

Modelling of the Mini Batch Distillation Column

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Abstract: Distillation is the most common method for separating components in chemical unit operation. This paper presents the mini batch distillation column modeling to facilitate a closed loop control design in the later stage. This mini plant is part of the development of a cyber physical system and will be a physical system controlled by DCS (Distributed Control System) with data transmission over the network in real time. In this study, ITB Honeywell mini plant for laboratory scale is used for separating binary mixtures, ethanol and water. An on-off condenser valve is chosen as an actuator to regulate the end product concentration. The plant is modeled by 2 approaches, i.e. White-box modeling approach by first principle, which produces nonlinear equations model of batch distillation column based on component mass balance, vapor-liquid equilibrium and others physical characteristics. Meanwhile, the Black-box approach uses input-output experimental data to produce a linear model of the plant. The modelling is for two different locations of the concentration sensor, i.e. at the inflow or outflow of the end-product tank. The nonlinear model is not yet appropriate for control design purpose. Unlike the nonlinear model, the linear models developed at two different control inputs show marginal to good fit to the experimental data at respective operating points. However, it seems better to use a piecewise linear modelling over process operating regions for concentration control design purposes of the batch distillation column. Further development of the nonlinear model shall be done by considering the influence of temperature variability in the model.

Keywords: Batch Distillation Column, First principle, white box model, black box model.

1. Introduction

In the recently established ITB Honeywell Laboratory at Institut Teknologi Bandung (ITB), a Cyber-Physical System (CPS) is developed and involves a mini (binary) batch distillation column as its subsystem. Cyber-Physical System (CPS) is a combined dynamic system between components that have physical characteristics (such as sensors, actuators, and processes) with components that are mostly digital systems with software, which have on/off, transmit/receive characteristics. From a modeling perspective, the dynamic of a physical system is time-driven, which is represented in a differential equation (for continuous systems). Whereas, digital systems with software operating under on/off conditions are event-driven. Thus, both characteristics in CPS form a hybrid system [1]. Equipped with ITB Honeywell's DCS (distributed control system) and instruments, the CPS with mini batch distillation column is depicted in Figure 1. Smart transmitters (xST) and controllers (xSC) are to bridge signals from sensors and to actuators with DCS as supervisory controller through a cloud TCP/IP network. For effective control development of our CPS, suitable model is required and this paper focuses on the modelling of the distillation column as a continuous time plant.

Distillation is one of the most important methods and the oldest separation process in chemical plant to separate components mixture, such as ethanol and water. It is therefore widely used in operation of process industries. In a refinery plant of petrochemical industries, for example, crude oil is processed to be petroleum fraction and its derivatives. Refinery is a distillation process to separate components mixture. The separation of the liquid components mixture into desired purity of each component fraction is one of the physical chemical processes

involved. By heating the liquid components mixture, a purification process known as distillation is carried out until one of the liquid component is evaporated based on relative boiling point and volatility of each components. Production of alcoholic beverages are other examples of distillation unit operation, [2, 3, 4].

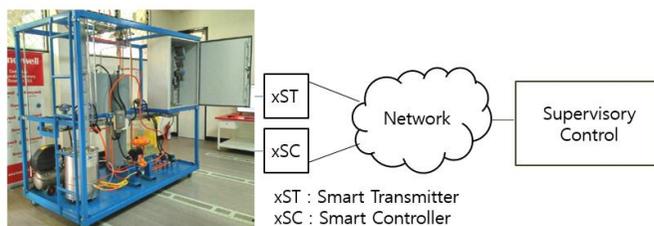


Figure 1. CPS with Mini Batch Distillation Column

Distillation processes may be classified into two general categories, i.e. batch distillation and continuous distillation processes. In batch distillation process, liquid components mixture is loaded into a heater pot and heated in one time distillation process and pure distillate component is then produced. In continuous distillation process, the distillation process occurs continuously because new liquid components mixture are fed into the feeding tray in a distillation column and then produce pure distillate and residue [5].

The modification from the batch distillation process to the continuous distillation process begins in the petrochemical industry and refineries that are slowly continuing to dominate all major industrial sections. Small-scale industries, however, still use batch distillation since it is more flexible than continuous distillation [6]. The batch distillation process is also often used for small-scale production of multi component distillates due to its minimal cost operation [7]. Research and development of the batch distillation process has been, therefore, more concerned with the process industry in recent years.

In terms of its column type, a distillation column may have internal tray column or packed column. The internal tray column consists of various designed trays that are used to hold up the liquid to facilitate better contact between vapour and liquid, hence better separation. Meanwhile, in the packed column, instead of trays, 'packings' are employed to strengthen contact between vapour and liquid. The distillation column consists of the following parts of processes [8], i.e. (i) a heater that is used to provide the heat in order to evaporate the liquid phase from the bottom of the distillation column, (ii) vertical packed column that combines vapor phase flows up to the top of column and liquid phase flows down to the bottom of column, (iii) condensers that are used to condensate and cool the vapor phase in the top of column, both being reflux to the bottom of the column again and being pure distillate product.

Modeling of a distillation column is a challenging problem due to its strong interactions inter-components, highly nonlinear physically and complex to control. However, for efficient and effective batch distillation process, one may need to have proper model of the process for control system analysis and design. Proper dynamic modeling of the process is important to predict its behavior and characteristics [9]. Several works on the modeling of distillation columns have been reported in the literatures. Modeling continuous distillation column based on mass balances and liquid-vapor equilibrium are reported in [8, 10]. Design of reduced order linear model based on linearized model of the continuous plant is also proposed in [11,12]. Moreover, a modelling by considering constant and variable relative volatility is presented in [5]. Meanwhile, nonlinear models of batch distillation columns are presented found in [13,14] based on dynamic composition, mass balance equation, energy balance and vapor liquid equilibrium. A linearized model from a nonlinear model is also published in [15] in continuous time state space form.

Nonlinear and linear models are developed here for a mini, packed type, binary, and batch distillation plant in our ITB Honeywell Laboratory. The modelling approach in [16] is revisited and the models therein are then refined with other experimental data. In addition, a linear

modelling approach is also undertaken to get several linear models at some different operating points. Unlike previously developed model in the literatures, an on-off opening of the reflux control valve is used as an actuating input (as in [16]) and the distillate concentration of ethanol is the output of the proposed model.

2. Description of The Mini Batch Distillation Column

The diagram of the mini binary batch distillation column plant in the ITB Honeywell laboratory is illustrated in Figure 2. The distillation process of the binary mixtures of ethanol and water occurs in a packed column type of distillation plant. The plant is about 3 meters tall and it has a heater pot with 22 liter capacity. It comprises two columns in series with 21 mm diameter and 90 mm tall each. A reflux valve located at the top part of the column is accessible for on-off control. Among other sensors for monitoring and feedback purposes installed, temperature sensors in the heater pod, top column, and cooling water are used to monitor. A sensor unit developed in [18] is used to measure the ethanol concentration. For modelling experiments, an Arduino based data acquisition is also developed to access the temperature sensors, concentration sensor, and the reflux valve as a control actuator

The objective of distillation column is to separate the binary mixtures and produce pure concentration of ethanol. The mixture of two liquid components is charged into the heater pot and then heated. Heating the liquids mixture up to a certain boiling point, the component of the mixture will change from the liquid phase to the vapor phase. The component with a lower boiling point will be evaporated first.

Ethanol has a lower boiling point, at 78.37°C , than water, at 100°C . The ethanol will then evaporate earlier than the water in the distillation process. As the temperature of the heater increases, the boiling point of the ethanol will be achieved and the ethanol component in the heater will decrease. In the end, the water is the only component left in the heater. In this study, the binary liquid mixture of ethanol and water will be separated by the distillation column.

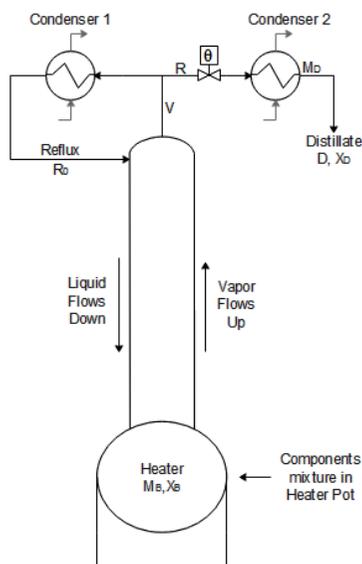


Figure 2. Diagram of Distillation Column

The amount of mass and the component concentration in the heater pot are denoted as M_B and x_B respectively. An on-off reflux valve is operated to determine a reflux ratio by changing the on-off valve's duty cycle. To get the desired product concentration x_D , it is necessary to control the valve. When the valve is fully open (on), some of the heated steam in the heater flows into Condenser 1 as a reflux with $R + R_0$ flowrate, i.e. the component that falls down to the

heater. R_0 is the leakage flowrate when the valve is fully open. The rest of the steam flows into Condenser 2 with D flowrate, which becomes the ethanol distillate. The amount of the reflux and the distillate is determined by the reflux ratio. The remaining water component in the heater will still be in the heater pot and the output of the plant is the distillate concentration x_D . An on-off opening of the reflux control valve is chosen as an actuator to influence the distillate concentration.

A chemical unit process, as in a distillation column, involves multi variables with strong interactions among its components and consists of highly nonlinear interacted physical and chemical processes therein. Knowing its uncertainties and complexity, the following assumptions are then taken to facilitate modelling, i.e. (i) the dynamic of the distillate concentration that is modelled are the concentration at the distillate tank when the ethanol liquid starts to evaporate, (ii) the temperature at the heater pot is assumed to have been regulated, (iii) the vapor flowrates from the bottom to the top column and vice versa are assumed constant.

For control purpose, the on-off valve is seen here as a linear actuator control by changing the duty cycle operation of the valve from 0 to 100% linearly with a suitably chosen time period. In this case, there is no negative control input.

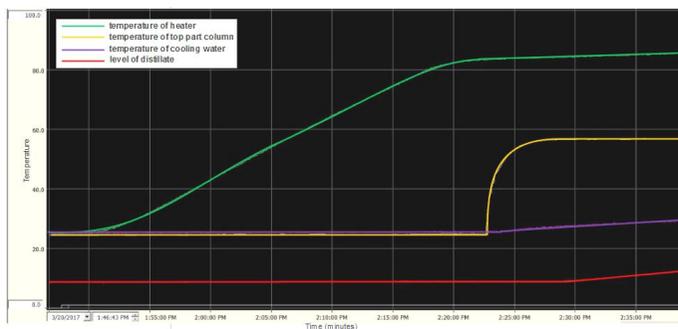


Figure 3. Measured temperature changes of the distillation column

The changes of the measured temperature in the distillation process are shown in Figure 3. The heater at the pot is firstly turned on and it will reach a relatively constant temperature (green line). After some time lag, the temperature of the top part of the column (yellow line) will slowly rise and reach a steady state in approximately 5 minutes. It is when the top column temperature reaches its steady state value that the dynamic behaviour of the ethanol distillate is modelled here.

3. Non Linear Modeling of The Mini Batch Distillation Column

A nonlinear dynamic model of ethanol water binary batch distillation cloumn may be developed as a set of differential equations by considering mass balance, physical properties, and vapor liquid equilibrium law of the distillation process.

A. Flowrate Relationship

Using the forementioned assumptions, the analysis of batch distillation column is simplified as a flowrate relationship of V , R , R_0 and D similar to the one described in [1] in which the flowrate V is the total flowrate at the top part of distillation column. The flowrate relation in [1] is refined as the following.

$$V = R + R_0 + D \tag{1}$$

$$D = \theta(V - R_0) \tag{2}$$

$$R = (1 - \theta)(V - R_0) \tag{3}$$

The reflux ratio θ takes the value of 0 to 1, which is equivalent to is 0 to 100% PWM (pulse width modulation) duty cycle. The value 0 means the reflux valve is fully closed and the value

1 means the fully opened. The longer the reflux valve is opened, the more vapor flow to distillate. Otherwise, if the reflux valve is closed, then there is no vapor flow to distillate.

B. Liquid Mass Holdup

In the batch distillation column, there are two liquid masses holdup to accommodate the liquid mixtures and the distillate result. M_B is the liquid mass holdup in the heater pot and M_D is the liquid mass holdup in Condenser 2. Based on the flowrate relation in equation (2), the following liquid holdup mass at in Condenser 2 is obtained.

$$M_D = C \int_{t_0}^t D(t) dt \tag{4}$$

$$M_D = C \int_{t_0}^t [\theta(V - R_0)] dt \tag{5}$$

with constant C as a correction factor. With initial mass hold up M_{B0} , the liquid mass holdup in the heater pot may then be represented as

$$M_B = M_{B0} - C \int_{t_0}^t [\theta(V - R_0)] dt \tag{6}$$

$$M_B = M_{B0} - M_D \tag{7}$$

The change of mass hold up in the heater pot may then be written as

$$\frac{dM_B}{dt} = -C [\theta(V - R_0)] \tag{8}$$

C. Mass Balance in the Condenser and Heater

The equations for mass balance in the condenser and the heater are modified from [11] and [17]. Using equations (1)-(3), for the mass balance in Condenser 2 (see Figure 3), one may obtain the following

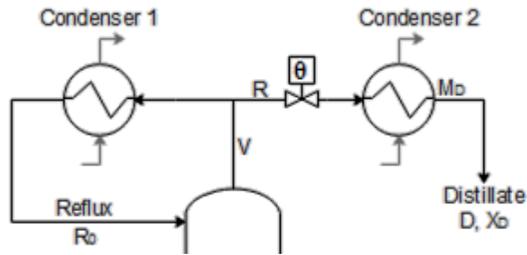


Figure 3. Mass Balance in the Condenser

$$M_D \frac{dx_D}{dt} = Vy_B - (R + R_0)x_D - Dx_D \tag{9}$$

$$M_D \frac{dx_D}{dt} = Vy_B - Vx_D \tag{10}$$

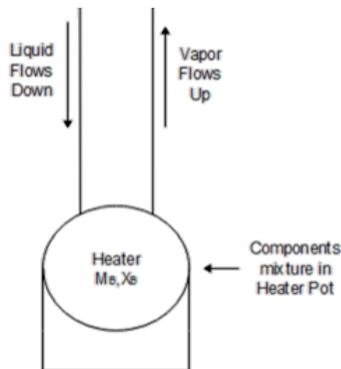


Figure 4. Mass Balance in the Heater

Using equation (7), equation (10) may be written as

$$\frac{dx_D}{dt} = \frac{Vy_B}{M_{B0}-M_B} - \frac{Vx_D}{M_{B0}-M_B} \quad (11)$$

Using equations (1)-(3), for the mass balance in the heater (see Figure 4), the following equations are obtained.

$$M_B \frac{dx_B}{dt} = (R + R_0)x_D - Vy_B \quad (12)$$

$$M_B \frac{dx_B}{dt} = (V - (V - R_0)\theta)x_D - Vy_B \quad (13)$$

$$\frac{dx_B}{dt} = \frac{Vx_D}{M_B} - \frac{Vy_B}{M_B} - \frac{(V-R_0)\theta x_D}{M_B} \quad (14)$$

D. Vapor Liquid Equilibrium

In the distillation process, the vapor and liquid at each temperature boiling point have difference composition. The relationship of vapor and liquid composition in equilibrium can be expressed by relative volatility as the following, [2, 7].

$$\alpha = \frac{y_B/x_B}{1-y_B/1-x_B} \quad (15)$$

$$y_B = \frac{\alpha x_B}{1+(\alpha-1)x_B} \quad (16)$$

For relatively constant α , using equation (14) and (16), one may obtain the following.

$$\frac{dy_B}{dx_B} = \frac{\alpha}{(1+(\alpha-1)x_B)^2} = \frac{dy_B/dt}{dx_B/dt} \quad (17)$$

$$\frac{dy_B}{dt} = \frac{\alpha}{(1+(\alpha-1)x_B)^2} \cdot \frac{dx_B}{dt} \quad (18)$$

$$\frac{dy_B}{dt} = \frac{\alpha}{(1+(\alpha-1)x_B)^2} \left\{ \frac{Vx_D}{M_B} - \frac{Vy_B}{M_B} - \frac{((V-R_0)\theta)x_D}{M_B} \right\} \quad (19)$$

E. Nonlinear State Equations

Using the defined variables listed in Table 1, a nonlinear model of the distillation column may then be formulated.

Table 1. Model Variables

Variable	Description	Units
V	Vapor flow rate	Mole/min
R	Reflux flow rate	Mole/min
R_0	Initial reflux flow rate	Mole/min
D	Distillate flow rate	Mole/min
x_D	Distillate composition of liquid phase	Mole fraction
x_B	Bottom composition of liquid phase	Mole fraction
y_B	Bottom composition of vapor phase	Mole fraction
M_D	Liquid holdup in the distillate	Mole
M_B	Liquid holdup in the bottom	Mole
M_{B0}	Initial liquid holdup in the bottom	Mole
α	Relative volatility	-
θ	Reflux ratio	-

The equations (8), (11), (14), and (19) can be expressed as a nonlinear dynamic system below.

$$\dot{x}(t) = f(x, t) + g(x, t)\theta(t) \quad (20)$$

$$y(t) = C(x, t) \quad (21)$$

or

$$\frac{dx}{dt} = \begin{bmatrix} f_1(x, t) \\ f_2(x, t) \\ 0 \\ f_4(x, t) \end{bmatrix} + \begin{bmatrix} 0 \\ g_2(x, t) \\ g_3(x, t) \\ g_4(x, t) \end{bmatrix} \theta(t) \quad (22)$$

$$y = [1 \ 0 \ 0 \ 0]x \quad (23)$$

where $x = [x_D \ x_b \ M_B \ y_B]^T$ and $f_1, f_2, f_4, g_2, g_3, g_4$ are suitable nonlinear functions of the states in x that may be obtained from equations (8), (11), (14), and (19).

F. Parameters and Initial Values

In the real mini batch distillation column and in the experiment, the units for the liquid mixtures are volume and volume %. Meanwhile, the units in the model and simulation are mole and mole fraction. Some calculations are then performed in order to change the units as follow.

Volume Percent

To determine the volume of each component, i.e. ethanol and water, percent volume formula is used. The concentration of a liquid mixtures component is expressed as a percentage of volume. The volume of ethanol per mixtures component volume is expressed as

$$\text{ethanol volume} = \% \text{ ethanol volume} \times \text{mixtures volume}$$

Initially, 3000 mL of binary liquid component mixture with 30% of ethanol, i.e. 900mL, is charged in the heater pot with 2100mL water.

Density

Density are characteristic of each substance. Density is the ratio between the mass of a substance (m) with its volume (V) and is expressed as $\sigma = m/V$ with $\rho_{\text{ethanol}} = 0.789 \text{ gr/cm}^3$ and $\rho_{\text{water}} = 1 \text{ gr/cm}^3$. Within the 3L of the mixture, the mass of the ethanol and the water are then 710.1 gram and 2100 gram respectively.

Number of Moles

Mole (n) is the unit used to measure the number of substances. One mole is defined as the number of substances containing the particles of the substance. Meanwhile, the mass of one mole of substance is called the molar mass (denoted as M_r). The number of moles contained in a substance can be denoted as $n = m/M_r$ with $M_{r_{\text{ethanol}}} = 46.07 \text{ gr/mol}$ and $M_{r_{\text{water}}} = 18.016 \text{ gr/mol}$. Within the 3L of the mixture, the number of mole for the ethanol and the water are then 15.414 mol and 116.563 mol respectively.

Table 2 Calculation Results

Description	Ethanol	Water
Volume	900 mL	2100 mL
Density (ρ)	0.789 gr/cm ³	1 gr/cm ³
Mass (m)	710.1 gr	2100 gr
Relative Molecular Mass (M_r)	46.07 gr/mol	18.016gr/mol
Number of Mole (n)	15.414 mol	116.563 mol
Mole Fraction (x)	0.1168	0.8832

Mole Fraction

The mole fraction represents the ratio of the number of moles of a component (solute or solvent) to the total number of moles present in the solution (mole of solute plus mole of solvent).

$$\text{ethanol mole fraction} = \frac{\text{mole of ethanol}}{\text{mole of all components}}$$

The fraction moles for the ethanol and the water may now be computed as 0.1168 and 0.8832. The previous calculation results are then summarized in Table 2.

For simulation, however, the initial liquid mixtures at the heater pot (M_{B0}) and leakage reflux flowrate (R_0) in the model are also required to set as initial conditions. In 3000 mL initial liquid mixture of 30% ethano, the initial liquid mixtures at the heater pot (M_{B0}) will then be 131.977 mol.

Leakage Reflux Flowrate

An experiment that lasted 24 minutes or 1440 seconds was firstly conducted with a fully open solenoid valve, i.e. $\theta=1$. The total volume obtained and the final ethanol distillate concentration is recorded. After conversion into mole units, the number of moles of the distillate is equivalent to 12.5 mol. In average, therefore, the flowrate of the distillate shall be the amount of distillate mole divided by the length of experiment time, i.e.

$$D = \frac{12.5 \text{ mole}}{24 \text{ minutes}} = 0.52 \frac{\text{mol}}{\text{min}} = 0.0087 \text{ mol/sec}$$

Using equation (1), the values of flowrate V and R_0 may be predicted. It is reasonable to assume that if $\theta=1$, i.e. $D=0.52 \text{ mol/min}$, then the reflux flowrate $R = 0$ and

$$V = R + R_0 + D = 0.52 + R_0.$$

In the distillation column, the diameter ratio between the top part of the column and the valve shall determine the amount of the flow rate $R + R_0$ with respect to the flowrate D . A diameter ratio in which $V = 5D$ is adopted and then

$$V = 5 \times 0.52 \frac{\text{mol}}{\text{min}} = 2.6 \text{ mol/min} = 0.043 \frac{\text{mol}}{\text{sec}}$$

Since $V = 0.52 + R_0$, one may then have

$$R_0 = V - 0.52 = 2.08 \text{ mol/min} = 0.0347 \text{ mol/sec}$$

G. Model Simulation

The model has reflux control valve (θ) as the input and the distillate concentration as the output (x_D). Using the calculated parameters and initial values in Table 3, the model simulation is constructed as a subsystem and depicted in Figure 5. A function is used to convert a mole fraction unit to a concentration unit as in the actual experiment. The transport delay is used to model the time delay as in the experimental results. The subsystem block is built from a set of nonlinear differential equations, i.e. equations (8), (11), (14), and (19), as presented in Figure 6.

Table 3 Parameter and Initial Values

Description	Symbol	Value
Mixture Liquid mass holdup in heater	M_{B0}	132 mole
Leakage reflux flowrate	R_0	0.035 mole/sec
Vapor flow rate	V	0.043 mole/sec
Relative volatility	α	1.5
Constants	C	1

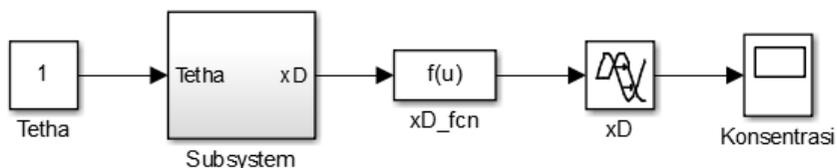


Figure 5. Simulation Block

For simulation, initial values of integrators are set as follows. The 0.8507 value of integrator x_D is obtained from the initial distillate of 96% ethanol concentration. Meanwhile, the 0.113 value of integrator x_B is obtained from the 30% liquid mixtures component in mole fraction unit. The initial value of integrator y_B is set to be quite close to the initial value of integrator x_D . Similarly, the integrator value M_B is made practically M_{B0} value since the mole value on the heater pot will decrease gradually with time from M_{B0} .

Referring to the simulation results in Figure 7, delay times of 135 seconds for input $\theta=1$ and 225 seconds for $\theta=0.5$ are set to coincide with the experimental data in which the contraction sensor was located at the outflow of the distillate tank. For $\theta=1$, after some delay, the concentration of ethanol component is high in the beginning, i.e. at $\pm 96\%$, and it decreases slowly with time until it reaches $\pm 20\%$ at the end of the simulation. For $\theta=0.5$, after some delay, the concentration of ethanol starts also at $\pm 96\%$ and it then decreases slowly with time until it reaches $\pm 30\%$ at the end of the simulation.

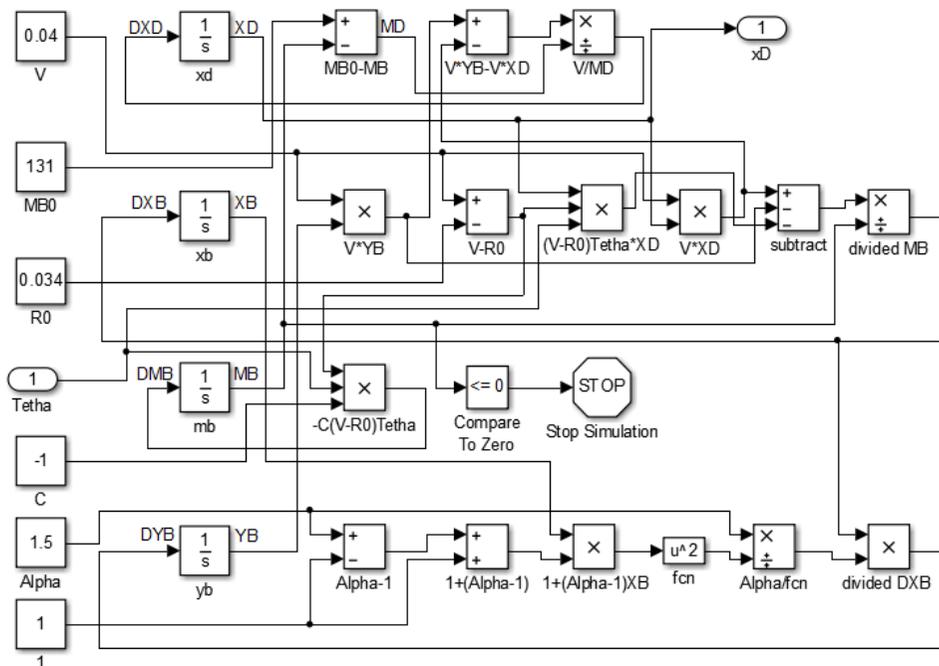


Figure 6. Subsystem Block

Table 4. Initial Value of Integrator

Variable	Initial Value
x_D	0.8507
x_B	0.113
y_B	0.84999
M_B	130.999

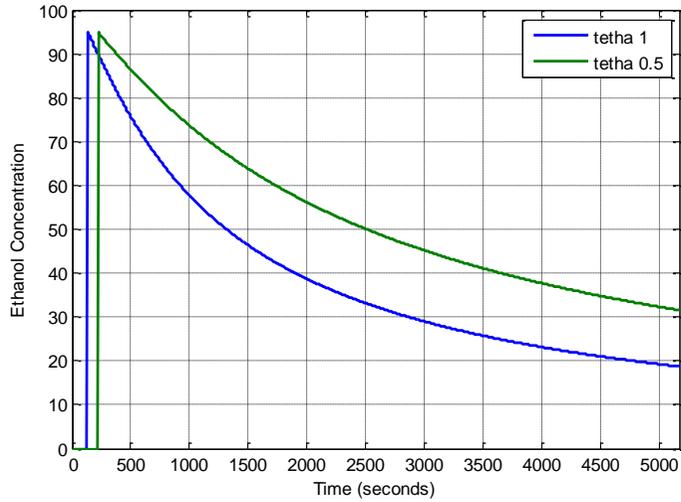


Figure 7. Simulation Result of Ethanol Concentration

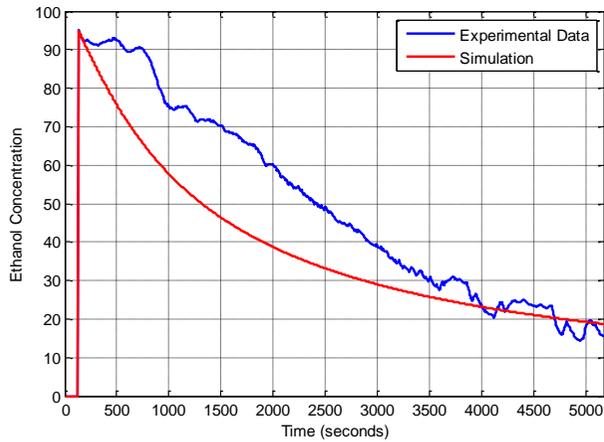


Figure 8. Validation for $\theta = 1$

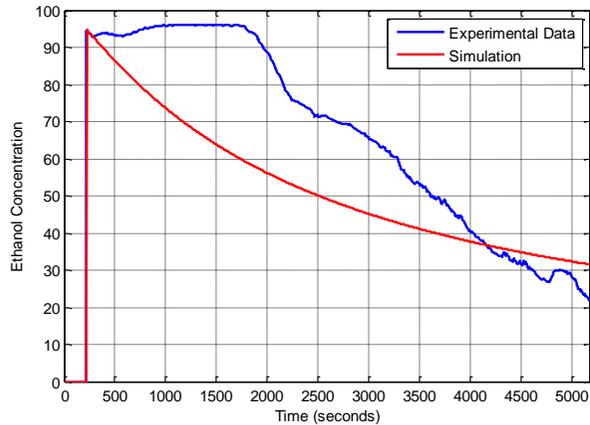


Figure 9. Validation for $\theta = 0.5$

Figure 8 and Figure 9 present the comparison for both $\theta=1$ and $\theta=0.5$ cases. Note that the experiment data was obtained by Arduino with necessary instrumentation with 5 seconds sampling time and 1 minute of PWM time period of input test. The two figures show that the concentration of the distillate starts at the same value at $\pm 9\%$. The model fit testing from simulation result towards the experimental data for each θ input was also performing using normalized RMSE and goodness of fit in Matlab. The model's goodness fit for $\theta=1$ is 42.4525% whereas for $\theta=0.5$ is 31.0251%, which are not very good. After some delays, however, the concentrations at both cases start to decrease until they reach at about similar values of ethanol concentration at the end of observed time period. The simulation results also do represent the same trend as the real experiment but with different transient due to some assumptions in modeling process.

4. Linear Modeling of The Distillation Column

A. Black Box Linear Model

The paper studies both linear and nonlinear models that may be later used for various control design options. It is well known that linear model may be used for complex system at a chosen operating point. Having developed the nonlinear model of the process, a linear modeling approach may also be taken as an approximate model for practical reason. The process is now assumed to contain linear systems dynamics. Proposed modeling structure is depicted in Figure 10.

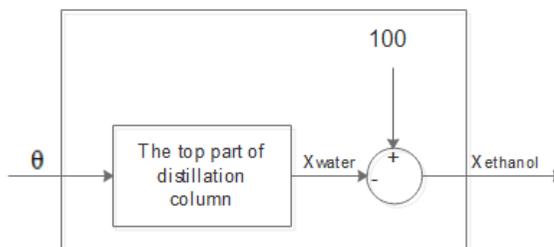


Figure 10. Diagram Block of Distillation Column System

B. Experimental Procedure

The experiment runs were done in a batch process in which the data was obtained by Arduino with 5 seconds sampling time and 1 minute of PWM time period of input test. The experiment data of the ethanol concentration is taken from a newly developed sensor in [18]. The water concentration is then computed from the ethanol concentration data. The computed water concentration will be later processed in the identification system software to obtain an LTI system model of the proposed model.

In this mini plant, a reflux valve is located at the top part of the column. Although the reflux valve is an on-off solenoid valve, a linear control is conducted by changing the duty ratio of the on-off reflux valve with 1 minute time period from 0 to 1. This is the input of the batch distillation column (θ) to determine the distribution of vapor flow from heater pot to the column as a reflux and or to a distillate tank. The model developed here is based on experimental data when the duty ratio of the valve is 0.5 and 1. The reflux valve is fully closed when $\theta=0$ in which no distillate produced and all vapour goes as a reflux. Otherwise, the valve is fully opened when $\theta=1$ in which ethanol distillate is produced with some leakage reflux as well.

The several experiments were run for each of two different concentration sensor locations, i.e. firstly at the inflow and secondly at the outflow of the distillate tank. Noting that the sensor may add some addition delay, locating the sensor at the inflow of the tank shall reduce the time delay of the plant and hence of its model. On the other hand, locating the sensor at the outflow of the tank shall represent not merely the newly coming product but rather accumulated end products.

For each sensor location, the data for ethanol concentration is obtained for both $\theta=0.5$ and $\theta=1$. Sample of the experiments data for those two cases are presented in Figure 11 to Figure 14.

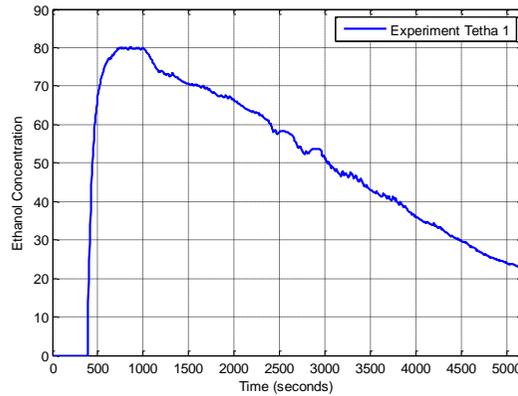


Figure 11. The First Experiment with $\theta = 1$

Figure 11 shows the distillate concentration in the case when the sensor is located at the outflow of the storage tank of the ethanol liquid product. There was about 360 seconds of delay before the ethanol is produced. The first ethanol concentration produced is about $\pm 75\%$ because the liquid in the tank may still mixed with the result from a previous experiment. However, the highest concentration achieved is about $\pm 80\%$ and then it continues to decline until $\pm 20\%$ after ± 5200 seconds.

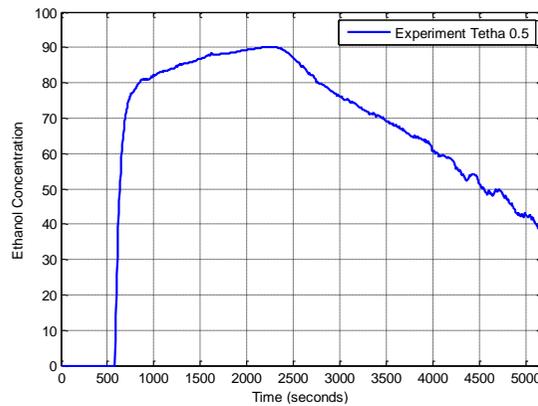


Figure 12. The First Experiment with $\theta = 0.5$

With the same sensor location as in Figure 11, Figure 12 shows the distillate concentration but with $\theta=0.5$. There was about 590 seconds of delay before the ethanol is produced. Similarly, the first ethanol concentration produced is also about $\pm 75\%$ due to the same reason. However, the highest concentration achieved is about $\pm 90\%$ and then it goes down to $\pm 40\%$ after ± 5200 seconds.

Figure 13 and Figure 15 show the distillate concentration when the location of concentration sensor is relocated at the inflow of the storage tank or below Condenser 2. This is to get a less delay time and higher starting ethanol concentration.

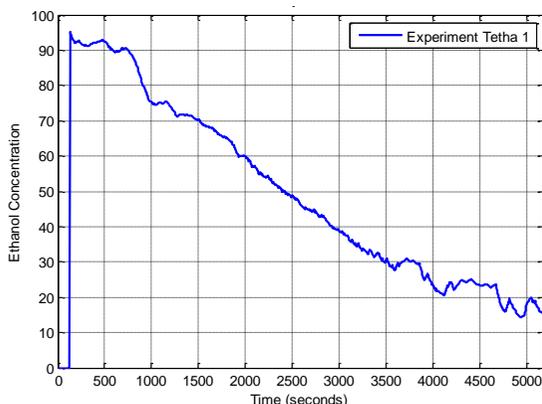


Figure 13. The Second Experiment with $\theta = 1$

It is seen in Figure 13 that there was about 135 seconds of delay before the ethanol is produced. The first ethanol concentration produced is high enough at about $\pm 95\%$ and it decreases to $\pm 15\%$ after ± 5200 seconds.

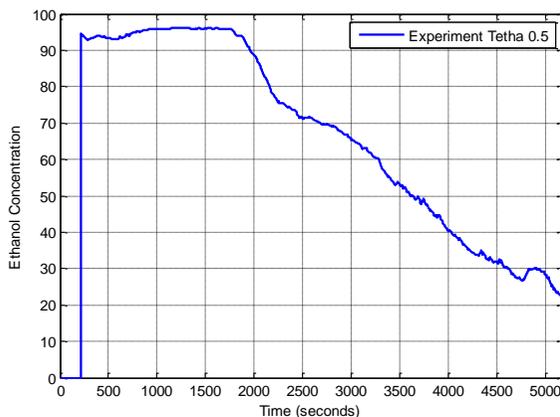


Figure 14. The Second Experiment with $\theta = 0.5$

Meanwhile, Figure 14 shows that there was a 225 seconds of delay. The first ethanol concentration produced is also high enough at about $\pm 95\%$ for about 1575 seconds before it then declines to $\pm 20\%$ after ± 5200 seconds.

The lower time delay in the model is the better for control design perspective later on, whereas the higher starting concentration is the better fit to the proposed model structure. For modelling purpose, the second experiments data are therefore used in order to get a linear model of the water concentration dynamics (see the proposed model structure in Figure 10) since the data shows lower time delay time and high starting concentration result of the ethanol than the data of the first experiments. The second experiment data for ethanol concentration is next used to compute the water concentration.

C. System Identification

The second experiment data for ethanol concentration is used to compute the water concentration. It is then processed by a system identification software tool to obtain linear models. The data from several experimental runs with longer observation time suggest that the linear model of the water concentration dynamics may be approximated as First Order Plus Delay Time (FOPDT) model. The first order model is preferred since second order approximation will give another pole location that is non dominant.

The FOPDT models will be obtained for two operating points, i.e. when $\theta=1$ and for $\theta=0.5$, from several experiment data. The obtained model fit for those two operating points are shown in Figure 15 and Figure 16 respectively.

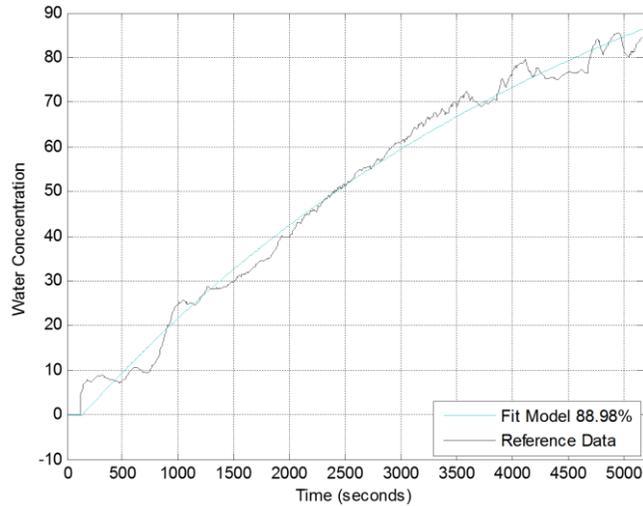


Figure 15. Fit model of water concentration for $\theta=1$

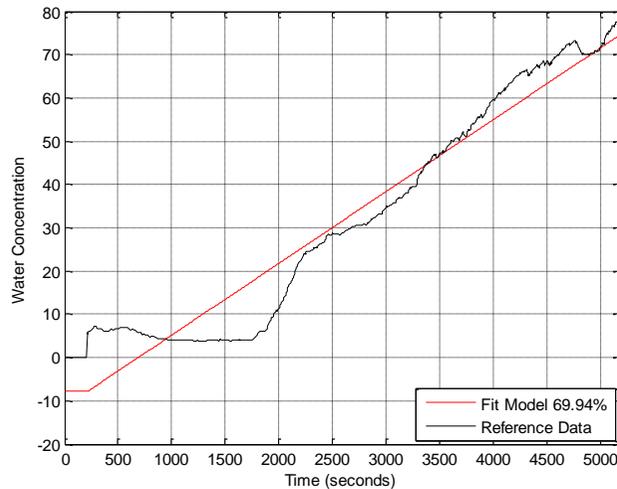


Figure 16. Fit model of water concentration for $\theta=0.5$

Figure 15 shows the FOPDT model for $\theta=1$ is about 88.98% fit with the reference data. With delay time of 135 seconds, the FOPDT transfer function is obtained as

$$P_{11}(s) = \frac{2.748 \times 10^{-4}}{s + 2.039 \times 10^{-4}} e^{-135s}$$

For control design purpose, one may need a state space model that has no time delay instead. In this case, the FOPDT transfer function may be firstly converted into an approximate transfer function with no delay time. First order Pade approximation may then be used to get a new transfer function without delay time as

$$P_{12}(s) = \frac{-2.748 \times 10^{-4}s + 4.071 \times 10^{-7}}{s^2 + 1.502 \times 10^{-2}s + 3.021 \times 10^{-7}} = \frac{-2.748 \times 10^{-4}(s - 1.481 \times 10^{-3})}{(s + 1.502 \times 10^{-2})(s + 2.014 \times 10^{-5})}$$

Meanwhile, for $\theta=0.5$, the FOPDT model is about 69.94% fit with the reference data (see Figure 16). With delay time of 225 seconds, the FOPDT transfer function is obtained as

$$P_{21}(s) = \frac{3.322 \times 10^{-4}}{s + 2.084 \times 10^{-13}} e^{-225s}$$

One may further simplify the transfer function by approximating it as a pure integrator with a delay time, i.e.

$$P_{21a}(s) = \frac{3.322 \times 10^{-4}}{s} e^{-225s}$$

For control design purpose that requires a linear model with no time delay, first order Pade approximation of $P_{21}(s)$ may be taken to get the following transfer function

$$P_{22}(s) = \frac{-3.322 \times 10^{-4}s + 2.953 \times 10^{-7}}{s^2 + 8.889 \times 10^{-3}s + 1.853 \times 10^{-15}} = \frac{-3.322 \times 10^{-4}(s - 8.889 \times 10^{-4})}{(s + 8.889 \times 10^{-3})(s + 2.084 \times 10^{-13})}$$

One may further simplify the transfer function by approximating one very slow first order part of $P_{22}(s)$ as an integrator. A simplified transfer function may be obtained as

$$P_{22a}(s) = \frac{-3.322 \times 10^{-4}(s - 8.889 \times 10^{-4})}{s(s + 8.889 \times 10^{-3})}$$

The linear models indicate that they are all stable with non minimum phase behaviour due to delay time.

5. Discussion

The mini distillation plant itself is well-known to have nonlinear dynamics. Being a batch process, controlling the plant is quite a challenge since there is no way to have continuous material intake to regulate the output, i.e. the end product concentration. The concentration will normally be lower after sometimes since the ethanol content of the initial product has been mostly transferred to the end product. However, there is a possible control effort that may be devised to get the end product having a prescribed concentration as long as possible before the plant should be stopped. It is in this motivation that a plant model will be needed to devise appropriate controller.

The first model to propose is the nonlinear model that is derived from the physical structure of the miniplant and based on known chemical and physical dynamics. The simulation shows that the nonlinear model is not in a good fit with the experimental data. The model fitness seems worse for different experimental input (θ), i.e. between $\theta = 1$ and $\theta = 0.5$. The quite low model fitness may be due to a combination of unmodelled nonlinearities, inaccurate pre calculated parameters of the nonlinear model, and inaccurate concentration sensor. Although nonlinearities have been considered in the model, this suggest that the model has not covered all operating points of the plant. However, the nonlinear model presents the same trend as the experimental data.

Comparing the linear models obtained from two different experimental input clarifies the nonlinear nature of the plant dynamics with its marginal model fit. It is evident that the time delay is longer for lower θ values, i.e. 135 sec for $\theta = 1$ in comparison to 225 sec for $\theta = 0.5$. The second FOPTD model is with lower model fitness to the experimental than the first model. This may suggest that a higher order model would be needed. Moreover, even if it was developed at a certain operating point of θ , the developed linear models could not cover the dynamics due to nonlinearity of other variables, such as temperature.

Even after moving the concentration transducer position, i.e. from the outlet into inlet of the end product tank, the nonlinear behaviour of the plant is understandably still evident. However, the time delay of the model is lower that may facilitate simpler or easier controller design. Similar to the case when the sensor is located at the outlet end-product tank, the two linear models obtained are local models at their respective operating points.

With 3 liters of mixture in the heater pot, the batch process of the distillation column in ITB Honeywell Lab usually run for about two hours even less. The ethanol concentration eventually decreases to very low value since almost all ethanol content in the liquid mixture in the heater pot has been vaporized out. In this case, the nonlinear behavior emerges and there shall be no

control effort capable to regulate the distillate concentration. It is then understandable that the linear models shall show the best model fit with the experimental data for certain period of time.

Although the linear models obtained do not have high fitness to the experimental data, one may select an appropriate model to start designing a controller at a certain operating point that gives favourable concentration of the end product. At minimum, a closed loop controller may be expected to give better results than a process without controller, i.e. open loop operation of the plant. For example, from experimental data and validation of the linear model plus delay of water concentration with $\theta = 0.5$, the graph shows that the concentration is relatively constant for around 1500 seconds. This batch distillation process process can not be regulated all the time. However, if a controller should be devised, it will then be beneficial to regulate the concentration as long as possible. In this regard, the linear model may be used to design a controller at an operating point with $\theta = 0.5$.

6. Concluding Remarks

The batch process in the mini distillation column has been modeled to facilitate a closed loop control design in the later stage. The challenge is not only due to the nonlinearities within the process but also the fact that there is no new material inflow to regulate the end product concentration. In this study, the on-off condenser valve is chosen as an actuator to regulate the end product concentration as long as possible. The valve is assumed to be driven by PWM like control signal at an appropriate time period.

In this context, attempts were done to obtain both nonlinear and linear models of the batch process. Under several assumptions, a nonlinear model has been obtained with low fitness to the experimental data at two different control input at the valve. This may be due to a combination of unmodelled nonlinearities, inaccurate precalculated parameters of the nonlinear model, and inaccurate concentration sensor. The nonlinear model is therefore not yet appropriate for control design purpose and needs further development, e.g. by considering the influence of temperature variability in the model.

Two sets of linear models with time delay have also been developed for two different locations of the concentration sensor, i.e. at the inflow or outflow of the end-product tank. The sensor location at the inlet brings in models with shorter time delays. Unlike the nonlinear model, the linear models developed at two different control inputs show marginal to good fit to the experimental data at respective operating points. The use of the linear models for closed loop control design is therefore by firstly determining an appropriate operating point before developing its associated linear model. For relatively shorter time of operation, a linear model involves an integrator shall give better model fit. Despite that, it seems better to use a piecewise linear modelling over process operating regions for concentration control design purposes of the batch distillation column.

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8. References

- [1]. Guan et al.: A Comprehensive Overview of Cyber-Physical Systems: From Perspective of Feedback System. *IEEE/CAA Journal of Automatica Sinica*, Vol. 3, No. 1, January 2016.
- [2]. W. L. McCabe, J. C. Smith and P. Harriot, *Unit Operation of Chemical Engineering*. New York: McGraw-Hill, Inc, 1993.
- [3]. J. Drgona, Z. Takac, M. Hornak, R. Valo, and M. Kvasnica, "Fuzzy control of a laboratory binary distillation column," *21st International Conference on Process Control (PC)*. Slovakia, pp. 120-125, June 2017.

- [4]. M. Heras-Certavez, J. Anzures-Marin, A. del Carmen Tellez-Anguiano, M. Del Carmen Garcia-Ramirez, J. Correa-Gomez, "Modeling a heating-power actuator for a distillation column boiler," *IEEE International Autumn Meeting on Power, Electronics and Computing (ROPEC)*. Ixtapa, Mexico, pp. 1-6, 2016.
- [5]. G. Orozco, B. Cortez, M. Heras, A. Tellez, J. Anjuez, "Analysis and comparison of Distillation Column Models Considering Constant and Variable Relative Volatility," *IEEE International Autumn Meeting on Power, Electronics and Computing (ROPEC)*. Ixtapa, Mexico, pp. 1-6, 2016.
- [6]. M. U. Diwekar, *Batch distillation: simulations, optimal design and control*. London: Taylor & Francis, 1995.
- [7]. N. A. Habobi, S. M. Yaseen, "Dynamics, simulation and control of a batch distillation column using Labview," *International Jurnal of Current Engineering and Technology*. Iran, vol. 6, pp. 303-309, 2016.
- [8]. P. Acharya, G. Dumpa, T. Kumar Dan, "Modeling and control of distillation column," *IEEE International Conference on Computation of Power, Energy Information and Communication (ICCPEIC)*. Pp. 123-128, 2016.
- [9]. D. Ugryumova, G. Vandersteen, B. Huyck, F. Logist, J. V. Impe, B. D. Moor, "Identification and modeling of distillation columns from transient response data," *2012 IEEE International Instrumentation and Measurement Technology Conference Proceedings*. pp. 2098-2103, 2012.
- [10]. S. Diaz, J. R. Perez-Correa, A. Cipriano, M. F. Fernandez, "Intelligent control applications on a binary distillation column," *IEEE International Conference on Automatica (ICA-ACCA)*. Chile, pp. 1-8, 2016.
- [11]. V. T. Minh, "Modeling and control of distillation column in a petroleum process," *5th IEEE Conference on Industrial Electronics and applications*. Pp. 123-128, 2010.
- [12]. V. T. Minh, A. M. Abdul Rani, "Modeling and control of distillation column in a petroleum process," *Mathematical Problems in Engineering*. Vol 2009. Article ID 404702, 14 pages, 2009.
- [13]. H. Binous, M. A. Al-Harathi, "Simple batch distillation of binary mixture," *Computer Applications in Engineering Education*. Vol 22 issue 4, pp. 649-657. Research Article, 2014.
- [14]. S. Pan, H. Su, T. P. Li, Y. Gu, "State estimation for batch distillation operations with a novel extended kalman filter approach," *Proceeding of the 48th IEEE Conference on Decision and Control (CDC) held jointly with 2009 28th Chinese Control Conference*. Shanghai, China, pp. 1884-1889, 2009.
- [15]. Z. Zou, D. Yu, Z. Hu, N. Guo, L. Yu, W. Feng, "State space modeling and predictive control of a binary batch distillation column," *Proceedings of the 6th World Congress on Intelligent Control and Automation*. Dalian, China, pp. 6253-6256, 2006.
- [16]. R. Maulidda, P.H. Rusmin., A.S. Rohman, E.M.I. Hidayat, D. Mahayana, Modeling and Simulation of Mini Batch Distillation Column. *5th International Conference on Instrumentation, Communication, Information Technology, and Biomedical Engineering 2017*, Bandung, November 6-7, 2017.
- [17]. W. L. Luyben, *Process Modeling, simulation and control for chemical engineers* 2nd edition. New York: McGraw-Hill, Inc, 1990.
- [18]. I. Saputra, P.H. Rusmin, E.M.I. Hidayat, A.S. Rohman, D. Mahayana, "Analysis of Implemented Density Sensor Models with Flow rate as Disturbance", *Proceedings of The 2017 International Seminar on Sensors, Instrumentation, Measurement and Metrology*, Surabaya, Indonesia, August 25-26, 2017.



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