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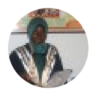

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Dynamics and Servo Control of Biodiesel Purity from a Reactive Distillation Process

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Abstract – This research work has been carried out to study the dynamics and apply some techniques to perform set-point tracking (servo) control on the purity of biodiesel obtained from a reactive distillation process via the esterification reaction between palmitic acid and methanol reported in the work of Giwa *et al.* (2015). The model used for the study of the system was developed with the aid of System Identification Toolbox of MATLAB as a first order transfer function. The input variable of the process model was the reboiler duty of the column while its output variable was the mole fraction of biodiesel obtained from the bottom section of the reactive distillation column of the process. The open loop simulation of the developed model revealed that the system was a stable one because its response could attain a steady state when simulated. Furthermore, the closed loop responses obtained from the simulations of the process using proportional (P), proportional-integral (PI) and proportional-integral-derivative (PID) controllers tuned with Cohen-Coon and Ziegler-Nichols techniques showed that the best controller for the system was PID controller tuned with Ziegler-Nichols method because, apart from the graphical comparisons made among the various controllers and techniques, the integral absolute error (IAE) and the integral squared error (ISE) of that combination (PID controller tuned with Ziegler Nichols technique) were found to be the lowest, implying that that was the best for the process considered.

Keywords: Biodiesel, reactive distillation, System Identification Toolbox, MATLAB, Cohen-Coon, Ziegler-Nichols.

1 INTRODUCTION

Production of biodiesel has been discovered to be carried out using a batch reactor or by employing an integrated method because Giwa *et al.* (2014) have found out that the product could be obtained in high purity from esterification reaction of fatty acids with alcohols (especially, methanol or ethanol) and Omota *et al.* (2003) proposed the use of batch reactor for the esterification reaction of the biodiesel production, even though, according to Gao *et al.* (2007), biodiesel production from the esterification reaction in the conventional batch reactor was having many problems due to low conversion, heavy capital investments and high energy costs, which thereby brought about the development of a novel technology referred to as "reactive distillation" (Kusmiyati and Sugiharto, 2010; Giwa *et al.*, 2014; Giwa *et al.*, 2015) for the production.

Reactive distillation is a process that combines both separation and chemical reaction in a single unit. It is very attractive whenever conversion is limited by reaction equilibrium (Balasubramhanya and Doyle III, 2000; Lai et al., 2007; Giwa and Karacan, 2012e; Giwa and Giwa, 2012; Giwa, 2013). It is an excellent alternative to conventional flowsheets with different reaction and separation sections (Al-Arfaj and Luyben, 2002; Giwa and Karacan, 2012d) as it combines the benefits of equilibrium reaction with distillation to enhance conversion (Giwa and Karacan, 2012a; Giwa and Karacan, 2012c). Combining reaction and distillation has several advantages such as shift of chemical equilibrium and an increase of reaction conversion by simultaneous reaction and separation of products, suppression of side reactions and utilization of heat of reaction for mass transfer operation (Giwa and Karacan, 2012b). The utilization of heat of reaction for mass transfer operation, which resulted into low external energy consumption of the process, actually, give rise to reduced investment and operating costs (Giwa, 2012). This is one of the main advantages of this reactive distillation process. However, this combination of reaction and distillation in a single unit makes the dynamics and control of the process a little bit challenging.

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The researches on the study of the dynamics and control of the process (reactive distillation) include that of Sneesby *et al.* (1997) in which the dynamic simulation and control aspects of reactive distillation for the synthesis of ethyl tert-butyl ether was presented and general recommendations for the control of the reactive column of this type including the need for addressing the control issues early in the design process were emphasized. Bock *et al.* (1997) developed a control structure for a reactive column with a recovery column by analysing the column's steady state and dynamic sensitivity of possible disturbances and manipulated variables. Sneesby *et al.* (1999) used an ethyl tert-butyl ether reactive distillation column as a case study to show how a two-point control configuration, which recognized the importance of both composition and conversion, could be developed and implemented for a reactive distillation process. Kumar and Daoutidis (1999) studied the dynamic behaviour and control of an ethylene glycol reactive distillation column by deriving a detailed tray-by-tray model that explicitly included the vapor-phase balances. They developed a nonlinear controller that yielded good performance with stability in the high-purity region with the aid of a physical insight into the nonminimum phase behaviour, and the superior performance of the developed controller over linear PI controllers was demonstrated through simulations. Monroy-Loperena *et al.* (2000) also studied the control problem of an ethylene glycol reactive distillation column with the control objective of regulating the ethylene glycol composition in the product by manipulating the reboiler boil-up ratio. They proposed a new idea for robust stabilization based on an analysis of the underlying input/output bifurcation diagram and on modelling error compensation techniques. Al-Arfaj and Luyben (2000) explored the closed-loop control of a reactive distillation column with two products and discovered that single-end temperature control could keep both products at or above specified purity values, even for large disturbances, if reactive-zone holdup was sufficiently large. Vora and Daoutidis (2001) studied the dynamics and control of an ethyl acetate reactive distillation system and designed model-based linear and nonlinear state feedback controllers, along with conventional single-input single-output (SISO) PI controllers. They demonstrated the superior performance of the nonlinear controller over both the linear controller and the conventional PI controller. Grüner *et al.* (2003) applied asymptotically exact input/output-linearization to an industrial reactive distillation column and found through simulation studies 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) investigated the nonlinearity of an ethyl tert-butyl ether reactive distillation column and developed a 2 x 2 unconstrained model predictive control scheme for product purity and reactant conversion control by using the process dynamics approximated by a first-order plus dead time model to estimate the process model for the model predictive controller. They found 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 showed experimentally that the controller performed well for large set-point changes and in the face of process disturbances. Furthermore, Giwa and Karacan (2012a) used two black-box models (AutoRegressive with eXogenous Inputs (ARX) model and AutoRegressive Moving Average with eXogenous Inputs (ARMAX) model) they developed using experimental data to study the dynamics of a reactive distillation column for ethyl acetate production, and found out that ARMAX model was better in performance because of its higher calculated fit value but that ARX model was faster in getting to steady state when a step input was applied to both models. The models they developed were not utilized to study the control of the process in their work. Also, Giwa and Karacan (2012b) developed dynamic models for a reactive packed distillation started from first principles for ethyl acetate production. Solving the developed models with the aid of MATLAB, comparisons were made between the experimental and theoretical results by calculating the percentage residuals for the top and bottom segment temperatures of the column, and the results revealed that there were good agreements between the experimental and theoretical top and bottom segment temperatures because the calculated percentage residuals were small. However, the models they developed were not used for the control of the column in the work. Giwa and Karacan (2012c) demonstrated the application of decouplers in the design of model predictive controllers for a reactive distillation for the production of ethyl acetate by taking top segment temperature, reaction segment temperature and bottom segment temperature as the controlled variables while reflux ratio, feed ratio and reboiler duty were selected as 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) because the integral squared error values calculated for the top segment and the reaction segment temperatures from the control carried out with NNDMPC were found to be less than those of the TFDMPC. Also, Giwa

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and Karacan (2012d) applied decoupling proportional-integral-derivative control to a reactive distillation column producing ethyl acetate 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.

As can be noticed from the literature review carried out, there are researches on the dynamics and control of reactive distillation process, but the ones on the production of biodiesel are very scarce. As such, this work has been carried out to bridge the gap by developing a transfer function model between the reboiler duty and the biodiesel mole fraction obtained from the bottom section of the reactive distillation column in which the process was taking place to study its dynamics. Furthermore, the developed transfer function model was used to investigate the control of the process by applying Cohen-Coon and Ziegler-Nichols tuning techniques.

2 METHODOLOGY

2.1 Biodiesel Production System Identification

The model of the biodiesel reactive distillation process used in this work was obtained by developing a transfer function between the reboiler duty (input variable) and the mole fraction of biodiesel obtained from the bottom section of the reactive distillation column (output variable) 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.* (2015). The model development was carried out using the *pem* command of the System Identification Toolbox inside MATLAB (Mathworks, 2015). The type of the transfer function model developed was chosen to be first order, as shown in Equation 1, so as to obtain a simple single-input single-output (SISO) model to represent the process.

$$G_p(s) = \frac{x_{biod}(s)}{Q(s)} = \frac{K_p e^{(-T_d s)}}{\tau s + 1} \quad (1)$$

2.2 Dynamics Study of Biodiesel Production System

After the transfer function of the process was developed with the aid of System Identification Toolbox of MATLAB, it was further modelled in Simulink, also contained in MATLAB, as shown in Figure 1, and its open-loop dynamics was studied by applying a unit step change to the input of the developed SISO model. The Simulink model was, actually, run using the codes written in M-File, also, of MATLAB.

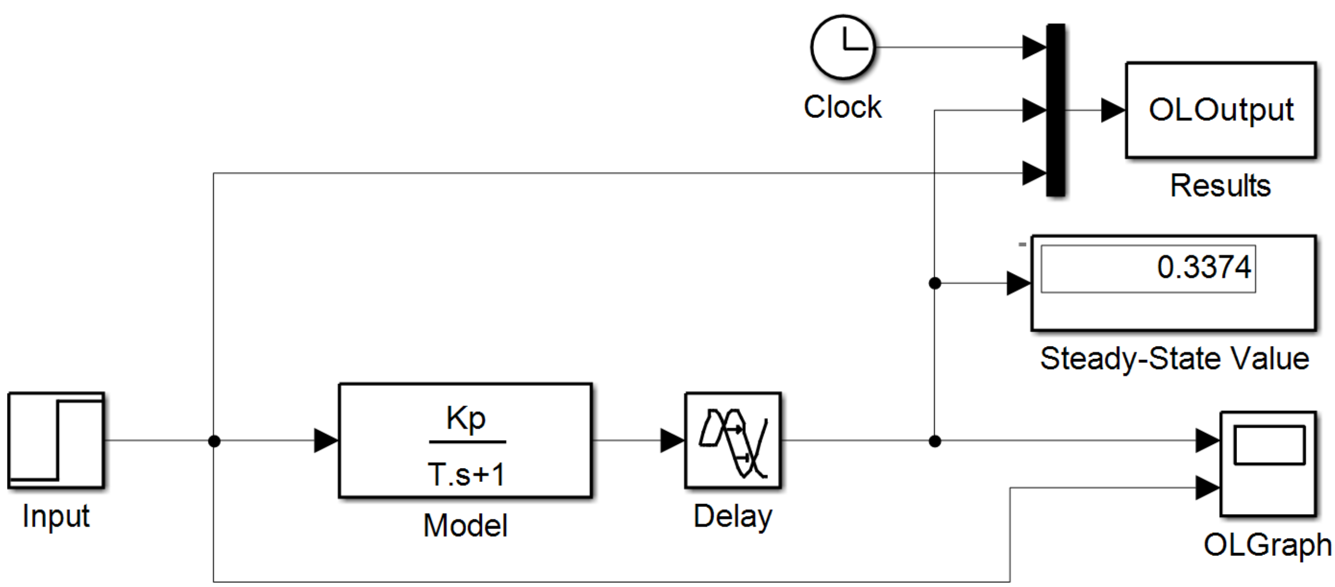

Figure 1. Simulink model of the single-input single-output (SISO) process

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2.3 Control Study of Biodiesel Production System

Furthermore, the control of the process for the biodiesel production with reactive distillation developed was simulated for set-point tracking (servo) by applying a positive and a negative steps of 0.1 each to the steady-state value of the controlled variable. The manipulated variable of the control study was the reboiler duty of the column. The models used for the control studies, which were also developed with the aid of Simulink, are given in Figures 2–4 respectively for the P-only, PI and PID control systems of the process.

The tuning parameters of the controllers were calculated using Cohen-Coon and Ziegler-Nichols tuning techniques. Given that the general transfer function of the controllers is as shown in Equation 2, the expression used to calculate the parameters of the two tuning techniques considered were as given in Table 1.

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

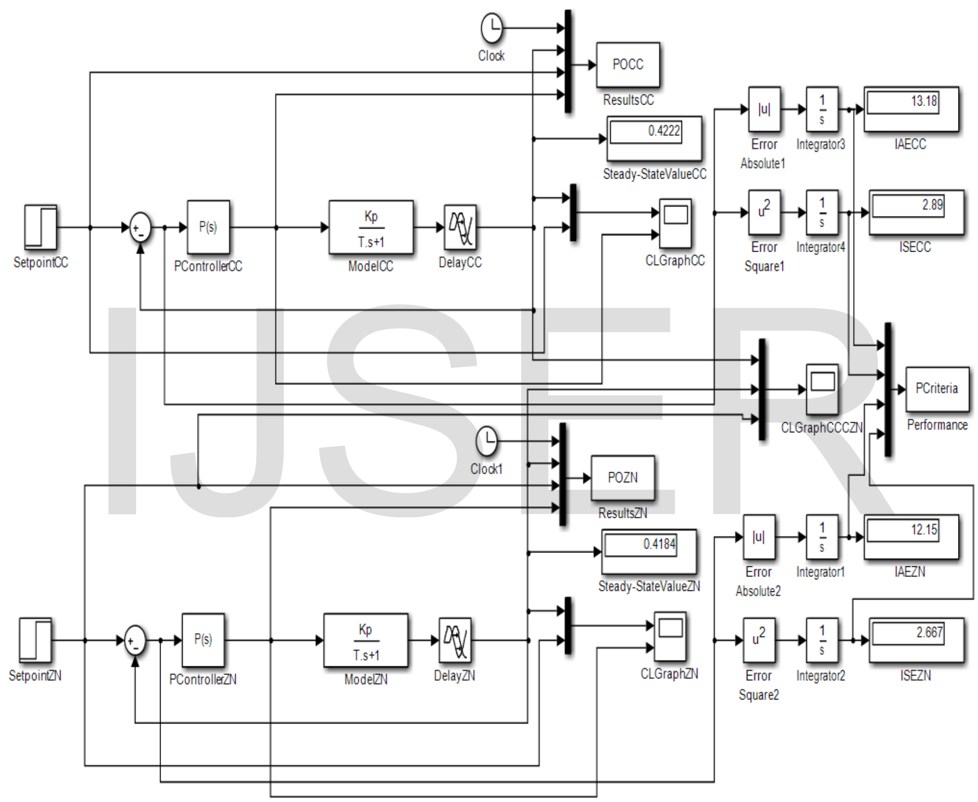

Figure 2. Closed-loop model of the process with P-only controller

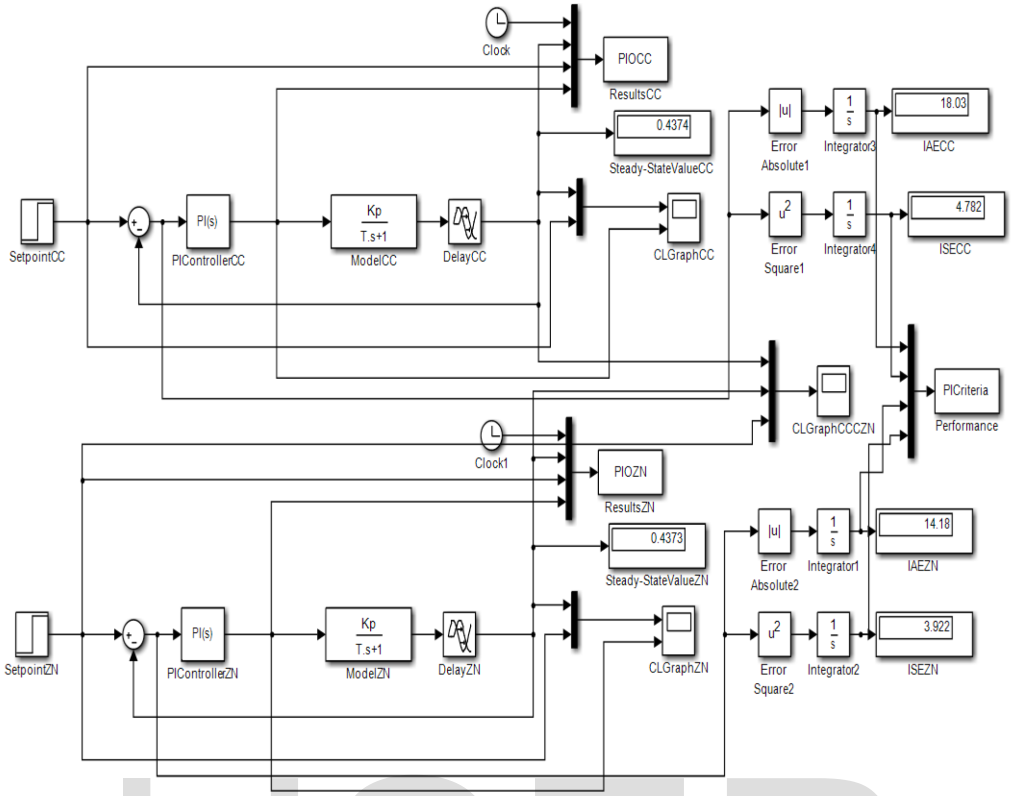

Figure 3. Closed-loop model of the process with PI controller

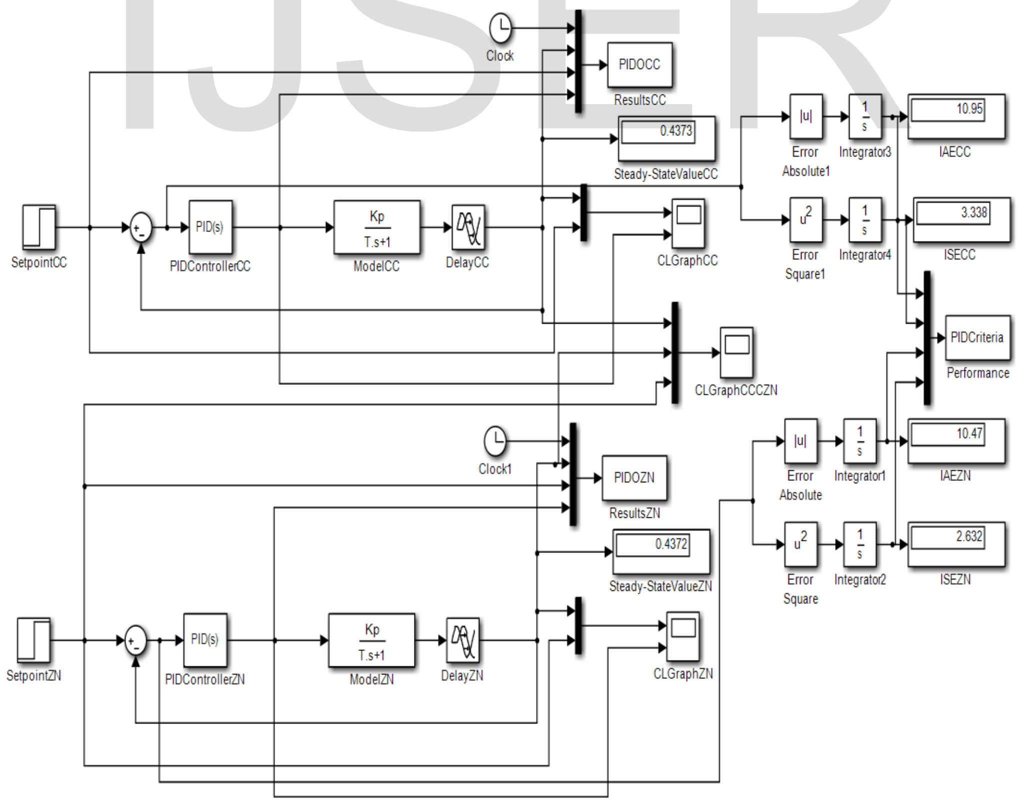

Figure 4. Closed-loop model of the process with PID controller

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

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

Source: Stephanopoulos, 1984

3 RESULT AND DISCUSSION

The developed transfer function obtained for the production of biodiesel in the reactive distillation column in which the esterification reaction between palmitic acid and methanol was taking place is given as Equation 3.

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

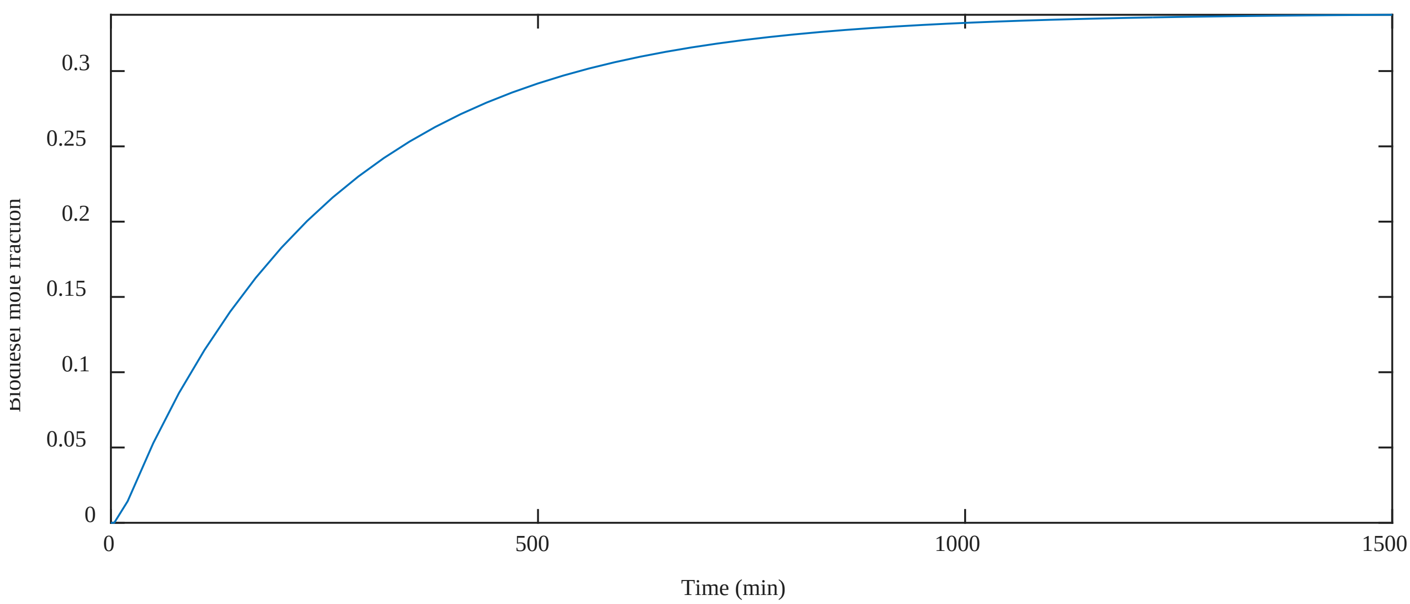

Figure 5. Open loop response of the process to a unit step change in the reboiler duty of the column

When the developed process model (transfer function) was simulated by applying a step increase to its input variable (reboiler duty), from the response given in Figure 5 that was obtained, it was observed that the steady-state value of the process, which was the mole fraction of biodiesel obtained from the bottom section of the reactive distillation column, was approximately 0.34. This observation was found to be in agreement with the behaviour of a first order system with delay. Furthermore, the response of the system was found to be slow in reaching the steady-state because it was found that the steady-state time was approximately 1500 min. In order to reduce the time taken by the system to get to the steady-state and to improve its performance, it was found that the development of a control system was necessary.

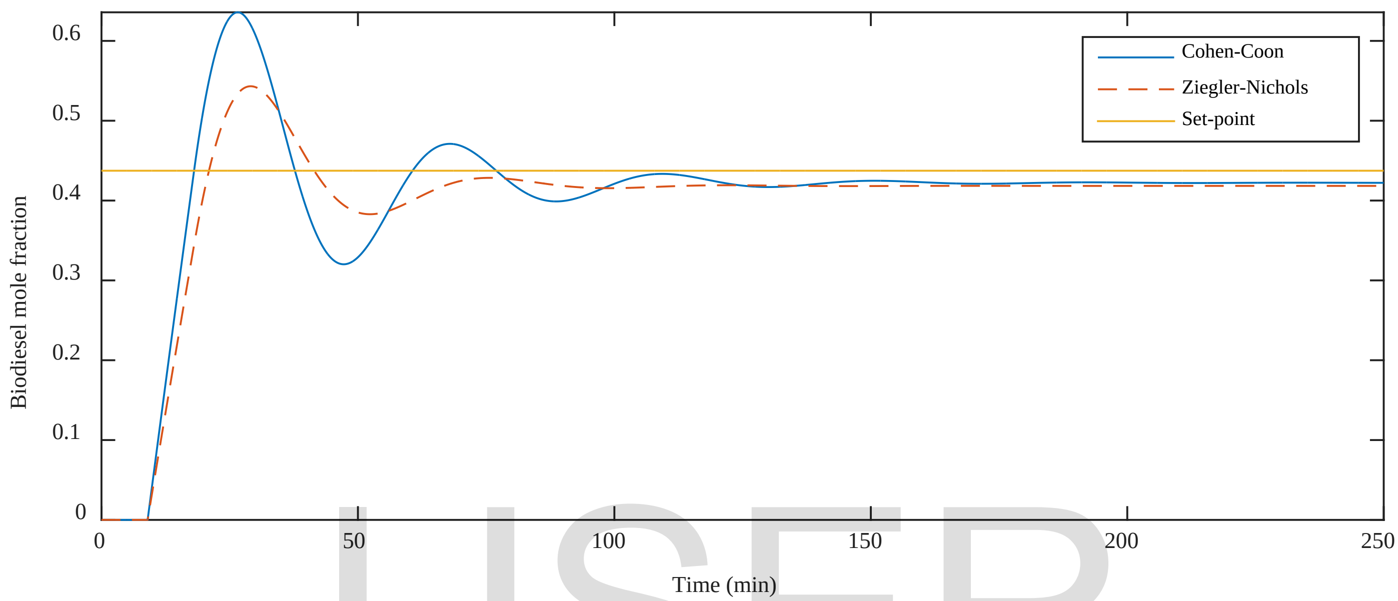

Figure 6. Closed loop response of P-only controlled biodiesel production system

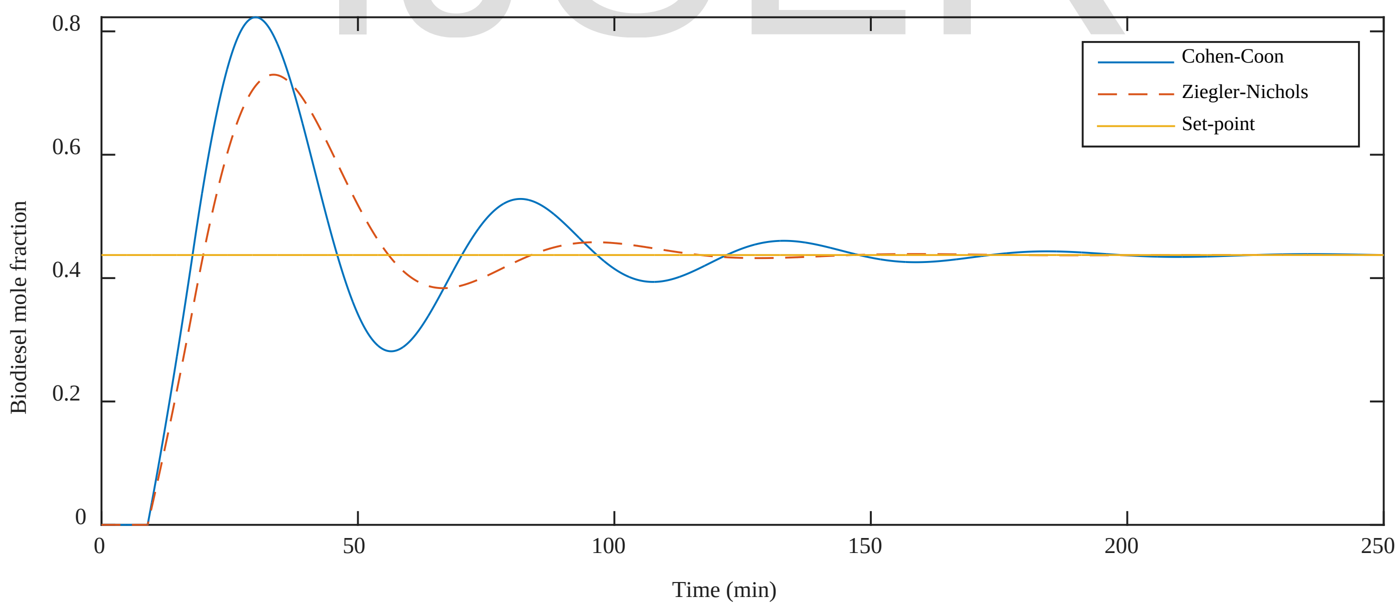

Figure 7. Closed loop response of PI controlled biodiesel production system

Shown in Figure 6 are the responses of the system to a P-only controlled system simulated with the aid of Simulink and written M-file using Cohen-Coon and Ziegler-Nichols tuning techniques. From the figure, it was observed that the response of the set-point tracking control simulation carried out using Cohen-Coon method was able to respond faster because it was able to cross the set-point quicker than that of the Ziegler-Nichols. However, the overshoot of the response of Cohen-Coon was found to be higher than that of the Ziegler-Nichols. The two responses were seen to be stable because they were able to get to steady states, but there were offsets. The offsets observed in the responses was normal because that (offset) is a characteristic feature of a P-only controller.

Given in Figure 7 are the responses obtained from the simulation of the process using Cohen-Coon and Ziegler-Nichols tuning techniques with PI controllers. According to the figure, the rise time of the response obtained from Cohen-Coon tuning technique was found to be less than that of Ziegler-Nichols; meaning that the response of Cohen-Coon was faster than that of Ziegler-Nichols in the case of this PI control system just as it was observed when P-only controller was used to control the system. Also observed from the response given in Figure 7 was that the offset noticed when P-only controller was used has been eliminated as the closed-loop responses of the process were seen to have got to steady states at the set point. This has also demonstrated an important feature of a PI controller in eliminating offsets in a control system. The observation made concerning the overshoot of the responses was similar to that obtained with P-only controller because, in this case also, the overshoot of the response of Cohen-Coon tuning technique was found to be higher than that of the Ziegler-Nichols.

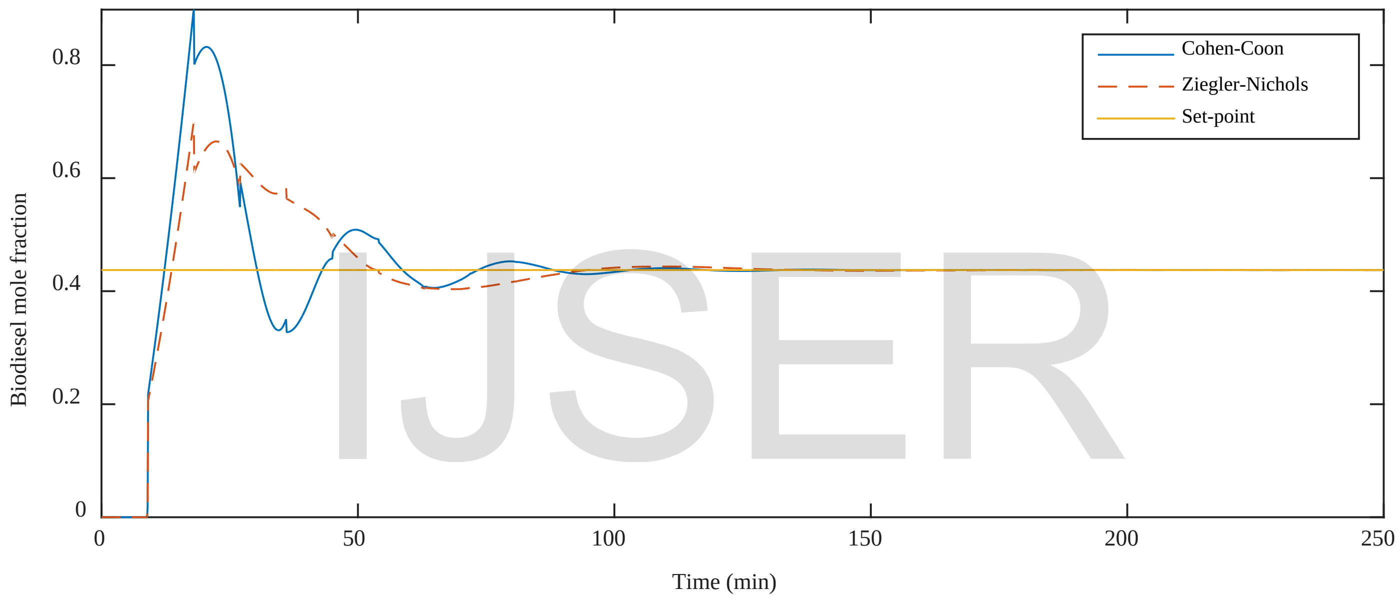

Figure 8. Closed loop response of PID controlled biodiesel production system

Table 2. Performances of the controllers and the tuning techniques

Controller	Cohen-Coon Tuning Technique		Ziegler-Nichols Tuning Technique	
	IAE	ISE	IAE	ISE
P	13.18	2.89	12.15	2.67
PI	18.03	4.78	14.18	3.92
PID	10.95	3.34	10.47	2.63

Figure 8 shows the responses of the process when Cohen-Coon and Ziegler-Nichols tuned PID controllers were applied to control it. From the figure, the rise time, the overshoot and the number of oscillations observed from the response of the Cohen-Coon tuning technique were found to be more than those of the Ziegler-Nichols. These observations were found to be similar to the ones obtained when Cohen-Coon and Ziegler-Nichols tuned P-only and PI controllers were used to control the system. Moreover, it was noticed from the responses given in Figure 8 that they were able to get to the steady state faster than those of the P-only and the PI controllers. In addition, the number of oscillations observed to occur in the PID controlled systems was less than that of the P-only and PI controllers, even though that of the PI controllers was more than that of P-only controllers (cf. Figures 6 and 7).

In order to compare the performances of the different controllers and the tuning techniques used in this work, the integral absolute error (IAE) and integral squared error (ISE) of each of the controllers and with each of the methods used were estimated and the results obtained were as given in Table 2. From the table, for the Cohen-Coon tuning method, it was observed that the controller with the lowest IAE was PID while the one with the lowest ISE was P-only controller despite its offset. From the results of the closed-loop simulations carried out using Ziegler-Nichols tuning technique, PID controller was found to be the one with the lowest IAE and ISE values. Considering the graphical responses of the control simulations and the estimated performance values, it was observed that the overall performance of PID controller with Ziegler-Nichols tuning technique was the best among the three (P, PI and PID) controller types considered in this work. The finding of the better performance of PID tuned with Ziegler-Nichols technique over Cohen-Coon was found to be in contrary to the finding of Giwa and Karacan (2012d) who obtained that the performance of Cohen-Coon was better for the system they considered. The findings have just pointed out that the performances of the methods might vary according to the particular process or system being considered.

4 CONCLUSION

The results obtained from the open loop simulation carried out on the process model developed, with the aid of System Identification Toolbox of MATLAB, for biodiesel production revealed that the system was stable because it could attain a steady state when simulated. Also, the closed loop responses showed that the best control of the system was achieved when PID controller tuned with Ziegler-Nichols method was applied because that was the combination that gave best performance criteria among the controllers used.

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NOMENCLATURE

τ	Time constant of the process (min)
τ_D	Derivative time of the controller (min)
τ_I	Integral time of the controller (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_p	Static gain of the process
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)
SISO	Single-input single-output
T_d	Dead time of the process (min)
TFDMPC	Transfer Function Decoupling Model Predictive Controller
x_{biod}	Bottom biodiesel mole fraction

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