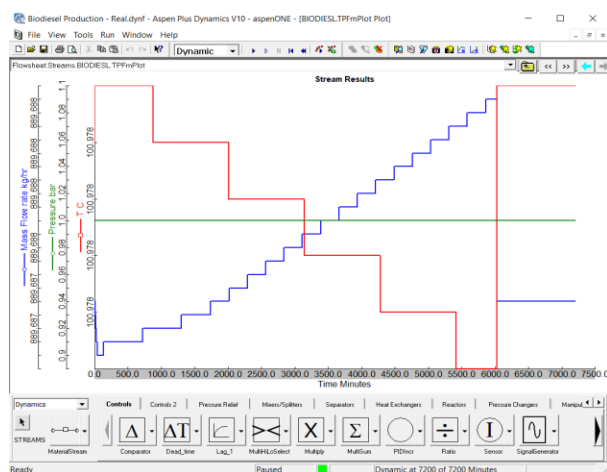


Figure 5b: Dynamic Response of Glycerol Production

$$x_{MOE}(s) = \frac{296.6e^{-0.002s}}{5336s + 1}D(s) + \frac{141e^{-0.002s}}{75360s + 1}R(s) \quad 14$$

Where:

$D(s)$ = Duty of the reboiler

$R(s)$ = Ratio of the reflux in Laplace transform pattern.

The transfer function model shows that the procedure has a big-time constant (τ_i) and small process gains (k_p) as a result of the difficulties in it due to the joining of reaction and separation processes in one production entity.

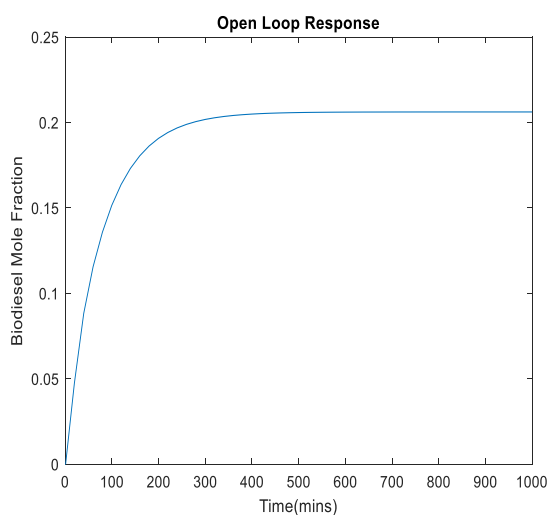


Figure 6: Open Loop Response of the Process to a Unit step-change in Decanter duty and Reflux ratio

A unit step change was applied using the developed transfer function via Simulink to both the manipulated and disturbance variables to derive the open loop response of the controlled output variable. A first-order system response was suggested (Figure 6) where a biodiesel mole fraction of 0.2 was accomplished at 400min at a steady state. This conformed with the value obtained in the procedure at a steady state. The change in decanter duty and reflux ratio could ensure stable dynamics in the system. However, it was essential to have a highly purified product faster than the settling period by this open-loop response, and this brought about system control.

ii. Controller Tuning Results

Control parameters K_c , τ_i and τ_d obtained were 8.31×10^3 , 4.00×10^{-3} , 1.00×10^{-3} for ZN, respectively, as well as 1.20×10^4 , 4.90×10^{-3} , and 0.70×10^{-3} for Cohen-Coon respectively, (Table 8), based on the script code developed from MATLAB, PID control method and the process transfer function relating methyl oleate mole fraction to decanter duty, the controller parameters gotten using both ZN and CC tuning methods (Table 8). The values of K_c (8.31×10^3) and τ_d were greater for ZN than CC but τ_i is greater for Cohen-Coon tuning method than Ziegler-Nichols. These values were used to control the system disturbance rejection and set-point tracking.

Table 8: Controller Parameters using Zeigler-Nichols and Cohen-Coon Tuning

Controller parameter	Zeigler-Nichols Tuning	Cohen-Coon Tuning
K_c	8.31×10^3	1.20×10^4
τ_i	4.00×10^{-3}	4.90×10^{-3}
τ_d	1.00×10^{-3}	0.70×10^{-3}

Where K_c is controller proportional gain, τ_i is integral time and τ_d is the derivative time

iii. Set-point Tracking Closes Loop Response

The closed-loop response model was put at a set point of 1.0-mole fraction of biodiesel and using the obtained controller parameters, there is no change in reflux ratio (disturbance variable) for trial and error, ZN and CC tuning methods for PID feedback control, using Simulink run through MATLAB script code as shown in Figures 7a-b. The PID control using both ZN and CC controller tuning techniques got to the desired set point of 0.8 biodiesel mole fraction (Figure 7a). However, the closed-loop response model could not be achieved because it exceeded a maximum mole fraction of 1 (Figure 7b). This indicated the necessity of applying a different approach to tune the PID controller of the process. The different PID controller parameters were obtained using the trial and error method, this is as shown in Table 9. It was observed that a set point of 0.8 methyl oleate mole fraction was achieved when the parameters gotten using the trial-and-error method were used to run the closed-loop model of the system using the MATLAB script code. This was achieved at about 100 mins without exceeding the maximum mole fraction of one.

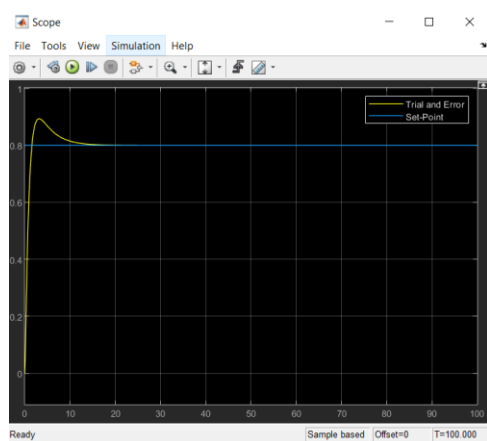


Figure 7a: Closed Loop Response of the Process Model to a Unit Step Change in Reflux Ratio (Trial and Error Method)

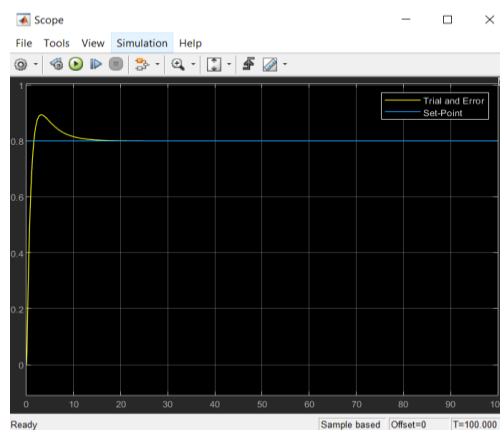


Figure 7b: Closed Loop Response of the Process Model to a Unit Step Change in Reflux Ratio (Set-point Tracking)

Table 9: Controller Parameters Obtained using Trial-and-Error Method

Parameter	Value
K_c	23.21
τ_i	5.75
T_d	-1.67

Where K_c is controller proportional gain, τ_i is integral time and τ_d is the derivative time

iv. Disturbance Rejection

It was observed from the result that using both ZN and CC controller parameters, the PID control approaches zero (Figure 8). This allowed the system to return to its previous value of zero mole fraction, a requirement for regulatory control of the system. Also, suppressed effects that the reflux ratio (disturbance variable) step-change could have on the process output.

PID control was tuned by CC and ZN, and the process output returned to the initial set point within about 100 mins. It was noted that the process response to both ZN and CC parameters had similar overshoots, however, the CC response decayed faster and has more oscillations. The CC tuning technique had a better performance compared to the ZN tuning technique in the regulatory control of this reactive distillation procedure for methyl oleate

production due to the lower value derived from ISE (1.126) and the IAE (3.909) (Table 10).

Table 10: Performance Metrics

Performance Criterion	Zeigler-Nichols Tuning	Cohen-Coon Tuning
ISE	1.269	1.126
IAE	4.09	3.909

ISE = Integral Square Error; IAE = Integral Absolute Error

D. Model Validation

The Aspen Plus simulation of the biodiesel production process was validated against the Chemcad version previously done by [12] (Figure 9). Approximately, 73% biodiesel (mole fraction of 0.729015) was formed at a 98% conversion rate from this study, while [12] could only get approximately 53% biodiesel (mole fraction of 0.5257) in the total product stream. Also, since glycerol is a very important product from this study, approximately 20% (mole fraction of 0.2062) of the output stream from this study represents glycerol production when compared to the previous study with just approximately 0.2% (mole fraction of 0.0015) [12].

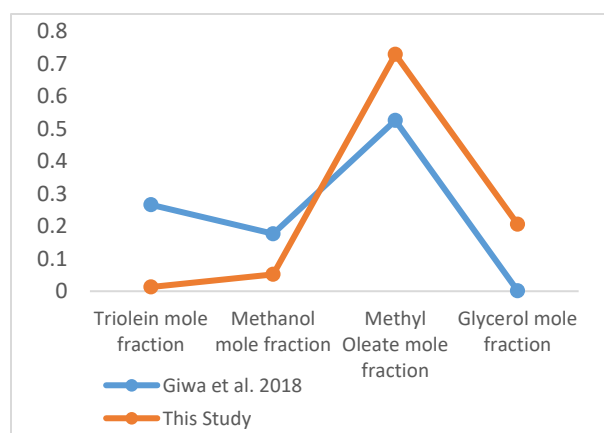


Figure 9 Comparison of the final products

More energy (≈ 5 kW) and time are required (Table 11) to achieve more conversion of triolein to methyl-oleate (biodiesel).

Table 11: Simulation Result for the Biodiesel Production Process

	[10]	This Study
Temperature (deg C)	116	101
Pressure (atm)	1	1
Vapour Fraction	0	0
Enthalpy (kJ/sec)	10441	14376
Triolein mole fraction	0.266	0.012985
Methanol mole fraction	1	0.0518
Methyl Oleate mole fraction	0.176	0.729015
Glycerol mole fraction	8	0.2062
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IV. Conclusions

Biodiesel was produced via a reactive distillation process from the transesterification reaction of Triolein and Methanol with a 98% conversion rate. Changes in decanter duty of the column and reflux ratio were seen, this subsequently resulted in stable dynamics response of methyl oleate mole fraction. PID control of the biodiesel process (mole fraction) was achieved. Cohen-Coon and Zeigler-Nichols tuning methods were found not to be suitable for controller tuning the process. The set-point tracking control of biodiesel mole fraction, using a set point of 0.8 and controller parameters of $K_c = 23.21$, $\tau_i = 5.75$, $\tau_d = -1.67$ was achieved. Cohen-Coon tuning gave better performance when compared with the Zeigler-Nichols tuning in the control of biodiesel mole fraction. PID control system was used to obtain high biodiesel purity in a reactive distillation column as a bottom product.

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