Dynamics and Regulatory Control of Biodiesel Purity from a Reactive Distillation Process

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Abstract – The open loop and disturbance rejection (regulatory) closed loop dynamic simulation of a reactive distillation process used for the production of biodiesel, details of which are given in the work of Giwa *et al.* (2015a), have been carried out in this work. The first-order-plus-dead-time transfer function model of the system used was developed with the aid of System Identification Toolbox of MATLAB. The input and output variables of the process were the reboiler duty of the column and the mole fraction of biodiesel obtained from the bottom section of the column, respectively while the disturbance variable was the reflux ratio. Both the open loop and the closed loop simulations of the system were achieved from the Simulink model of the system developed and run via written *m-file* codes. The results obtained from the open loop dynamic simulation of the process revealed that the disturbance variable had effects on the output (biodiesel mole fraction obtained from the bottom section) of the process because the steps applied to it made the output not to be at the desired set point. Furthermore, the application of P, PI and PID controllers tuned with Cohen-Coon and Ziegler-Nichols techniques showed that the process could be made to behave in a desired manner. However, the response given by the simulation of the system with P-only controller was found not to settle at the desired set-point value, while the simulations with PI and PID were able to bring the output to the desired reference value. Moreover, the performance of PID controller tuned with Cohen-Coon technique was found to be the best among the ones considered for this case of regulatory control of the process because its IAE and ISE were obtained to be the lowest. This finding was observed to be in contrary to that of Giwa *et al.* (2015b) who obtained that the best controller for biodiesel production using reactive distillation process was PID controller tuned with Ziegler-Nichols. However, it was discovered that their own work considered set-point tracking only but not disturbance rejection. This has, therefore, shown that a particular controller type may not be able to handle both set-point tracking and disturbance rejection in a best way in all cases.

Keywords: Biodiesel, reactive distillation, System Identification Toolbox, MATLAB, disturbance rejection control.

INTRODUCTION

Biodiesel is an alternative fuel that is currently receiving attention owing to the limited availability of conventional petroleum diesel and, also, due to environmental concerns. This material can be used to replace petroleum diesel without any modification because of their similar properties (Simasatitkul *et al*., 2011; Giwa *et al.*, 2014; Giwa *et al.*, 2015a). Furthermore, it has a number of advantages as it can be derived from a renewable domestic resource. In addition, it reduces emission of carbon dioxide apart from being nontoxic and biodegradable (Wang *et al.*, 2004; Jaya and Ethirajulu, 2011; Giwa *et al.*, 2014; Giwa *et al.*, 2015a).

Biodiesel can be obtained in high purity by carrying out an esterification reaction of a fatty acid and an alcohol via a reactive distillation process (Giwa *et al.,* 2014). The use of reactive distillation process is preferred for the production of biodiesel in order to overcome the problems associated with the use of conventional batch reactor, which include low conversion, heavy capital investments and high energy costs (Kusmiyati and Sugiharto, 2010; Giwa *et al.*, 2014; Giwa *et al.*, 2015a; Giwa *et al.*, 2015b).

Generally, reactive distillation is defined as a process that combines both separation and chemical reaction in a single unit (Giwa and Giwa, 2012). It is found to be more advantageous than a conventional process having reaction and separation sections separately (Al-Arfaj and Luyben, 2002a; Giwa and Karacan, 2012b; Giwa, 2013a; Giwa and Karacan, 2012d; Giwa and Karacan, 2012e; Giwa and Karacan, 2012f; Giwa and Karacan, 2012g; Giwa, 2012; Giwa and Giwa, 2013a; Giwa, 2013a; Giwa *et al.*, 2013; Giwa and Giwa, 2013b; Giwa, 2014). It has been used in a small number of industrial applications for many years, but the last decade has shown an increase in both its research and applications (Al-Arfaj and Luyben, 2002b; Giwa *et al.*, 2015a). In reactive distillation, the temperature levels for both reaction and vapour-liquid equilibrium must overlap (Al-Arfaj and Luyben, 2002a; Giwa *et al.*, 2015a). By carrying out chemical reaction and separation in one process, the operating and investment costs can be minimized. Some additional benefits offered by reactive distillation technology include: (i) increased yield, because of overcoming chemical and thermodynamic equilibrium limitations, (ii) improved selectivity via suppression of side reactions (Giwa and Karacan, 2012c), (iii) reduced energy consumption, due to effective utilization of reaction heat, in the case of exothermic reactions, (iv) avoidance of hot spots by simultaneous liquid evaporation, (v) ability to separate close boiling components (Prakash et al., 2011; Giwa *et al.*, 2015a) and (vi) ability to avoid azeotropes (Giwa and Karacan, 2012a). Due to these advantages and with growing process understanding, the chemical process industry has developed an increasing number of processes based on reactive distillation (Bock *et al.*, 1997; Giwa *et al.*, 2015a). However, this process is not extensively used in industry because it is perceived that understanding its dynamics will be problematic and that its operation and control are more difficult than those of the conventional systems.

In order to address those issues concerning the dynamics and control of the process, different investigations have been carried out on it by some researchers. For instance, Sneesby *et al.* (1997) carried out the dynamic simulation and control of reactive distillation process used for ethyl *tert*-butyl ether synthesis. They presented recommendations for the control of the reactive column of this type such as the need for early addressing of the control issues in the design process. Bock *et al.* (1997) developed a structure for the control of a reactive column with recovery by analysing the steady state and dynamic sensitivity of the column with respect to possible disturbances and manipulated variables. Sneesby *et al.* (1999) worked on an ethyl tert-butyl ether reactive distillation column as a case study to demonstrate how a two-point control configuration recognizing the importance of both composition and conversion could be developed and implemented for a reactive distillation process. Kumar and Daoutidis (1999) investigated the dynamic behaviour and control of a reactive distillation column used for the production of ethylene glycol. They derived a detailed tray-by-tray model that explicitly included the vapour-phase balances. Also developed in their work was a nonlinear controller that yielded good performance with stability in the high-purity region. The superior performance of the developed controller over linear PI controllers was demonstrated in the work through simulations. Monroy-Loperena *et al.* (2000) studied the control problem of an ethylene glycol reactive distillation column in order to regulate the ethylene glycol composition in the product by manipulating the reboiler boil-up ratio. A new idea for robust stabilization based on an analysis of the underlying input/output bifurcation diagram and on modelling error compensation techniques was proposed in the work. Al-Arfaj and Luyben (2000) studied the closed loop control of a reactive distillation column with two products and discovered that single end temperature control could be used to keep both products at or above specified purity values, even in the presence of large disturbances, because the reaction zone holdup was sufficiently large. Vora and Daoutidis (2001) studied the dynamics and control of an ethyl acetate reactive distillation process and designed model-based linear and nonlinear state feedback controllers along with classical single-input single-output (SISO) proportional-integral (PI) controllers. The superior performance of the nonlinear controller over both the linear and the classical PI controllers was demonstrated in the work. Grüner *et al.* (2003) carried out the simulation of an industrial reactive distillation column unto which asymptotically exact input/output-linearization was applied and discovered that, in comparison with a well-tuned linear controller, it showed a superior performance with respect to set-point changes and disturbances, even in the presence of unknown input delays. Khaledi and Young (2005) studied the nonlinearity of a reactive distillation column producing ethyl *tert*-butyl ether and developed a 2 x 2 unconstrained model predictive control scheme for product purity and reactant conversion control using the process dynamics approximated by a first-order-plus-dead-time model as an estimate of the process model of the controller. They found from the study carried out that the controller was very efficient for disturbance rejection and set-point tracking. Völker *et al.* (2007) designed a multivariable controller for a medium-scale semi-batch reactive distillation column and demonstrated that the controller performed well for large set-point changes and in the face of disturbances. Furthermore, Giwa and Karacan (2012a) used two black-box models (AutoRegressive with eXogenous Inputs (ARX) and AutoRegressive Moving Average with eXogenous Inputs (ARMAX) models) they developed using experimental data to study the dynamics of a reactive distillation column used for ethyl acetate production, and discovered that the performance of ARMAX model was better because of its higher calculated fit value. They also found that ARX model was faster in getting to steady state upon the application of a step input to the two models. However, the models developed in their work were not utilized to study the control of the process. Giwa and Karacan (2012c) developed dynamic models for a reactive packed distillation starting from first principles and solved them (the developed models) with the aid of MATLAB. The comparisons made between the experimental and the theoretical results obtained revealed that there were good agreements between them because the calculated percentage residuals were small. Also, the models they developed were not used for the control of the column in the work. Giwa and Karacan (2012d) studied the application of decouplers in the design of model predictive controllers for a reactive distillation process used for the production of ethyl acetate. In the work, top segment temperature, reaction segment temperature and bottom segment temperature were taken as the controlled variables while reflux ratio, feed ratio and reboiler duty were the manipulated variables of the control system. The results obtained from the work showed that the performance of neural network decoupling model predictive controller (NNDMPC) was better than that of transfer function decoupling model predictive controller (TFDMPC) as the integral squared error values calculated for the top segment and the reaction segment temperatures from the control simulation carried out with NNDMPC were found to be less than those of the TFDMPC. Moreover, Giwa and Karacan (2012e) applied decoupling proportional-integral-derivative control to a reactive distillation column for set-point tracking and disturbance rejection using tuning parameters calculated with Ziegler-Nichols and Cohen-Coon techniques, and the results obtained from the simulations of the work showed that decoupling PID control with Cohen-Coon tuning technique was better than that of Ziegler-Nichols, for the process considered in the work. Giwa *et al.* (2015b) studied the dynamics and set-point tracking control of a reactive distillation process used for biodiesel production by taking the biodiesel purity obtained from the bottom section of the column as the controlled variable, the reboiler duty as the manipulated variable and considering Cohen-Coon and Ziegler-Nichols tuning techniques. From the comparisons made among the controllers considered by them, they were able to discover that the best one for the system was PID controller tuned with Ziegler-Nichols method because its integral absolute error (IAE) and integral squared error (ISE) were found to be the lowest.

It can be seen from the literature review carried out that the dynamics and control of a reactive distillation process for biodiesel production has been carried out, but the control there was a servo (set-point tracking) type. It is important that the behaviour of the system to the presence of a disturbance be known so as to know how it should be handled in that case because, even, when trying to make a system to follow a particular set point, any disturbance can set into it anytime. Therefore, this work was carried out to study

the dynamics and perform regulatory (disturbance rejection) control of a reactive distillation process used for the production of biodiesel from an esterification reaction.

METHODOLOGY

Transfer Function Modelling of the Process

The process model used in this work was formulated by adding the transfer function relation between the output variable (reflux ratio) and the disturbance variable developed using the data generated from the prototype plant setup with the aid of Aspen HYSYS and reported in detail in the work of Giwa *et al.* (2015a). The process model formulation was done with the aid of the System Identification Toolbox contained in MATLAB (MathWorks, 2015). The type of the transfer function model of the disturbance relation was also chosen to be the same as that of the main process transfer function, that is, first-order-plus-dead-time, and this made the model of the process to be as shown in Equation (1).

$$x_{biod}(s) = \frac{K_p e^{(-T_{dp}s)}}{\tau_p s + 1} Q(s) + \frac{K_d e^{(-T_{dd}s)}}{\tau_d s + 1} R(s) \qquad (1)$$

Simulink Modelling and Open Loop Simulation of the Process

After obtaining the transfer function of the process, as given in Equation (1), it was thereafter modelled in Simulink, also contained in MATLAB, by combining the different appropriate blocks required, and the developed Simulink model for the open loop case of the system is as shown in Figure 1. Furthermore, the open loop dynamics of the process was studied by applying step changes to the disturbance variable of the developed model, while keeping the main input variable of the process at its steady state value, and running it using the codes written in *m-file* of MATLAB.
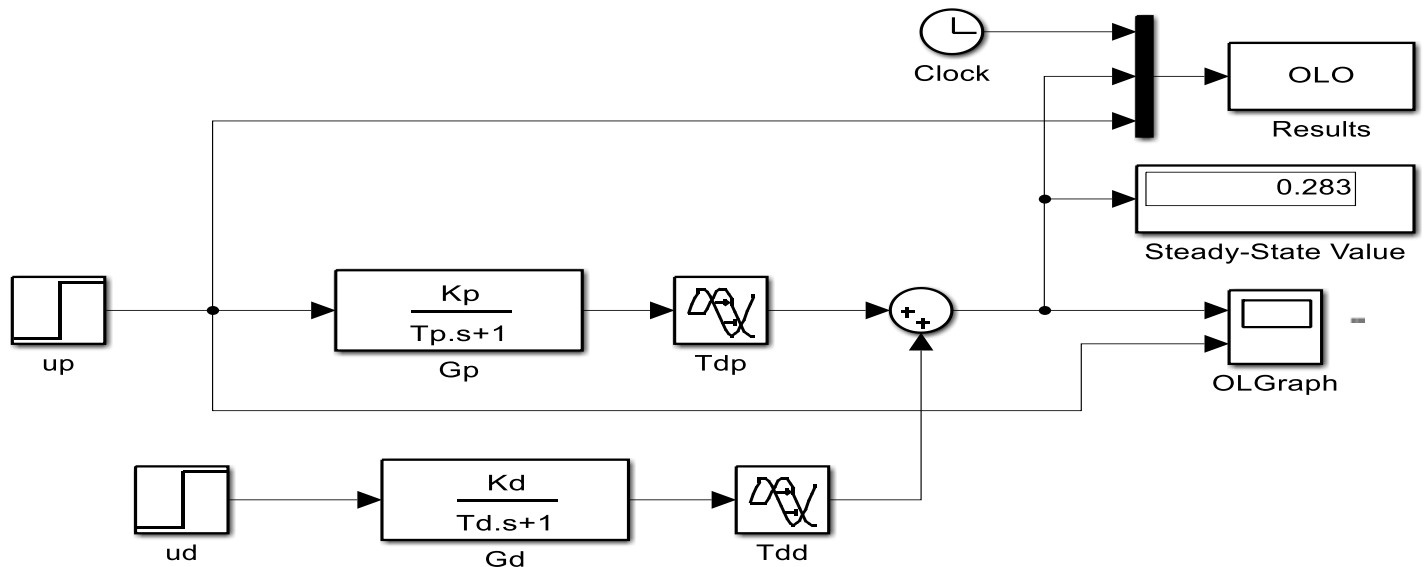

Figure 1. Open loop model of the process

Simulink Modelling and Closed Loop Simulation of the Process

After developing the model of the process and studying its open loop behaviours, its closed loop simulations were also carried out using the closed loop models, shown in Figures 2 – 4, developed with the aid of Simulink for P-only, PI and PID controllers, respectively. The developed closed loop models of the process were simulated for regulatory (disturbance rejection) with the controllers (P-only, PI and PID) tuned with both Cohen-Coon and Ziegler-Nichols techniques by applying a unit step change to the steady state value of the disturbance variable. The controlled, manipulated and disturbance variables of the closed loop models were biodiesel bottom mole fraction, reboiler duty and reflux ratio, respectively.

The calculations of the values for the tuning parameters of the controllers, using Cohen-Coon and Ziegler-Nichols tuning techniques, were carried out by taking the transfer function of the controllers to be as shown in Equation (2) and applying the expressions given in Table 1.

$$G_c(s) = K_c\left(1 + \frac{1}{\tau_I s} + \tau_D s\right) \qquad (2)$$
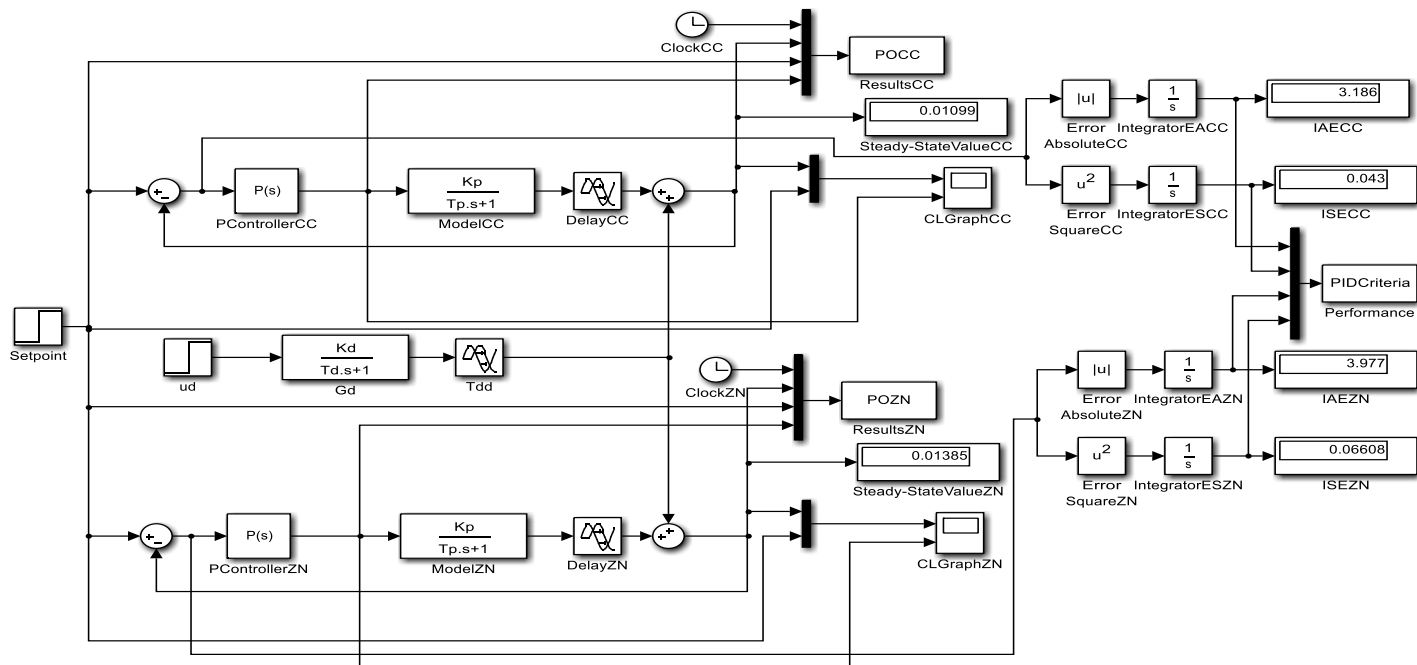

Figure 2. Closed loop model of the process with P-only controllers tuned with Cohen-Coon and Ziegler-Nichols techniques
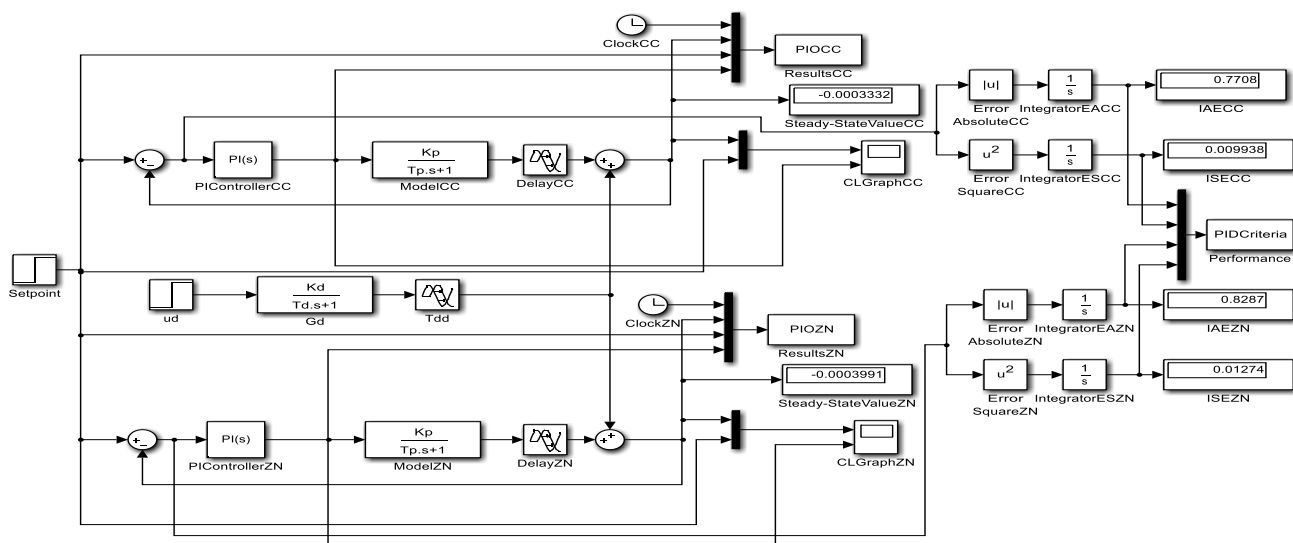

Figure 3. Closed loop model of the process with PI controllers tuned with Cohen-Coon and Ziegler-Nichols techniques

Figure 4. Closed-loop model of the process with PID controllers tuned with Cohen-Coon and Ziegler-Nichols techniques

Table 1: Cohen-Coon and Ziegler-Nichols tuning parameter expressions

Type of Control	Cohen-Coon Tuning Technique	Ziegler-Nichols Tuning Technique
Proportional (P)	$K_c = \dfrac{1}{K_p}\dfrac{\tau}{T_d}\left(1+\dfrac{T_d}{3\tau}\right)$	$K_c = \dfrac{K_u}{2}$
Proportional-Integral (PI)	$K_c = \dfrac{1}{K_p}\dfrac{\tau}{T_d}\left(0.9+\dfrac{T_d}{12\tau}\right)$ $\tau_I = T_d\dfrac{30+3T_d/\tau}{9+20T_d/\tau}$	$K_c = \dfrac{K_u}{2.2}$ $\tau_I = \dfrac{P_u}{1.2}$
Proportional-Integral-Derivative (PID)	$K_c = \dfrac{1}{K_p}\dfrac{\tau}{T_d}\left(\dfrac{4}{3}+\dfrac{T_d}{4\tau}\right)$ $\tau_I = T_d\dfrac{32+6T_d/\tau}{13+8T_d/\tau}$ $\tau_D = T_d\dfrac{4}{11+2T_d/\tau}$	$K_c = \dfrac{K_u}{1.7}$ $\tau_I = \dfrac{P_u}{2}$ $\tau_D = \dfrac{P_u}{8}$

Source: Stephanopoulos, 1984

RESULT AND DISCUSSION

The resulting model obtained for the process after the incorporation of the disturbance transfer function model term was as given in Equation (3),

$$x_{biod}(s) = \frac{0.3382e^{(-8.999s)}}{248.43s+1} Q(s) + \frac{0.283e^{(-1.993s)}}{151.87s+1} R(s) \qquad (3)$$

From Equation (3), it was observed that all the parameters (static gain, time constant and dead time) of the disturbance transfer model were, in magnitude, less than those of the main process transfer function model of the process. The lower time constant of the disturbance model was found to be an indication of the fact that the disturbance would respond faster than the main process if the same unit step change is applied to the two of them.

Shown in Figures 5 – 7 are the open-loop responses of the process to 1, 2, and 3 step unit(s) change in the reflux ratio, which was the disturbance variable of the process. As can be seen from the figure, the process was able to respond to the changes in the reflux ratio, but not as desired. Normally, it was expected of the output of the system to remain at zero, which was its set point in this case, even, in the presence of any disturbance. However, the observations made from the results (Figures 5 – 7) indicated that the simulation of the model with the applied step input changes were correct, as can be noticed from the magnitudes of the output variable (biodiesel mole fraction) of the three simulations carried out.

As such, it was clear from the deductions made from the figures that the process had to be controlled so as to keep its output at its desired set point (in this case of regulatory control, zero).

Based on this, the results obtained from the control of the process carried out with a unit step change in the disturbance variable using proportional (P), proportional-integral (PI) and proportional-integral-derivative (PID) controllers were as given in Figure 8 – 10. In each of the figures, the closed-loop dynamic responses of the process to a unit step change in the disturbance variable obtained using the three controller types tuned with Cohen-Coon and Ziegler-Nichols techniques are shown.
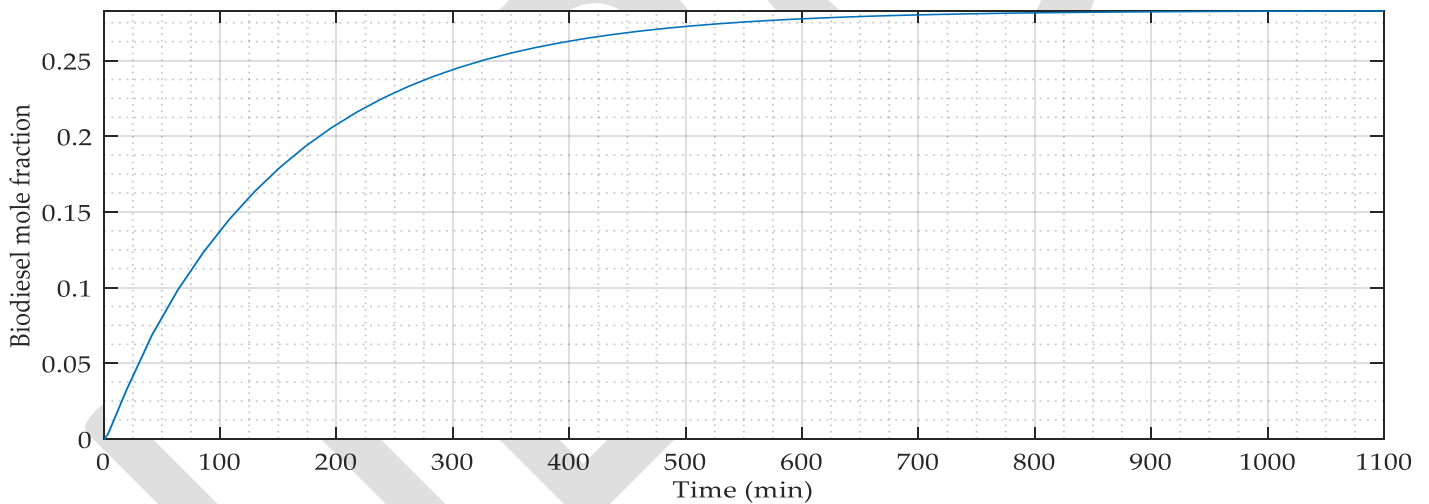

Figure 5. Open loop response of the process to 1 unit step change in the reflux ratio of the process
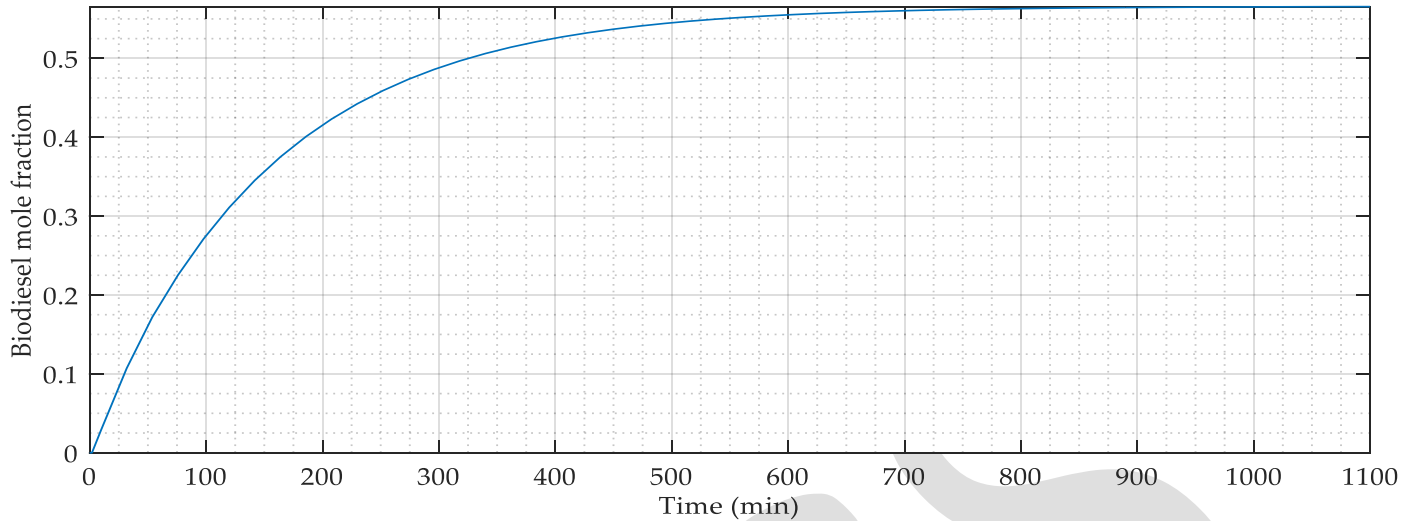
Figure 6. Open loop response of the process to 2 units step change in the reflux ratio of the process
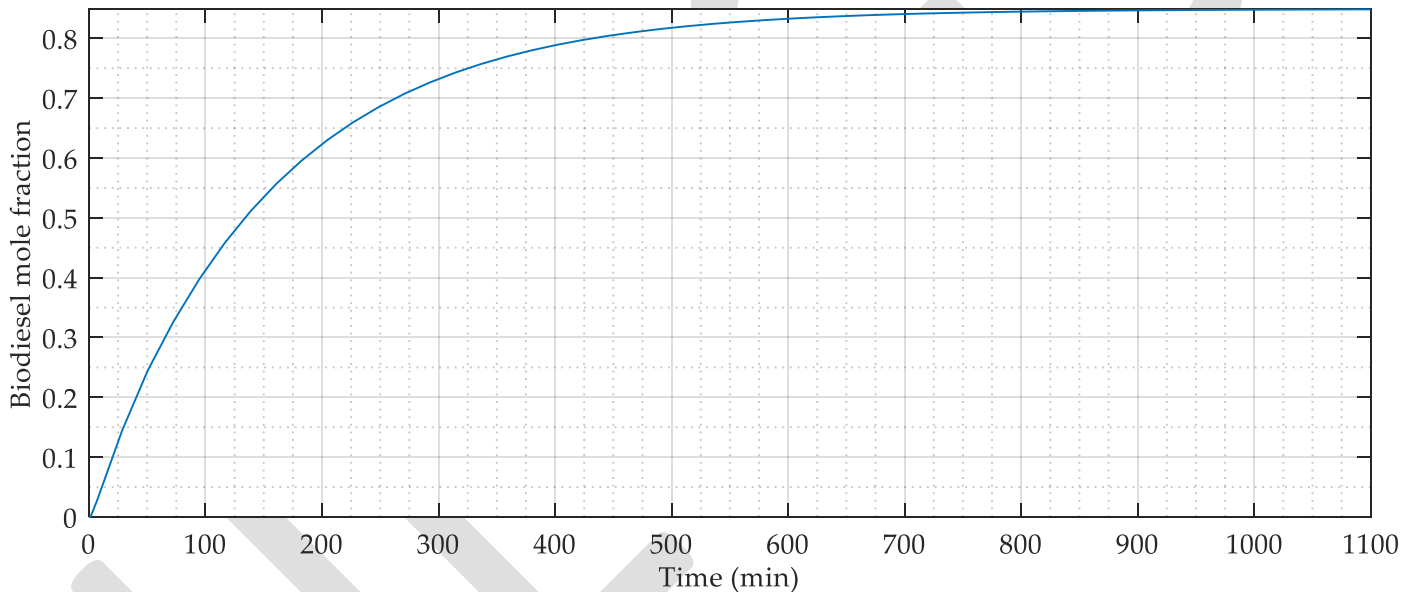

Figure 7. Open loop response of the process to 3 units step change in the reflux ratio of the process

From Figure 8, it was clear that the performance of P-only controller was not good for the process because it was not able to make the system settle at the desired set point at the end of the simulation time of 450 min chosen. Looking at the results, there seemed to be offsets associated with the responses of the P-only controllers tuned with Cohen-Coon and Ziegler-Nichols techniques, but the response of the Cohen-Coon tuning technique was found to be closer to the desired set-point of the system than that of the Ziegler-Nichols. From the responses given in Figure 8, it was very obvious that the performance of Cohen-Coon was better than that of Ziegler-Nichols for this case of the P-only controller in handling this process of biodiesel production for the regulatory control.
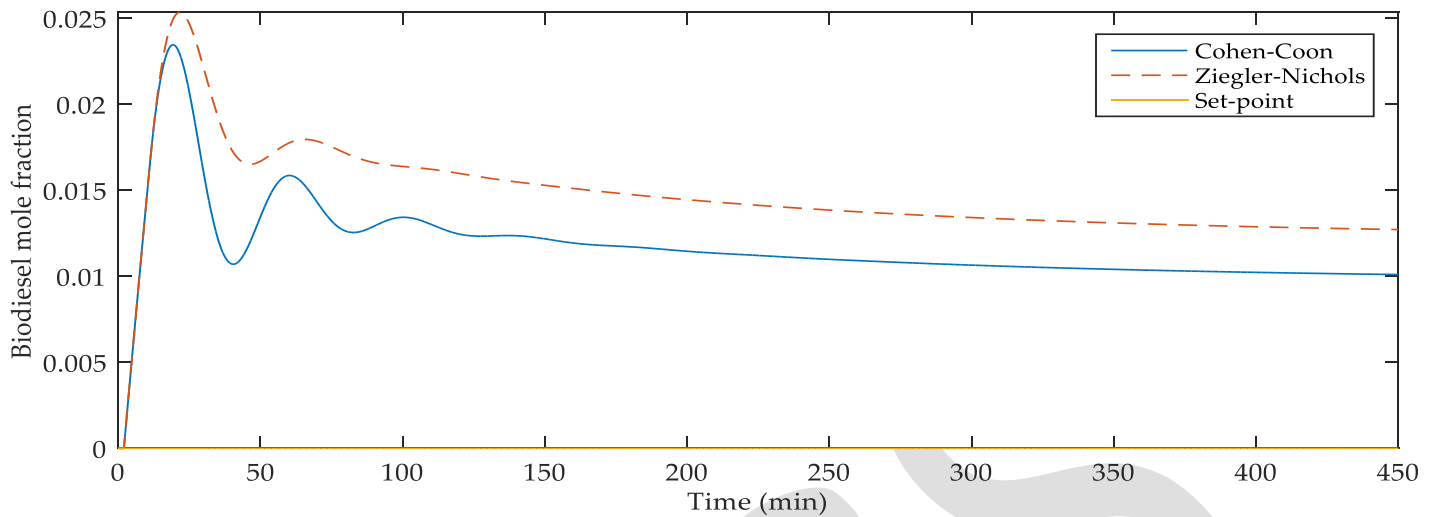
Figure 8. Closed loop responses of P-only controlled process

Shown in Figure 9 are the closed loop dynamic responses of the system obtained when Cohen-Coon and Ziegler-Nichols tuned PI controllers were used to control it. From the figure, it was seen that the performances of the two controllers (PI controller tuned with Cohen-Coon and Ziegler-Nichols techniques) were better than those of the P-only controllers considered before. Moreover, it was also noticed that even though the overshoot of the PI controller tuned with Ziegler-Nichols method was higher than that of the one tuned with Cohen-Coon technique, its number of oscillations was found to be lesser and decay faster. All in all, the responses of both of them were found to get stabilized at the desired set point within the simulation time of 450 min considered.
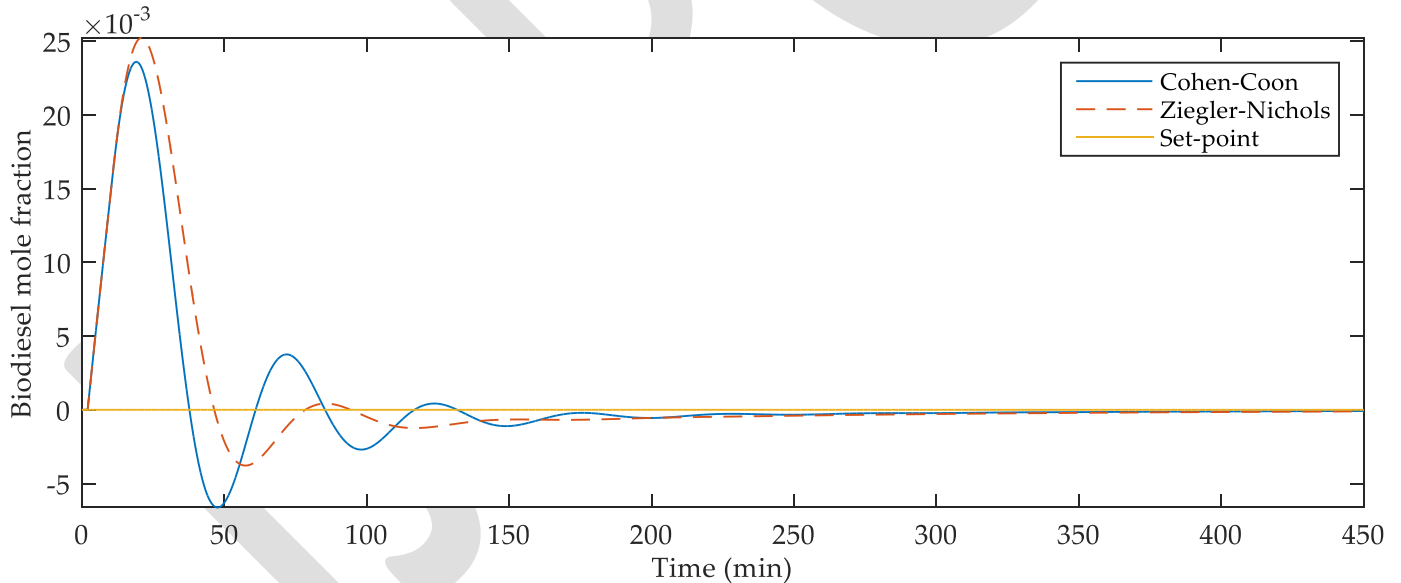

Figure 9. Closed loop responses of PI controlled process

Although the performances of the Cohen-Coon and Ziegler-Nichols tuned PI controllers were found to be better than those of the P-only controllers and satisfactory, it was still deemed necessary to get the responses of the system when controlled with Cohen-Coon and Ziegler-Nichols tuned PID controllers, and given in Figure 10 are the responses obtained.
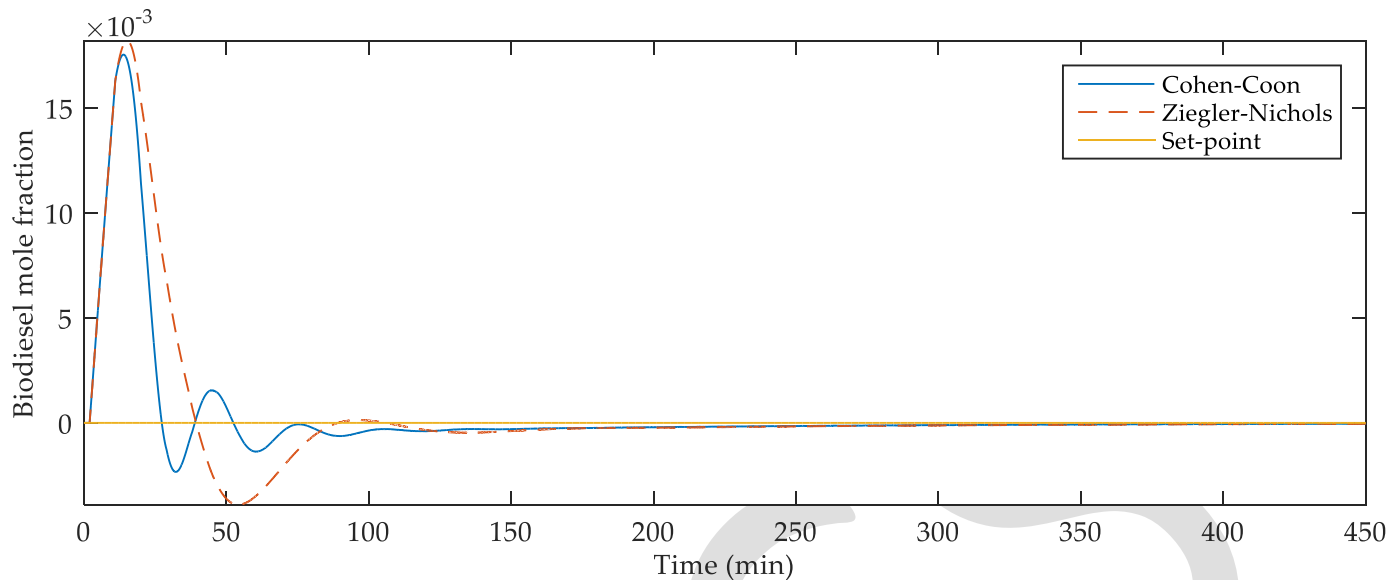
Figure 10. Closed loop responses of PID controlled process

From the figure (Figure 10), it was seen that the response with the higher overshoot was the one obtained from the simulation carried out using PID controller tuned with Ziegler-Nichols technique, just as it was obtained in the case of the PI controller investigated earlier, but its magnitude was found to be less than that given by the PI controller tuned with the same technique (Ziegler-Nichols). Also noticed from the results given in Figure 10 was that less oscillations were found to occur with the use of PID controllers.

In order to get a clearer picture on the performances of the controllers considered, their performance criteria values were calculated, and given in Table 2 are the results obtained from the estimation of the integral absolute error (IAE) and the integral squared error (ISE) of the controllers (P, PI and PID) tuned with Cohen-Coon and Ziegler-Nichols techniques.

Table 2. Performance criteria values of the controllers

Controller	Cohen-Coon Tuning Technique		Ziegler-Nichols Tuning Technique	
	IAE	ISE	IAE	ISE
P	5.28	0.06	6.61	0.10
PI	0.80	0.01	0.87	0.01
PID	0.37	0.00	0.53	0.01

From the results given in Table 2, for this case of disturbance rejection (regulatory) control study of the reactive distillation process, the performances of the controllers tuned with Cohen-Coon technique were discovered to be better than those of the ones tuned with Ziegler-Nichols technique because most of their corresponding IAEs and ISEs were found to be lower.

CONCLUSION

The results obtained from the open loop dynamic simulation of the reactive distillation process used for biodiesel production have shown that the disturbance variable had effects on the output of the process because the steps applied to it made the output not to be at the desired set point. Furthermore, the application of P, PI and PID controllers tuned with Cohen-Coon and Ziegler-Nichols techniques revealed that the process could be made to behave as desired. However, the response given by the simulation of the system with P-only controller was found not to settle at the desired set-point value, while the simulations with PI and PID were able to bring the output to the desired reference value within the time considered. Moreover, the performance of the PID controller tuned with Cohen-Coon technique was found to be the best among the ones studied for this case of the regulatory control of biodiesel production using reactive distillation because its IAE and ISE were found to be the lowest. This finding was, actually, found to be in contrary to that of Giwa *et al.* (2015b) who obtained that the best controller for the biodiesel production system was PID controller tuned with Ziegler-

Nichols. However, their own work considered set-point tracking only. This has, therefore, shown that a particular controller type may not be able to handle both set-point tracking and disturbance rejection best in all cases.

NOMENCLATURE

τ_D	Derivative time of the controller (min)
τ_d	Time constant of the disturbance process model (min)
τ_I	Integral time of the controller (min)
τ_p	Time constant of the main process model (min)
$G_c(s)$	Controller transfer function
$G_p(s)$	Process transfer function
IAE	Integral Absolute Error
ISE	Integral Squared Error
K_c	Proportional gain of the controller
K_d	Static gain of the disturbance process model
K_p	Static gain of the main process model
K_u	Ultimate gain
NNDMPC	Neural Network Decoupling Model Predictive Controller
P	Proportional
PI	Proportional-Integral
PID	Proportional-Integral-Derivative
P_u	Ultimate period (min/cycle)
Q	Reboiler duty (kJ/s)
R	Reflux ratio
SISO	Single-input single-output
T_{dd}	Dead time of the disturbance process model (min)
T_{dp}	Dead time of the main process model (min)
TFDMPC	Transfer Function Decoupling Model Predictive Controller
x_{biod}	Bottom biodiesel mole fraction

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