MODELLING AND SIMULATION OF FERMENTATION PRODUCT PURIFICATION USING BATCH DISTILLATION

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MODELLING AND SIMULATION OF FERMENTATION PRODUCT PURIFICATION USING BATCH

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the award of the degree of
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SUPERVISOR'S DECLRATION

I have hereby declared that I have checked this thesis and my opinion, this thesis is adequate in terms of scope and quality for the award of degree of Bachelor of Chemical Engineering.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have only acknowledge. The thesis has not been accepted for any and is not concurrently submitted for award of other degree.

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DEDICATION

Special dedication to my supervisor, Dr. Anwaruddin Hisyam for your Time, Guidance, and Support.

And,

To my beloved parents (Bakar bin Mamat & Timah bt Abdul Rasid) and friends, that encouraged and fully supports me throughout completing this thesis.

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MODELLING AND SIMULATION OF FERMENTATION PRODUCT PURIFICATION USING BATCH DISTILLATION

ABSTRACT

Batch distillation has important for industries recent years. This is mainly due to its flexibility and capability to produce high purity product. The simulation of batch distillation columns during steady state operations is a very challenging modelling problem because of the complex dynamic behaviour. In order to verify this issue, simulation of this batch distillation column is to be performed by using computer aided design software and mathematical model so that the results can be validated. This study is required to model and simulate of fermentation product purification using batch distillation. In this study, simple mathematical models are proposed for the simulation of the dynamic behaviour during steady state operations from product of fermentation. Simulation of simple batch distillation was done by using mathematical method and computer simulator, MATLAB ®. The results from these simulations were used as a basis to validate the results obtained from experimentally. As conclusion, distillate and bottoms composition for ethanol drop gradually over time by using graphical method and the trend from simulation showed that the distillate and bottoms composition for ethanol increase and decrease over the time respectively. The declining trend was due to the unsteady state nature of batch distillation, where the lighter component at the bottoms will deplete over time. As using computer software simulation, it able to integrate the model equations for fermentation product purification based on suitable numerical methods and it also gain the understanding due to simulation.

MODELLING AND SIMULATION OF FERMENTATION PRODUCT PURIFICATION USING BATCH DISTILLATION

ABSTRAK

Kebelakangan beberapa tahun ini, penyulingan berkelompok mempunyai kepentingan bagi industri . Hal ini adalah disebabkan oleh fleksibiliti dan keupayaan untuk menghasilkan keaslian produk yang tinggi. Simulasi kolum penyulingan kelompok semasa operasi keadaa mantap adalah masalah pemodelan yang sangat mencabar kerana dalam keadaan dinamik kompleks. Dalam usaha untuk mengesahkan isu ini, simulasi dalam penyulingan kelompok harus dilakukan dengan menggunakan komputer perisian reka bentuk dibantu dan model matematik supaya keputusan boleh disahkan. Kajian ini diperlukan untuk membina model dan simulasi pembersihan produk penapaian menggunakan penyulingan kelompok. Dalam kajian ini, model matematik mudah telah dicadangkan untuk simulasi kelakuan dinamik semasa operasi keadaan mantap daripada hasil penapaian. Simulasi penyulingan kelompok telah dilakukan dengan menggunakan kaedah matematik dan simulator komputer, MATLAB ®. Keputusan daripada simulasi ini telah digunakan sebagai asas untuk mengesahkan keputusan yang diperolehi daripada eksperimen. Kesimpulannya, hasil sulingan etanol dan hasil etanol dalam bekas pemanas adalah turun secara beransur-ansur dari semasa ke semasa dengan menggunakan kaedah graf dan trend daripada simulasi menunjukkan bahawa komposisi sulingan dan etanol dalam pemanas bagi etanol masing-masing adalah meningkat dan berkurangan dari masa ke masa itu. Hal ini oleh sifat keadaan tak mantap kumpulan penyulingan, di mana komponen ringan di dalam pemanas akan berkurang apabila lebih masa.Dengan menggunakan simulasi komputer perisian, ia dapat mengintegrasikan persamaan model untuk pembersihan produk penapaian berdasarkan kaedah berangka dan ia juga mendapat kefahaman kerana simulasi.

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A and n: regression coefficient for chemical compound respectively

H	Enthalpy of vaporisation, KJ/mol
H_e	enthalpy of vaporisation of ethanol, KJ/mol
H_w	enthalpy of vaporisation of water, KJ/mol]
Lo	Initial charge, kmol
Lo	initial charge, mol
Lt	total holdup at bottom at any time, kmol
Lt	Total holdup at bottom at any time, kmol
Q	heat input, KW
R	Reflux ratio
T	Temperature, K
Tc	Critical temperature, K
V	*vapour* boil-up rate, kmol/s
x_e	mole fraction of ethanol, mole
x_o	Initial bottom composition of lighter component
x_t	Bottom composition of lighter component at any time
x_w	Distillate mole fraction of water

CHAPTER 1

INTRODUCTION

1.1 Introduction

Batch distillation process is an important separation technique for many centuries that used in many areas of pharmaceutical and especially in chemical industries. It uses for separating or purifying liquid mixtures and waste removal in chemical process industries. Fermentation is one of the chemical processes in this industry. Where, local vinegar is formed from fermentation that can be used for separating via simulation. Therefore, it has common issues for the researchers in order to design alternative and suitable column configurations, to develop of mathematical models in line with the development of numerical methods and to use of artificial software networks in dynamic modelling, optimisation and control

In addition, batch distillation processes can easily handle variations both in the feed composition and in the product specifications. This variation of batch distillation processes provides the ability to manage with a market characterized by short product life times and strict specification requirements. Although the fixed cost required for constructing a batch column is lower than a series of continuous column, but the operating costs of batch distillation is higher due to more energy

consumption. Furthermore, the lowest capital cost design does not necessarily make for the most economical solution due to the low performance and high operating costs associated with a high reflux column (Kian & Sorensen, 2012). Since, batch distillation is often attractive for fermentation product purification. The flexibility of batch distillation allow for configuring the column of conventional column (used in this study), inverted column, middle vessel column and multi-vessel column, which are given in Figure 1.1.

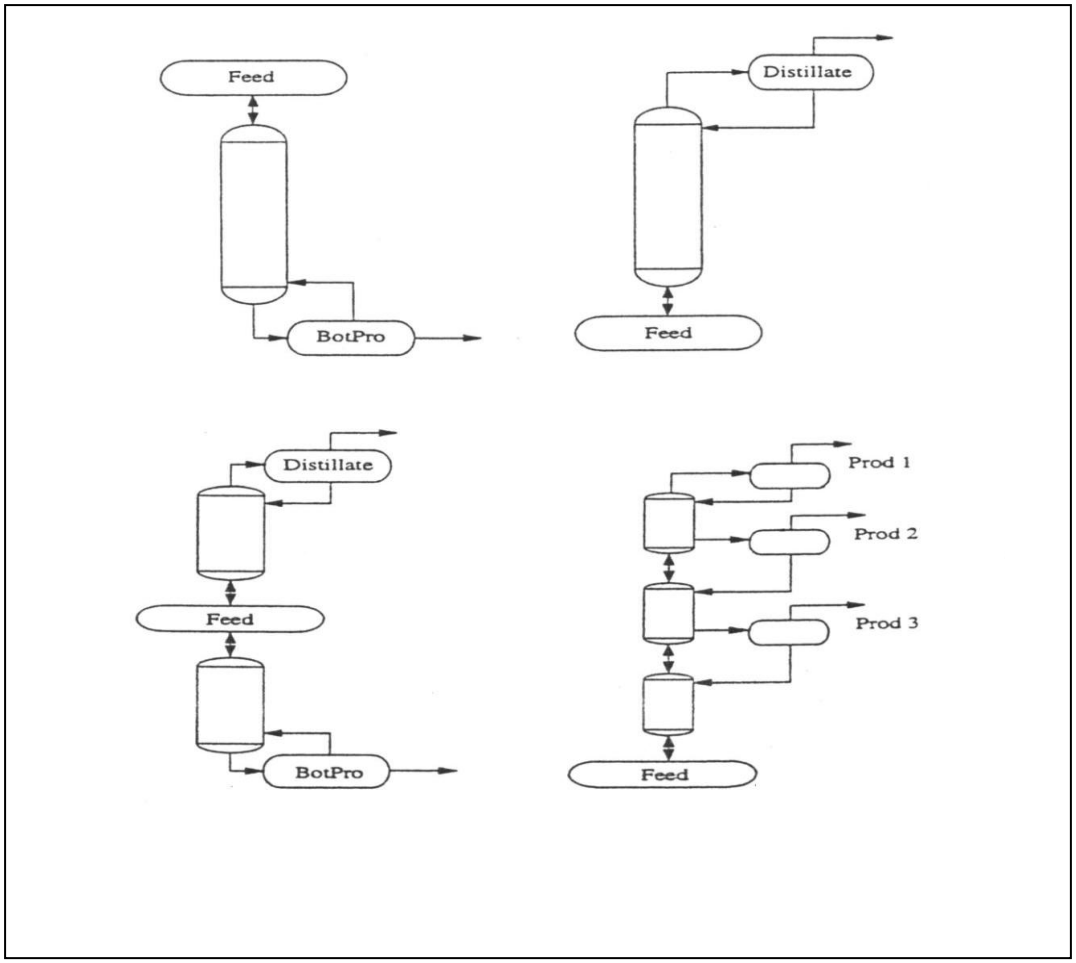

Figure 1.1 Examples of Ways to Configure The Batch Distillation Column.

(Source: Mujtaba.2004)

Current technology has made simulation of a batch distillation much easier and less complicated compared to a few decades. Simulation is usually the first step to conduct feasibility study of a design before it can be implement into a big industrial scale plant. Not only feasibility study, simulation give a better understanding of a process, approximation of the cost to setup and potential challenges that the process may face upon setting up the design to industrial scale.

Graphical method is used to conduct the simulation of process involves in an existing plant. It can help engineers to predict the behaviour of a process using basic engineering relationships such as mass and energy balances, phase and chemical equilibrium, and reaction kinetics. Besides, MATLAB® is also one of alternative software for running this simulation.

1.2 Statement of Project Problem

Simulating the actual operation of convectional column has been the subject of much research for recent century. The main interest was to develop a model that could be the best prediction on batch distillation column. It is difficult to teach batch distillation without using computer because in the process time is varying and has complex numeric integration techniques and process provide flexibility in operating and configuring column.

Batch distillation operates in unsteady where the compositions of the mixture are changing with time. Thus, it is difficult to control the process at all the process. In batch distillation a fixed amount of charge is added to the still for a long time to separate the mixture. Then, top product composition will varies with time. The

separation time depends on amount of bottom product composition, number of trays and reflux ratio.

Thus, it needs to consider the ways of configuration based on parameter which needs to be set as basis for a particular design. For a Rigorous model in batch distillation, a detailed analysis of characteristic of differential mass and energy balance associated with the complete dynamics of a multi component (Diweker, 2005). He said that the system of equations presented for batch distillation is more difficult to solve than that for continuous distillation due to several factors.

Simple Rayleigh Model is well- known as the best ways on the modelling of batch distillation (Mujtaba, 2004). It is good with the development of high speed digital computers which is the main issues in modelling. It can detailed energy balances, column holdup, accurate physical properties in order to simulate the actual operation of batch distillation.

In order to verify this issue, simulation of this batch distillation column is to be performed by using computer aided design software and mathematical model so that the results can be compared and validated. It is also necessary to understand the behaviour of batch distillation separation of multi component system.

1.3 Research Objective

Simulation on batch distillation column is done by using computer software simulation to examine the trend of distillate and bottoms composition at constant reflux and regulating reflux for fermentation of product purification. Both of the results from the simulation will be compared to the results obtained experimentally

(by others) using an existing column in research study had done by others. Thus, experimental results can be validated.

Secondly is to simulate the parameter of fermentation product purification including reflux ration and column pressure. Here, parameter such as composition product will change with time of separation.

Lastly is to develop a mathematical model in order to study of fermentation product purification.

1.4 Research Questions

1.4.1 Which one the best model of batch distillation process for product purification?

1.4.2 What is the trend of distillate and bottoms composition?

1.4.3 What is the parameter studied that affect the product purification via simulation?

1.5 Scope of Research

In order to achieve the objective of the project, some boundary or scope need to be specified. This project covers the simulation and modelling of separation of fermentation product purification using batch distillation and computer software simulation.

Firstly, the binary mixture used in this project is ethanol-water. It is chosen for it’s highly availability and also it is the most common used in the industries, where it is cheap and non-toxic.

Next, this study will focus on comparison the result via simulation and the results obtained experimentally had done by other student.

1.6 Expected Outcomes

This study will be able to examine the trend of distillate and bottoms composition at constant reflux and regulating reflux for fermentation of product purification using batch distillation. These are important results towards the further development of a reference method for product purification. In many cases it was found that the models had to be simplified because of several reasons. There are capabilities of the computer software, availability of suitable numerical methods to integrate the model equations and gain in accuracy of product purity.

It is also expected that high understanding on running the simulation for the industry plant especially using the batch distillation.

1.7 Significance of Research

The most significant input in this study is to increase in the production of high value added, low volume specially chemicals and biochemical for high purity. The research studies highlighted the importance of considering all the design and operational process available in order to gain a comprehensive economical close into the batch distillation process. It is maybe can optimum the production of time, capital costs, mixture characteristic and process allocation. The flexibility of batch

distillation processes provides the ability to manage with a market characterized by short product life times and strict specification requirements.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Batch distillation is the oldest separation process which is an important unit operation in the batch processing industry and is widely used (Mujtaba, 2004). The earliest batch distillation is known as Rayleigh distillation, was presented in by Lord Rayleigh (1902). In his model, there is only a condenser, a column and a still pot. No reflux is returned to the still and no stage or packing material inside the column. Hence, Rayleigh distillation is simply a one stage distillation, and the analysis is based on material and component balance (Diwekar, 1996). Due to the increasing of production on high value added, low volume specialty chemicals and biochemical, the batch distillation has been generated a new processing technologies (Diwekar, 1996). The flexibility of batch distillation, combined with inherent unsteady nature of the process, quite challenging design and operation problems. As result, many chemical engineers used batch distillation as their practical in batch processing operation.

2.2 Batch Process

Batch distillation column can be operated with three reflux policies in order to meet certain product specification. The reflux policies that can be employed to a batch distillation are either constant reflux, regulating reflux or optimum reflux. For constant reflux, distillate purity for lighter component will drop over time. In order to maintained the purity of distillate over time, regulating reflux policy can be employed. Optimum reflux policy is used when maximum profit from the operation is required; it is a trade off policy between constant reflux and regulating reflux policies (Diwekar, 1996). In Mujtaba (2004) book states, batch distillation operated on conventional batch distillation and continuous batch distillation.

In conventional batch distillation process, a bottom receiver or reboiler which charged with the feed to be processed and provide the heat transfer surface area. Then, a rectrifying column superimposed on the reboiler, coupled with either a total condenser or partial condenser system. This operation involves carrying out the fractional until desired amount has been distilled. During the process, the overhead composition is varies and usually number of cuts are made. The desired from the cuts are recycling to subsequent batches to obtain further operation.

2.3 Advantages of Batch Distillation

The main advantages of batch distillation based over on a continuous distillation are the uses of single column as opposed to multiple columns and its flexible operation. Besides, the alternative operation does not take into the

production of off specification material. Next, only one column and one sequence are necessary to separate all the components in a mixture. Continuous distillation column also can operate for long time (Mujtaba, 2004).

Whereas, Shahidatul (2008) thesis states batch distillation is less energy efficient than continuous distillation and it offers possibility of separating multi-component mixtures into high purity products using single columns. So, it is less expensive compared to continuous distillations that required (n-1) columns. It is flexible and robust to variation in feed composition and specification. It is important for production of seasonal or low capacity chemicals. Batch distillation also can be used for liquids with contaminants such as solids, tars and resins.

2.4 Batch Distillation Operation

Based on Fernholz G. et al., (2000) and Mujtaba (2004), batch distillation is operated in the following manner which is start-up period, production period and shutdown peroid. For start-up, the reboiler is filled with a specified amount of mixture and heated until the complete column is filled with boiling mixture. Then, the manipulated variables in each time interval are set to their optimal values. Lastly, the heat supply and the feed flow are stopped and the system is cooled down. The start-up and shutdown procedures are not included in the optimization because validated models for these phases are not yet available

2.5 Column Configuration

According to Mujtaba (2004) and Diweker (1996), batch distillation column can be operating in various configurations. There are two types of configuration which are conventional column configuration and unconventional column configuration. But, the alternative configuration that has found more advantages is unconventional column distillation. Unconventional configuration consists of inverted batch distillation, middle vessel batch distillation column, multivessel batch distillation column and continuous column. Both writers agree that it is multivessel batch distillation column of configuration to obtain purer product at the of a total reflux operation.

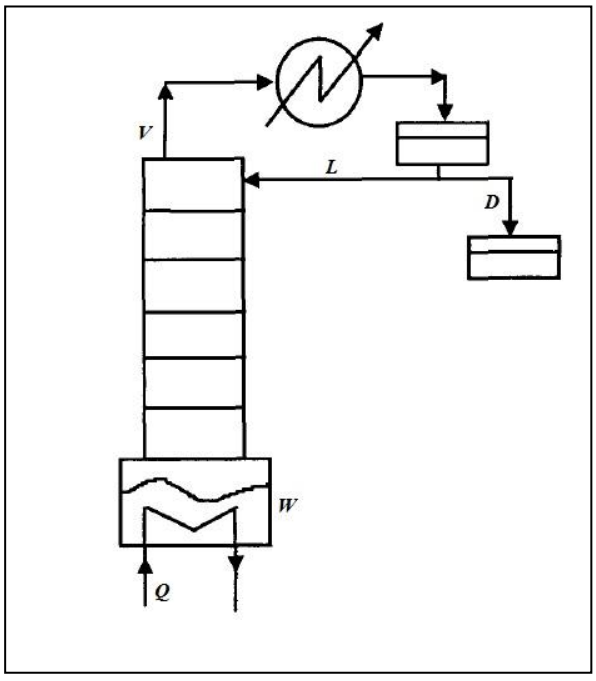

Figure 2.1 Conventional Batch Distillation Column

(Source: Mujtaba, 2004)

2.5.1 An Inverted Batch Column

In this column configuration, the feed mixture is charged into the top reflux drum, and the products are withdrawn at the bottom reboiler. A minimum boiling point azeotrope can be break in inverted batch column compare to the rectifier. Batch time in the inverted column configuration is also better than the regular column for separations where the light component in the feed is present in a small amount. (Diweker, 1996)

Meanwhile, Mujtaba (2004) states in these types, the feed charge and the condenser flux drum are combined and it operates in an all stripping mode with a small holdup reboiler. Its operation is same as the conventional batch column except that products are withdrawn from the bottom. This type of operation is supposed to eliminate the thermal decomposition problem of high boiling point products.

2.5.2 Middle Vessel Column

According to Diweker (1996), this column configuration consists of a middle vessel between two sections of the batch column. The feed is initially charged into the middle vessel, and the products are simultaneously withdrawn from the top and the bottom of the column. The middle vessel column can be an ideal configuration for ternary batch systems. This column configuration is very flexible and effective; hence, it obtains very pure components in the top, bottom, and middle vessel columns.

As mentioned by Mujtaba (2004), in this type, the separation is divided as in the usual continuous distillation column into rectifying and stripping section with a feed tray in the middle. The important features if these types are the feed is supposed to a suitable location in the middle of the column, liquid on the feed tray is recycled to the feed tank and the products can be withdrawn simultaneously from top and

bottom of the column. This type is absolutely very flexible in the sense that it can be easily converted to a convectional by changing the location of the feed and by closing or opening appropriate valve in the product.

2.5.3 Multivessel Batch Distillation Column

In this type, the charges in each vessel will be purified as the distillation proceeds if the column operates at total reflux. But, it depends on the number of plates in each section, vapor boil-up, the amount of initial charge in each vessel and the duration of operation to achieve purity (Mujtaba, 2004).

Based on Diweker (1996), this new emerging column configuration can have better separation performance than continuous distillation for systems having a larger number of products. An optimal operation policy for this column can be achieved using variable holdup modes. In this type, the batch time is affected the liquid flow rates in order to minimize distillate than that of the constant holdup mode. Moreover, this column is operated under total reflux conditions. This operation policy can be the ideal operation policy of batch distillation, especially for the middle vessel and multivessel columns. The total reflux mode is commonly used for the last multivessel column because multiple products can be accumulated in each vessel according to their relative volatilities. The initial feed distribution of column performance also been improved using various operating modes in terms of a mean energy consumption rate on the rectifier.

The multivessel column is also studied by Skouras S. & Skogestad S. (2004). This is the first study where the dynamics in the middle vessel of multivessel configurations are discussed where two different of multivessel configurations and a rectifier column are compared to each other .The multivessel batch column can be

viewed as a generalization of a batch rectifier and a batch stripper. The column has both a rectifying and a stripping section. Therefore a light and a heavy fraction are possible to obtain simultaneously from the top and the bottom of the column, while an intermediate fraction is also recovered in the middle vessel. In the multivessel column a ternary mixture can be separated simultaneously in one such close operation. No product change-overs are required and all products are accumulated in the three vessels at the end of the process.

2.6 Modelling And Simulation

According to Mujtaba(2004) simulation is the actual operation which consists of simulation of start-up period and simulation of product period. Many research has been done by simulating the operation of convectional column including by Mujtaba(2004), Diweker(1996) and Ronnie (2011). In simulation, it is important to develop a model before run the simulation. The model should consist of mass and energy balance, hydraulic model, physical properties and etc.

Meanwhile, modelling of batch distillation begins with simple Rayleigh Model (Rayleigh, 1902) and the well-known. In modelling, it requires to know whether and how to include energy balance, hydraulic model, accurate physical properties to simulate the actual operation batch distillation. It found that the model had to be simplified first because of size and complexity of the problem, capabilities of computer, and availability of suitable numerical methods to integrate the model equations, gain in accuracy in the prediction of real operation (Mujtaba, 2004).

Mehlhorn et al. (1996) investigated mass transfer and tray hydraulics issues in batch distillation modelling. They have integrated non-equilibrium and equilibrium models to get more accurate model for batch distillation columns with perforated plates. They handled the uncertainties in the mass transfer which was described within the equilibrium-tray model by means of tray efficiency and by using the non-equilibrium-tray model supported by the mass transfer coefficient and the mass transfer area. The developed model was experimentally verified both for binary and multi component systems.

CHAPTER 3

METHODOLOGY

3.1 Computer Software Simulation

Computer software simulation is done by using commercial software (MATLAB ®) and validated using graphical method. MATLAB provides several types of functions for performing mathematical operations and analyzing data, such as matrix manipulation, linear algebra, polynomials and interpolation, ordinary differential equations, partial differential equations, sparse matrix operations, 2D and 3D plotting and much more. The conventional batch distillation that used in this research is simple batch distillation (Rayleigh distillation). In this model, the vapour is removed from the still during a particular time interval and is condensed in the condenser. The more volatile components are richer in the vapour than in the liquid remaining in the still (Diwekar, 2005).

3.2 Study of Parameters

Certain parameters will take place in order to achieve the objective. The parameters consist of:

i) Vapour boil up rate
ii) Liquid boil up rate
iii) Time constraints
iv) Reflux ratio
v) Condenser hold up
vi) Reboiler hold up

3.3 The Assumptions of a Distillation Column

In forming a model for the distillation column, general assumptions about the operation of a distillation column must be made. The assumptions a model makes are the major distinctions between the models found in the distillation literature. A complete set of dynamic equations would be of a daunting size considering that a stage contains many forms of energy transformations and that a distillation column or a set of columns can contain hundreds of stages. Of course, simplifying assumptions are desirable in such a complex apparatus.

In this simple distillation process, it is assumed that the vapor formed within a short period is in thermodynamic equilibrium with the liquid. Hence, the vapour composition (x_D) is related to the liquid composition (x_w) by an equilibrium relation of the form $x_D = F(x_D)$

Others assumptions can be assumed are the condenser, the trays and chemical reactions. The most general models also allow side streams, feeds, and heat transfer

on each stage, allowing for a specific complex column to be modelled. The first assumption is heat flow. If the column is adiabatic, the only heat exchanges by conduction that need be modelled are those in the condenser and reboiler. Secondly, the thermal capacitance of the column metal is negligible. Both of these assumptions are used by almost all distillation models. Next, Vapour holdup which is mass and energy on each stage is negligible. The molar holdup of the vapour is usually much less than the molar holdup of the liquid, thus, the vapour holdup effects are considered small. Lastly, vapour boil-up, liquid boil-up rate, and total molar hold-up are assumed constant.

3.4 Start up Conditions

Before simulation can be done, the batch distillation need to start up periods consists of the following three steps:

i) Preheating the still charge to its bubble point
ii) Filling the column and condenser holdups
iii) Running the column without distillate withdrawal and bringing the unit to a steady state

These all step are done by others researches. At the end of the total reflux condition, the given charge distributes itself throughout the column. This effect is known as capacitance affect which the concept of equilibrium time. The equilibrium time is the time required from the time the column is charged to obtain steady state.

For second step can be achieved in different ways as shown below:

i) Charge the feed from top so initially the pot composition as well as the composition on each plate is equal to the feed composition

ii) Charge the feed to the still and operate the column without reflux. This results in the composition on each plate being equal to the vapour composition which is in equilibrium with the feed composition

While simulating the complete dynamics, the equilibrium time needs to estimate because it cannot be obtained from steady state model.

3.5 Mathematical Model Equations

A simple batch distillation model of the batch distillation operation involves solving several differential equations. The first detailed analysis of batch distillation represented by Mujtaba (2004). He points the equations for the condenser are presented. Then, the equation for the accumulator, followed by equations for the plates in the column and the reboiler are presented. The trays are counted from the top to the bottom.

Before running simulation, the boil-up rate of batch distillation operation needs to be calculated. This can be achieved by predicting the latent of vaporisation of the component involved at certain temperature by using modified Watson's equation (Coker, 2007).

$$He = A(1 - \frac{T}{TC})^n \quad (3.1)$$

Where,

H = enthalpy of vaporisation, KJ/mol
Tc = critical temperature, K
T = temperature, K
A and n = regression coefficient for chemical compound

Once the latent heat of vaporisation was found and together with the heat input of the system, boil-up rate of the system can be calculated by using:

$$Q = (x_e H_e + x_w H_w)V \quad (3.2)$$

Where,

Q = heat input, KW
x_e = mole fraction of ethanol
x_w = mole fraction of water
H_e = enthalpy of vaporisation of ethanol, kJ/mol
H_w = enthalpy of vaporisation of water, kJ/kmol
V = vapour boil-up rate, kmol/s

3.5.1 Rayleigh Model

Simulation of binary distillation with constant reflux ratio can be done by using Rayleigh equation in conjunction with graphical method (Kim & Diweker, 2005). Batch distillation can be analysed mathematically by using Rayleigh equation.

Originally, a charge of L_o of moles of components A and B with composition of x_o mole fraction of A is placed in the still. At any given time, there are L mole of liquid left in the still with composition x and the composition of the vapour leaving in equilibrium is y. A differential amount dL is vaporized. Thus,

$$-dL = dxL \quad (3.3)$$

The differential material balance for lighter component can be written as:

$$Ldx + xdL = ydL \quad (3.4)$$

Rearranging and integrating,

$$\int_{Lt}^{Lo} \frac{dL}{L} = \ln \frac{Lo}{L} = \int_{xt}^{xo} \frac{dx}{(y-x)} \quad (3.5)$$

Where,

Lo= initial charge, kmol
Lt = total holdup at bottom at any time, kmol
xo = initial bottom composition of lighter component
xt = bottom composition of lighter component at any time

The equilibrium curve gives the relationship between *y* and *x*. The integration of equation (3.6) can be done by calculating values of *f(x) = 1/(y-x)* and numerically or graphically integrating equation (3.6) between xt and xo. The average composition of total material distilled, y_{av} can be obtained by material balance:

$$L_oX_o = L_tX_t + (L_o\text{-}L_t)\, y_{av} \quad (3.6)$$

The bottoms holdup at any time can be found by using

$$Lt = Lo - \frac{Vt}{R+1} \quad (3.7)$$

Where:

Lt = total holdup at bottom at any time, kmol
Lo = initial charge, kmol
V = boil rate, kmol/s
R = reflux ratio

3.5.2 Simple Model Type III

3.5.2.1 For condenser section, that is for j=1,

The condenser characteristic the first tray and the holdup tank contain an amount of liquid H_C which kept constant at all times. The component balance for the holdup tank is:

$$\frac{dxli}{dt} = \frac{V}{HC}\ (y2i - xli) \tag{3.8}$$

The reflux ratio is defined as:

$$L=rV \tag{3.9}$$

3.5.2.2 For Reboiler section, that is for, j=1

The amount of mixture left in the reboiler depends on the liquid and the vapour flow rate through the column. The total mass balance is written as:

$$\frac{dHN}{dt} = L - V \tag{3.10}$$

The component mass balance

is

$$H_N \frac{dxi}{dt} = L(x,i) - V(y - x, i) \tag{3.11}$$

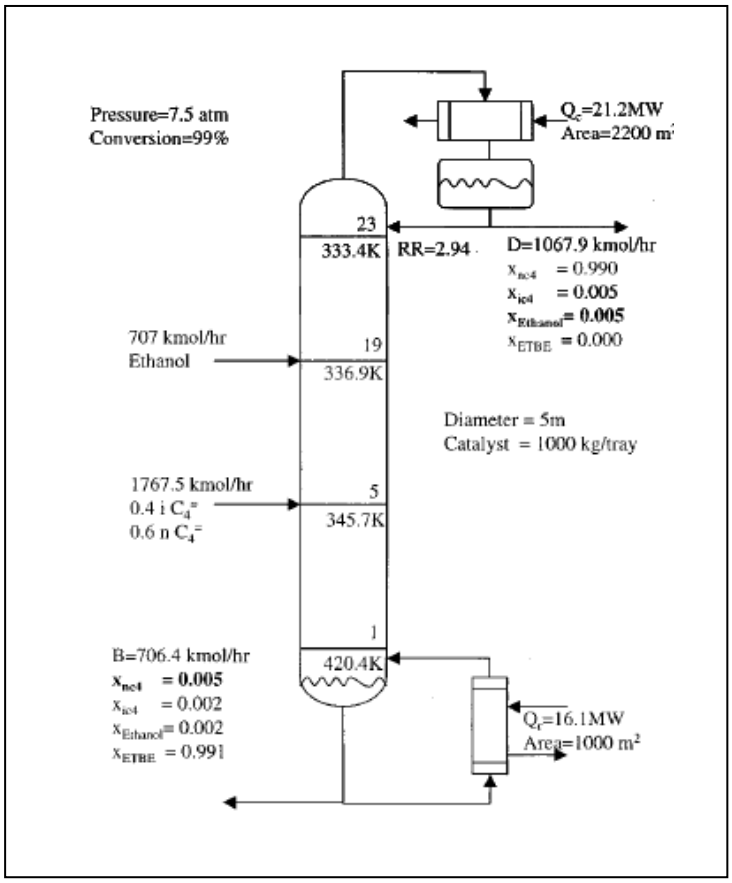

Figure 3.1 Example of Batch Distillation Column Scheme

(Source:Mujtaba, 2004)

3.6 Experimental Result Done By Others

The experimental results use in this research is obtained from previous experiment by others.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Determination of Flow Rate

The vapour boil-up rate, *V*, was estimated by using Equation (3.1) and Equation (3.2). The critical temperature, *T* and regression coefficient *A* and *n* for ethanol and water given by Coker (2007) is as per Table 4.1.

Table 4.1 Critical Temperature *Tc*, and Regression Coefficient *A* and *n* for Ethanol and Water

Compound	A	n	Tc (K)
Ethanol	43.122	0.079	516.25
Water	52.053	0.321	647.13

(Source: Coker, 2007)

The feed mixture of ten litres of 50 v/v% of ethanol in water was converted to mole fraction first.

Mole of ethanol, n_e:

$$ne = \frac{5x10^{-3}m^3\ x\ 789kg/m^3}{46.07kmol/kg}$$

= 0.0856kmol (85.6 mol)

Mole of water, n_w :

$$nw = \frac{5x10^{-3}m^3\ x1000kg/m^3}{18.02kmol/kg}$$

= 0.2775kmol (2775mol)

Total initial holdup, *Lo*

= 0.0856 + 0.2775= 0.3631 kmol

Mole fraction of ethanol,*xe*:

$$Xe = \frac{0.0856}{0.0856+0.2775}$$

= 0.2357 kmol

Mole fraction of water, *xw* :

Xw = 1 - 0.2357

= 0.7643 kmol

Using heat input of 1.85KW and temperature at 85°C, the vapour boil-up rate, V is found to be:

$$He = A(1 - \frac{T}{TC})^n$$

$$= 43.122(1- \frac{85+273.15}{516.25})^{0.079}$$

$$= 39.27341 \text{ KJ/mol}$$

$$Hw = A(1 - \frac{T}{TC})^n$$

$$= 52.053(1-\frac{85+273.15}{647.13})^{0.321}$$

$$= 40.18427 \text{ KJ/mol}$$

$$Q = (x_e H_e + x_w H_w)V$$

$$V = \frac{Q}{(x\text{eHe} + x\text{wHw})}$$

$$= \frac{1.85}{(0.2357x39273.41) + (0.7643\ x\ 40184.27)}$$

$$= 4.6285\text{x}10^{-5} \text{ kmol/s}$$

$$= 2.777\text{x}10^{-3} \text{ kmol/min}$$

In this experiment, the temperature at the pot (reboiler) is always maintained at 85^{o}C. Thus, the boil-up is assumed to be constant throughout the distillation.

The liquid hold-up at the bottoms, L at any time then calculated using equation (3.9) with the reflux ratio of 4.

$$R = \frac{L}{V}$$

$$L = RV$$

$$= 0.8(2.777\text{x}10^{-3})$$

$$= 2.2216\ \text{x}10^{-3} \text{kmol/min}$$

4.2 Determination of Distillate Composition(*Y*) and Bottom Composition(*X*)

4.2.1 Graphical Method

Reflux of 80% is equal to reflux ratio of 4. Using time equal to 10 minutes as example, the hold-up at the bottom L_{10} can be calculated by using

$$Lt = Lo - \frac{Vt}{R+1}$$

$$= 0.3635 - \frac{2.777x10^{-3}}{4+1}$$

L_{10} = 0.357946 kmol

The relationship between *y* and x were obtained from vapour-liquid equilibrium data for ethanol and water and tabulated in the Table 2. Then, a graph of 1/(y-x) against x was plotted.

Table 4.2 Relationship between *x*, *y* and 1/(*y*-*x*)

Mole Fraction		
Liquid (*x*)	Vapour (*y*)	1/(*y*-*x*)
0.019	0.17	6.622517
0.072	0.389	3.154574
0.097	0.438	2.932551
0.124	0.47	2.890173
0.166	0.509	2.915452
0.234	0.545	3.215434
0.261	0.558	3.367003
0.327	0.583	3.90625
0.397	0.612	4.651163
0.508	0.656	6.756757
0.52	0.66	7.142857
0.573	0.684	9.009009
0.676	0.739	15.87302
0.747	0.782	28.57143

By using the initial condition t=10minutes as example the initial total hold up at the bottoms that calculated is 0.3631 kmol with the ethanol and water mole fraction of 0.2357 kmol and 0.7643 kmol respectively. Value of y and x were

calculated by using equation (3.5) and equation (3.6). The integration of equation (3.6) can be done by calculating values of $f(x) = 1/(y-x)$ and numerically or graphically integration equation (3.5) between x_t and x_o. The area under the curve will give value of ln (L_1/L_2) and to avoid graphically integration, an equation to fit the curve was obtained by using Microsoft Excel as shown in the Figure 4.1. The unknown is x_1, the composition of liquid L_2 at the end of the differential distillation. To solve this numerical integration, the equilibrium data can be fitted to a polynomial function as shown in Figure 4.1.

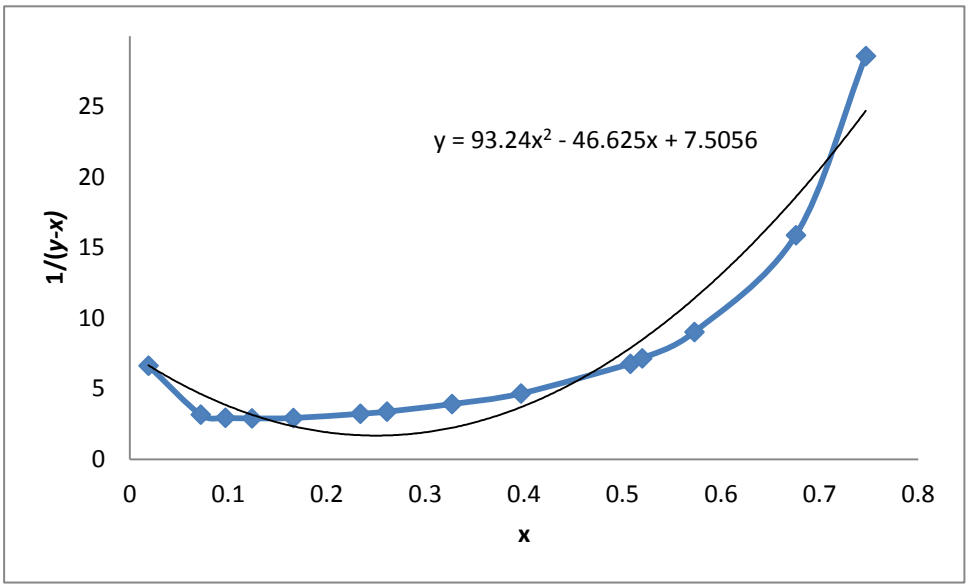

Figure 4.1 Numerical Integration for Ethanol-Water

$$\ln \frac{L0}{L} = \int_{xt}^{xo} \frac{dx}{(y-x)}$$

$$\ln \frac{0.3631}{0.3579} = \int_{x10}^{x0.2357} 93.24x^2 - 46.625x + 7.5056$$

$$0.0143 = [\frac{93.24}{3} x^3 - \frac{46.625}{2} x^2 + 7.5056x]_{x10}^{0.2357}$$

$$x_{10} = 0.2267$$

Thus, the average composition of total material distilled, y_{av}, can be obtained by equation (3.6)

$$L_oX_o = L_tX_t + (L_o\text{-}L_t)\, y_{av}$$

$$y_{av} = \frac{\text{LoXo} - \text{LtXt}}{(\text{Lo} - \text{Lt})}$$

$$y_{av} = \frac{0.3631(0.2357) - 0.3579(0.2267)}{(0.3631 - 0.3579)}$$

$$= 0.8608 \text{ kmol}$$

Calculations at time 20, 40, 60, 80 and 100 minutes are done based on the same method as above. The results from the calculation are tabulated in the Table 4.3 and Table 4.4.

Table 4.3 Composition of Distillate and Bottom from Graphical Method

Time,t	Mole Fraction at distillate		Mole Fraction at bottom	
(minutes)	Ethanol	Water	Ethanol	Water
0	0	0	0.2357	0.7643
10	0.860751	0.139249	0.2267	0.7733
20	0.805029623	0.194970377	0.2184	0.7816
40	0.78010242	0.21989758	0.2009	0.7991
60	0.752164099	0.247835901	0.1842	0.8158
80	0.721925082	0.278074918	0.1686	0.8314
100	0.692557599	0.307442401	0.1539	0.8461

Table 4.4: Distillate and Bottom Holdup Composition

Time,t (minutes)	0	10	20	40	60	80	100
Hold up at distillate	0	0.00515	0.01071	0.02182	0.03292	0.04403	0.005514
Hold up at bottom	0.3631	0.35795	0.35239	0.34128	0.33018	0.31907	0.30796

The profile of mole fraction at distillate and bottom were plotted as shown in Figure 4.2 and Figure 4.3

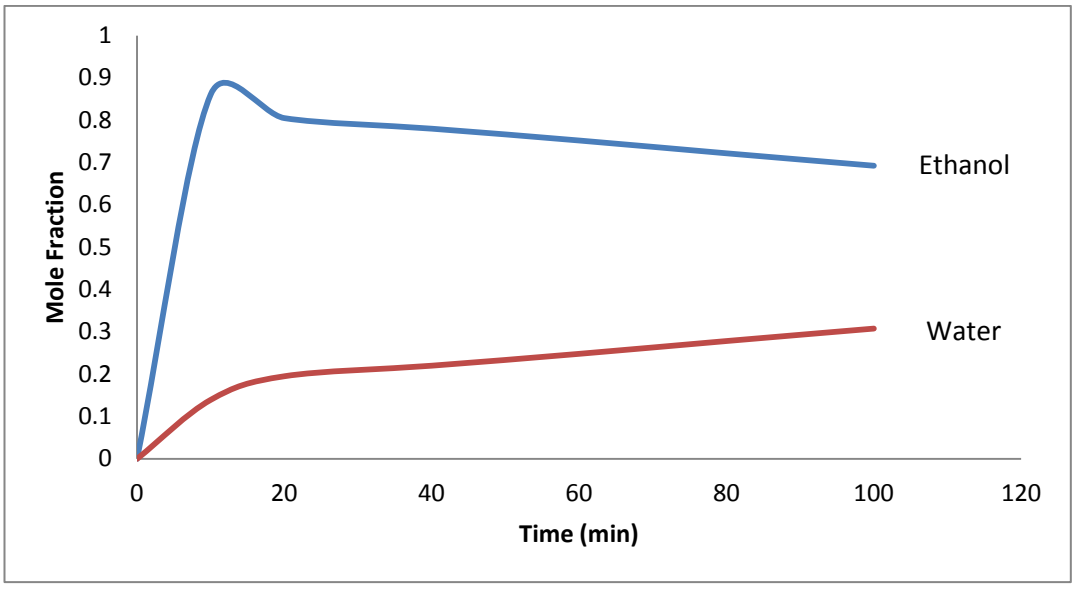

Figure 4.2 Theoretical Dynamic Response of Mole Fraction of Component in the Condenser

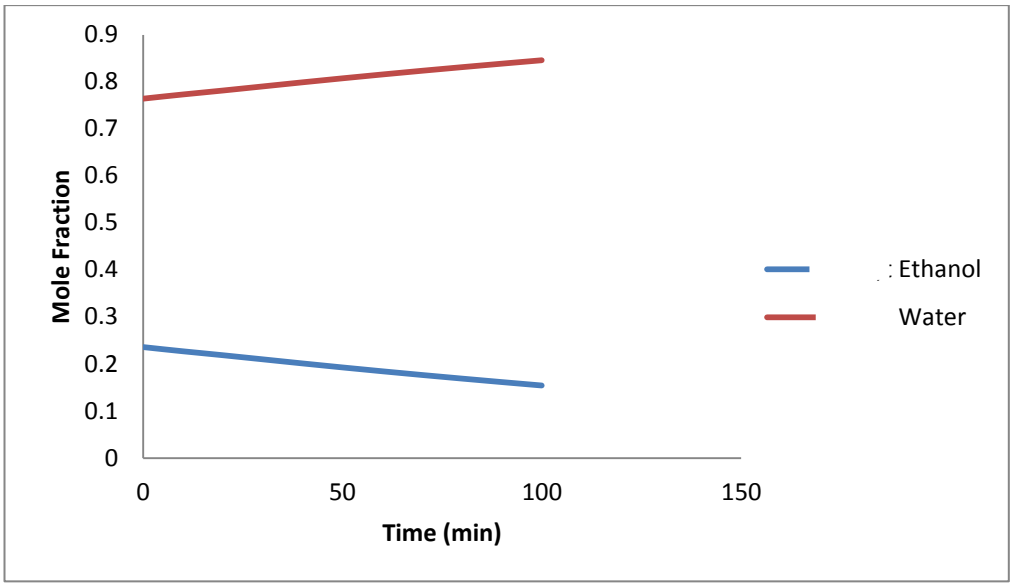

Figure 4.3 Theoretical Dynamic Response of Mole Fraction of Component in the Reboiler

4.3 Comparison Between Simulation of The Model By Using MATLAB® Software and Graphical Method

The system parameters were defined as shown in the Table 4.5 before running simulation in MATLAB®. Using the data in Table 4.5 for the experimental study to simulate the developed model equations with the aid of MATLAB, the dynamics responses obtained for the distillate and bottoms segment compositions at constant reflux as well as composition profiles of the mixture as shown in Figure 4.3 and Figure 4.4.

Table 4.5 Parameter Value for Modelling

Parameter	Input
Vapour boil- up rate, V	$2.777x10^{-3}$ kmol/min
Liquid boil- up rate, L	$2.216x10^{-3}$kmol/min
Condenser holdup, Hc	0.3631 kmol
Reboiler holdup, H_N	0.3631 kmol
Reflux ratio	4

During the simulation, the reflux ratio was set to be constant (R=4). Model equations were solved by an explicit Euler method and it was possible to discuss simulation parameters and their influence on composition and process dynamics. Since the model equations developed in this work for Simple Model-Model Type III composed of both ordinary and partial differential equations, the partial differential equation were converted to ordinary differential equations using the backward difference approach to make the model equations uniform in nature. The resulting ordinary differential equations were then solved using ode15s and ode23 command of MATLAB.

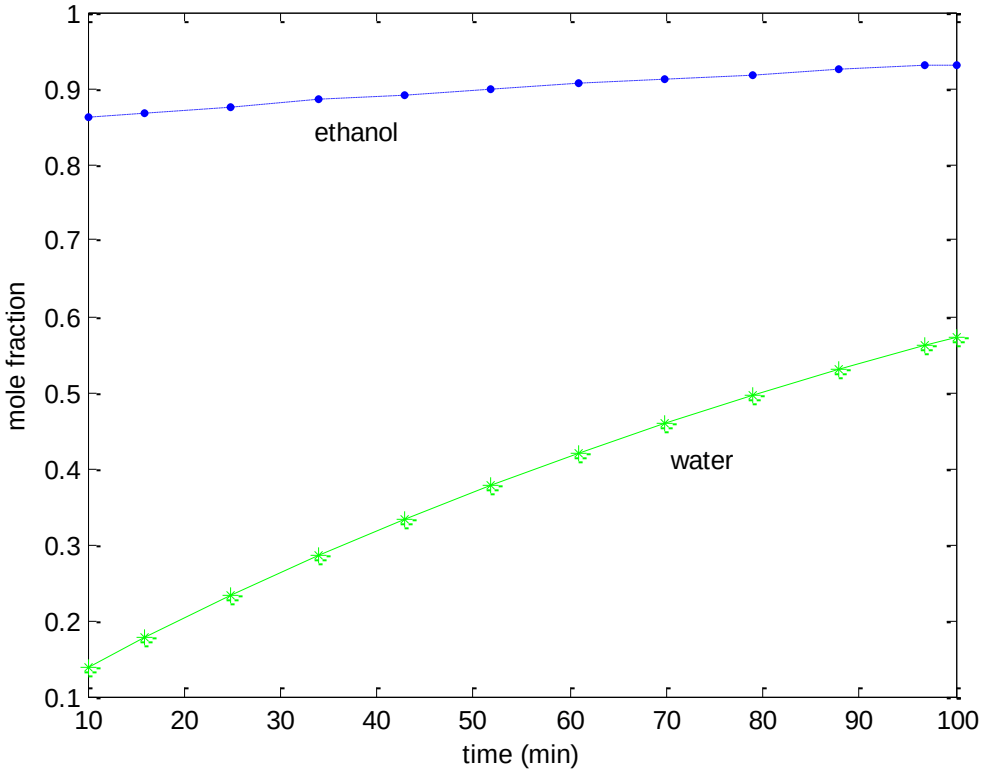

Figure 4.4 Theoretical Dynamic Responses of Mole Fractions of Components in the Condenser by Simulation.

Figure 4.4 shows the dynamics responses of mole fractions of components in the segment of the condenser. As can be seen from Figure 4.4, the dynamics response of the mole fraction of ethanol and water were increase as time increase. At condenser, mole fraction of ethanol was higher than mole fraction of water at time of 10 minutes. It is because boiling point of ethanol is lower than water, thus ethanol vaporised first. But from graphical method and experiment, it showed that mole fraction of ethanol will decreased after 30 minutes. In contrast, by simulation the mole fraction of ethanol continuously increasing without dropped. It recognized that some assumption made in simulation model is error or not valid to the modelling.

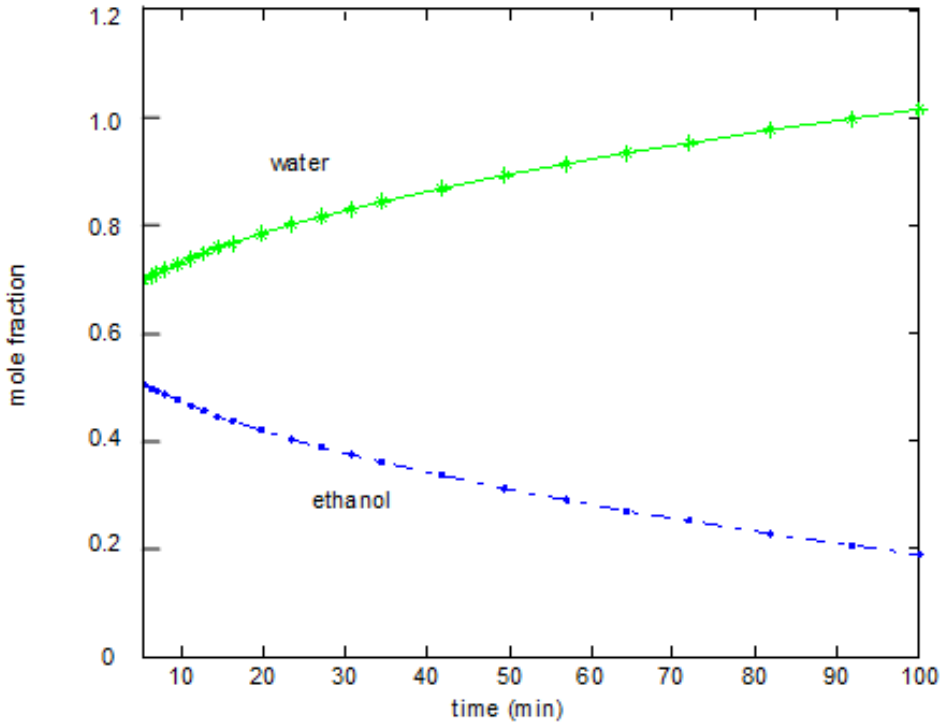

Figure 4.5 Theoretical Dynamic Responses of Mole Fractions of Components in the Reboiler by Simulation.

Considering Figure 4.5, it was discovered that the mole fraction of ethanol was low in the reboiler compared to the water in the condenser. This is because ethanol being more volatile than water. Meanwhile the mole fraction of water was increasing over the time. At bottom, the mole fraction for both was validated from graphical method under difference mole fraction.

4.4 Experimental Results by Others

Experiment results which previously done by others was extracted to obtain the composition of ethanol at the condenser. The experiment was conducted for 120 minutes under atmospheric pressure. Table 4.7 shows the results for experiment with constant reflux of 4. Measurement for volume was not taken due to very low amount of distillate and bottoms hold up is not available.

Table 4.6 Experiment Results of Distillate

Time	Mole Fraction	
(min)	Water	Ethanol
0	0	0
5	0.32434	0.675657
10	0.8528	0.147201
20	0.83392	0.166076
40	0.76873	0.231272
60	0.90976	0.090245
80	0.92858	0.07142
100	0.91371	0.086294
120	0.85349	0.146509

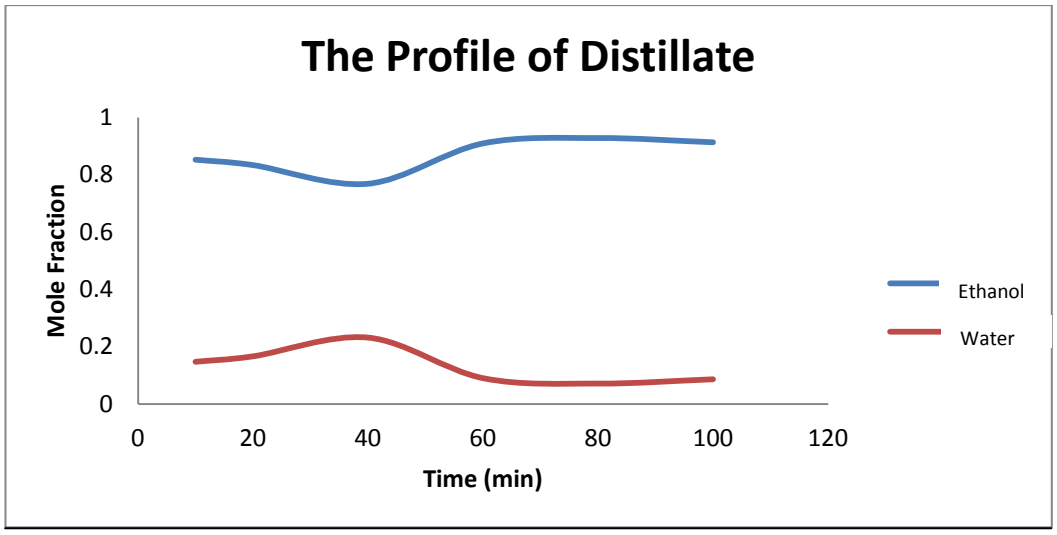

Figure 4.6 Theoretical Dynamic Responses of Mole Fractions of Components in the Condenser by experiment.

4.4.1 Comparison between Results Graphical Method, Simulation by MATLAB and Experiment

The results from graphical method and experiments show a theoretical trend where ethanol purity at distillate is decreasing over time. But it was opposite for MATLAB simulation. This trend is due to unsteady state operation of batch distillation where the composition of more volatile at the bottoms is depleting over time and resulting in varying distillate composition at constant reflux (Kim & Diwekar, 2005).

Comparison of the results shows that the purity of distillate obtained by graphical methods is almost similar to results from experiment. The graphical results, however, deviated from the results from experiment at 20 minutes above. This deviation indicated that the experimental results are flawed, with error of 14.7 % for ethanol and 124.5 % for water composition at distillate. This large value of error is

unacceptable for experimental results to be recognized even with some assumption made in simulation model.

Table 4.7 Composition for Error between Graphical Method with Experiment for Ethanol Distillate Composition

	Composition		Percentage Error (%)
Time	Graphical Method (basis)	Experiment	Experiment
10	0.860751	0.8528	1
20	0.805029623	0.83392	4
40	0.78010242	0.76873	1
60	0.752164099	0.90976	21
80	0.721925082	0.92858	29
100	0.692557599	0.91371	32
		Mean	14.7

Table 4.8 Composition for Error between Graphical Method with Experiment for Water Distillate Composition

	Composition		Percentage Error (%)
Time	Graphical method (basis)	Experiment	Experiment
10	0.139249	0.147201	5
20	0.194970377	0.166076	17
40	0.21989758	0.231272	5
60	0.247835901	0.090245	175
80	0.278074918	0.07142	289
100	0.307442401	0.086294	256
		Mean	124.5

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

Simulation of batch distillation was successfully done using graphical method and MATLAB software. At constant reflux, the distillate and bottoms composition for ethanol drop gradually over time by using graphical method. The declining trend was due to the unsteady state nature of batch distillation, where the lighter component at the bottoms will deplete over time. On the other hand, the bottoms and distillate composition for water increases over time as the lighter component is removed from the reboiler. The trend from simulation showed that the distillate and bottoms composition for ethanol increase and decrease over the time respectively

The result obtained from this study has shown there were quite good agreements between the distillate and bottom compositions estimated by graphical method and experimental study. Their percentage absolute errors for ethanol distillate and ethanol bottom were 14.7% and 124.5% respectively. It was not high that was not set as the criterion for validity of the model equations. Therefore, the experiment results have been found not accurate results in representing the batch distillation. Responding to this error, troubleshooting was carried out on the existing

column and faulty instruments were identified. The reflux splitter was found to be faulty and was not functioning properly, causing incorrect split of reflux and distillate.

Comparison between the distillate compositions obtained from graphical method with MATLAB shows only slight error for ethanol. It has two methods supported each other and show confident in the results obtained. The experiment results and graphical method have the same result. However, for the bottom result it gave same profile behaviour for simulation and graphical method.

Finally, the comparison with experimental results, graphical result and simulation result gave, the analysis data and the revaluation of the hypothesis initially adopted. In this study the trend of distillate and bottoms composition for fermentation product purification using batch distillation had been studied. As using computer software simulation, it able to integrate the model equations for fermentation product purification based on suitable numerical methods and it also gain the understanding due to simulation.

5.2 RECOMMENDATION FOR FUTURE RESEARCH

A future project for this research is controlling the reflux ratio for binary mixture batch distillation to obtain constant distillate composition that can be carried out using graphical method. Other recommendations for further research include:

i) Upgrade column with real-time computer-monitoring and control systems
ii) Quantify the effect of heat loss on the internal reflux ratio stage by stage

iii) Develop a more accurate model to predict the performance of the separation of binary mixture of ethanol-water.

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APPENDENCES

APPENDIX A

Table A1 Result of variable by using EXCEL

Time	Lt	ln(0.3631/Lt)	xt	yav
10	0.357946	0.014296141	0.2267	0.8607512
20	0.352392	0.029934086	0.2184	0.8050296
40	0.341284	0.061963303	0.2009	0.7801024
60	0.330176	0.095052433	0.1842	0.7521641
80	0.319068	0.129274032	0.1686	0.7219251
100	0.30796	0.164708374	0.1539	0.6925576

APPENDIX B Result of Graphical Method

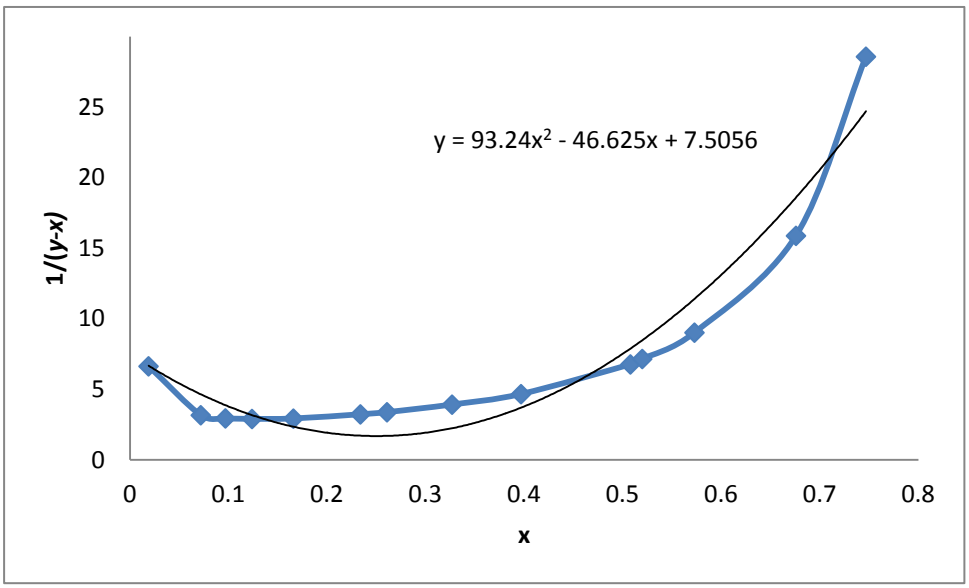

Figure B1 Numerical Integration for Ethanol-Water

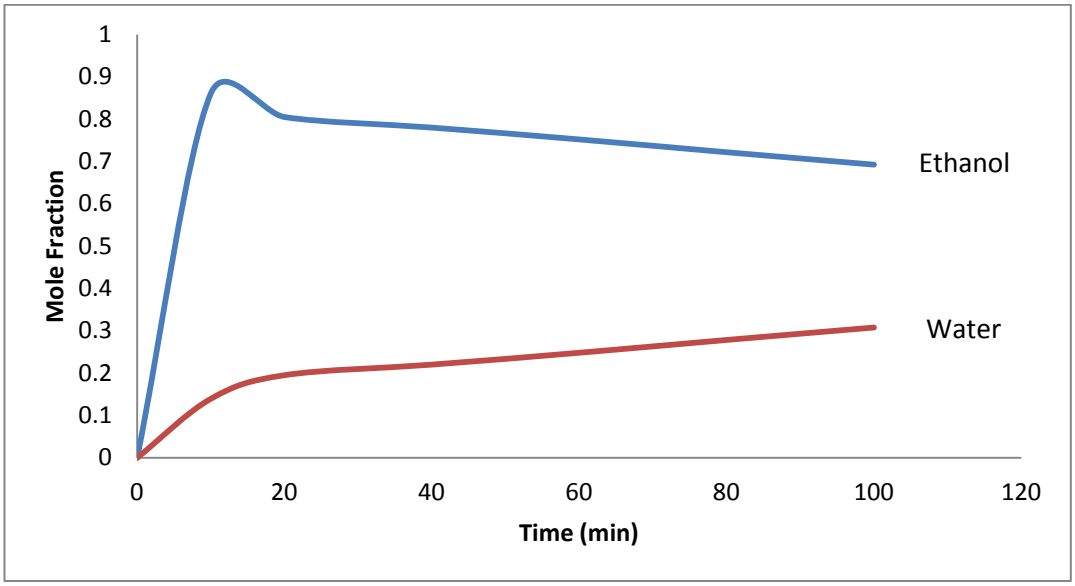

Figure B2 Theoretical Dynamic Response of Mole Fraction of Component in the Condenser

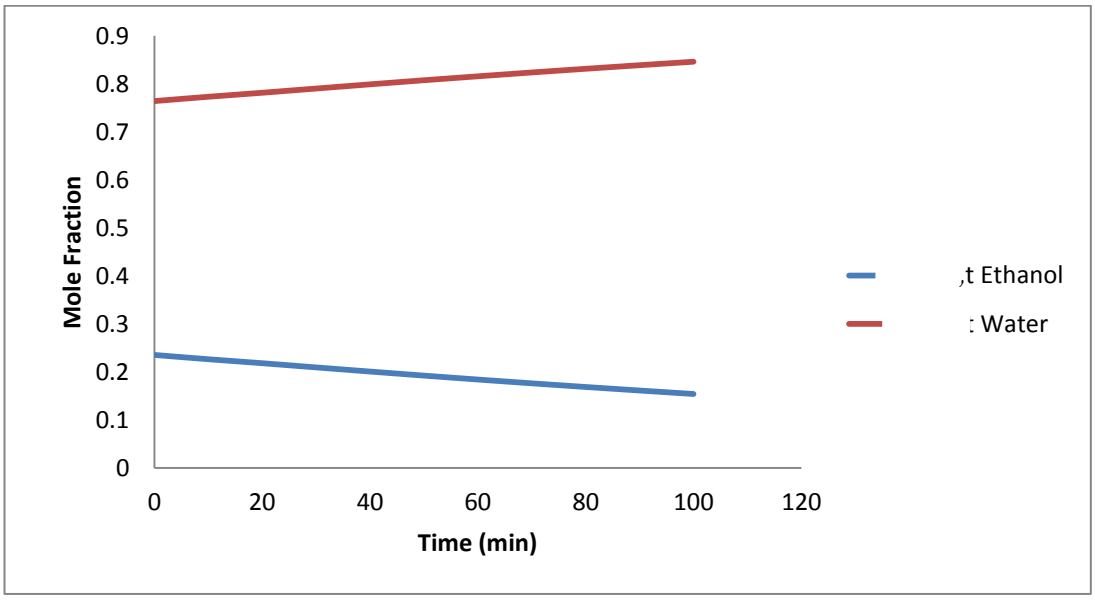

Figure B 3 Theoretical Dynamic Response of Mole Fraction of Component in the Reboiler

APPENDIX C Simulation Report by Using MATLAB

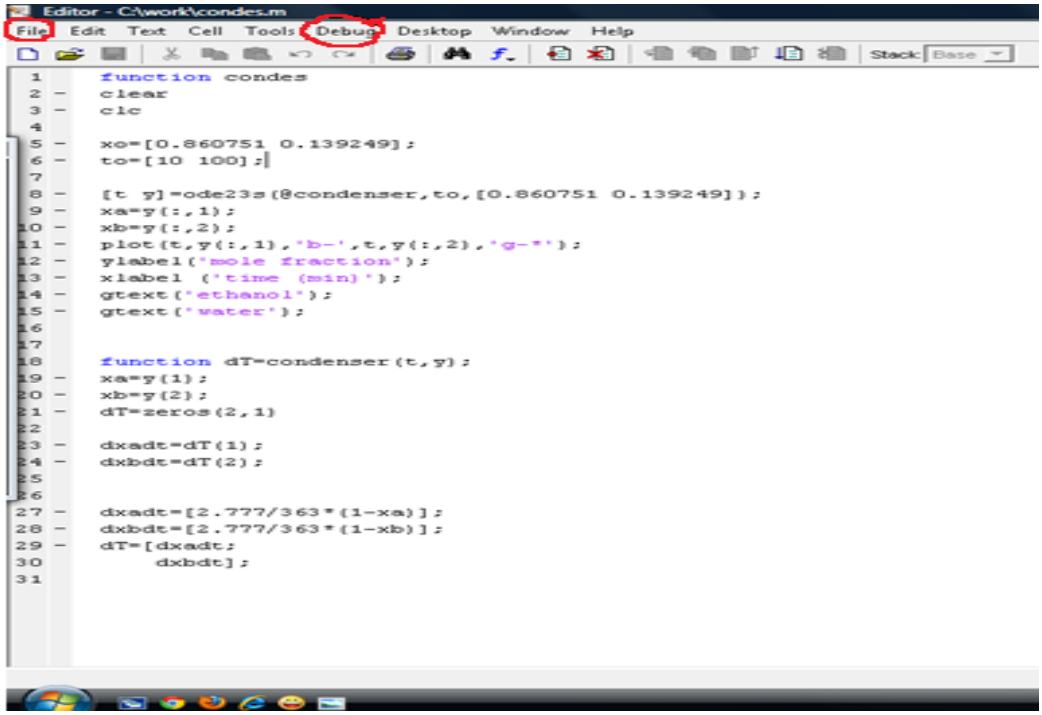

Figure C1 Coding of MATLAB at condenser

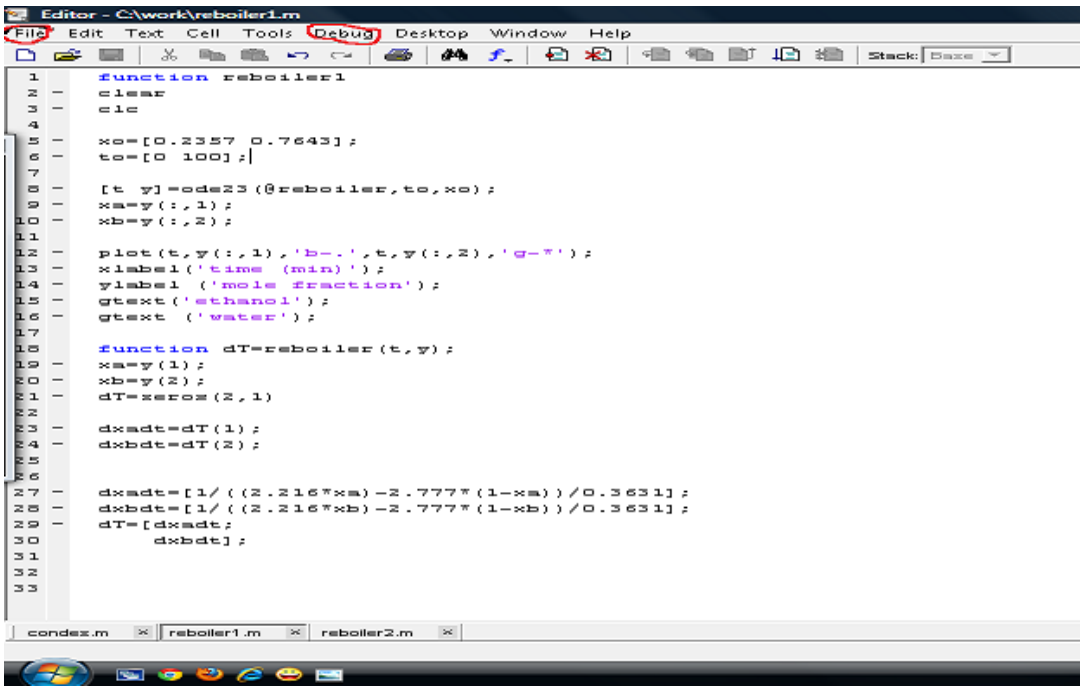

Figure C2 Coding of MATLAB at Reboiler

APPENDIX D Result of Simulation

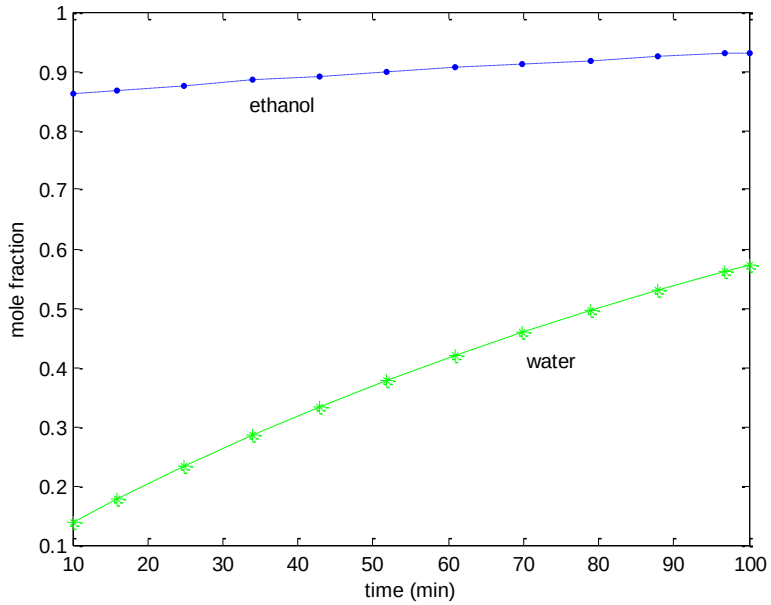

Figure D1 Theoretical Dynamic Responses of Mole Fractions of Components in the Condenser.

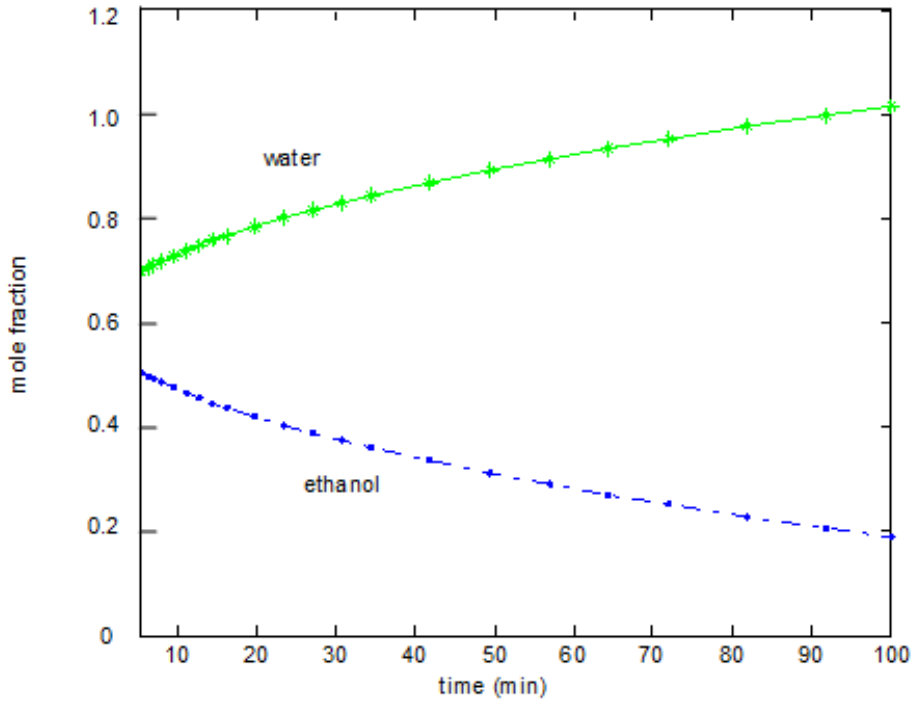

Figure D2 Theoretical Dynamic Responses of Mole Fractions of Components in the Reboiler .