OPTIMAL BATCH DISTILLATION USING PC-BASED

A Thesis
Submitted to the College of Engineering
of Al-Nahrain University in Partial Fulfillment of
the Requirements for the Degree of
Master of Science
in
Chemical Engineering

by

RAND QUSAY KADHIM AL-KHAFAJI
(B.Sc. IN CHEMICAL ENGINEERING 2012)

Shaban 1436
May 2015

Supervisor Certification

I certify that this thesis entitled **"Optimal Batch Distillation Using PC-Based"** was prepared under my supervision at college of Engineering/Al-Nahrian University in partial fulfillment of the requirements for the Degree of **Master of Science in Chemical Engineering.**

Naser Habobi

Supervisor

Assit.Prof. Dr. Naseer A. Al Habobi

3/6/2015

PhD in Chemical Engineering

In view of the available recommendation, I forward this thesis for debate by the Examining Committee.

Naseer Habobi

Assit. Prof. Dr. Naseer A. Al Haboubi

Head, Department of Chemical Engineering

Committee Certification

We the Examining Committee, after reading this thesis **"Optimal Batch Distillation Using PC-Based"** and examining the student **"Rand Qusay Alkafaji"** in its content, find it is adequate as a thesis for the degree of **Master of Science in Chemical Engineering.**

Wadood

Prof. Dr. Wadood Taher Mohammed

Chairman

Qusay

Prof. Dr. Qusay F. Alsalhy

Member

3/6/2015

Sarmad T. Najim

Dr. Sarmad Talib Najim

Member

3/6/ 2015

Naseer Habobi 14/6/2015

Assit. Prof. Dr. Naseer A. Al Habuobi

Supervisor and Member

Approved by the College of Engineering /Al-Nahrain University

Prof. Dr. Jassim Abbood Abbas Aldabbagh

Dean, College of Engineering / Al-Nahrain University

Abstract

Batch distillation is becoming increasingly important as a result of the expansion in the fine chemical and pharmaceutical industries characterized by small amounts of the products with high added value. This thesis aimed at finding optimal way of operating batch distillation column using PC-based.

The operation strategies for batch distillation were studied methanol and water mixtures with a constant heat duty of 288 W At the beginning of the batch operation, total reflux policy is required. The duration of the total reflux period was chosen as follows: when the distillate temperature (i.e., composition) reached the desired value. The initial volume of the mixture was 1L and all the experiment feed mixture (methanol-water) have the same initial composition, 25%mole of methanol and 75%mole of water. The column works at atmospheric pressure with 10 trays. Batch distillation was operated by using LabVIEW program, the major findings were data acquisition device (DAQ) NI-ELVIS II for acquiring temperatures and generate signals for control purposes.

Zieglar-Nichols tuning rules and Routh's stability criterion were applied to determine the parameters of the implemented PID controller. It was found that $k_p = 3.4001\ \tau_I = 11.754, and\ \tau_D = 5.881\ s.$ the simulation results show good agreement with the experimental result

This thesis addresses different aspects of operation and control of batch distillation columns, which are constant reflux ratio and constant overhead composition. It has been recognized that a first order may represent process dynamics of batch distillation. Where the transfer function of the experimental system have gain $k_P = 2.5,$ and time constant $\tau_I = 46.67\ s.$ It was found that using constant overhead strategy can reduce the distillation time of up to 20% in comparison with constant reflux operation mode.

List of Contents

Content **Page**

Chapter One: Introduction

Chapter Two: Literature Review

Chapter Three: Batch distillation monitoring and control

Chapter Four: Experimental work

Chapter Five: Result and discussion

Chapter Six: Conclusion and Recommendation for future work

Nomenclature

Symbol	Definition	Unit
B	Amount remaining in the reboiler	mole
B_t	Amount remaining in the reboiler at time t	mole
D	Total distillate	mole
D_t	Total distillate at time t	mole
F	Total feed	mole
G_c	Transfer function of the controller	-
G_p	Transfer function of the process	-
G_m	Transfer function of the measurement	
G_v	Transfer function of the actuator	
H_{mix}	Enthalpy of ,mixing	kJ
H_C	Molar compartmental holdup	mole
H_j	Molar holdup on the plate j	mole
H_m	Middle vessel molar holdup	mole
H K	Heavy key component	-
I_B	Enthalpy of liquid reboiler	J/ mole
k_p	Process gain	-
k_c	Controller gain	-
k_{cr}	Critical gain	-
L_j	Liquid stream leaving plate j	mole/s
L_{j-1}	Liquid stream entering plate j	mole/s
n	Number of component	-
N	Number of theoretical plates	-
N_{min}	Minimum number of theoretical plates	-
Q_B	Reboiler heat duty	J/s
R	Reflux ratio	-
x_B	Liquid composition of the component 1 in a binary mixture	-
x_{B0}	Initial reboiler composition of the component 1 in a binary mixture	-
x_D	Distillate composition of the component 1 in a binary mixture	-

x_F	Feed composition of the component 1 in a binary mixture	-
x_j	Liquid composition of the component 1 in a binary mixture leaving plate j	-
x_{j-1}	Liquid composition of the component 1 in a binary mixture entering plate j	-
V	Vapor boil up rate	mole/s
V_B	Vapor stream leaving the reboiler	mole/s
V_j	Vapor stream leaving plate j	mole/s
V_{j+1}	Vapor stream entering plate j	mole/s
y_B	Vapor composition of the component 1 in a binary mixture which is in equilibrium in the reboiler	-
y_j	vapor composition of the component 1 in a binary mixture leaving plate j	-
y_{j+1}	vapor composition of the component 1 in a binary mixture entering plate j	-

Greek symbols

α	Relative volatility of component 1 in binary mixture	-
λ	Latent Heat of vaporization	kJ/mole

List of Tables

List of Figures

CHAPTER ONE

INTRODUCTION

1.1 INTRODUCTION

Distillation columns constitute a major part of most chemical engineering plants and remains as the most important separation technique in chemical process industries around the world. Therefore, improved distillation control can have a significant impact on reducing energy consumption, improving product quality and protecting environmental resources. The recent increase in the production of high value-added, low-volume specialty chemicals and biochemical has generated a renewed interest in batch processing technologies. Batch distillation is an important unit operation in the batch processing industry and is widely used [Diwekar, 1995].

In comparison to continuous distillation, batch distillation offers flexible and economically attractive options for equipment reduction, because the same equipment can be used for different products depending on market demand. In batch distillation control research focus on the development of efficient feedback control configurations. Within the control theory framework, the problem can be formulated as a tracking problem where a commonly optimal temperature or composition profile becomes the reference trajectory for a feedback controller [Quintero, 1991].

The flexibility of batch distillation combined with the inherent unsteady nature of the process poses challenging design and operation problems, however, gives rise to challenging control problems that are due to the non-stationary, nonlinear and finite-time duration nature of the underlying dynamics. Because in a batch distillation operation there is no steady-state, there is no normal condition at which traditional input/output linear models can be constructed and controllers can be tuned. Accordingly, satisfactory control responses have not, thus far, been obtained [Nooraii, 1999].

Batch distillation techniques are used extensively in the fine chemicals and pharmaceutical industries to achieve a variety of processing objectives. These include [Edwards, 2001]:-

- Removal of impurities (unwanted) components from reaction mixes.
- Water removal and drying.
- Solvent change between reaction stages on multistage syntheses.
- Concentration prior to crystallization.
- Heat removal control for reactions reflux.
- Solvent recovery

The simplest form of batch distillation involves a single flash separation and is used where a large difference in volatility exists between the components. Such a distillation doesn't need a fractionating column between the batch still, normally a stirred jacketed reactor, and the condenser. Simple batch distillation, also referred to as pot-to-pot, provides one theoretical plate of separation. When the difference in volatility between components is small or when operating over narrow composition ranges, a rectification section is necessary between the still and the condenser. Overhead facilities are required to provide control of reflux ratio and layer separation when handling heterogeneous azeotropes. Batch distillation has been a common technique for separation and purification of small volumes of high-value specialty chemicals in the course of the decades [Lopes, 2010].

In operation, the system is brought to steady state under total reflux. Overheads are continuously withdrawn in accordance with the reflux control strategy. Cuts (fractions) are taken by switching to different accumulators (receivers), following a time, temperature or overhead composition strategy [Block, 1967].

Within a batch process, batch distillation is a ubiquitous way of physical separation of binary and multicomponent mixtures. Many different components and multiple product fractions can be separated in the same batch column, and at a much lower capital cost than would be associated with the relevant train of continuous columns. The streams coming from a batch column may be themselves the desired products; in such a case, they can be directly placed in the market. Alternatively, the distillation may serve as a way of recovering one or more components in order to recycle them to a certain stage of the batch process, or as a method of removing an undesired component from aby product stream in order to comply with the regulations on by product discharge. However, no matter what the objective of the distillation, the purity requirements are normally strict, and a significant economic penalty exists if the specification requirements are not met. The development of accurate methodologies for tight composition control in batch columns is necessary [Edwards, 2013].

1.2 Aim of this work

1. To study the optimal operation strategies for batch distillation column with the NI LabVIEW Control Design and Simulation Module.
2. To analyze the system and its stability using bode diagram
3. Design the required controller (PID) to improve the process response.
4. Using a simulation to explain the dynamic behavior of batch distillation column.
5. Applied the implemented model and compare the experimental result with the simulation result.

1.3 Motivation

- Batch distillation is becoming increasingly important as a result of the expansion in the fine chemical and pharmaceutical industries.
- Batch distillation has the advantage of being able to recover a number of products from a feed charge in a single column.
- Batch distillation offers a maximum of flexibility to answer the random demand for chemical products [Edwards, 2013].

CHAPTER TWO

THEORETICAL ASPECT AND LITERATURE REVIEW

This chapter will provide introduction to batch distillation, with some of basic topics to give a brief description for batch distillation, material balance around batch distillation column, the difficulties in controlling batch distillation and the previous researches in this field.

2.1 Batch Distillation

Distillation is the oldest separation process and the most widely used unit operation in industry. Distillation began as a simple still in laboratory, initially filled with a feed mixture which evaporates and leaves the liquid. The vapor, which is richer in the more volatile component, is collected in the condenser at the top. The simple distillation still is an example of a batch operation. The concept of reflux and the use of accessories such as packing or plates to increase the mass transfer convert this simple still into a batch distillation column. [Hansen and Jorgensen, 1986]

Batch distillation has been a common technique for separation and purification of small volumes of high-value specialty chemicals in the course of the decades. The recent increase in the production of high-value-added, low-volume specialty chemicals and biochemical has generated a renewable interest in batch distillation [Lopes, 2010].

The flexibility offered by batch distillation gives it an advantage over continuous distillation. At the same time, this flexibility and the inherent unsteady state nature of batch distillation poses challenging design and operational problems [Diwekar, 1995].

Many different components and multiple product fractions can be separated in the same batch column, and at a much lower capital cost than would be associated with the relevant train of continuous columns. The streams coming from a batch column may be themselves the desired products; in such a case, they can be directly placed in the market. Alternatively, the distillation may serve as a way of recovering one or more components in order to recycle them to a certain stage of the batch process, or as a method of removing an undesired component from aby product stream in order to comply with the regulations on by product discharge [Edwards, 2013].

2.1.1 Important of batch distillation

Batch distillation columns are flexible separation processes that are becoming widely used. The main reason is that production amounts are usually small with minimum raw material inventories; this often results in an economic incentive [Barolo and Berto, 1998]. In comparison to continuous distillation, batch distillation offers flexible and economically attractive options for equipment reduction, because the same equipment can be used for different products depending on market demand. Recent trends in batch distillation control research focus on the development of efficient feedback control configurations. Within the control theory framework, the problem can be formulated as a tracking problem where a commonly optimal temperature or composition profile becomes the reference trajectory for a feedback controller [Quintero, 1991].

2.2 Material balance around batch distillation column

Figure (2.1) represents a schematic of a batch distillation column, where the total batch is fed to the re-boiler at the bottom of the column. The column consists of perforated plates that permit the upward flow of vapor. The liquid from the top is refluxed back across each plate, and the vapor rising from the top plate passes to a condenser. Under the simplification of negligible holdup, the overall material balance and the material balance for the more volatile component around the complete column, shown in Figure 2.1, result in the following equations [Coward, 1967]:-

$$x_D\, dB = d\,(B\, x_B) = B\, d\, x_B + x_B\, dB \tag{2.1}$$

$$\int_F^B \frac{dB}{B} = \int_{xF}^{xB} \frac{dxB}{x_D - x_B} \tag{2.2}$$

$$\ln\left(\frac{B}{F}\right) = \int_{x_F}^{x_B} \frac{d\ x_B}{x_D - x_B} \tag{2.3}$$

If the equilibrium relationship is a straight line (which is the case in ideal dilute mixtures) then the Rayleigh equation, Equation (2.3) reduces to the following form.

$$\frac{x_D - x_B}{x_{D0} - x_F} = \left(\frac{B}{F}\right)^{m-1} \tag{2.4}$$

Where m is the slope of the equilibrium line and x_{D0} is the initial composition of the more volatile component in the distillate.

Similarly, for a system following the ideal behavior given by Raoult's law, the equilibrium relationship between the vapor composition y (x_D) and liquid composition x (x_B) of the more volatile component in a binary mixture can be approximated using the concept of constant relative volatility (α), and is given by:

$$y = \frac{\alpha x}{(\alpha-1)x+1} \tag{2.5}$$

Substitution of the above equation in equation (2.3) results in:

$$\ln\left(\frac{B}{F}\right) = \frac{1}{\alpha-1} \ln\left[\frac{x_B\ (1-x_F)}{x_F\ (1-\ x_B)}\right] + \ln\left[\frac{1-x_F}{1-x_B}\right] \tag{2.6}$$

There are four stream involved: liquid stream L_{j-1} and vapor stream V_{j+1} are entering and liquid stream $L_j$and vapor stream $V_j$are leaving the j-th plate. if the liquid composition and the vapor composition of the more volatile component are denoted by $x\ and\ y$, respectively, and the liquid and the vapor enthalpies by $I\ and\ J$ respectively, the material and the energy balance for the control volume can be written as follows: [Diwekar, 1995]

Material balance:

$$L_{j-1} + V_{j+1} = L_j + V_j \tag{2.7}$$

$$L_{j-1}x_{j-1} + V_{j+1}y_{j+1} = L_j\ x_j + V_j y_j \tag{2.8}$$

Energy balance:

$$L_{j-1}I_{j-1} + V_{j+1}J_{j+1} = L_j\ I_j + V_jJ_j + losess + H_{mix} \tag{2.9}$$

Where H_{mix} is the heat of mixing,

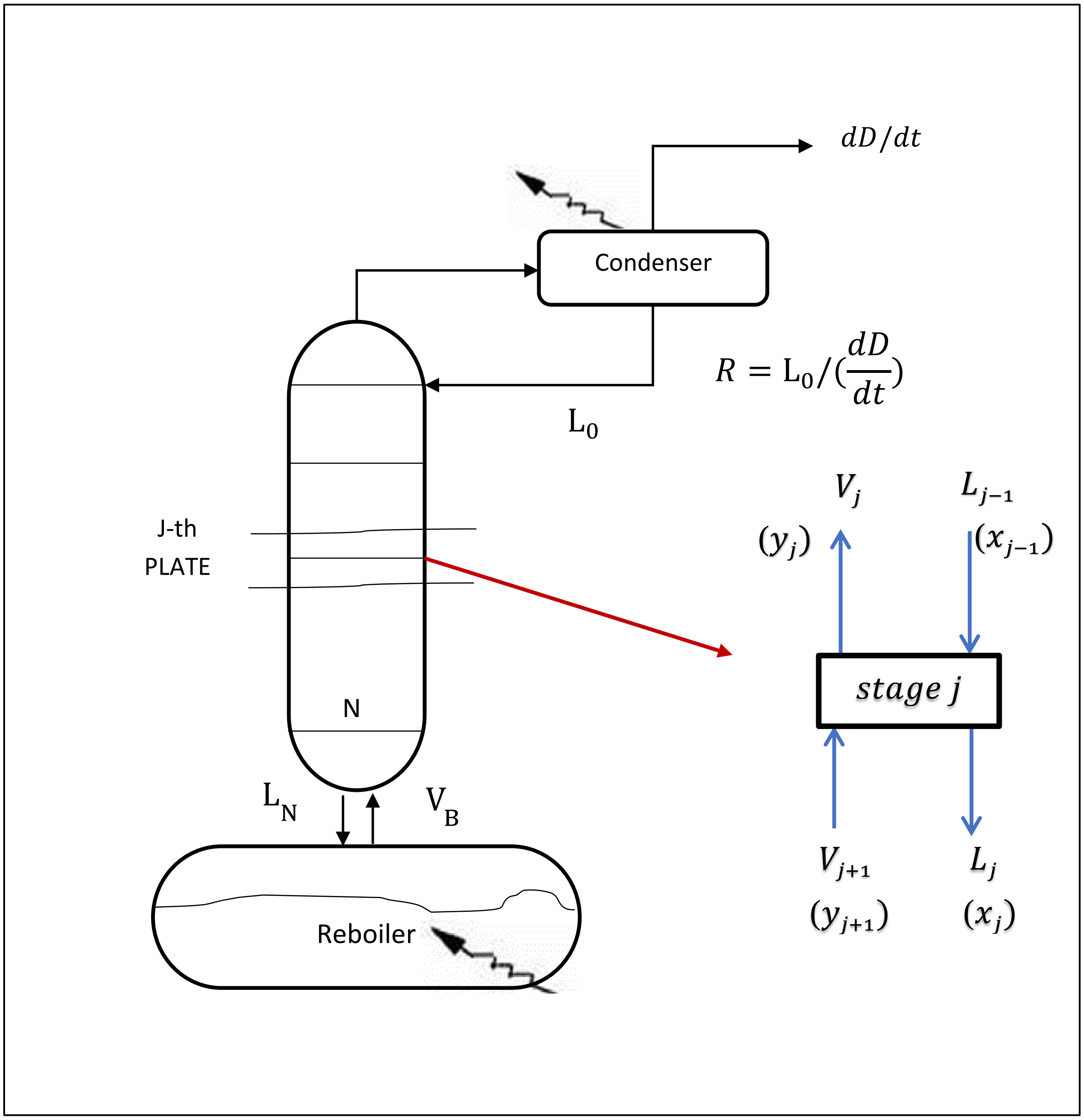


Figure 2.1 Schematic of a batch distillation column and plate balance.

If the molar latent heat of vaporization is assumed to be constant $(J_{j+1} = J_j)$ and the system is assumed to be ideal $H_{mix} = 0, losses = 0$), then the energy balance equation (2.9) reduces to $L_{j-1} = L_j = L$ and the $V_{j+1} = V_j = V,$ which is the equimolar overflow assumption. The equimolar overflow assumption allows one to extend the popular McCabe and Thiele graphical method (McCabe and Thiele, 1925) for continuous distillation to batch as shown below.

In the McCabe and Thiele method of the material balance is considered around the condenser and the j-th plate. This leads to the following equations. The overall material balance around the top section and the j-th plate:

$$V = L + \frac{dD}{dt} = (dD/dt)(R + 1) \tag{2.10}$$

Where L represents the liquid refluxed from the condenser and R represents the reflux ratio defined as $R = \frac{L}{dD/dt}$ and dD/dt is the distillation rate.

The material balance for more volatile component considering the envelope around the top section of the column and the plate j in Figure (2.1) is:

$$y_j = \frac{L}{V} x_{j-1} + \frac{dD/dt}{V} x_D \tag{2.11}$$

By substituting for V from equation (2.10), equation (2.11) can be expressed in terms of the reflux ratio R, as follows,

$$y_j = \frac{R}{R+1} x_{j-1} + \frac{1}{R+1} x_D \tag{2.12}$$

McCabe and Thiele proposed that since the above equation represents a straight line connecting $y_j \ and \ x_{j-1}$, it can be drawn on the same diagram as that of the equilibrium curve, shown in Figure (2.2). Recall that the equilibrium curve represented the relation between $y_j \ and \ x_j$, which in general may be expressed as:

$$y_j = f(x_j) \tag{2.13}$$

Thus the line equation (2.12) will pass through multiple points, in Figure 2.2

In equation (2.12), if $x_{j-1} = x_D$ (the equation for the top plate of the column) then:

$$y_j = \frac{R}{R+1} x_D + \frac{1}{R+1} x_D \tag{2.14}$$

Therefore the equation (2.12) represents line through point $y_j = x_{j+1} = x_d$ with slop $\frac{R}{R+1}$.

In the case of batch distillation, the still composition x_B dose not remains constant as observed in continuous distillation. This necessitates the use of recursive scheme several times. If the scheme is used, while keeping the reflux ratio constant throughout the operation just like a normal continues distillation column, the composition of the distillate keep changing. On the other hand, the composition of an important key component distillate composition can be maintained as constant by changing the reflux [Mayur, 1970]. Therefore the two basic methods of operating batch distillation column are:

-Constant reflux variable product composition

-Variable reflux constant product composition

These are described as following sections.

2.2.1 Constant reflux

Smoker and Rose (1940) presented an analysis of constant reflux operation of batch distillation for the first time in the context of binary system. They used the Rayleigh equation in conjunction with the McCabe-Thiele method to capture the dynamics of the batch distillation column. In their procedure, the right hand side of Rayleigh equation (2.1) is integrated graphically by plotting $\frac{1}{x_D - x_B}$ versus x_B and the area under the curve between the feed compositions x_F and the still composition x_B gives the value of the integral.

To establish the relation between the distillation composition x_D and the still composition x_B. Several values of x_D, are selected, and operating lines with the same slope $\frac{R}{R+1}$ are drawn through the intersection of these x_D values and the diagonal. Once these lines are drawn, steps are drawn between the operating line and the equilibrium curve to get the bottom composition as shown in figure (2.2).

From the overall material balance it can be seen that the rate of decrease of the still amount, dB/dt is the same as the distillate rate dD/dt, i.e.

$\frac{dB}{dt} = -dD/dt$ Hence, from equation (2.10) the rate of change of B is given by:

$$\frac{dB}{dt} = -\frac{V}{R+1} \tag{2.15}$$

By integrating the above equation, equations below are obtained:

$$\int_0^t dt = \int_B^F \frac{R+1}{V} dB \tag{2.16}$$

$$t = \frac{R+1}{V}(F - B) = \frac{R+1}{V} D \tag{2.17}$$

Similarly one can also obtain estimates of energy requirements from this preliminary analysis. The heat supplied to re-boiler is essentially used for providing the reflux for column. Therefore, the total heat supplied to the re-boiler can be calculated using:

$$Q_R = \int_0^D \lambda R \, dD = \lambda R D \tag{2.18}$$

Where λ represented the average latent heat of vaporization of the mixture.

In this case, the reflux ratio (or the reflux rate) is kept constant throughout the distillation, which results in a continuously changing distillate composition. This is inherently an open-loop strategy. Therefore, unless a proper feedback from the plant is considered, the average product composition is known only at the end of the batch. Therefore, if an off specification product were obtained, the batch would have to be blended or rerun, with a significant economic loss. [Barolo, 1998]

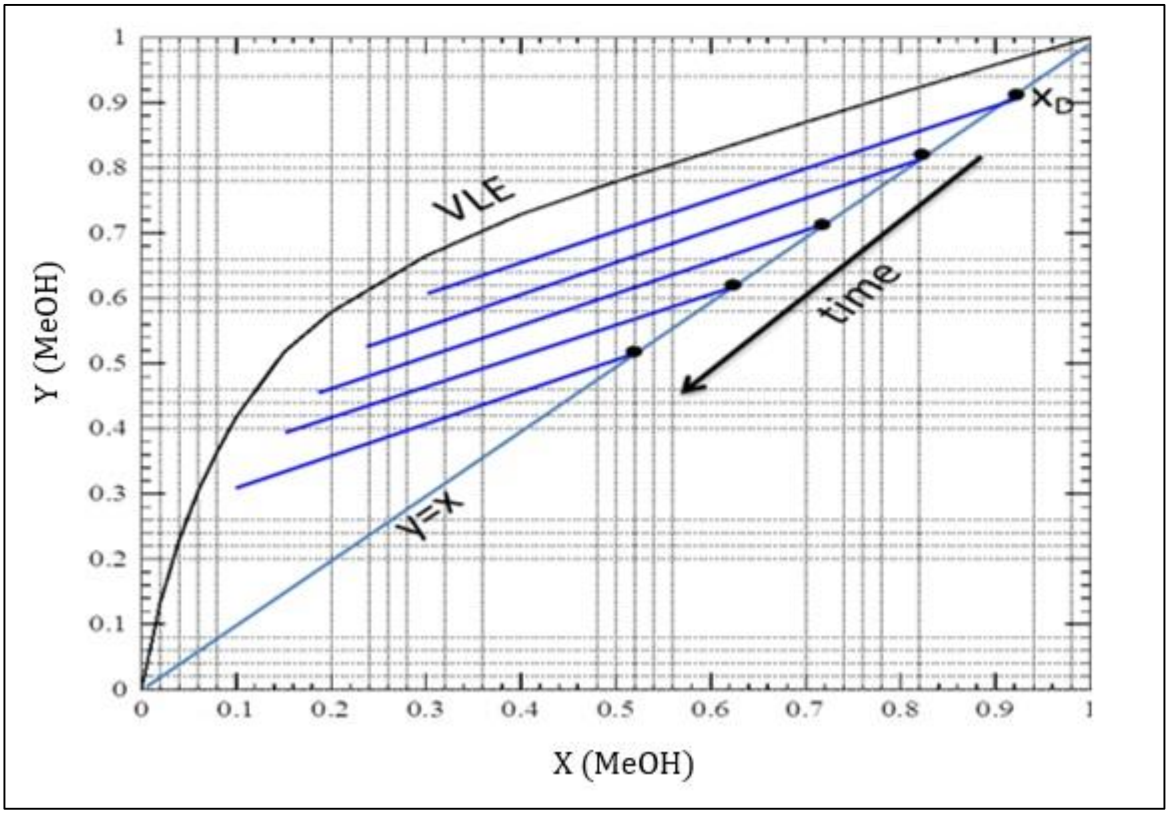


Figure 2.2 Vapor liquid equilibrium for the constant reflux mode.

2.2.2 Variable reflux

In the variable reflux mode of operation, in order to maintain the composition of a key component at constant, the reflux ratio is changed continuously. Bogart (1937) presented an analysis of variable reflux conditions for a binary system. The steps involved in the calculation procedure are similar to those in the case of constant reflux mode; however, in the variable reflux case the reflux ratio is varied instead of the distillate composition at each step. Moreover, the Rayleigh equation, though valid for variable reflux condition, takes a simplified form as shown below [Coward, 1967].

Rayleigh equation:

$$\ln\left(\frac{B}{F}\right) = \int_{x_F}^{x_B} \frac{dx_B}{x_D - x_B} \tag{2.19}$$

Since the distillate composition remains constant throughout the operation, the Rayleigh equation reduces to:

$$\frac{B}{F} = \frac{x_D - x_F}{x_D - x_B} \tag{2.20}$$

The second step is to establish the relation between R and x_B. Several values of R are selected, and operating lines are drawn through the fixed point $(x_{D,}\, x_D)$ with the slope equal to $\frac{R}{R+1}$, and steps are drawn between the operating line and the equilibrium curve to get the bottom composition. The stopping criterion for the variable reflux case can be still composition at the end of the fraction, or the total amount of product collected in the condenser or re-boiler. Similar to the case with constant reflux, the batch time could be a stopping criterion and can be introduced in the calculation procedure as follows.

Integrating the overall material balance equation (2.15) results

$$\int_0^t dt = \int_B^F \frac{R+1}{V}\, dB \tag{2.21}$$

Obtaining the differential of $B$with respect to x_B from equation (2.20) and substituting the results into the above equation, we get

$$t = \int_{x_B}^{x_F} \frac{R+1}{V} \frac{F(x_D - x_F)}{(x_D - x_B)^2}\, dx_B \tag{2.22}$$

Similarly one can also obtain the estimates of heat supplied to the re-boiler Q_R from the amount of material refluxed for the given batch, as shown below.

$$Q_R = \int_0^D \lambda R dD \tag{2.23}$$

The graphical integration technique was used earlier in the case of the constant reflux condition to obtain the left hand side of the Rayleigh equation and not calculate the batch time or heat requirement. Since the reflux is variable, one has to resort to graphical integration to calculate the batch time and the heat requirements. The Rayleigh equation is straightforward in the case of variable reflux and, therefore, can be integrated without resorting to the graphical procedure.

Constant-distillate-composition operation is more difficult. In fact, to keep the distillate composition to the desired value throughout the whole batch, one needs to continuously adjust (increase) the reflux rate. On the other hand, the product composition can be controlled accurately during batch production, i.e., this mode is inherently a feedback operation. [Barolo, 1998]

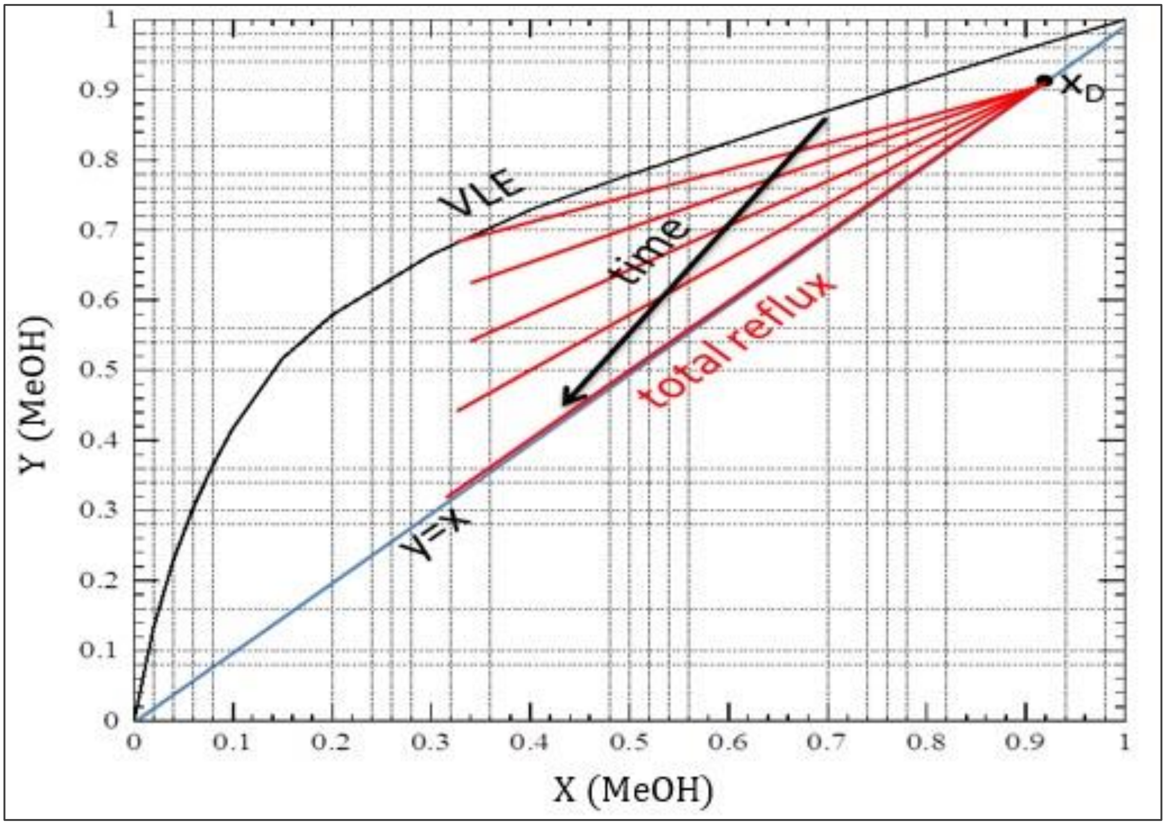


Figure 2.3 Vapor liquid equilibrium for the constant composition mode.

2.3 Control of batch distillation

Optimizing the operation of a batch distillation process is known as an optimal control problem, which has received considerable attention in the literature. The control variable frequently used in the optimization process of batch distillation is the reflux ratio and it is convenient to use an objective function directly related to the control variable. The flexibility offered by batch distillation, gives rise to challenging control problems that are due to the non-stationary, nonlinear and finite-time duration nature of the underlying dynamics. Because in a batch distillation operation there is no steady-state, there is no normal condition at which traditional input/output linear models can be constructed and controllers can be tuned. Accordingly, satisfactory control responses have not, thus far, been obtained [Quintero, 1999].

In general, the most common problems for optimal control of the batch distillation process are:

- **Maximum distillate problem**: To maximize the amount of distillate for a fixed concentration of the most volatile key component for a known operating time [Converse. and Gross, 1963; Diwekar et al., 1987; Farhat et al. 1990; Diwekar, 1992; Logsdon and Biegler, 1993; Zavala-Loría et al., 2006; Zavala-Loría and Coronado-Velásco, 2008].

-**Minimum time problem**: To minimize the operating time necessary to produce a certain amount of distillate that meets a specified concentration of the most volatile key component [Coward, 1967; Robinson, 1970; Mayur and Jackson, 1971; Hansen and Jorgensen, 1986; Mujtaba and Macchieto, 1988; Diwekar, 1992].

- **Problem of maximum profit**:To maximize profit function for a specified concentration of product (Kerkhof and Vissers, 1978; Logsdon at al., 1990; Diwekar, 1992).

- **Problem of minimum energy**: Energy requirements are minimized to produce a certain amount of distillate to meet a specified concentration of the most volatile key component (Furlonge et al., 1999; Mukherjee et al., 2001).

2.4 Previous Work

Batch distillation is a well-known unit operation that is widely used in the fine chemistry, pharmaceutical, biochemical, and food industries to process small amounts of materials with high added value. The success of batch distillation as a method of separation is undoubtedly due to its operational flexibility. A single batch column can separate a multicomponent mixture into several products within a single operation. Batch columns can be operated in three different ways: at constant-reflux ratio, at constant distillate composition, and at total reflux. A combination of these three basic modes can be used to optimize the performance of the separation (Leegwater,1992).

The use of such inferential composition estimators (or software sensors) has long been suggested to assist the monitoring and control of continuous distillation columns. Several applications have been reported in the literature, for both simulated and experimental columns (Joseph & Brosilow, 1978, Yu & Luyben, 1988; Lang & Gilles, 1990; Mejdell & Skogestad, 1991; Baratti, Bertucco, Da Rold, & Morbidelli, 1995; Chien & Ogunnaike, 1997; Kano, Miyazaki, Hasebe, & Hashimoto, 2000). However, theissue of composition estimation in batch distillation columns has received very little attention. Quintero-Marmol, Luyben, and Georgakis (1991) and, Quint!ero-Marmol and Luyben (1992) compared the performances of a steady-state composition estimator, a quasi-dynamic estimator (QDE), and an extended.

Luenberger observer (ELO) for a ternary batch column. They found that the ELO provided the best performance. However, they noted that the observer was quite sensitive to the accuracy of assumed vapor–liquid equilibria and to the assumed initial compositions; moreover, the estimator’s performance rapidly degraded when the tray temperature measurements (i.e., the observer’s inputs) were affected by noise. Similar issues were noted by Barolo and Berto (1998), who used the composition estimates generated by an ELO within a nonlinear strategy for composition control in a conventional batch rectifier. They also observed that the estimator accuracy tended to degrade if the tray hydraulics was taken into account and the number of trays was large. Han and Park (2001) used Luyben’s QDE to estimate the distillate composition, and to control the estimated composition profile of a batch rectifier.

Over the past decades, the optimal design and operation of batch distillation have received significant attentions (Logsdon, Diwekar and Biegler, 1990; Mujtaba and Macchietto, 1996; Sharif, Shah and Pantelides, 1998; Low and Sørensen, 2003). The previous works have provided some general insights on the comparative performances of various batch distillation systems and indicated that the interdependent design and operating control variables should be optimized simultaneously. Furthermore, different configuration of batch distillation column was also taken into account as an optimization parameter with the method of a disjunctive model consisting a given set of equipment modules and the corresponding logical relationships provided by the design context (Oldenburg, Marquardt and Leineweber, 2004; Low and Sørensen, 2005). With the development of manufacture and control in chemical industry, more efficient and energy saving techniques are applied in batch distillation process.

Predictive and adaptive control schemes have been proposed to confront the time-varying nature of the batch distillation process. [Li and Wozny 2001], proposed that a predefined optimal policies can be tracked via an adaptive control strategy, to realize the optimum of multiple-fraction batch distillation. To maximize the thermodynamic efficiency of the process in a fixed time to produce a quantity of distillate complying with a specified concentration of the most volatile key component [Zavala-Loría, 2004; Zavala-Loría and Coronado-Velásco, 2008].

As a result, the number of possible batch distillation column configurations and complex operating strategies proposed in the literature continue to increase, such as the side withdrawals, thermally linked streams or other complex designs. The state space based superstructure has been successfully used for the synthesis of continuous distillation process to reduce both the utility and capital costs (Zou et al., 2012), take the unique characteristics of batch distillation process (dynamic behavior and tray-vessel configuration) into account to develop the superstructure. To be specific, the superstructure of batch distillation system is viewed as a system of two interconnected parts. So there is an urgent need to propose a completed superstructure of batch distillation system that encompasses all possible configurations and operating strategies to reduce the energy consumption and capital investment, and eventually achieve the profit maximization. The incompletion of the superstructure may preclude a series of design alternatives, where the optimal solution may actually lies.

Due to high energy costs, the study of energy consumption is of great interest for industry the analysis of thermodynamic efficiency in a batch distillation column has been presented by [Kim and Diwekar 2000, and Zavala et al. 2007]. Most study on the optimal operation of batch distillation are theoretical. Only few experimental works have been carried out to test the practical feasibility of constant composition.

CHAPTER THREE

BATCH DISTILLATION MONITORING AND CONTROL

3.1 PC-based control

Batch distillation is difficult to set up the correct conditions for the fractionation column to operate at a high theoretical plate efficiency.

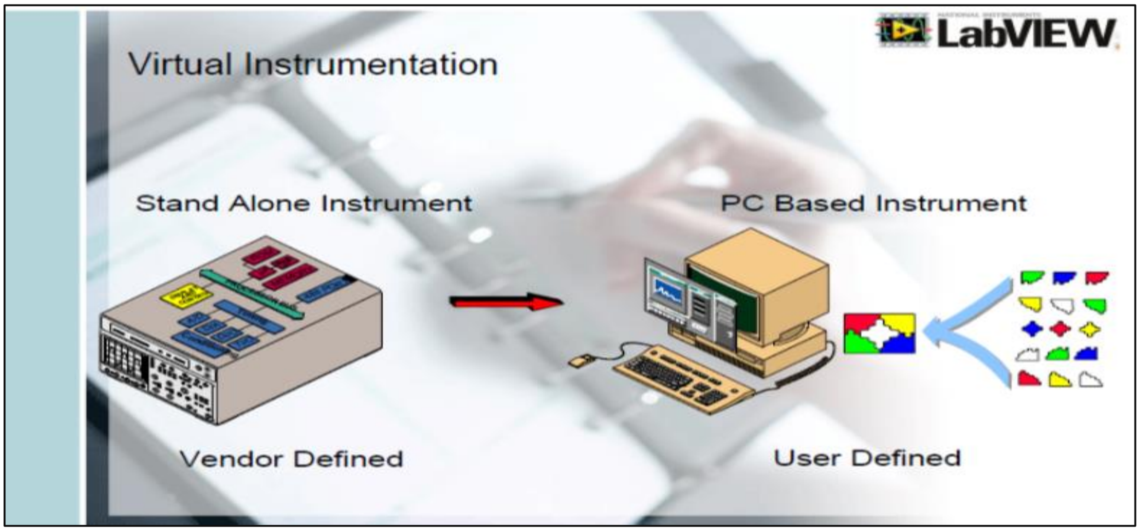


Figure 3.1 PC-Based instrument.

Three problems arise [Chipperfield, 1985]

1) It is difficult to control the boil-up rate so that the fractionating column does not flood, and so that the reflux return rate is satisfactory.

2) It is difficult to control the temperature of the heating jacket surrounding the fractionation column so that local boiling or local condensation does not occur, with catastrophic loss of fractionation efficiency

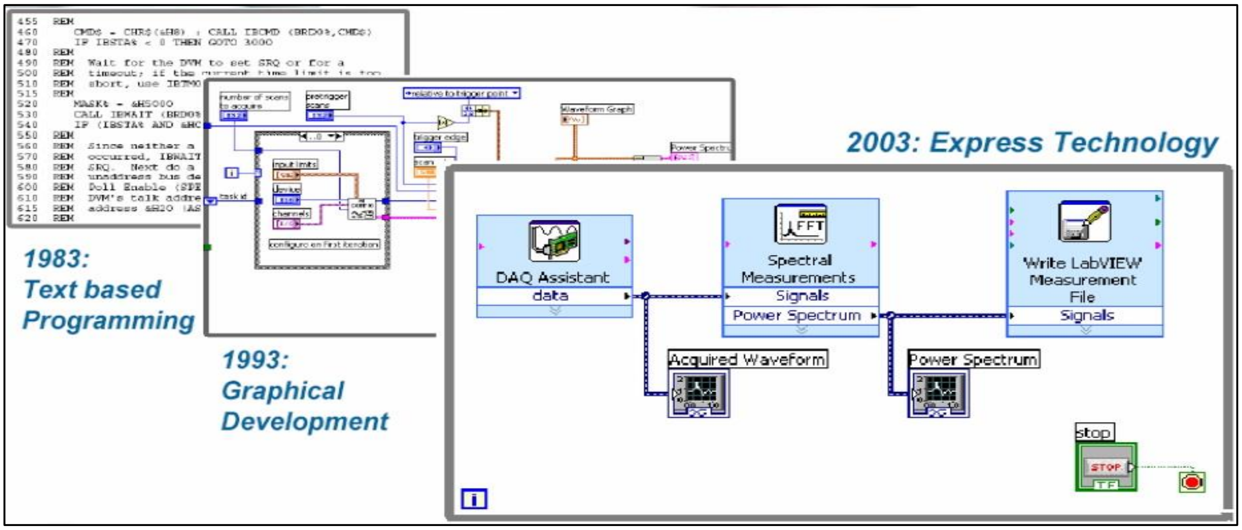
3) Distillation is a slow process and requires continuous attention.

All of these problems can be overcome if the column is monitored and controlled by PC-Based because is used to adjust the boiling rate, the temperatures of the column, and the reflux/take off ratio for optimum separations over a wide range of temperatures. As well as acting as a controller, the computer also monitors the distillation process and displays a schematic diagram of the distillation apparatus showing the temperatures at the still pot, top plate and the condenser.

3.2 Virtual Instruments versus Traditional Instruments

Traditional instruments: Vendor defined, Function-specific; stand-alone with limited connectivity. Hardware is the key, Expensive, Closed, fixed functionality, slow turn on technology (5–10 year life Minimal economics of scale).

Virtual Instruments: User-defined, Application-oriented system with connectivity to networks, peripherals, and applications. Software is the key, low cost, reusable, Open, flexible functionality leveraging off familiar computer technology (Maximum economics of scale). Software minimizes development and maintenance costs (figure3.2).



```
455   REM
460      CMD$ = CHR$(&H8) : CALL IBCMD (BRD0%,CMD$)
470      IF IBSTA% < 0 THEN GOTO 3000
480   REM
490   REM  Wait for the DVM to set SRQ or for a
500   REM  timeout; if the current time limit is too
510   REM  short, use IBTMO
515   REM
520      MASK% = &H5000
530      CALL IBWAIT (BRD0%
540      IF (IBSTA% AND &HC
550   REM
560   REM  Since neither a
570   REM  occurred, IBWAIT
580   REM  SRQ.  Next do a
590   REM  unaddress bus de
600   REM  Poll Enable (SPE
610   REM  DVM's talk addre
615   REM  address &H20 (AS
620   REM
```

Figure 3.2 Programming Evolution

3.3 Batch distillation column online monitoring and control by using LabVIEW

The observation and control of the variables is carried out using virtual instruments while the connection between the virtual instruments and the batch distillation model is solved using the Simulation User Interface. This way has a flexible and versatile tool, being an efficient, economic and simple way to monitoring and control the distillation column programmed in LabVIEW. Details of the LabVIEW front panel designed for this application are depicted in next Figures.

LabVIEW is flexible graphical programming system for universally usage and provides an easy intuitive handling for nearly every kind of coding language. Especially the possibility to switch between the graphical coding and user interface allows it to build up programs in a very time efficient on-site, so it is particular suitable for engineers and scientists. LabVIEW gives thousands of successful users a faster way to program instrumentation, data acquisition, and control systems. By using LabVIEW to prototype, design, test, and implement instrument systems, so can reduce the system development time and increase productivity by a factor of 4 to 10 [Bitter, 2007].

The auto-control of the system has been built from data acquisition device known as NI – ELVIS II which was very crucial since it played a big role to accomplished objectives of this research. The DAQ has ability to generate signal to trigger device for open/closed action of magnetic solenoid for reflux flow control. The DAQ also has ability to acquire analogue signal from external via sensors which are connected to it, the temperature sensors (thermocouple type K) inserted in the batch distillation column responsible for sensing the temperature from the column through the NI DAQ to the lab server by using DAQ’s analogue inputs channels so that output voltage of the product will with drawn at the top of the column. During the column operations, the distillate flow rate profile, the temperature profiles (concentration profiles) along the column and the power for heating were measured online all this done by LabVIEW program.

3.4 Description of the LabVIEW based monitoring system

Applications created with LabVIEW are referred to as virtual instruments (VIs) created as block diagrams. Input and output interfacing with the VI is performed in another window called the front panel. The graphical icon based source code and interfacing creates very user-friendly applications and eliminates typing in lengthy character-based code. Besides, LabVIEW enables to interface directly to instruments, sensors and actuators. This visual computing environment has been applied to a wide variety of control problems. This thesis has utilized this platform to provide a powerful toolset for process identification and control of nonlinear systems. This tool has been applied to nonlinear lab-scale batch distillation column, with up to several control loops.

Also LabVIEW environment has been employed as a graphical user interface for monitoring and controlled batch distillation column, by visualizing both the closed loop performance and the user selected control conditions. The proposed control scheme offers high speed of response for changes in set points and null stationary error for dual composition control and shows robustness in presence of externally imposed disturbance**.**

3.5 Strategies to operate the batch distillation using Pc-Based (LabVIEW)

Batch distillation column coupled to PC was used to evaluate the performance of the identification and control software developed in LabVIEW. The column have selected 10 trays column with heated electrically boiler and water cooling condenser, for separating a mixture of methanol and water.

The system is equipped with temperature sensors for plant state sensing, together with plant actuators for flow and temperature control, turning on the heating, bringing the column to equilibrium under total reflux conditions (start-up procedure). At the end of the startup period, composition and temperature profiles are established. These initial profiles basically depend on the geometry of the column and the composition of the mixture charged into the boiler. During the operation period, the quality of the top product collected depends on the reflux flow rate. The reflux ratio is varied using the solenoid valve to change the relative quantities of material returning to the column and flowing to product storage.

3.5.1Constant reflux ratio(C R R)

C R R operation start after the total reflux period the reflux ratio was turned from infinity to finite reflux ratio (constant number), batch distillation under constant reflux ratio, R is kept constant and time varying distillate composition continued until the distillate composition x_d dropped below a certain pre-specified level, for this operation mode the block diagram and the front panel are shown in figures 3.3 and 3.4 respectively.

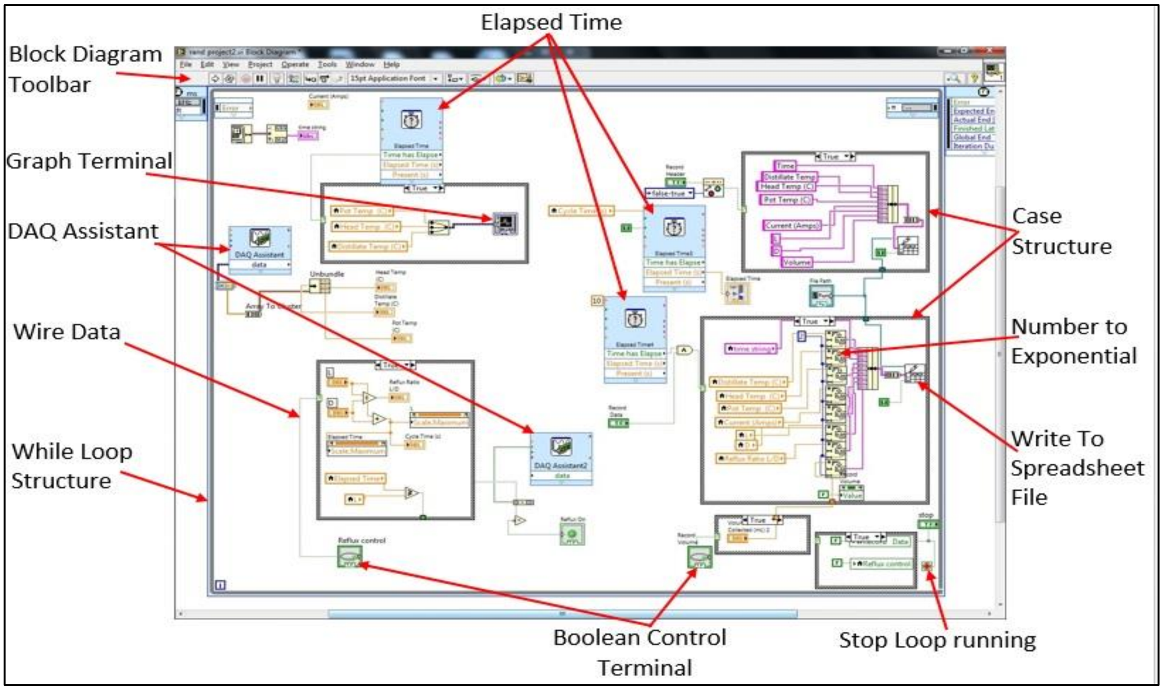


Figure 3.3 Block diagram for constant reflux ratio of batch distillation by LabVIEW.

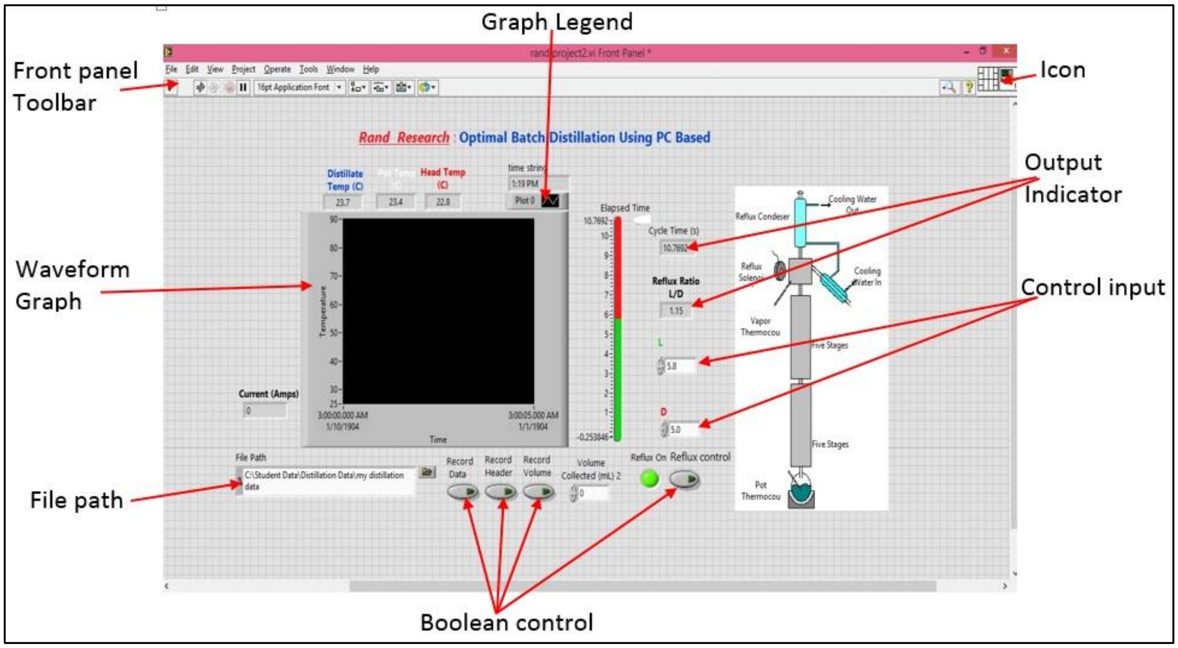


Figure 3.4 Front panel for constant reflux ratio of batch distillation by LabVIEW.

3.5.2 Constant distillate composition

The constructed PC-based controller (LabVIEW) is implemented with the batch distillation system to control the distillate concentration. This is achieved by changing the reflux ratio as manipulating variable to control on temperature (concentration) of the distillate (top plate), for this operation mode the block diagram and the front panel are shown in figures 3.5 and 3.6 respectively.

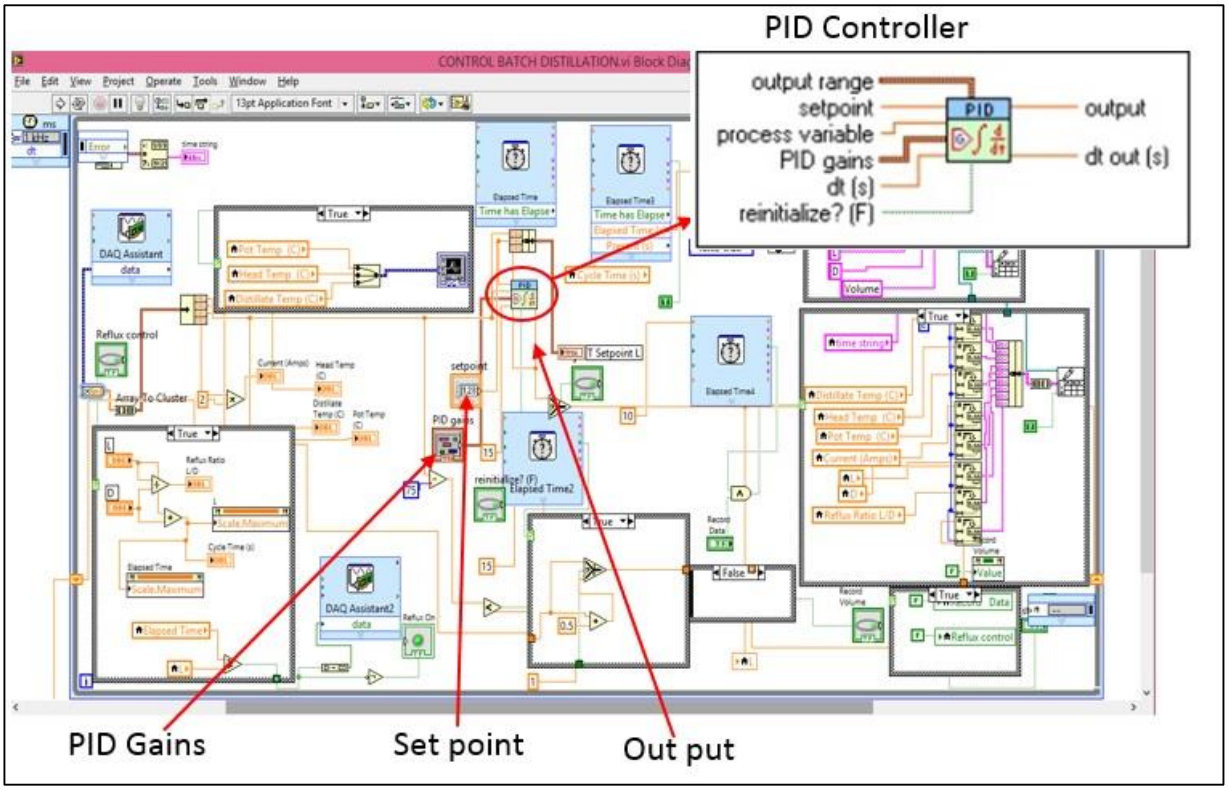
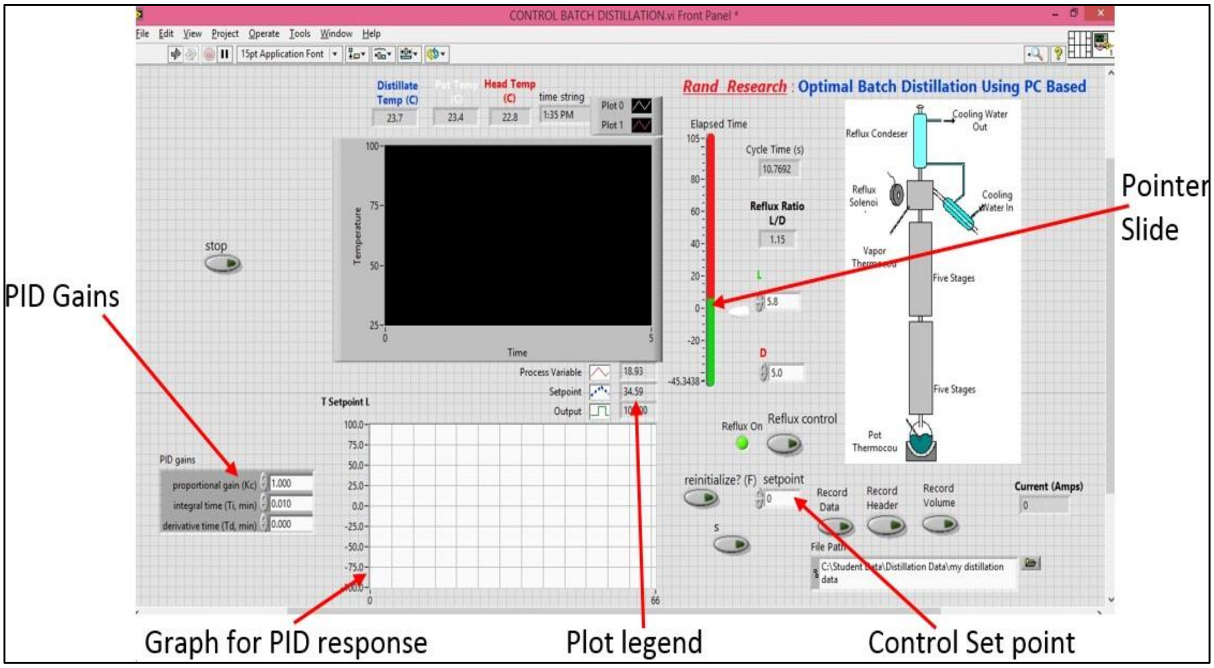


Figure 3.5 Block diagram for constant concentration of batch distillation by LabVIEW.



Distillate Temp (C) 23.7
Pot Temp (C) 23.4
Head Temp (C) 22.8
time string 1:35 PM
Cycle Time (s) 10.7692
Reflux Ratio L/D 1.15
L 5.8
D 5.0
Process Variable 18.93
Setpoint 34.59
Output 100.00
proportional gain (Kc) 1.000
integral time (Ti, min) 0.010
derivative time (Td, min) 0.000
setpoint 0
Current (Amps) 0
File Path C:\Student Data\Distillation Data\my distillation data

Figure 3.6 Front panel for constant concentration of batch distillation by LabVIEW.

3.6 Modeling of batch distillation column

Batch distillation is the process of separating a specific quantity of a liquid mixture into products. The feed mixture is charged to the equipment and one or more products are withdrawn. The composition of both the initial charge and distillate changes with time, there is no steady state. The relative amount of the lower boiling components in the charge decreases as distillation proceeds. Several models are available for solving batch distillation problems [Mellicham, 2003].

3.6.1 Simple model

This model is based on the assumptions of constant relative volatility and equimolal overflow and includes detailed plate to plate calculations. The model is for a multistage, multicomponent system with a constant reflux ratio [Edgar, 2003].

Mass balance

The equations for the condenser are done first, then the equations for the accumulator. This is then followed by the equations for the accumulator, followed by the equations for the plates in the column and the reboiler .

Accumulator and condenser

a) For the accumulator one can write the following total balance equation,

$d/dt\ (H_a) = L_D$ (3.1)

Where H_a is the amount of product in the accumulator and L_D is the incoming liquid from the condenser.

Doing a component balance, for component i, one can write,

$d/dt\ (H_a x_{ai}) = L_D x_{1i}$ (3.2)

Equation (3.2) is expanded as $d/dt\ (x_{ai}) = L_D/H_a\ (x_{1i} – x_{ai})$ (3.3)

b) For the condenser, one can write the following total balance equation,

$d/dt\ (H_c) = V – L_D$ (3.4)

Where H_c is the holdup liquid in the condenser, and V is vapor from tray 2.

Doing a component balance one can write, $d/dt\ (Hcx_{1i}) = Vy_{2i} – L_Dx_{1i}$. Solving this will yield the following equation,

$$d/dt\ (x_{1i}) = V/H_c\ (y_{2i} – x_{1i}) \quad (3.5)$$

The internal reflux ratio is defined as $L = rV$. $V = L_D + L$, which gives,

$$L_D = V\ (1-r) \quad (3.6)$$

Internal plates and reboiler

The same types of material balances can be done for the internal plates and the reboiler to give,

a) For the internal plates:

$$dx_{j,i}/dt = V/H_j\ (y_{j+1,i} – y_{j,i}) + L/H_j(x_{j-1,i} – x_{j,i}) \quad (3.7)$$

$j = 2$, to $n-1$ and $i = 1$ to n_c, assuming a constant molar plate holdup.

The vapor liquid equilibrium relationship is written as:

$$y_{j,\,i} = \alpha_i x_{j,\,i} / (\sum (\alpha_k x_{j,k})) \quad (3.8)$$

b) For the reboiler

$$H_N\ (dx_{N,\,i}/dt) = L\ (x_{N-1,\,i} - x_{N,\,i}) – V\ (y_N – x_{N,\,i}) \quad (3.9)$$

Given the number of trays, equations can be written for each stage and solve simultaneously for $x_{i,\,n}$, the vapor liquid equilibrium relationship is then used to compute values for $y_{i,\,n}$.

This model can be used to compute tray by tray compositions, for the batch column. Hence it is a model which provides quick estimates of the performance of a batch column, in terms of component distributions. However, its results are only valid if the following assumptions are satisfied:

a) Constant molar holdup for condenser and internal plates.

b) Total condensation without sub-cooling.

c) Negligible vapor holdup

d) Perfect mixing of vapor and liquid on the plates

e) Negligible heat losses

f) Theoretical plates

g) Feed mixture at its bubble point

h) Adiabatic column

i) Negligible pressure drop across the column

3.6.2 Rigorous model

The rigorous model is based on the assumptions of equilibrium stages, perfect mixing of liquid and vapor phases at each stage, negligible vapor hold up, constant liquid hold up, M, on a stage and in the condenser system and adiabatic stages in the column. The stages are numbered from top to bottom.

To initiate operation feed is charged to the reboiler, to which heat is supplied. The vapor leaving stage 1 at the top of the column is totally condensed and passed to the reflux drum. At first, a total reflux condition is established for a steady state fixed overhead vapor flow rate. Depending upon the amount of liquid hold up in the column and in the condenser system, the amount and composition of the liquid in the reboiler at total reflux differs to some extent from the original feed.

Starting at some time, $t = 0$, distillate is removed from the reflux drum and sent to an accumulator at a constant molar rate, and a reflux ratio is established. The heat transfer rate to the reboiler is adjusted so as to maintain the overhead vapor molar flow rate. Model equations based on total material balances, component material balances, and energy balances are derived for the overhead condensing system, the column stages and reboiler [Postalcıoğlu, 2005].

Mass and energy balance

Total material balance:

$$V_1 - L_0 - D = dM_0/dt \tag{3.10}$$

Component material balance:

$$V_1 y_{i,1} - L_0 x_{i,0} - D x_{i,D} = d/dt\,(M_0 x_{i,0}) \tag{3.11}$$

Energy balance:

$$V_1 h_{v1} - (L_0 + D)\,h_{L0} = Q_0 + d/dt\,(M_0 h_{L0}) \tag{3.12}$$

The derivative terms are accumulation due to hold up.

For phase equilibrium at stage 1, $y_{i,1} = K_i, x_{i,1}$ (3.13)

These yield the following sets of equations:

Overhead condensing system

Component balance:

$dx_{i,0}/dt = [(L_0 + D + dM_0/dt)/M_0]\ x_{i,0} + [(V_1K_{i,1})/M_0]$ (3.14)

for i = 1 to C components.

Total mole balances: $V_1 = D(R+1) + dM_0/dt$ (3.15)

Enthalpy balance: $Q_0 = V_1\ (h_{v1} – h_{L0}) – M_0\ (dh_{L0}/dt)$ (3.16)

Column stages

Component balance:

$dx_{i,\,j}/dt = [L_{j-1}/Mj]\ x_{i,\,j-1} – [(L_j + K_{i,\,j}V_j + dM_j/dt)/M_j]\ x_{i,\,j} +$

$[(K_{i,\,j+1}V_{j+1})/M_j]\ x_{i,\,j+1}$, for i = 1 to C (3.17)

Total mole balance: $L_j = V_{j+1} + L_{j-1} – V_j – dM_j/dt$ (3.18)

Enthalpy balance: $V_{j+1} = 1/\ (h_{vj+1} – h_{Lj})\ [V_j\ (h_{vj} – h_{Lj}) – L_{j-1}(h_{Lj-1} – h_{Lj})$

$+ M_j\ (dh_{Lj}/dt)]$, for j = 1 to n (3.19)

Reboiler

Component balance: $dx_{i,\,N+1}/dt = (L_N/M_{N+1})\ x_{i,\,N} – [(V_{N+1}K_{i,\,N+1} + (dM_{N+1}/dt))/M_{N+1}]$
$x_{i,\,N+1}$, for i = 1 to C (3.20)

Enthalpy balance: $Q_{N+1} = V_{N+1}(h_{vN+1} – h_{LN+1}) – L_N(h_{LN} – h_{LN+1})$

$+ M_{N+1}(dh_{LN+1}/dt)$ (3.21)

$L_0 = RD$ (3.22)

Phase Equilibrium relationships for the column stages and the reboiler can be written, $y_{i,j} = K_{i,j}x_{i,j}$, under the restriction that $\sum y_{i,j} = 1$, for i = 1 to C and j = 1 to N+ 1

These equations can be solved under the following conditions:

a) Constant Volume Hold Up (CVH)

b) Constant Molar Hold Up (CMH)

They constitute initial value problems for a system of ordinary differential equations. Analytical solutions are difficult, so these systems of equations are generally solved numerically.

This technique can be used for predictive modeling purposes once the assumptions that are used in its deriving are met. The accuracy of the model will depend on the thermodynamic models used for vapor liquid equilibrium data and for predicting enthalpy. The fact that enthalpy calculations are used in this model makes it a better model for predictive purposes than the Short Cut Model and the Simple Model. This is because if the conditions of equimolal overflow are not met, the enthalpy calculations that are used as part of the iterative process for calculating compositions will still ensure that good results are obtained.

3.6.3 Rigorous model with chemical reactions

The assumptions made for the rigorous model are used for the rigorous model with chemical reactions. Further assumptions are that there are no chemical reactions in the vapor phase and in the condenser accumulator. Reaction kinetics data, which can be written as, [Edgar, 2003]

$r_{ji} = r_{ji}(k_r, x_D)$ (3.23)

$\Delta n_j = \sum r_{ji}$ (3.24)

Mass and Energy balance equations:

a) Condenser and Accumulator

Accumulator total mass balance: $dHa/dt = L_D$ (3.25)

Accumulator component balance: $d/dt\ (Hax_{ai}) = L_D x_{Di}$ (3.26)

Condenser Hold up Tank component balance: $d/dt\ (H_c x_{Di}) = V_2 y_{2i} +$

$r_{1i} H_c - L_c x_{Di}$ (3.27)

Energy balance: $d/dt\ (H_c h_1^L) = V_2 h_2^v - L_c h_1^L - Q_c$ (3.28)

b) Internal Plates

Total mass balance: $dH_j/dt = L_{j-1} + V_{j+1} - L_j - V_j + \Delta n_j H_j$ (3.29)

Component mass balance: $d/dt\ (H_j x_{j,i}) = L_{j-1} x_{j-1,i} + V_{j+1,i} - L_j x_{ji}$

$- V_j y_{ji} + r_{ji} H_j$ (3.30)

Energy Balance: $d/dt\ (H_j h_j^L) = L_{j-1} h_{j-1}^L + V_{j+1} h_{j+1}^v - L_j h_j^L - V_j h_j^v$ (3.31)

Equilibrium relationship: $y_{ji} = k_{ji} x_{ji}$, under the restriction that $\sum y_{ij} = 1$

c) Reboiler

Total mass balance: $dH_N/dt = L_{N-1} - V_N + \Delta n_N H_N$ (3.32)

Component mass balance: $d/dt\ (H_N x_{Ni}) = L_{N-1} x_{N-1,\ i} - V_N y_{Ni} + r_{Ni} H_N$ (3.33)

Energy balance: $d/dt\ (H_N h_N^L) = L_{N-1} h_{N-1}^L - V_N h_N^v + Q_R$ (3.34)

This is just an extension of the rigorous model. From the material and energy balances, thermodynamic data and kinetic data, compositions of distillates, residues and tray compositions can be computed.

3.7 Implemented model of batch distillation

The process under consideration is binary distillation column which separates a mixture for methanol/water.

The model referred to single composition control, in this work single input/single output (SISO) system is used with feedback loops and PI controller, schematics of the process found in Figure 3.7.

In this system we select the controlled and the manipulating variables as shown in table below:-

Table 3.1 the manipulated and controlled variable of
the batch distillation column.

Manipulated variable	Controlled variable
Reflux ratio	Overhead MeOH composition

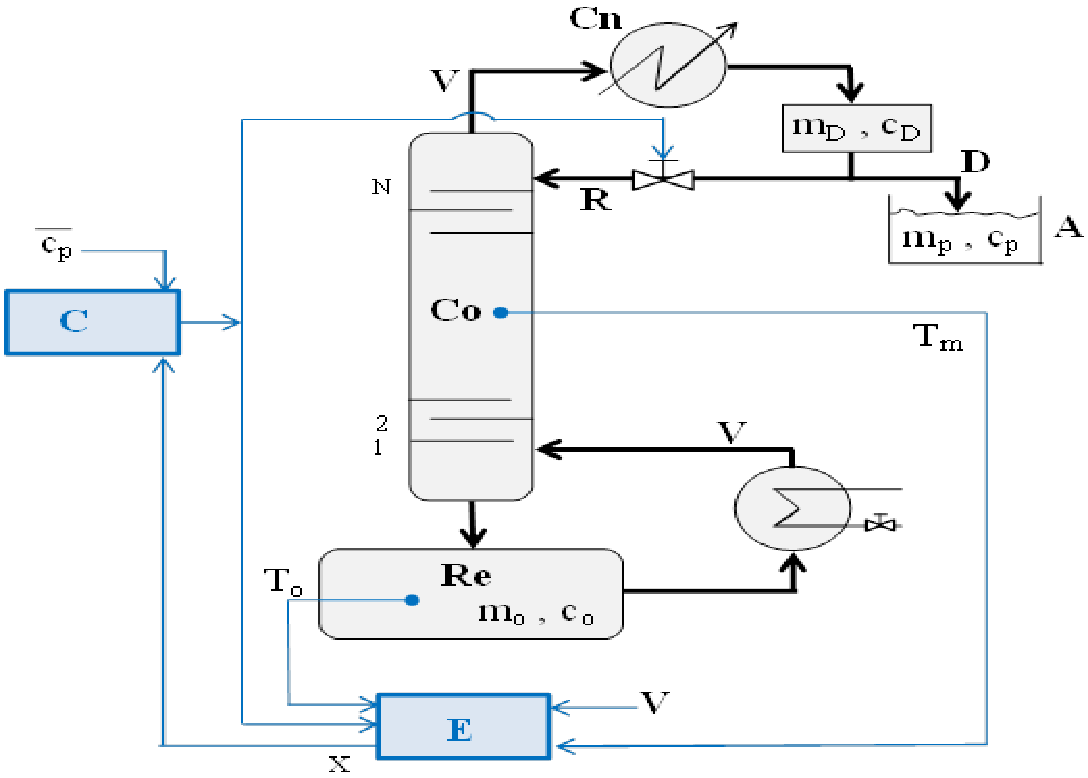


Figure 3.7 Batch distillation column and control system: accumulator (A), condenser (Cn), column (Co),controller (C), estimator (E), reboiler (Re),control (u = R), and measurement [$y = (T_0,..,T_m)'$].

3.7.1 Obtaining transfer function

The analytical determination for the system's transfer function is difficult. The individual component values not been known, or the internal configuration of the system may not accessible. In such cases, the frequency response of the system, from input to output obtained experimentally and using the frequency response plot experimentally, by using a sinusoidal force or signal generator at the input of the system and measure the output steady state sinusoidal amplitude and phase angle [Luyben, 1997].

3.7.2 The transfer function models

There are three processes before doing the simulator, first, the transfer function between the input and output of the open loop process must be determined, and then process transfer function can be computed. The next step to determine the dynamic of the process, and the third is based on the use of an external computer simulator [Fagely, 1990].

In this work, the transfer function models for column by step forcing the manipulative variable. An alternative industrial approach would be to step force the actual column and record the overhead composition X_D response. The gain in the transfer function not fitted but were determined from the difference between the initial and final composition divided by the step change value [Luyben, 1997].

3.7.3 Control strategies

Feed control in general is achievement and maintained of desired condition by using an actual value of this condition and comparing it to reference value (set point), and using differences between those to eliminate any difference between them. Most controller use negative feedback, which measured process output (controlled variable), subtracted from a desired value (set point) to generate an error signal. The controller recognize the error signal and manipulates a process input (control element), to reduce the error [Luyben, 1997]. Most important type of industrial fed back controllers; include P, PI and PID controller. In frequency response there will be G_C's, which are add to transfer, function G of system to get the total loop transfer function of a system [Fagely, 1990].

3.7.4 summery of modeling equation

The basic mathematical model for the description of a batch distillation process is a large system of differential algebraic equations of the form:

$$f(\dot{x}, x, t, R) = 0, t \in [0, t_f] \tag{3.35}$$

Where t denotes time, t_f is the period of batch, x is the controlled variables (such as flows, pressures, temperatures, etc.), while R denotes as the manipulated variables (e.g., reflux ratio).

A typical operation of a batch distillation processes can be described as follows. The mixture is added into the reboiler, and certain distillate composition, denoted as x, is made to track a prescribed trajectory $x_r(t)$ by manipulating the reflux ratio R. At first, the distillate composition $x(t)$ starts from the initial condition $x(0)$, such that a control policy must ensure composition tracking in the sense that $x(t) \rightarrow x_r(t)$ as closed and soon as possible. As mentioned earlier, in batch distillation processes operation there is no steady state, so there is no normal condition at which a linear input/output model can be constructed by step response methods. To avoid the absence of a steady state, one may linearize the system dynamics around a trajectory. Considering situations of this nature, the use of empirical input/out models seems to be a reasonable practical solution for the design of controllers for batch distillation. [Seborg, 2003]

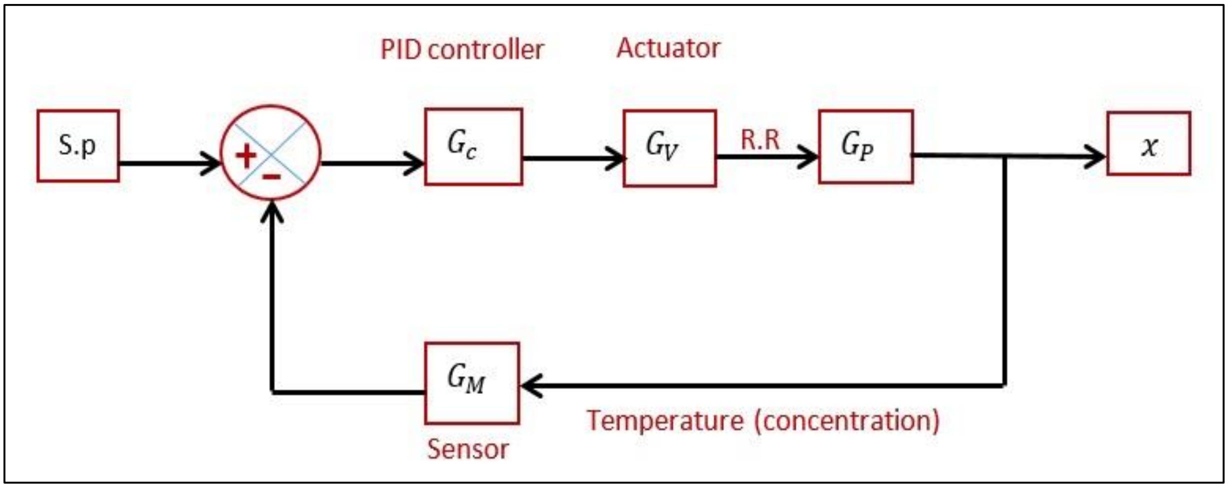


Figure 3.8 Closed loop control system.

First order model

$$G_P = \frac{K}{\tau s+1} \tag{3.36}$$

$$\Delta x = G_P \Delta R \tag{3.37}$$

For step change response

$$\Delta R = \frac{m}{s} \tag{3.38}$$

$$\Delta x = \frac{K}{\tau s+1} * \frac{m}{s} \tag{3.39}$$

$$\dot{x} = \left(1 - e^{-\frac{t}{\tau}}\right) K\, m \tag{3.40}$$

When $t = 5\tau$

$$\dot{x} = 0.99Km \tag{3.41}$$

The simplest linear input/output model that retain this dynamic characteristic of batch distillation processes is an integrating process with a process gain

$$\Delta x = K\Delta R \tag{3.42}$$

Where K is a (time-varying) gain of high-frequency. The aim of an identification procedure should be to obtain estimates of the single parameter k_p. In principle, the process gain K can be obtained as

$$K = \frac{\partial x}{\partial R} = \frac{\partial}{\partial t}\left(\frac{\partial x}{\partial R}\right) \tag{3.43}$$

Control loop

Closed loop control system with disturbance input is shown in Figure 3.8. Gp is the transfer function of the process and Gv is the transfer function of the actuator while Gc is transfer function of the controller which is PID controller, The transfer function of the PID controller is given below

$$G_c = k_p[1 + \frac{1}{\tau_I} + \tau_D] \tag{3.44}$$

3.8 Auto-Tuning Techniques

PID controllers have been used for industrial processes because of their simplicity and robustness. But it is difficult and time consuming to define the controller parameters accurately. Therefore, it becomes impracticable. So, auto-tuning techniques are developed to define these controller parameters. It has auto-tuning and by means of that PID parameters are computed and transferred to the controller. Thus PID controller has been faster, more practical and reliable [Bolat, 2005]. There are many auto-tuning methods such as Ziegler Nichols Step Response, Relay and ISTE. To implement real time temperature control of the distillate, a PC based is used, Designed controllers using auto-tuning techniques can be done by simulated in LabVIEW simulation by using mathematical model of the batch distillation first.

3.8.1 Ziegler-Nichols tuning

The process of selecting the controller parameters to meet given performance specifications is known as controller tuning. Ziegler and Nichols suggested rules for tuning PID controllers (meaning to set values K_p, τ_I andτ_D,) based on experimental step responses or based on the value of K, that results in marginal stability when only proportional control action is used.

The Ziegler-Nichols Step Response Method a simple way to determine the parameters of a PID regulator based on step response data was developed by Ziegler and Nichols and published in 1942. The method uses only two of the parameters shown in Fig. 3.9, namely, a and τ. The regulator parameters are given in Table 1. The Ziegler-Nichols tuning rule was developed by empirical simulations of many different systems [Wittenmark, 1995].

$$G(s) = \frac{k}{1+sT} e^{-s\tau} \tag{3.45}$$

$$a = k \frac{\tau}{T} \tag{3.46}$$

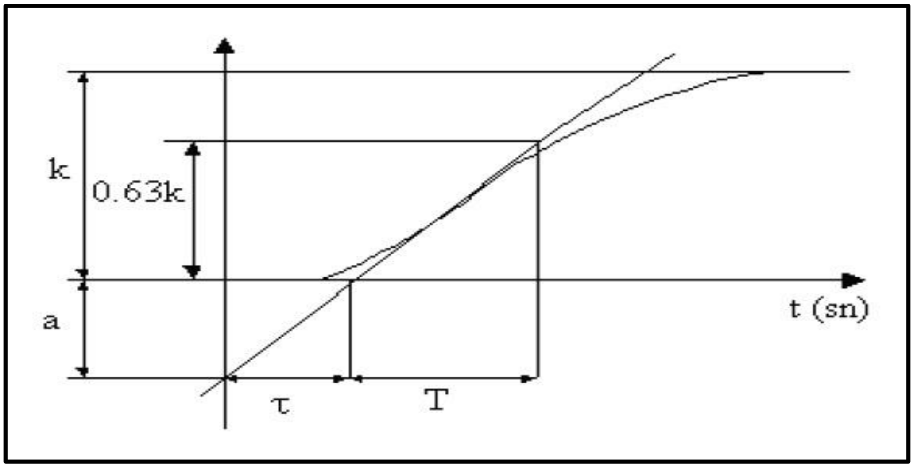


Figure 3.9 Unit step response.

Table 3.2 Regulator parameters obtained by the Ziegler-Nichols step response method.

Controller	K_P	T_I	T_D
P	1/a	-	-
PI	0.9/a	3 τ	-
PID	1.2/a	2 τ	τ /2

In the Ziegler-Nichols tuning rule based on critical gain K_{cr}, and critical period P_{cr}, (Second Method), first set $\tau_I = \infty$ and $\tau_D = 0$. Using the proportional control action only, increase K_p, from 0 to a critical value K_{cr}, at which the output first exhibits sustained oscillations. (If the output does not exhibit sustained oscillations for whatever value K_p, may take, then this method does not apply.) Thus, the critical gain K_{cr}, and the corresponding period P_{cr}, are experimentally determined (see Figure 3.10). Ziegler and Nichols suggested setting the values of the parameters K_p, τ_I and τ_D according to the formula shown in Table 3-2 [Ogata, 2002].

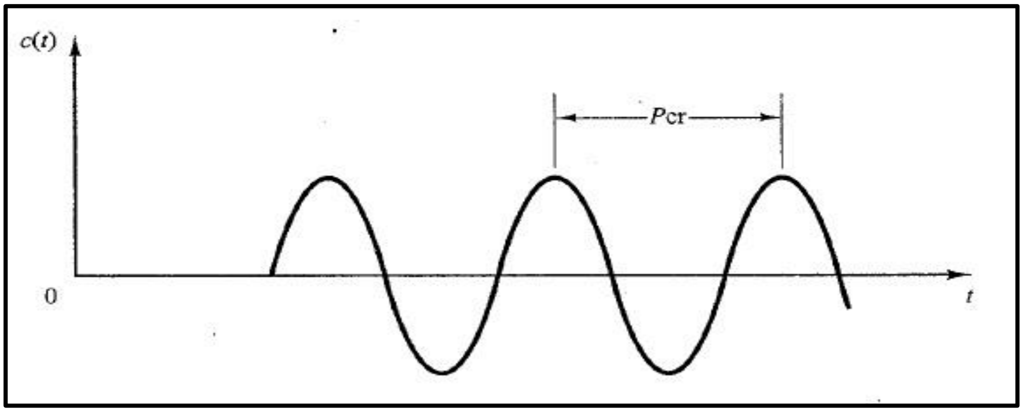


Figure 3.10 Sustained oscillation with period P_{cr}.

Table 3-3 Ziegler-Nichols tuning rule based on critical gain K_{cr}, and critical period P_{cr}, (Second Method)

Type of Controller	K_p	T_i	T_d
P	$0.5K_{\mathrm{cr}}$	∞	0
PI	$0.45K_{\mathrm{cr}}$	$\frac{1}{1.2}P_{\mathrm{cr}}$	0
PID	$0.6K_{\mathrm{cr}}$	$0.5P_{\mathrm{cr}}$	$0.125P_{\mathrm{cr}}$

Notice that the PID controller tuned by the second method of Ziegler-Nichols rules gives:

$$G_c(s) = k_p[1 + \frac{1}{\tau_I s} + \tau_D s] \tag{3.47}$$

$$G_c(s) = 0.5k_{cr}[1 + \frac{1}{0.5\, P_{cr}\, s} + 0.125\, P_{cr} s] \tag{3.48}$$

Thus, the PID controller has a pole at the origin and double zeros at $s = -\frac{4}{P_{cr}}$. Note that if the system has a known mathematical model (such as the transfer function), then the root-locus method can be used to find the critical gain *Kc,* and the frequency of the sustained oscillations w_{cr}, where $\frac{2\pi}{w_{cr}} = P_{cr}$. These values can be found from the crossing points of the root-locus branches with the jw axis.

3.8.2 Relay tuning method

The relay method is attractive since a control-relevant excitation signal is generated automatically, and many tuning rules exist to utilize the resulting process information [Zhuang and Atherton, 1993]. The relay feedback is an efficient method of obtaining the critical point of a process with the critical point made available. PID types of controllers are easily tuned using classic Ziegler-Nichols rules and variants. The arrangement for a relay feedback auto-tuner is shown in table 3.3 [Halevi, 1997].

Table 3.4 PID parameters used in relay tuning method.

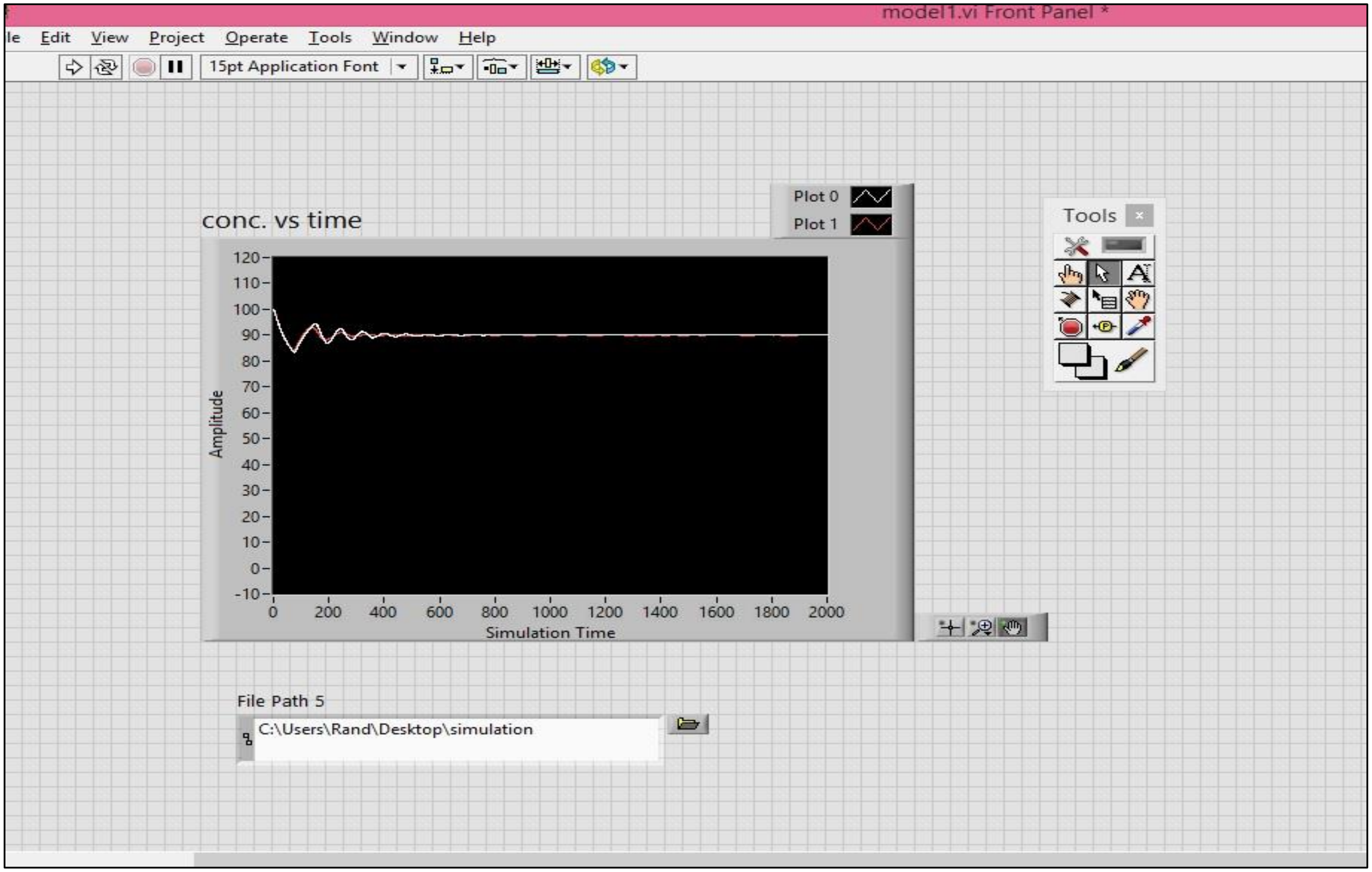
Controller	K_P	K_I	K_D
P	$0.5\ K_C$	-	-
PI	$0.4\ K_C$	$1.25 / T_C$	-
PID	$0.6\ K_C$	$2 / T_C$	$0.12T_C$

Kc can be regarded as the equivalent gain of the relay for transmission of sinusoidal signals with amplitude a. This parameter is called ultimate gain. It is the gain that brings a system with transfer function G(s) to the stability boundary under pure proportional control. The period $Tc=2\pi/\omega u$ is similarly called the ultimate period.

3.8.3 Simulation by LabVIEW

The experimental work has been done in LabVIEW software. LabVIEW has a dedicated software which only deals with the Control Design and Simulations of the different plant models and control system. This part of LabVIEW is very similar to the SIMULINK of the MATLAB by Math Works Inc. SIMULINK contains different toolboxes which deals with simulations of the different system such as Hydraulic Systems, Electrical Systems etc. LabVIEW also has different toolboxes which include PID Toolbox, and different toolbox which deals with simulations of the plant such as Linear Systems, Nonlinear System, and Simulation Loop. Figures 3.11 and 3.12 shows the front panel and block diagram respectively for the simulation of batch distillation that used in this research

Some situations do not lend themselves to precise mathematical treatment. Others may be difficult, time-consuming, or expensive to analyze. In these situations, simulation may approximate real-world results; yet, require less time, effort, and/or money than other approaches.



File Path 5

C:\Users\Rand\Desktop\simulation

Figure 3.11 front panel simulation constant concentration of batch distillation by LabVIEW.

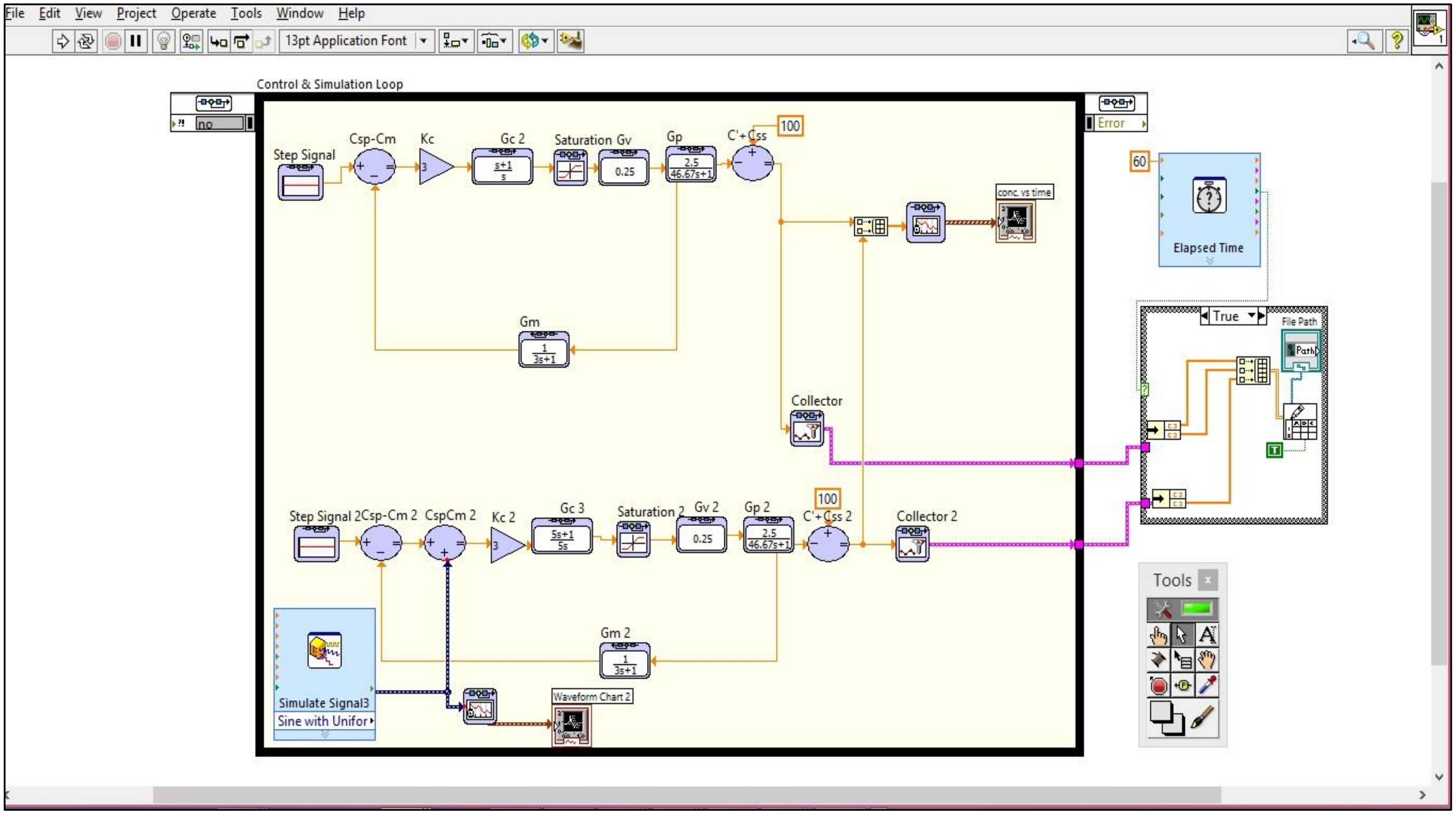


Figure 3.12 block diagram simulation constant concentration of batch distillation by LabVIEW.

CHAPTER FOUR

EXPERIMENTAL WORK

In this chapter there will be description of the batch distillation, experimental work and the strategies used to operate the batch distillation. The experimental rig was designed and built in the laboratories of Chemical and Biomolecular Engineering department at Case Western Reserve University in the United States of America.

4.1 Studied System

Methanol – water mixture were used in the experimental work. Methanol is a liquid chemical with the formula CH_3OH (often abbreviated MeOH). It is colorless, volatile, flammable, and poisonous. Methanol is made from the destructive distillation of wood and is chiefly synthesized from carbon monoxide and hydrogen. Its principal uses are in organic synthesis, as a fuel, solvent, and antifreeze.

Methanol economy is a suggested future economy in which methanol replaces fossil fuels as a means of energy storage, ground transportation fuel, and raw material for synthetic hydrocarbons and their products. It offers an alternative to the proposed hydrogen economy or ethanol economy. This well-known binary mixture has been chosen in order to avoid sample taking in the column during the experiments. In fact, temperature measurements allow to know the exact tray composition by the way of vapor-liquid equilibrium diagram. A water-methanol mixture has also been chosen for the volatility variations it presents. Furthermore, this binary mixture in agreement with assumption of (vapor density is negligible compared to the liquid density).

4.2 Apparatus of Batch Distillation implemented

A schematic diagram and photo of batch distillation column are shown in Figures (4.1) and (4.2) respectively. The still pot is typically a 1000 cm^3, two-necked round bottom flask. The side neck is connected to a thermocouple type K, the center neck is connected to the fractionation column. The still-pot heater is a 380 Watt fitted around the still-pot. A variable transformer (Variac) is connected to the still-pot heater to control the power supplied to the still.

The fractionation column consists of two parts, each part containing 5 trays with dimensions 40 cm × 2 cm id. The column supplied with silver vacuum jacket and has an observation strip. The reflux head of the fractionation column contain a magnetic solenoid to control the reflux ratio. A vertical condenser connected to the top of the reflux head. A photo of the reflux head is shown in figure (4.3). Another condenser in connected to the side of the reflux head to cool the distillate and the product collected in a 250 cm^3 conical flask.

The auto-control of the system has been built from data acquisition device known as NI – ELVIS II which was very crucial since it played a big role to accomplished objectives of this research. The DAQ has ability to generate signal to trigger device for open/closed action of magnetic solenoid for reflux flow control. The DAQ also has ability to acquire analogue signal from external via sensors which are connected to it, the temperature sensors (thermocouple type K) inserted in the batch distillation column responsible for sensing the temperature from the column through the NI DAQ to the lab server by using DAQ's analogue inputs channels so that output voltage of the product will with drawn at the top of the column.

During the column operations, the distillate flow rate profile, the temperature profiles (concentration profiles) along the column and the power for heating were measured online all this done by LabVIEW program.

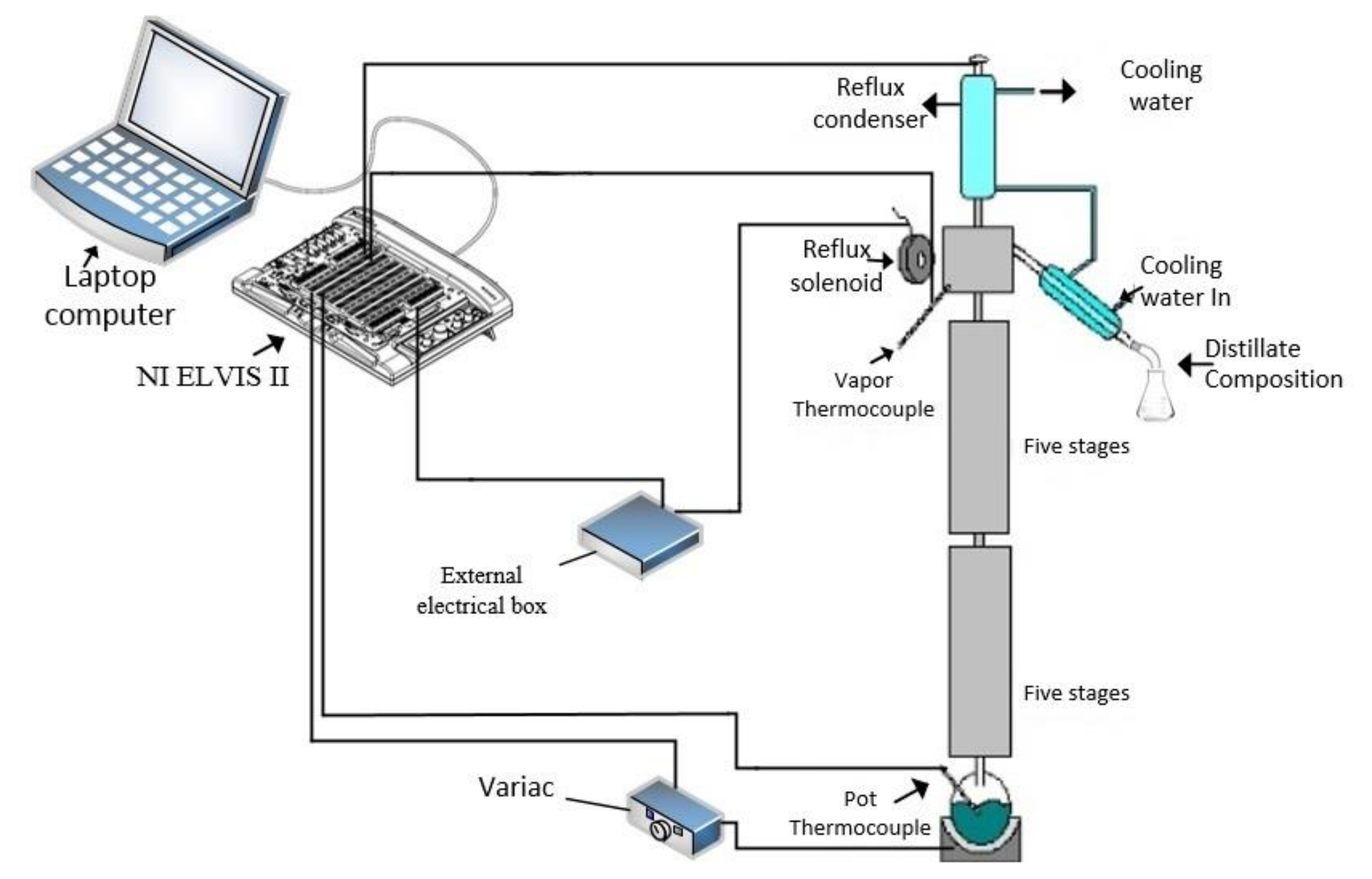


Figure 4.1 Schematic diagram of batch distillation column.

Figure 4.2 Experimental system.

4.3 Description of Electrical Circuit

The electrical circuit consisted of the following:

1. Thermocouples
2. Magnetic solenoid
3. Still-pot heater
4. Data Acquisition Adaptor (DAQ)

Description of the above items given as follows:

1. Thermocouples: Three thermocouples were used in the batch distillation system to measure the temperatures in the reboiler, distillate, and vertical condenser. Each of these thermocouples was connected to specific channel in the DAQ.

2. Magnetic solenoid: The condensate produced on the surface of the vertical condenser flows down from its end to a swinging funnel. Normally the liquid flows back into the column, but when attracted be electromagnet the funnel rod is deflected and the condensate falls to the receiver.

   The magnet solenoid is connected to the DAQ through a 4 channels, type LFA300, Range extender.

3. Still-pot heater: The heating mantel was connected to a Variac (Barnsted Electro thermal, MC228X1) which was plugged to the wall. The Variac allows controlling the voltage supplied to the heater in the range of 0-120 V.

   External electrical box was connected to the DAQ card to read the power supplied to the heater.

4. Data Acquisition Card (DAQ): National Instruments – Educational Laboratory Virtual Instruments (NI – ELVIS II) was used to connect the measurement sensors to a PC through a USB connection, and to control the system. Figure (4.4) illustrate the instrument connection to ELVIS.

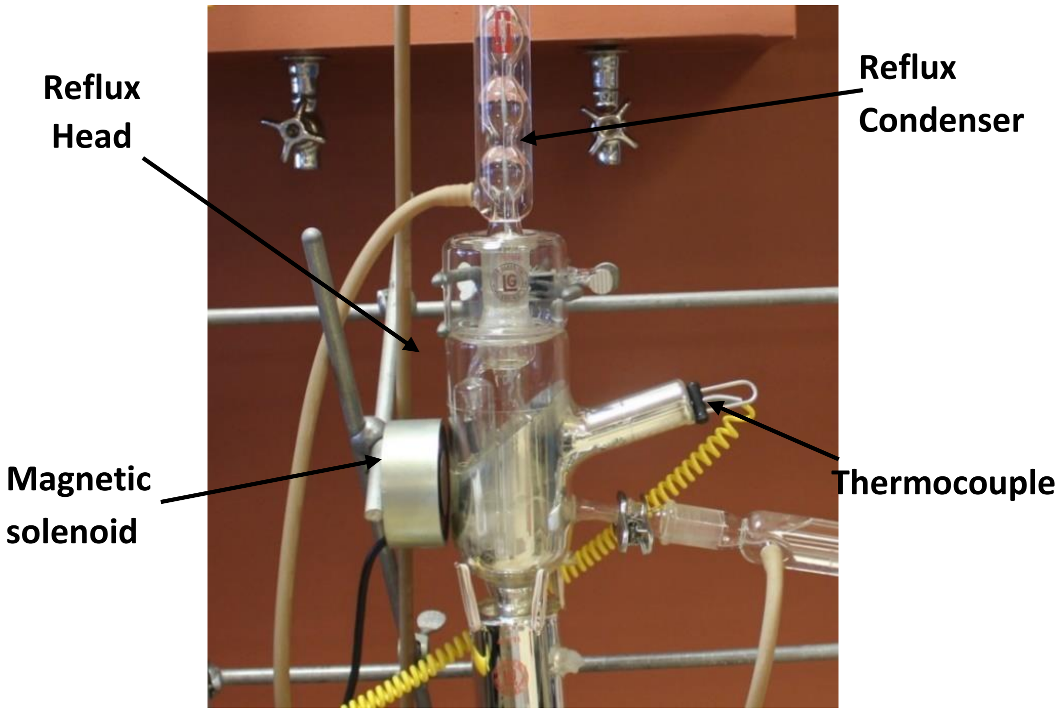


Figure 4.3 Reflux head

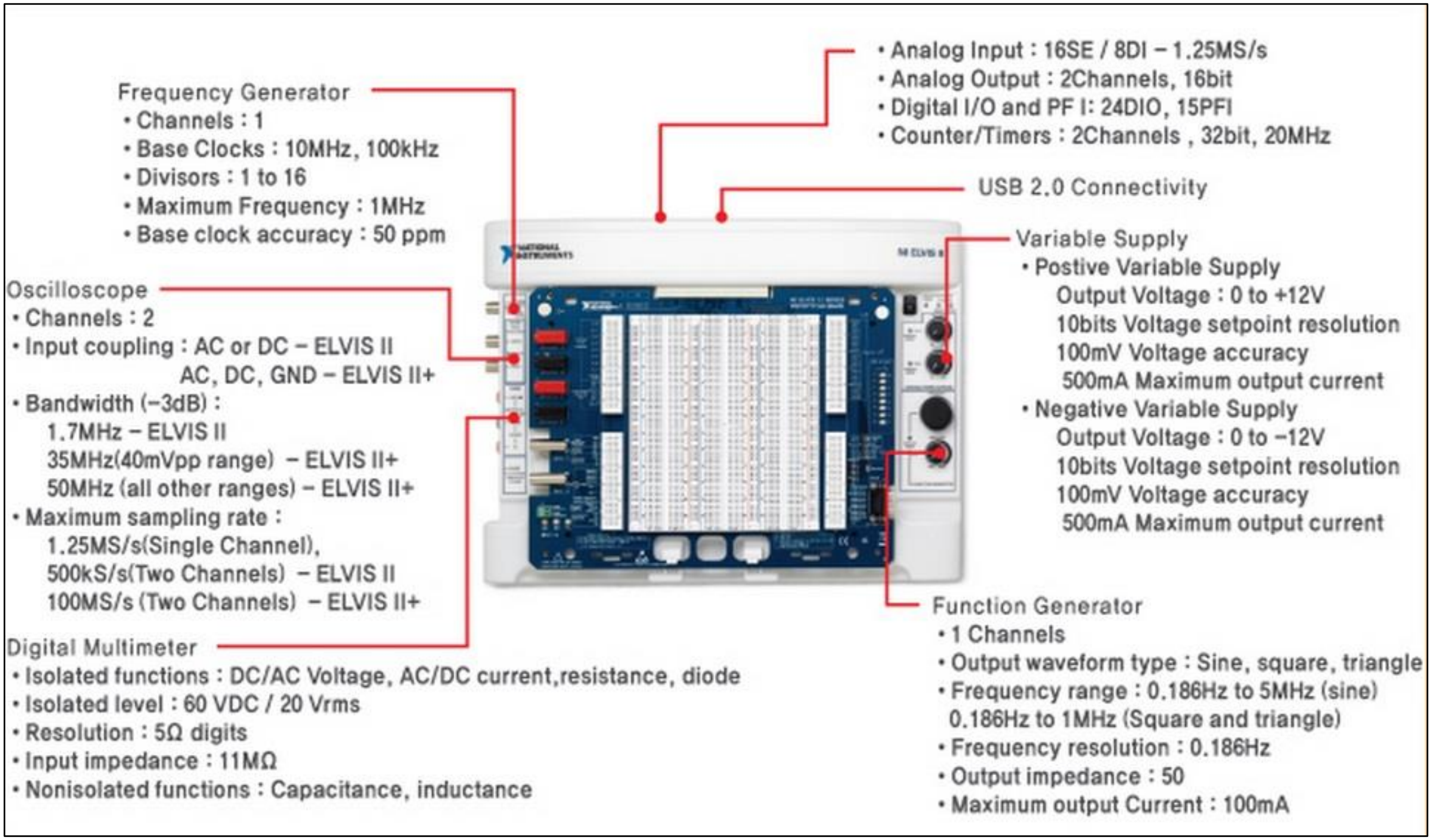


Figure 4.4 NI – ELVIS connections.

4.4 Experimental work

Experiment with methanol and water mixtures have been made with a constant heat duty of 288 W for each experiment still pot heater used for this purpose. At the beginning of the batch operation, total reflux policy is required. The duration of the total reflux period was chosen as follows: when the distillate temperature (i.e., composition) reached the desired value. After that, the column was worked at two strategies (a) constant reflux ratio with range (1.5-10) and (b) constant distillate composition. The initial volume of the mixture was 1L and all the experiment feed mixture (methanol-water) have the same initial composition,25%mole of methanol and 75%mole of water. The column works at atmospheric pressure with 10 trays.

4.4.1 Experiment procedure

1. Turn on PC-Control, run the LabVIEW program for batch distillation
2. Turn on cooling water and set flow meter to desired value also turn on the still pot heater on power 288 W.
3. Charge with a mixture of methanol and water.
4. Confirm magnetic solenoid for reflux is off.
5 Wait until temperature at the top stage finds equilibrium.
6. Insert the desired set point value for experiment (depend on the strategy that used which are constant reflux or constant concentration) in LabVIEW.
7. Record time and temperature profile.
8. Turn on magnetic solenoid for reflux.
9. Record time and temperature profile.
10. Take sample.
11. Open distillate value and allow collected distillate to accumulate in large

graduated cylinder.

12. Repeat step 11 for additional samples allowing T_{top} to change at least a full degree Celsius before taking another sample.
13. Measure and record the mass, kg, and density of the total distillate.
14. Shutdown/Recharging Procedure. Turn off magnetic solenoid reflux and Turn off still put reboiler.
15. Measure and record total remaining mass of bottoms.
16. Allow column to air cool for 1 hour. The cooling water should be running throughout.
17. If no other experiments are scheduled, turn off cooling water.

All the experiments the distillation still was charged and heated up under total reflux unit steady-state conditions were achieved. After, that two strategies used to operate the batch distillation:

4.4.2 Constant reflux ratio

C R R operation start after the total reflux period the reflux ratio was turned from infinity to finite reflux ratio (constant number), batch distillation under constant reflux ratio, R is kept constant and time varying distillate composition continued until the distillate composition x_d dropped below a certain pre-specified level. Each 5 minutes samples were taken to analysis by Refractometer.

4.4.3 Constant distillate composition

Conventional strategies for constant composition operation of batch distillation columns usually comprise a total–reflux startup, which is carried out until the process approaches the steady state. Then a controller is switched on in order to drive the distillate composition to desired value and maintain it at the desired level (by manipulating reflux ratio). Finally the operation is stopped when the reflux ratio is reached the pre-specified value. For the determination of liquid phase composition, samples were taken to analysis by Refractometer. The system in this operation has been divided into temperature (concentration) control and monitoring, and reflux control so when all these combined batch distillation operating at constant distillate composition is obtained

CHAPTER FIVE

RESULTS AND DISCUSSION

In the previous chapter, the experimental work for the batch distillation system using PC- based with constant reflux ratio and variable reflux ratio was considered.

In this chapter the result of the vapor and liquid flow rates, temperature in (reboiler, plates and condenser), and liquid composition profile, using PC-based (LabVIEW) for batch distillation to separate Methanol – Water mixture are presented and compared with the result obtained by LabVIEW simulation.

This thesis addresses different aspects of operation and control of batch distillation columns. Operating mode of constant reflux ratio and variable reflux ratio with distillate composition were studied.

The conventional operation of batch distillation are either constant reflux or constant over-head composition. In a constant reflux operation, the reflux ratio R remains constant in the whole process until the concentration of the overhead goes down to such a point that distillate amount equals to the prescribed. Where as in a constant overhead operation, the composition in the condenser raises to prescribed composition; the reflux ratio then adjusted so as to insure that the composition of distillate remains constant.

5.1 Constant reflux ratio

For fixed heat duty, when the reflux ratio increases, the time of the experiment (the time required to collect prescribed amount of the distillate) and purity of methanol obtained in the distillate also increased, while the distillate flow rate decreases. Figures 5.1-5.10 respectively shows the experimental result for constant reflux ratio of 0.5, 1.5, 2, 3, 4, 5, 6, 7, 8, and 10 respectively, the first 20 minutes in these figures represent the total reflux condition.

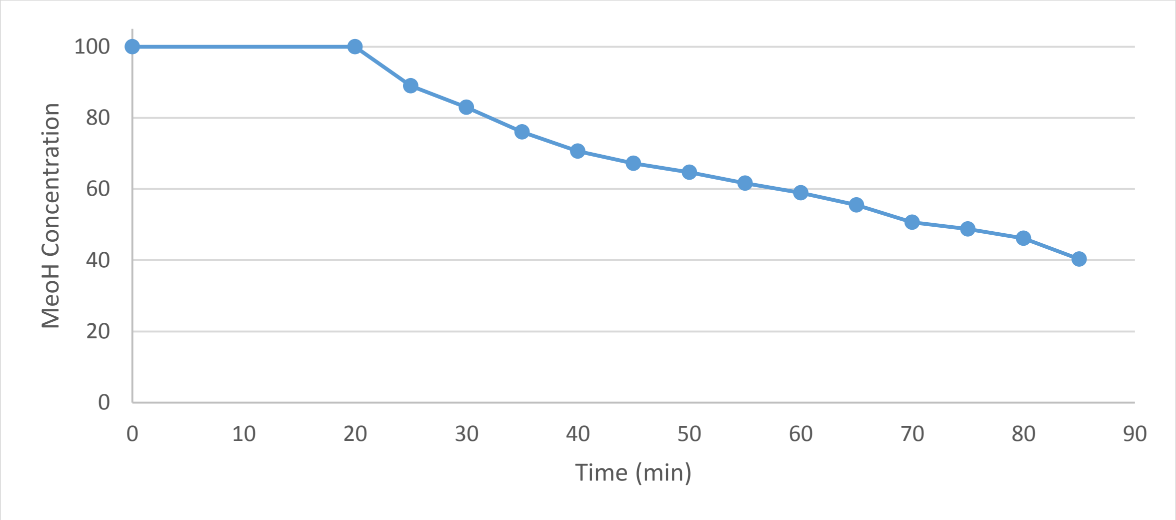


Figure 5.1 concentration profile for batch distillation
with constant reflux ratio (R=0.5)

Figure 5.1 show the change of x_D when the value of reflux ratio =0, also we can see that its need to 85 min to obtain 400 ml of methanol that had concentration $x_D = 65\%$.

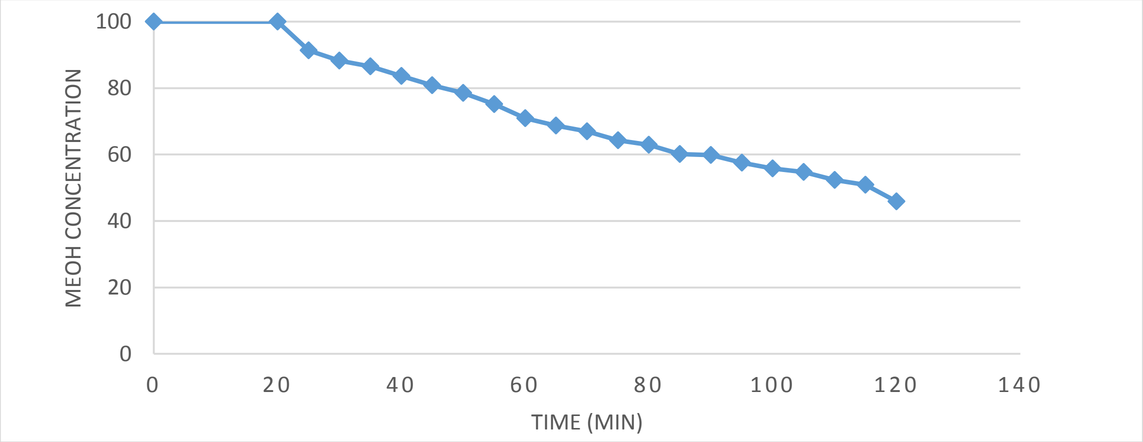


Figure 5.2 concentration profile for batch distillation
with constant reflux ratio (R=1.5).

Figure 5.2 show the change of x_D when the value of reflux ratio =1.5, also we can see that its need to 120 min to obtain 400 ml of methanol that had concentration $x_D = 70.35\%$.

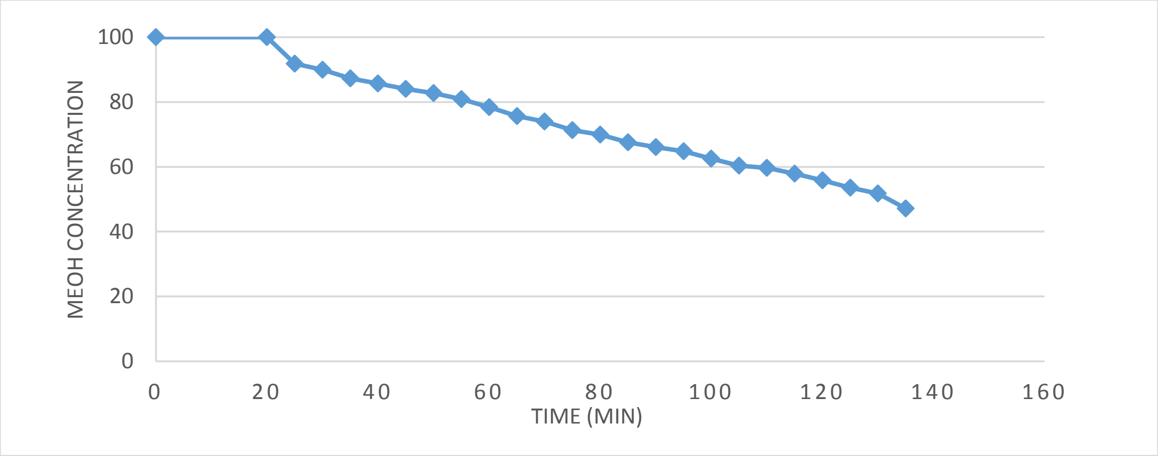


Figure 5.3 concentration profile for batch distillation with constant reflux ratio (R=2)

From figure 5.3 we can see the change of x_D when the value of reflux ratio =2, so the figure show that its need to 135 min to obtain 400 ml of methanol that had concentration $x_D = 72\%$

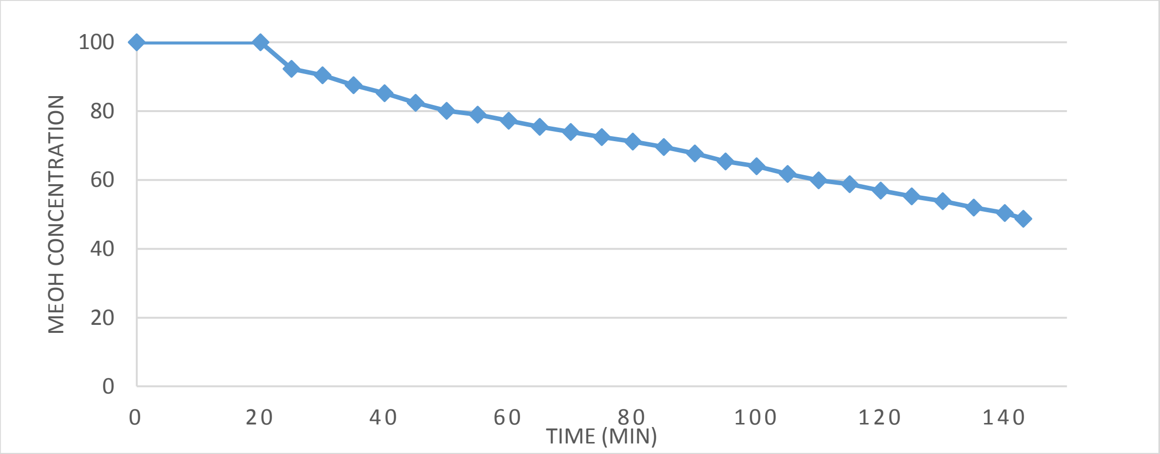


Figure 5.4 concentration profile for batch distillation with constant reflux ratio (R=3)

From figure 5.4 we can see the change of x_D when the value of reflux ratio =3, also we can see that its need to 143 min to obtain 400 ml of methanol that had concentration $x_D = 73.8\%$

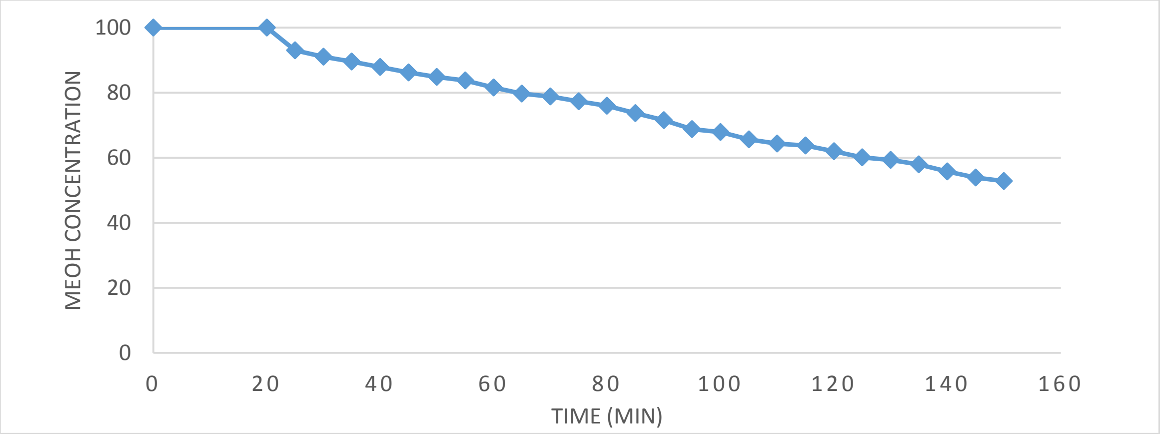


Figure 5.5 concentration profile for batch distillation with constant reflux ratio (R=4)

Figure 5.5 show the change of x_D when the value of reflux ratio =4, so from figure we can see that its need to 150 min to obtain 400 ml of methanol that had concentration $x_D = 75.2\%$

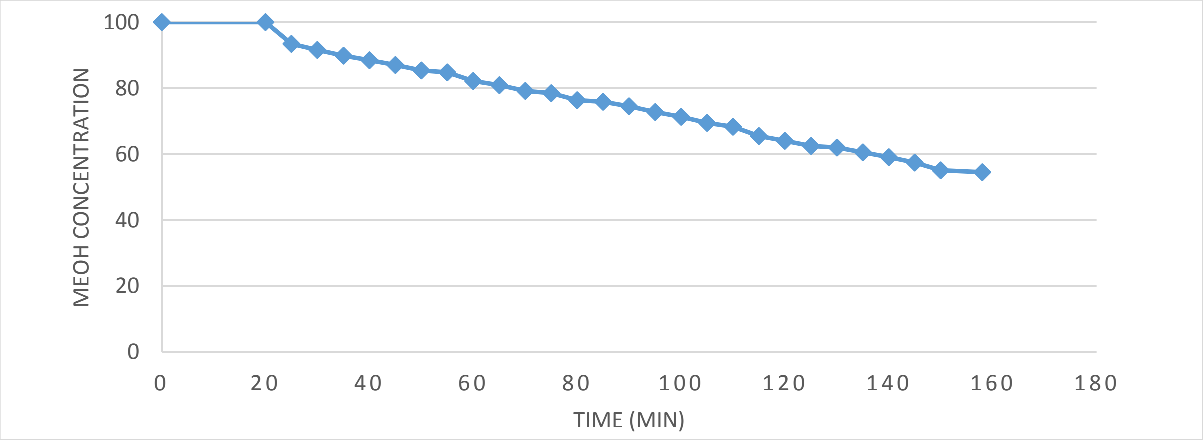


Figure 5.6 concentration profile for batch distillation with constant reflux ratio (R=5)

From figure 5.6 we can see the change of x_D when the value of reflux ratio =5, also we can see that its need to 158 min to obtain 400 ml of methanol that had concentration $x_D = 77.1\%$

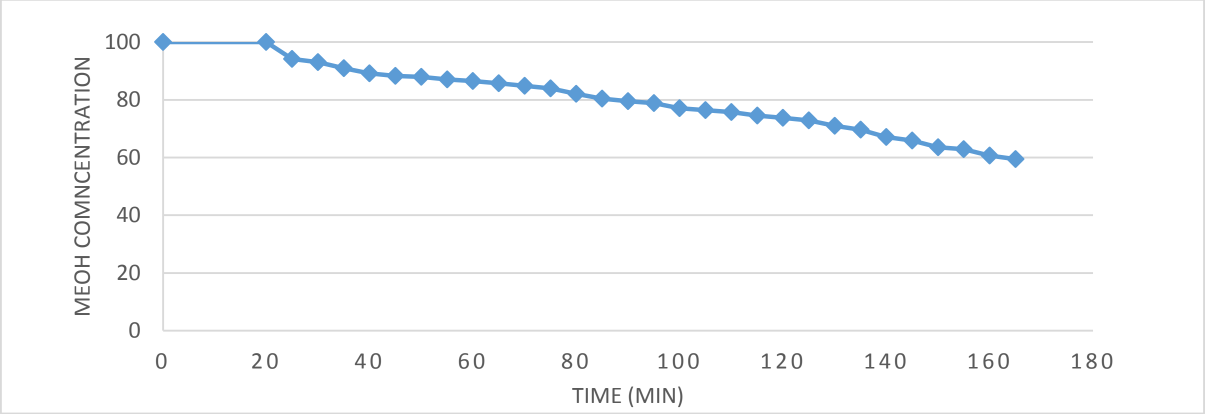


Figure 5.7 concentration profile for batch distillation with constant reflux ratio (R=6)

From figure 5.7 we can see the change of x_D when the value of reflux ratio =6, also we can see that its need to 165 min to obtain 400 ml of methanol that had concentration $x_D = 79.6\%$

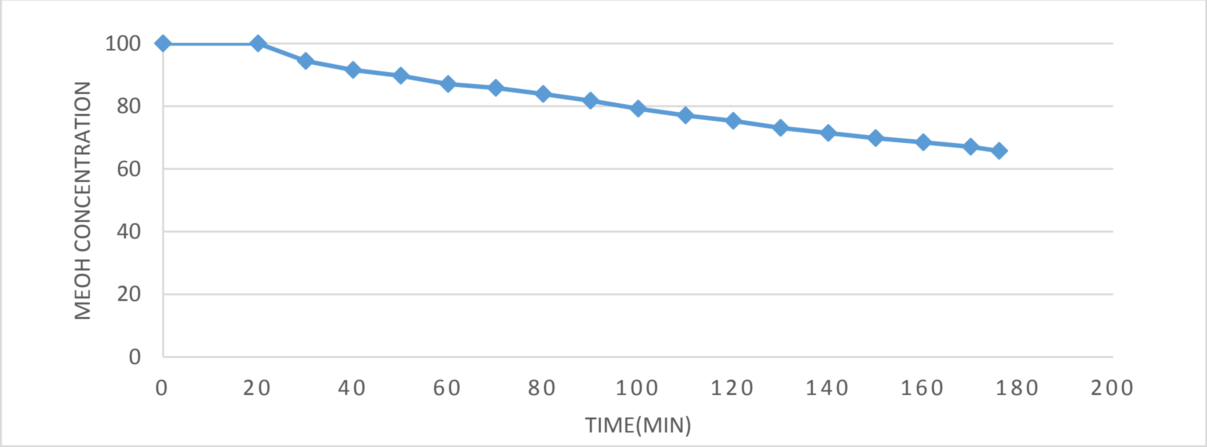


Figure 5.8 concentration profile for batch distillation with constant reflux ratio (R=7)

Figure 5.8 shows the change of x_D when the value of reflux ratio =7, also its need to 176 min to obtain 400 ml of methanol that had concentration $x_D = 81.55\%$

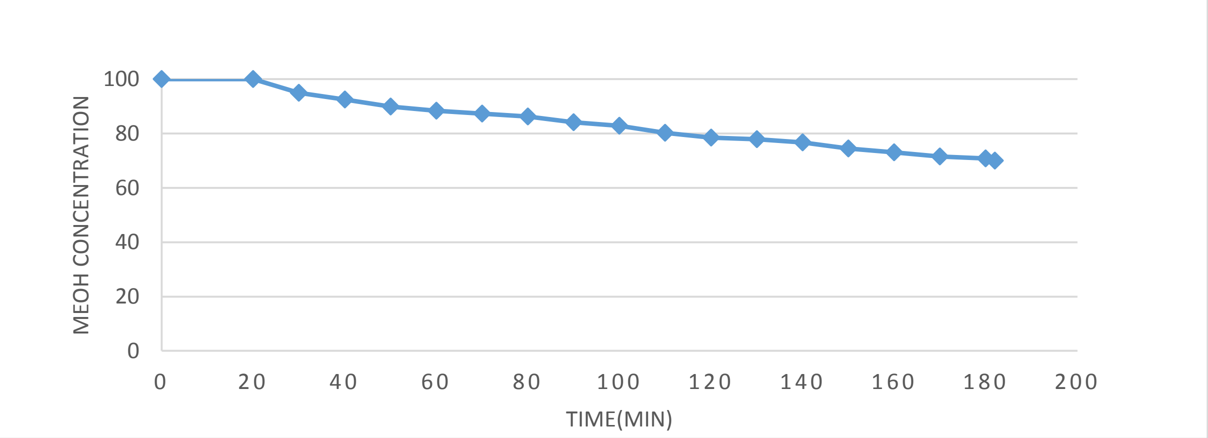


Figure 5.9 concentration profile for the batch distillation with constant reflux ratio (R=8)

figure 5.9 shows the change of x_D when the value of reflux ratio =8, also from figure we can see that its need to 182 min to obtain 400 ml of methanol that had concentration $x_D = 83.4\%$

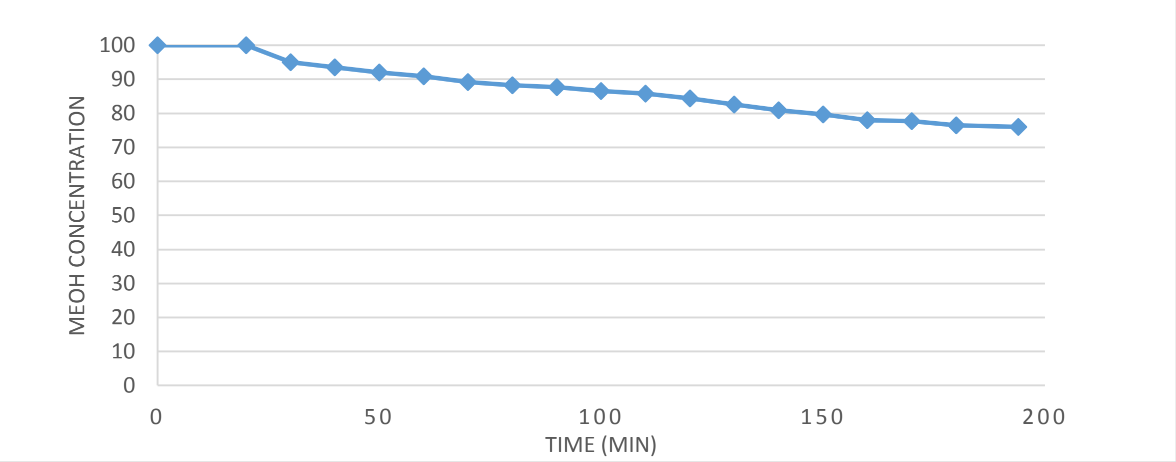


Figure 5.10 concentration profile for the batch distillation with constant reflux ratio (R=10)

From figure 5.10 can be seen the change of x_D when the value of reflux ratio =10, so from figure can see that its need to 194 min to obtain 400 ml of methanol that had concentration $x_D = 89.6\%$

Figures above show the concentration profiles for methanol along the top plate (distillate) for constant reflux ratio in different range and constant heat duty of 288W. After 20 minutes the column has been stabilized (total reflux). The temperature profiles agree with concentration profile along the column, and the distillate concentration is 100% this indicate that pure methanol is obtained. After this time concentration of methanol start to decrease and mixture enriched in water are obtained. So experiments shows that it need more time to collect 400 ml of methanol when reflux ratio is increased while in the same time higher purity of methanol is obtained.

Table 5.1 summarized the experimental results obtained with fixed heat duty of 288 W to collect 400 ml of distillate. When the reflux ratio increase, the time of the experiment increase and also the concentration of methanol obtained increase. While distillate flow rate decreases with time.

From table 5.1, it is clear that at higher reflux ratio can get a higher distillate concentration of methanol but the time required to get the 400 ml of distillate will be higher too. It can be seen that the experiment at R=10 permits the maximum recovery of methanol concentration which is uneconomical aspect.

5.1 Table experimental results for batch distillation, at constant reflux ratio.

Reflux ratio R	Time (min) to collect 400 ml of distillate	% MEOH concentration In distillate
10	194	89.6
8	182	83.4
7	176	81.55
6	165	79.6
5	158	77.1
4	150	75.2
3	143	73.8
2	135	72
1.5	120	70.35
0.5	85	65

Fig. 5.11 represent a comparison between different reflux ratios. The time for total reflux (20 minutes) in beginning of each experiment was subtracting in the figure.

.

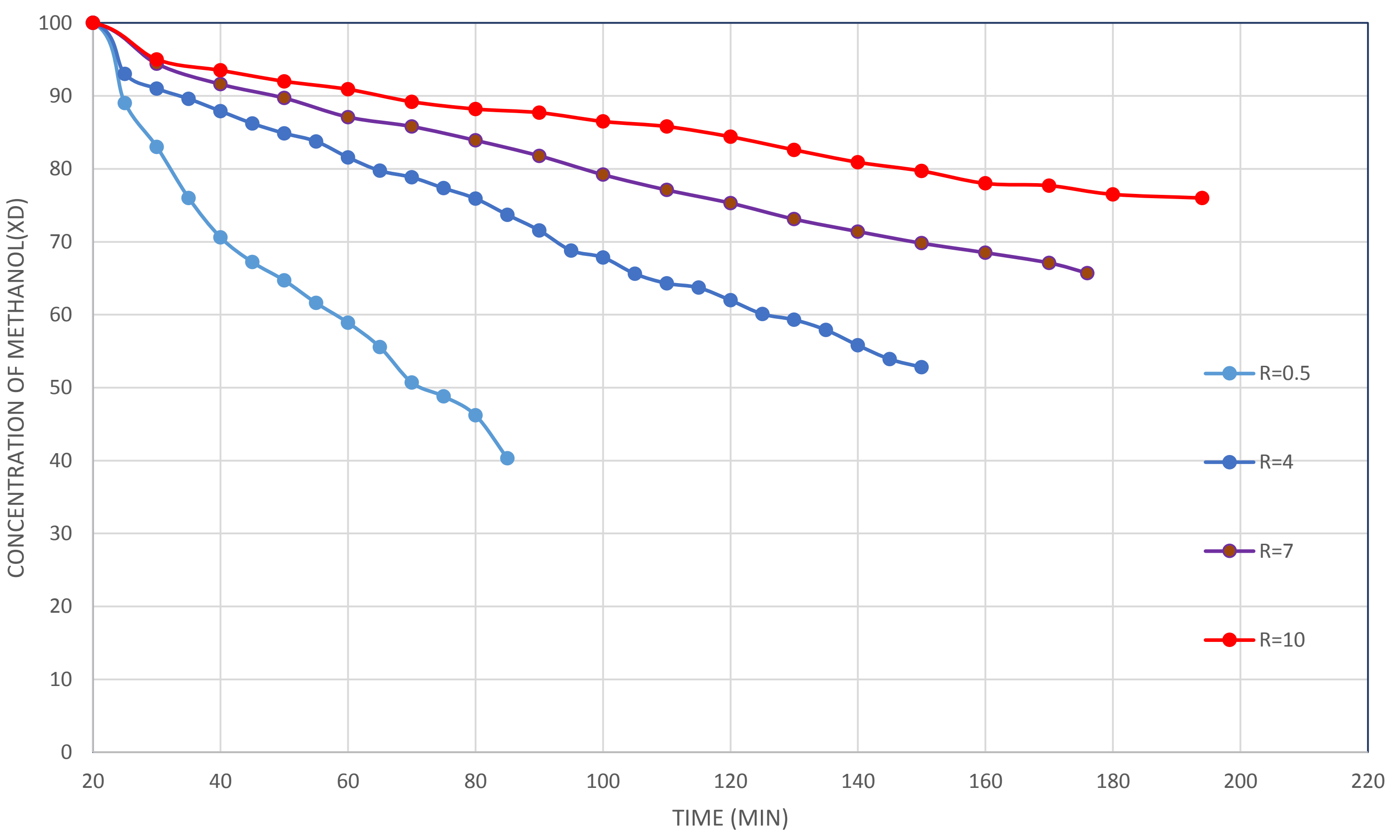


Figure 5.11 Experimental results with a constant reflux ratio experiment. Total reflux period = 20 min

5.2 Constant distillate composition

The constructed PC-based controller (LabVIEW) is implemented with the batch distillation system to control the distillate concentration. This is achieved by changing the reflux ratio as manipulating variable to control on temperature which is represent a (concentration) of the distillate (top plate).

5.2.1 Dynamic behavior for implemented model

It is necessary to identify model parameters from experimental data. The simplest approach involves introducing a step change test into the process and recording the response of the process. Then open-loop test for step changes in reflux ratio are applied 0-2, 2-4, and 4-6.

The response of the top distillate concentration is shown in figure 5.12. It has been recognized that a first order may in general represent process dynamics of batch distillation. The calculations are carried further to estimate the steady-state gain and the process time constant. Figures 5.13 shows the open-loop test for the three step changes in manipulating variable reflux ratio from 0 to 6.

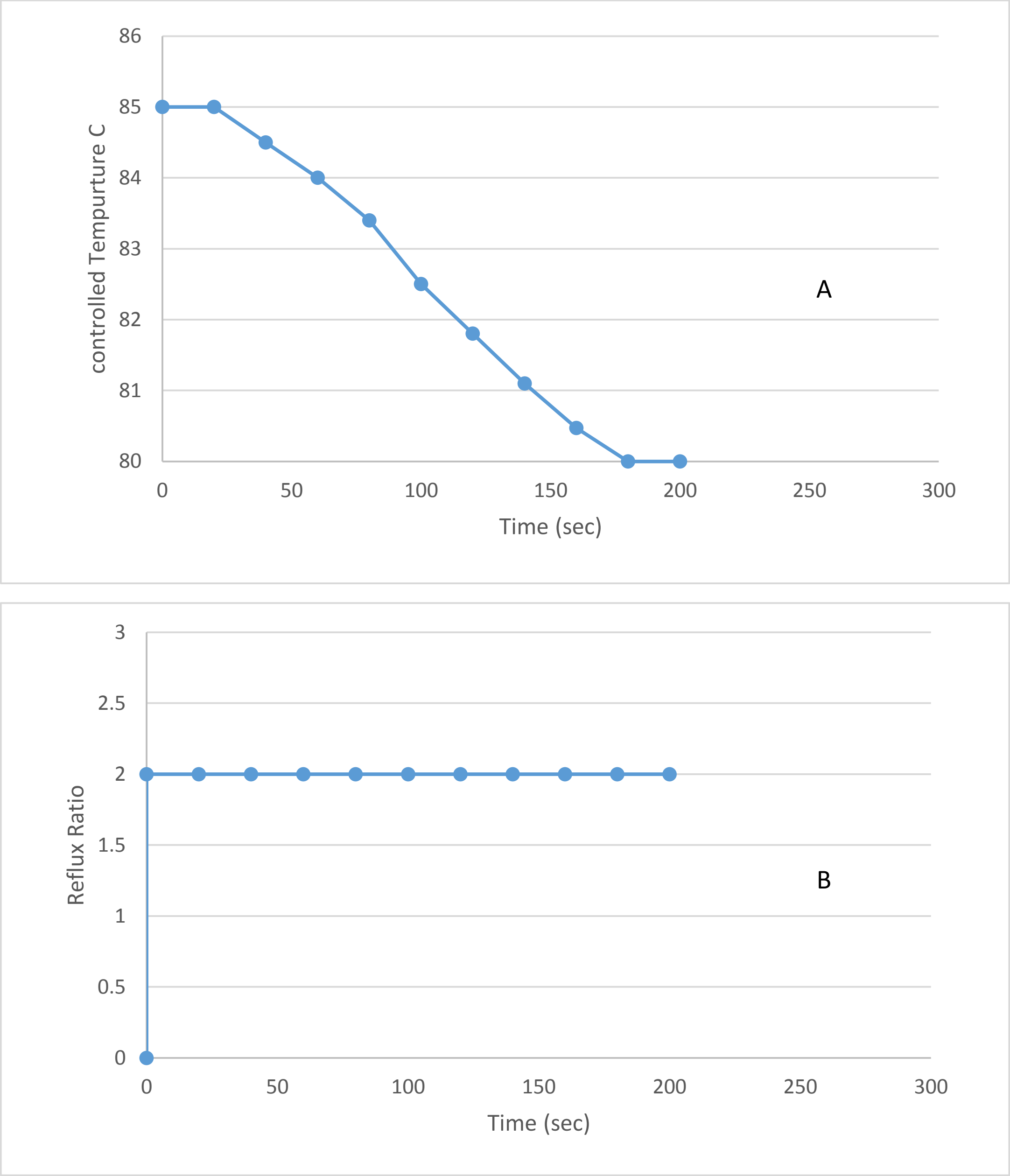


Figure 5.12 A and B response of the top distillate temperature to step change for open loop test for manipulating reflux ratio 0-2.

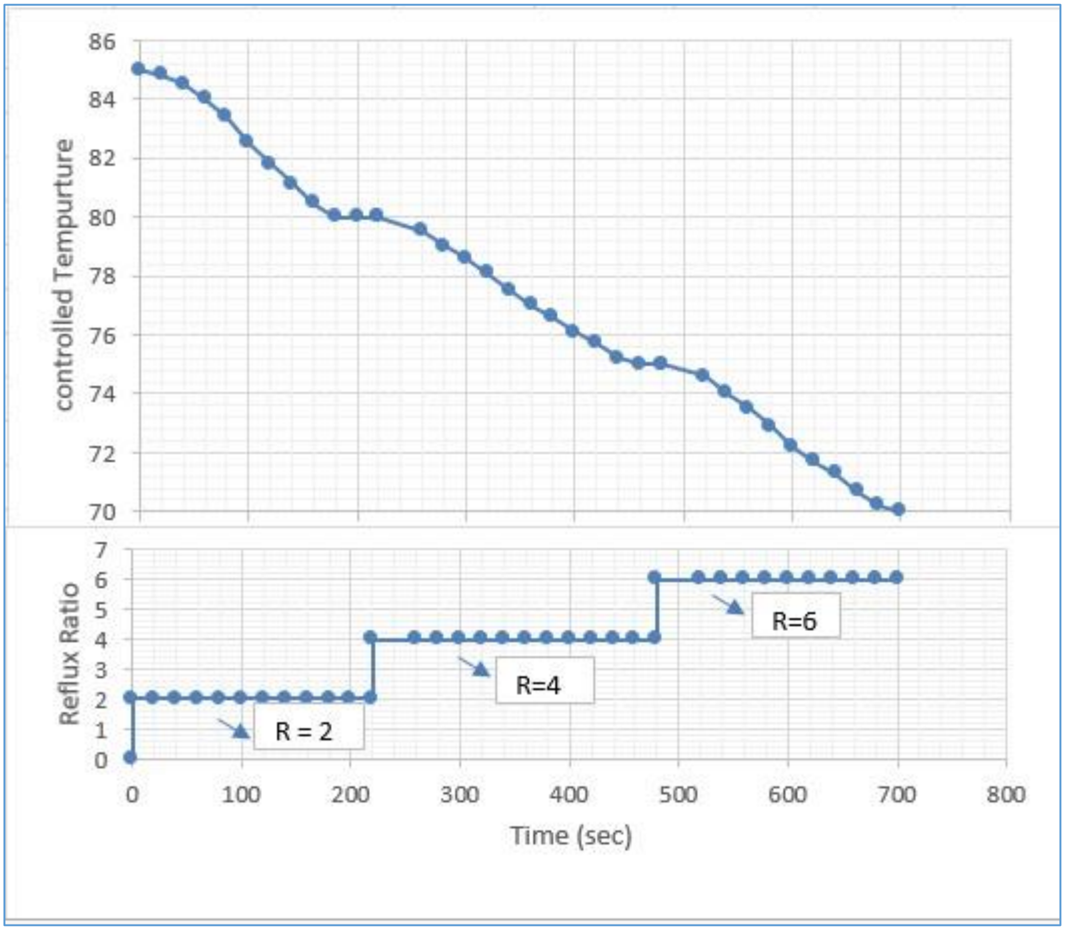


Figure 5.13. Open-loop test for the three step changes in manipulating variable reflux ratio from 0 to 6

Using the experimental data in figure 5.13, transfer function and PID controller are designed to maintain composition specifications for the distillate product taking the reflux flow rate as manipulated variable.

The transfer function of first order model can be represented by [Seborg, Edgar and Mellichamp, 2003]:

$$G_P = \frac{K}{\tau s+1} \tag{5.1}$$

$$\Delta x = G_P \Delta R \tag{5.2}$$

For step change response

$$\Delta R = \frac{m}{s} \tag{5.3}$$

$$\Delta x = \frac{K}{\tau s+1} * \frac{m}{s} \tag{5.4}$$

$$\dot{x} = \left(1 - e^{-\frac{t}{\tau}}\right) K\, m \tag{5.5}$$

When $t = 5\tau$

$$\dot{x} = 0.99Km \tag{5.6}$$

The simplest input/output model that retain this dynamic characteristic of batch distillation processes is an integrating process with a process gain

$$\Delta x = K\Delta R \tag{5.7}$$

In principle, the process gain Kcan be obtained as

$$K = \frac{\partial x}{\partial R} = \frac{\partial}{\partial t}(\frac{\partial x}{\partial R}) \tag{5.8}$$

$K = 2.5$, $\tau_{av} = 46.67\ sec$

Transfer function of the process can be deduced as follow:

$$G_P = \frac{2.5}{46.67\ S+1} \tag{5.9}$$

Transfer function of measurement (sensor) and actuator (solenoid valve) were found experimentally:

$$G_M = \frac{K_M}{\tau_m S+1} = \frac{1}{3\,S+1} \tag{5.10}$$

$$G_V = K_V = 0.25 \tag{5.11}$$

5.2.2 Analyze the transfer function by using frequency response (open loop system)

In designing a closed-loop system, the frequency-response characteristic of the open-loop transfer function adjusted by using several design criteria in order to obtain acceptable transient-response characteristics for the system [Richard and Robert, 2001].

Analysis the system by bode plot diagram depended on the gain margin and phase margin and from that the stability of the system can be test.

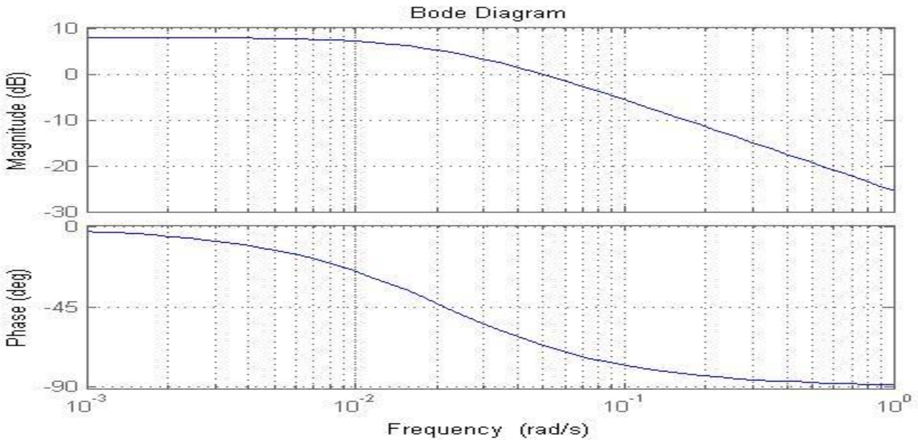


Figure 5.14 Frequency response of overhead composition to reflux ratio

From figure 5.14 can be seen that the open loop part is unstable because the phase margin is positive and the gain margin is negative (phase margin =114 degree and gain crossover frequency =0.0491 rad/sec). So the relation between reflux ratio and overhead composition is unstable and any change in reflux ratio will effect on composition and the system will be unstable.

5.2.3 The closed loop transfer function in frequency domain dynamics

Frequency response based on converting system differential equation to transfer function. Thus, generating mathematical model of the system that algebraically relates representation of the output to the input, replacing the differential equation with an algebraic equation not only simplifies the representation of individual subsystem but also simplifies modeling interconnected subsystems [Norman, 2000].

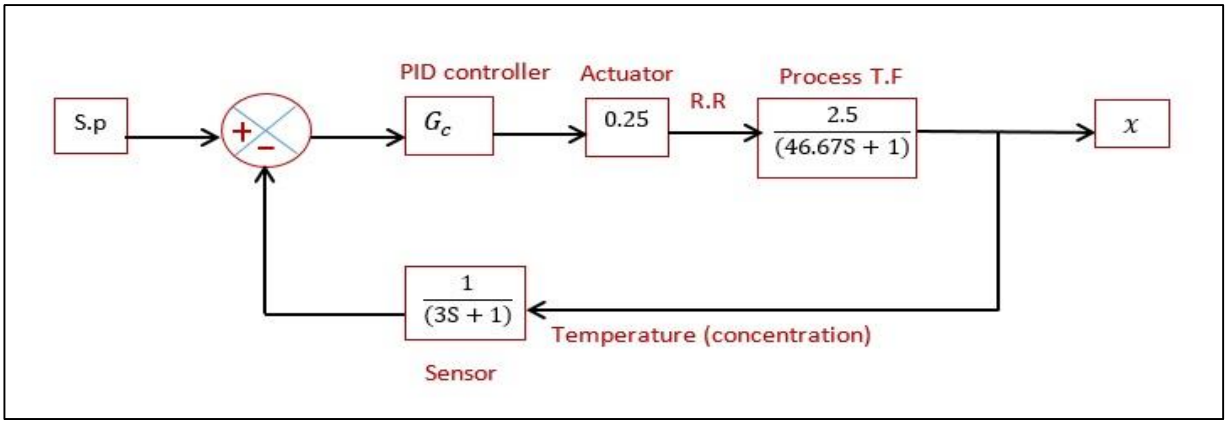


Figure 5.15 Closed control loop for the implemented system

The control system shown in Figure 5.15 in which a PID controller is used to control the system. The PID controller has the transfer function:

$$G_c(s) = k_p[1 + \frac{1}{\tau_I s} + \tau_D s] \tag{5.12}$$

Although many analytical methods are available for the design of a PID controller for the present system, Ziegler-Nichols tuning rules can, of course, be applied to process whose dynamics are known. (If the plant dynamics are known, many analytical and graphical approaches to the design of PID controllers are available, in addition to Ziegler-Nichols tuning rules), applying a Ziegler-Nichols tuning rule for the determination of the values of parameters k_p,τ_I and τ_D. Then obtain a unit-step response curve and check to see if the designed system exhibits approximately 25% maximum overshoot. If the maximum overshoot is excessive (40% or more), make a fine tuning and reduce the amount of the maximum overshoot to approximately 25% or less. Since the process has an integrator, we use the second method of Ziegler-Nichols tuning rules. By setting $\tau_I = \infty$ and τ_D =0. The closed-loop transfer function obtained as follows:

$$\frac{x_{(s)}}{R_{(s)}} = \frac{K_M G_C G_V G_P}{1+G_M G_C G_V G_P} \tag{5.13}$$

The value of K_P, that makes the system marginally stable so that sustained oscillation occurs can be obtained by use of Routh's stability criterion. Since the characteristic equation for the closed-loop system is:

$$1 + G_M G_C G_V G_P = 0 \tag{5.14}$$

$$14\,S^3 + 46.67\,S^2 + S + 0.625\,K_P = 0 \tag{5.15}$$

The Routh array becomes as follows:

$$\begin{array}{lll} S^3 & 14 & 1 \\ S^2 & 46.67 & 0.625\ K_P \\ S^1 & \frac{46.67-14*\ 0.625\ K_P}{46.67} & \\ S^0 & 0.625\ K_P & \end{array}$$

Examining the coefficients of the first column of the Routh table, can be found that sustained oscillation will occur if $K_P = 5.6765$. Thus, the critical gain K_{cr}, is

$$K_{cr} = 5.6765 \tag{5.16}$$

With the gain K_P, set equal to Kc,$(= 5.6765)$, the characteristic equation becomes:

$$14\ S^3 + 46.67\ S^2 + S + 3.547 = 0 \tag{5.17}$$

To find the frequency of the sustained oscillation, we substitute s = jw into this characteristic equation as follows:

$$14\ (jw)^3 - 46.67\ w^2 + jw + 3.547 = 0 \tag{5.17}$$

$$3.547 - 46.67\ w^2 = 0 \tag{5.18}$$

$$j(w - 14\ w^3) = 0 \tag{5.19}$$

From which can be found the frequency of the sustained oscillation to be $w = 0.26726$. Hence, the period of sustained oscillation is:

$$P_{cr} = \frac{2\pi}{w} = 23.509 \tag{5.20}$$

Referring to Table 3-2, determine k_p, τ_I and τ_D as follows:

$$k_p = 0.6\, K_{cr} = 3.40536 \tag{5.21}$$

$$\tau_I = 0.5\, P_{cr} = 11.754 \tag{5.22}$$

$$\tau_D = 0.124\, P_{cr} = 5.877 \tag{5.23}$$

The transfer function of the PID controller is thus

$$G_c(s) = 3.40536[1 + \frac{1}{11.754\, s} + 5.877\, s] \tag{5.24}$$

$$= \frac{20.01}{s}[0.17s + 0.0144 + s^2] \tag{5.25}$$

Next, let us examine the unit-step response of the system. The closed-loop transfer function $\frac{x_{(s)}}{R_{(s)}}$ is given by

$$\frac{x_{(s)}}{R_{(s)}} = \frac{12.5 + 2.126\, s + 0.18}{14\, s^4 + 49.67\, s^3 + 13.5\, s^2 2.126\, s + 0.18} \tag{5.26}$$

The unit-step response of this system can be obtained with MATLAB. The resulting unit-step response curve is shown in Figure 5.16. The maximum overshoot in the unit-step response is approximately 36.4%.The amount of maximum overshoot is excessive. It can be reduced by fine tuning the controller parameters.

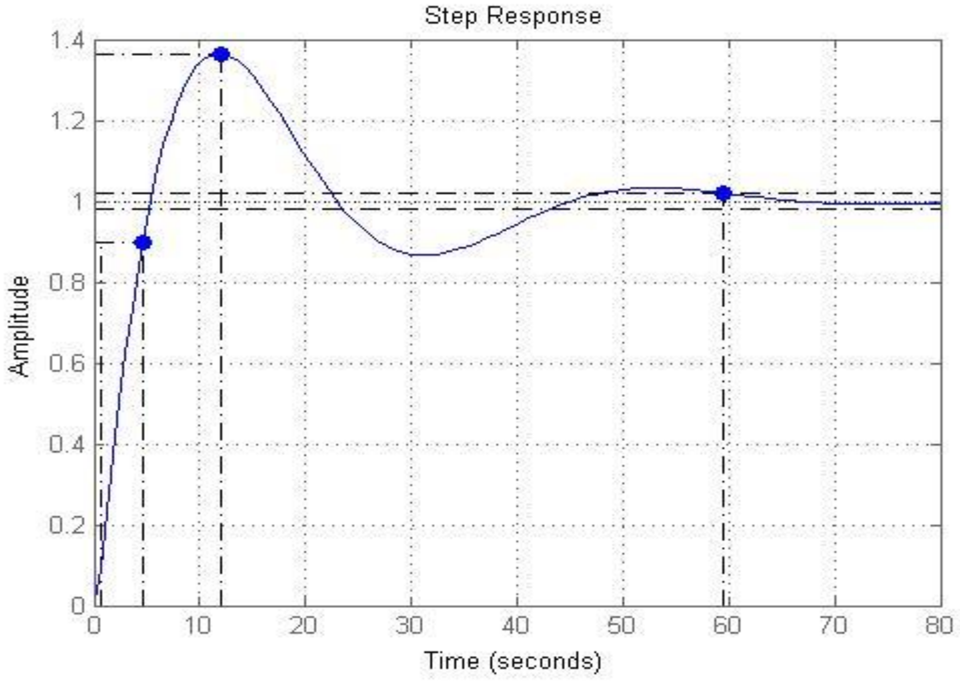


Figure 5.16 Unit-step response of the implemented system with PID controller having parameters K_P= 3.40536, τ_I= 11.754, and τ_D= 5.877.

5.3 Control on system performance using simulation

Simulation is the imitation of the operation of a real-world process or system over time. The act of simulating system first requires that a model be developed; this model represents the key characteristics or behaviors/functions of the selected physical or abstract system or process. The model represents the system itself, whereas the simulation represents the operation of the system over time [Bitter, 2007].

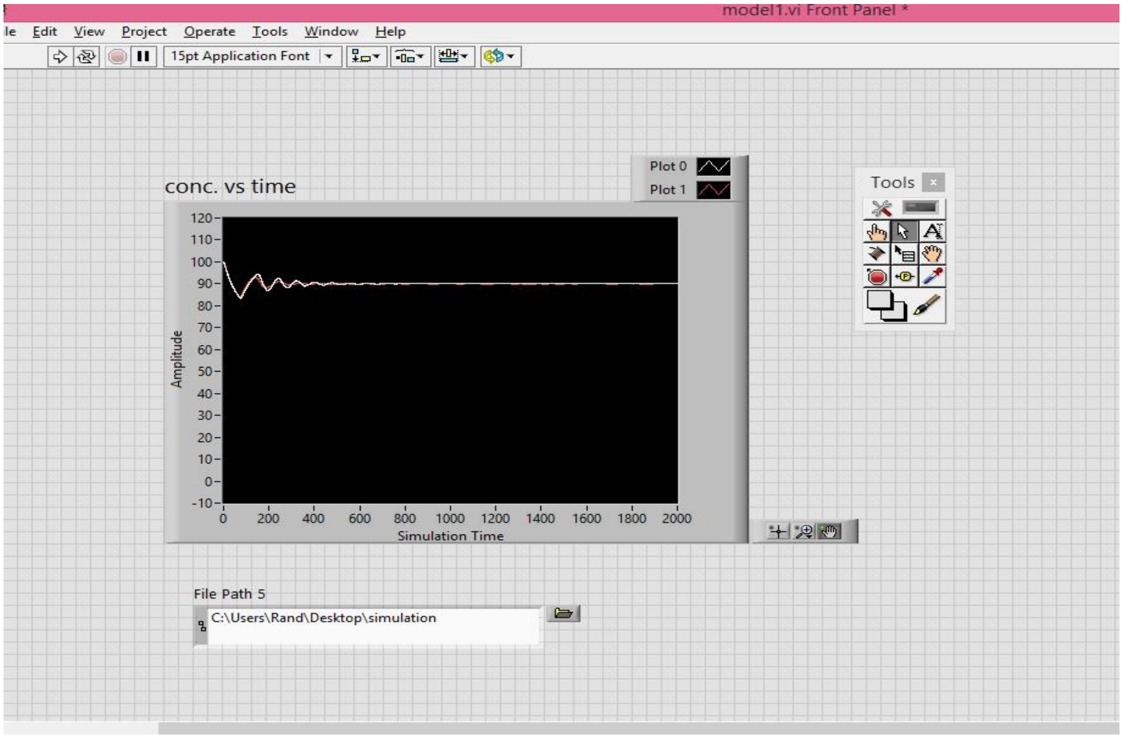
As mentioned before to control on distillate composition operation and obtained constant composition online with the experiment running, PID controller is used. To verify the optimal condition for controlling on distillate concentration optimal PID parameters should be obtained, this step done by simulation, LabVIEW –Simulation is used to simulate the system depicted in figures (5.17 and 5.18).

Although it required some guesswork and trial and error to estimate of the required parameters, we were able to simulate, at least qualitatively, the batch process described above using LabVIEW simulation.

The investigation reveals that the optimum solution is PI controller with the following parameters:

$K_c = 1\,, \qquad K_I = 5, \qquad K_D = 0$

Figures 5.19-5.27 Shows, respectively, the simulation data that correspond to reflux ratio change from simulation results by using LabVIEW simulation program.



File Path 5
C:\Users\Rand\Desktop\simulation

Figure 5.17 Front panel of the simulation for distillate concentration for batch distillation by LabView.

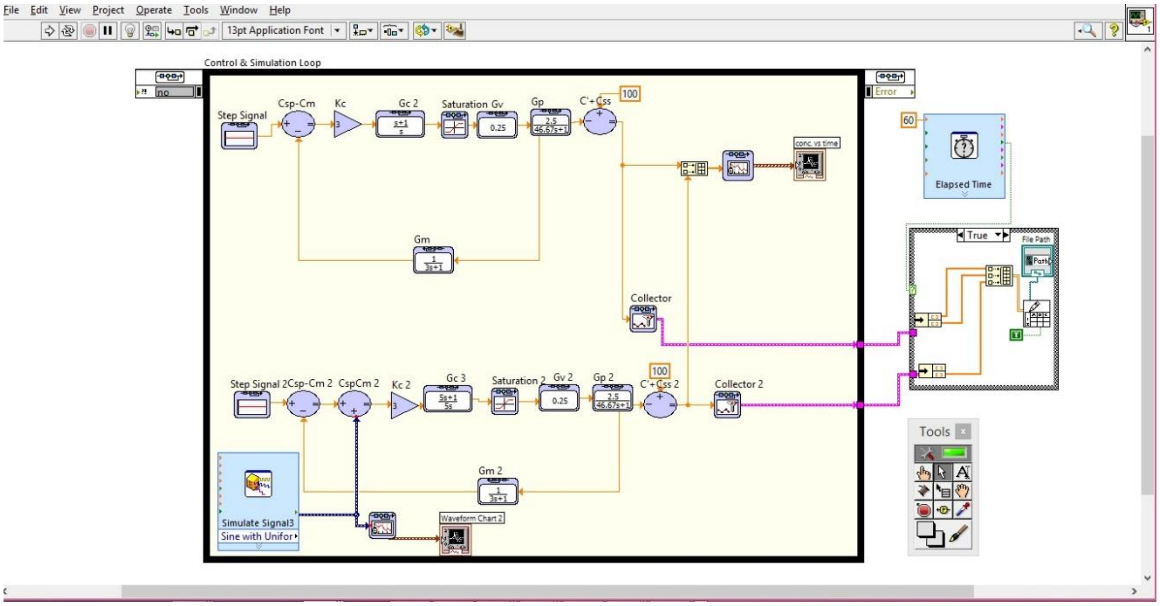


Figure 5.18 Block diagram of the simulation for distillate concentration for batch distillation by LabVIEW.

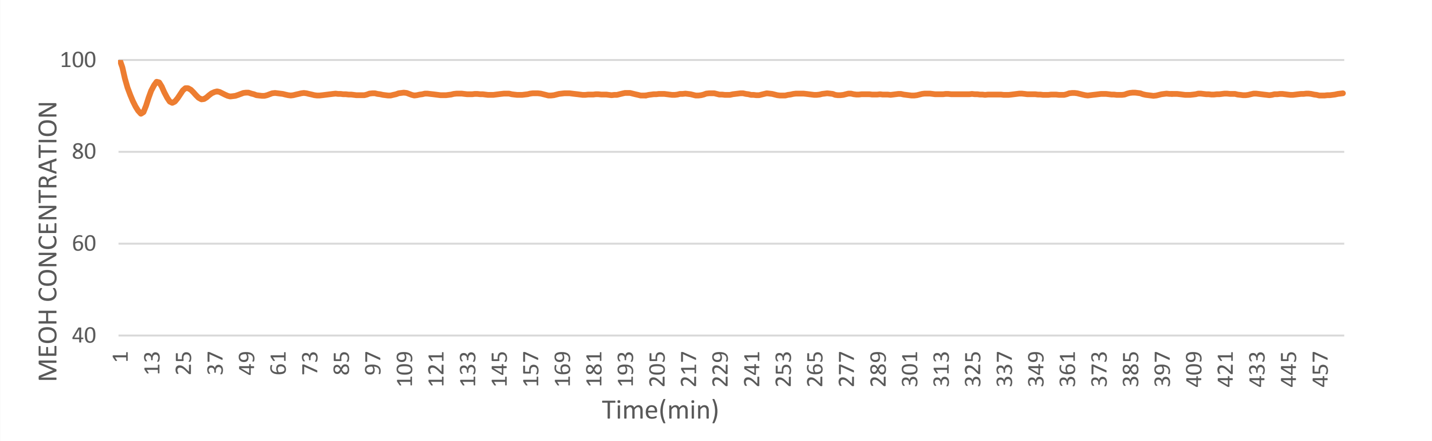


Figure 5.19 Concentration profile for Simulation with set point (95%).

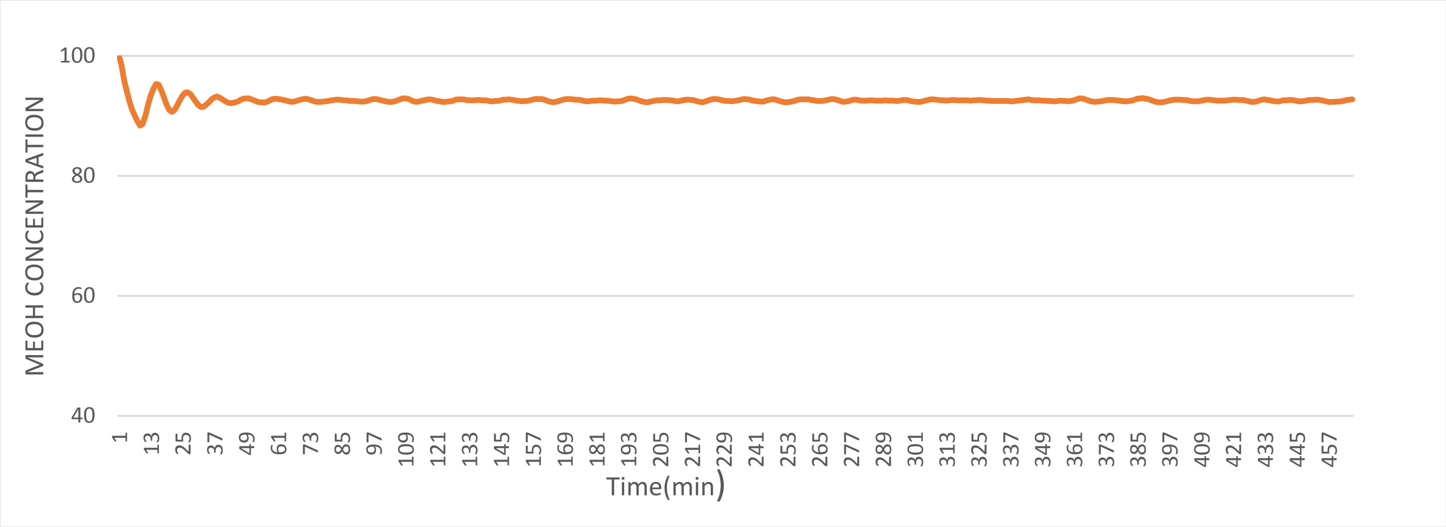


Figure 5.20 Concentration profile for Simulation results with set point (93%).

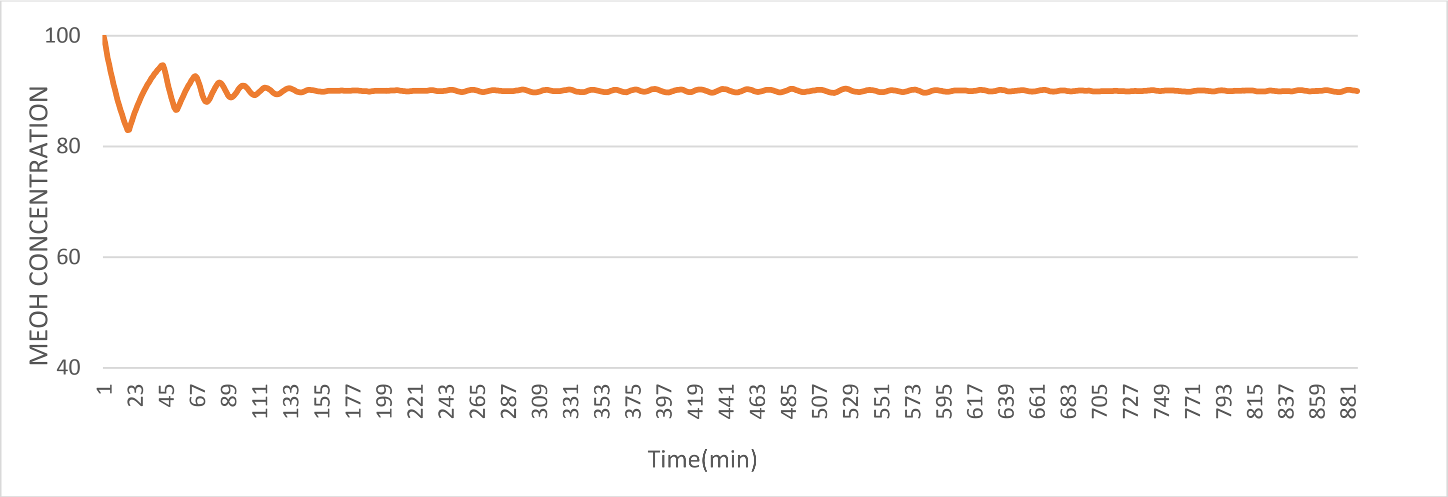


Figure 5.21 Concentration profile for Simulation with set point (90%).

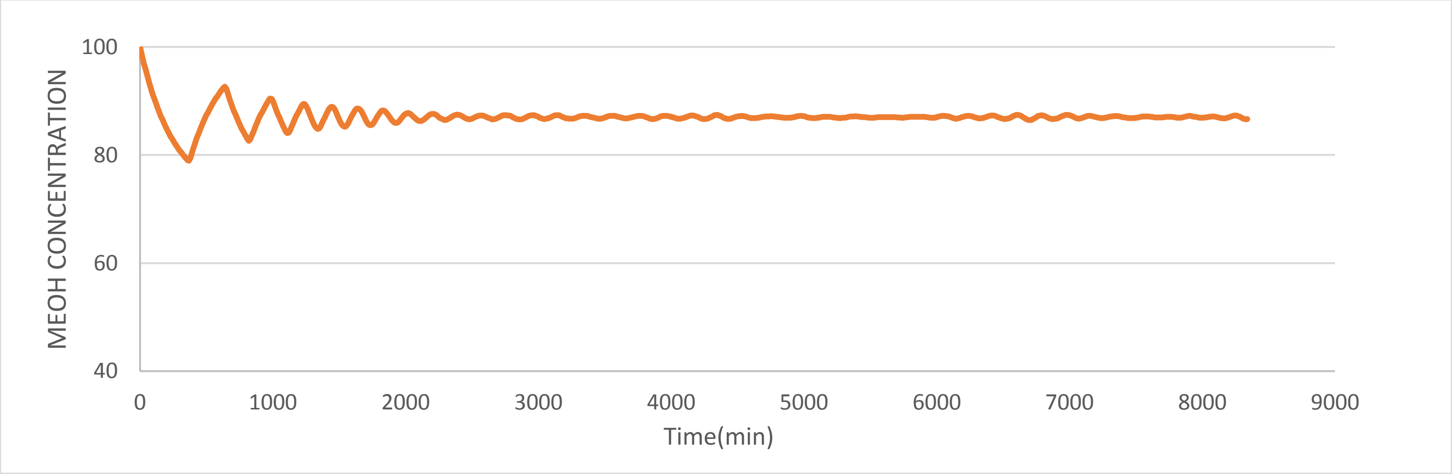


Figure 5.22 Concentration profile for Simulation with set point (87%).

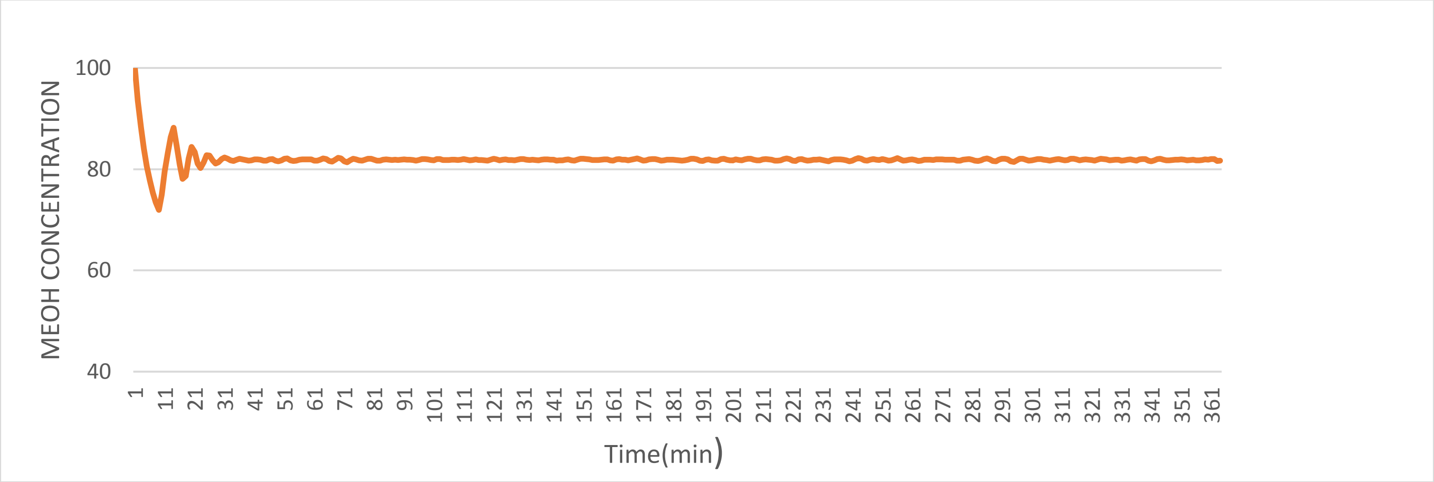


Figure 5.23 Concentration profile for Simulation with set point (82%)

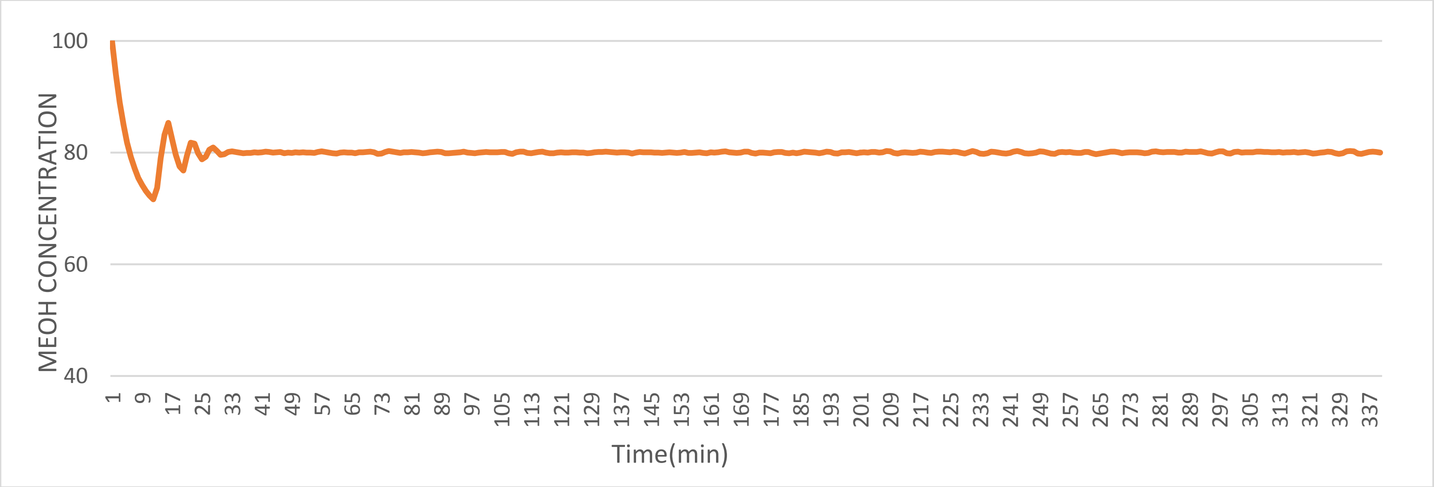


Figure 5.24 Concentration profile for Simulation with set point (80%).

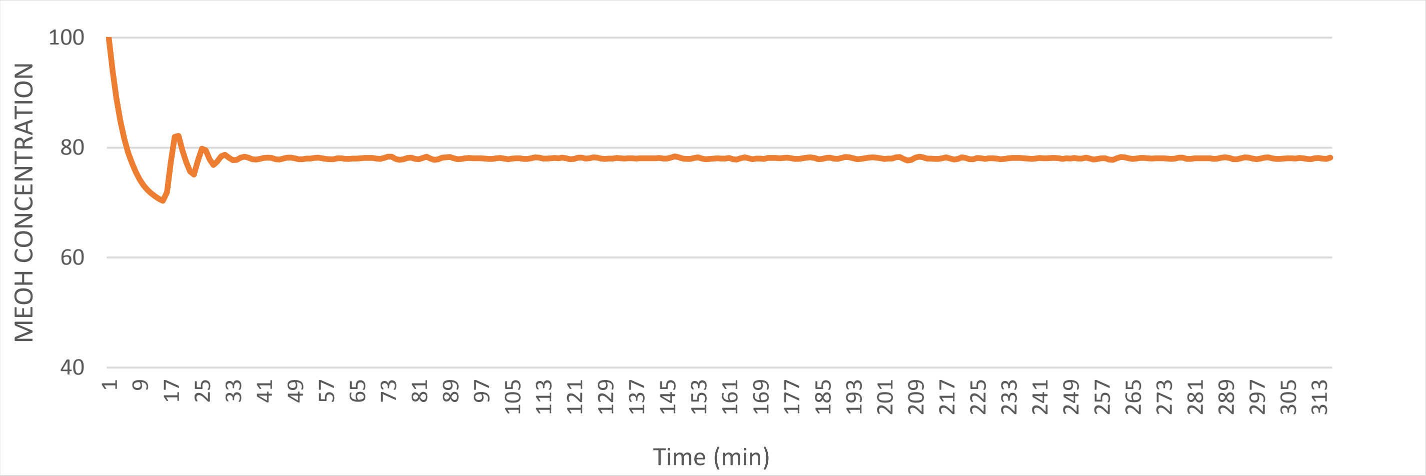


Figure 5.25 Concentration profile for Simulation set point (78%).

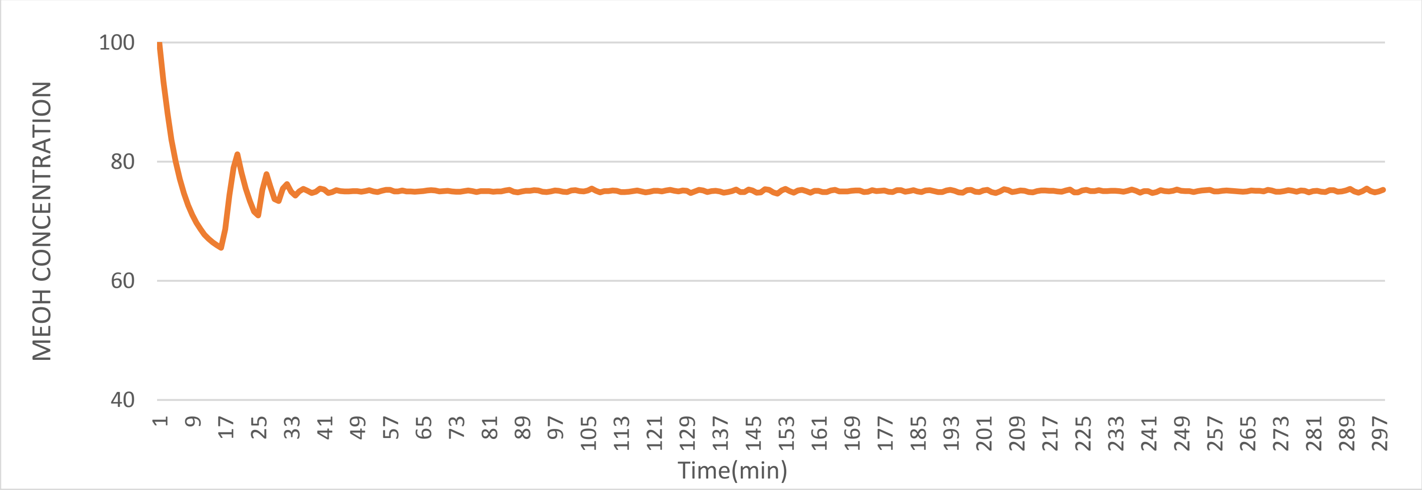


Figure 5.26 Concentration profile for Simulation with set point (75%).

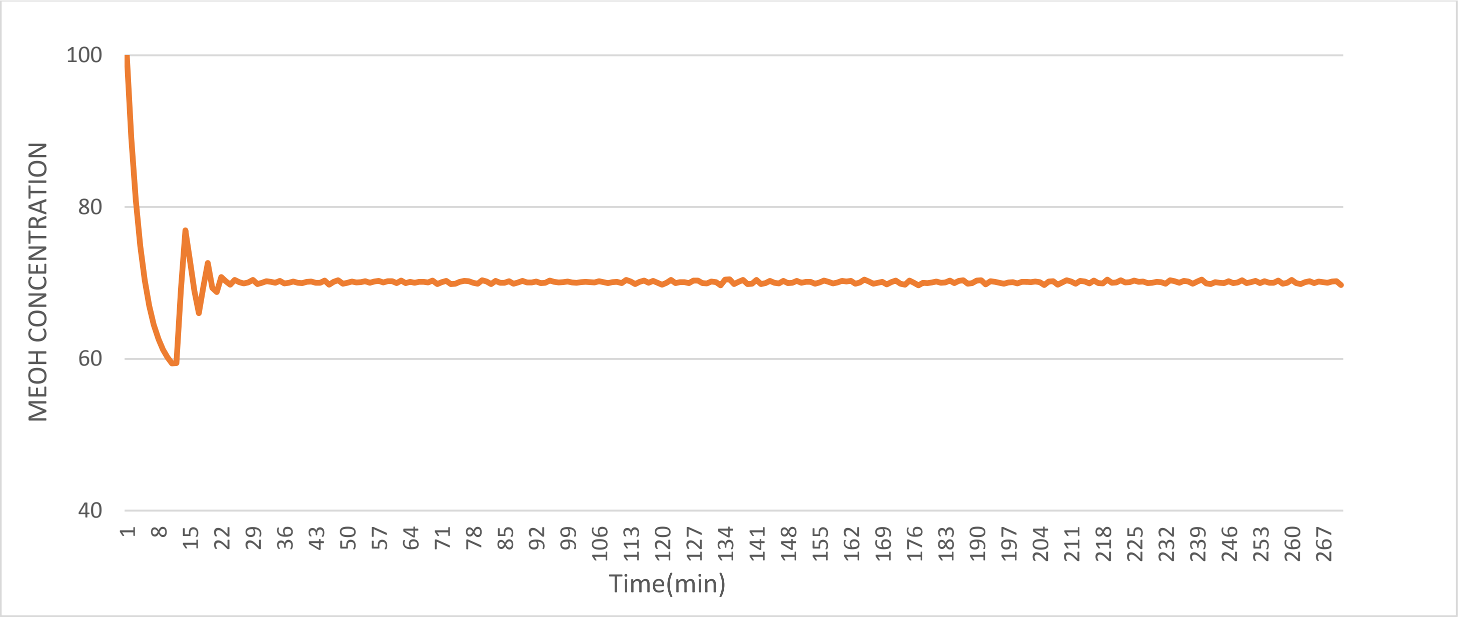


Figure 5.27 Concentration profile for Simulation with set point (70%)

All times responses for all figures from 5.19 to 5.27 are represented in table 5.2:

Table 5.2 Time responses from simulation results

Figure	Rise Time(s)	Peak Time(s)	Settling Time(s)
5.19	60	140	930
5.20	80	180	920
5.21	80	180	915
5.22	120	370	900
5.23	80	160	480
5.24	100	240	520
5.25	130	280	540
5.26	120	300	450
5.27	80	220	360

According to the simulation result K_p is set at 1 and $\tau_I = 5$ while $\tau_D = 0$, batch distillation column experiments were runs of methanol/water using the implemented PID model for the control strategy in this batch run K_p is taken as 1, $\tau_I = 5$ and $\tau_D = 0$, the minimum and maximum limit for reflux ratio is set at 0.5 and 10.

The concentration profile of the top tray and the reflux ratio profile of batch distillation experiments are illustrated in Figures 5.28-5-37. From these figures can see that the changing of the top tray composition is kept within 4%, which infers that, the top tray composition, the main control target is also kept within a desired narrow range.

Clearly, although the temperature is maintained to the set point, the distillation composition drifts away from the specification. This is because the temperature set point is chosen in such a way that the composition specification is met from the early steps of the operation; that is, at relatively law values of reflux ratio. However, as long as the reflux ratio increases, the fractionating capacity of the column increases and therefore the temperature set point should be increased in order to achieve the same distillate composition. If the set point is not increased, at the end of the batch less product is actually obtained, because the product is purer than required.

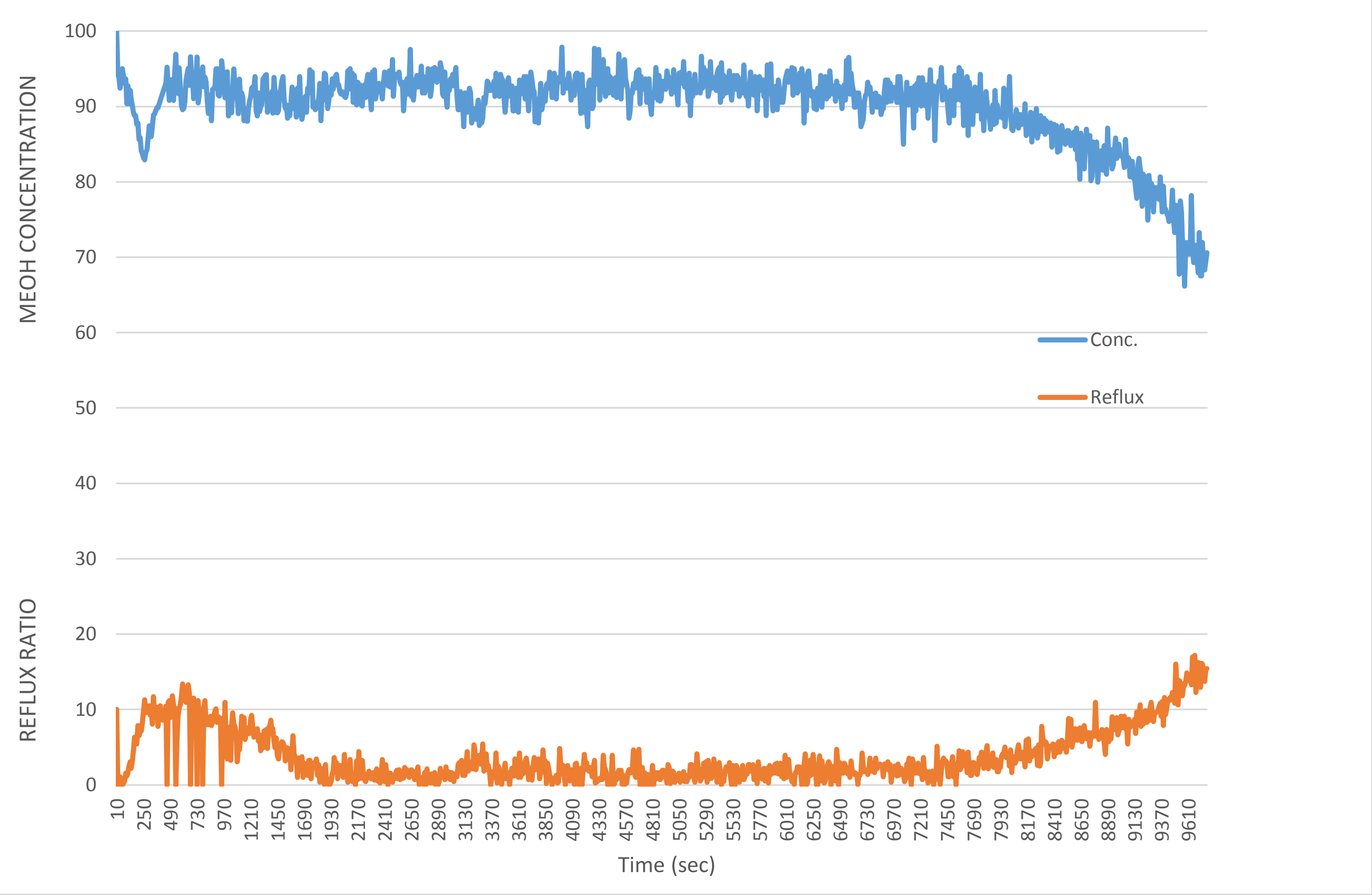


Figure 5.28 concentration profile and reflux ratio profile for experimental results with set point (95%).

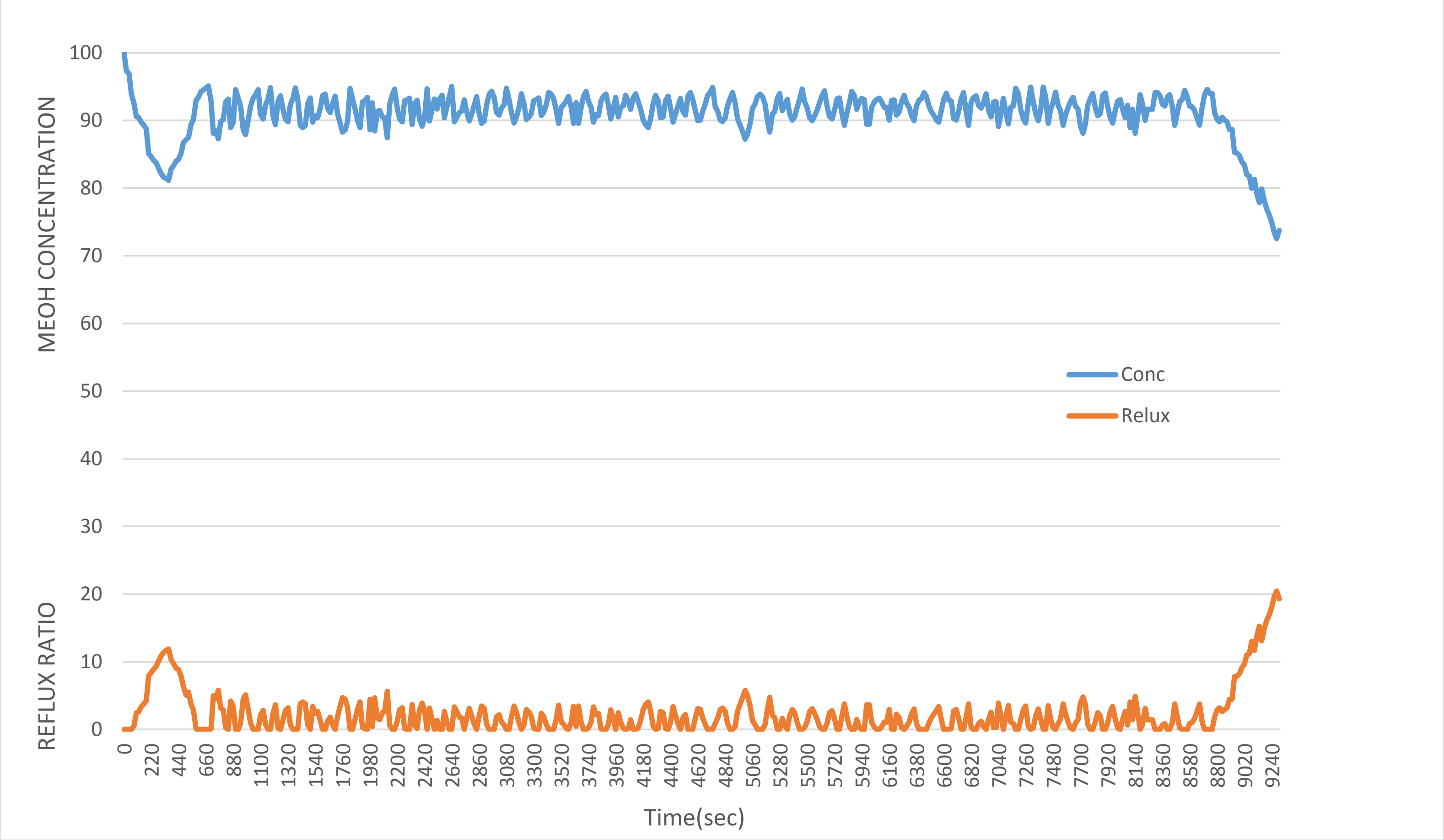


Figure 5.29 concentration profile and reflux ratio profile for experimental results with set point (93%)

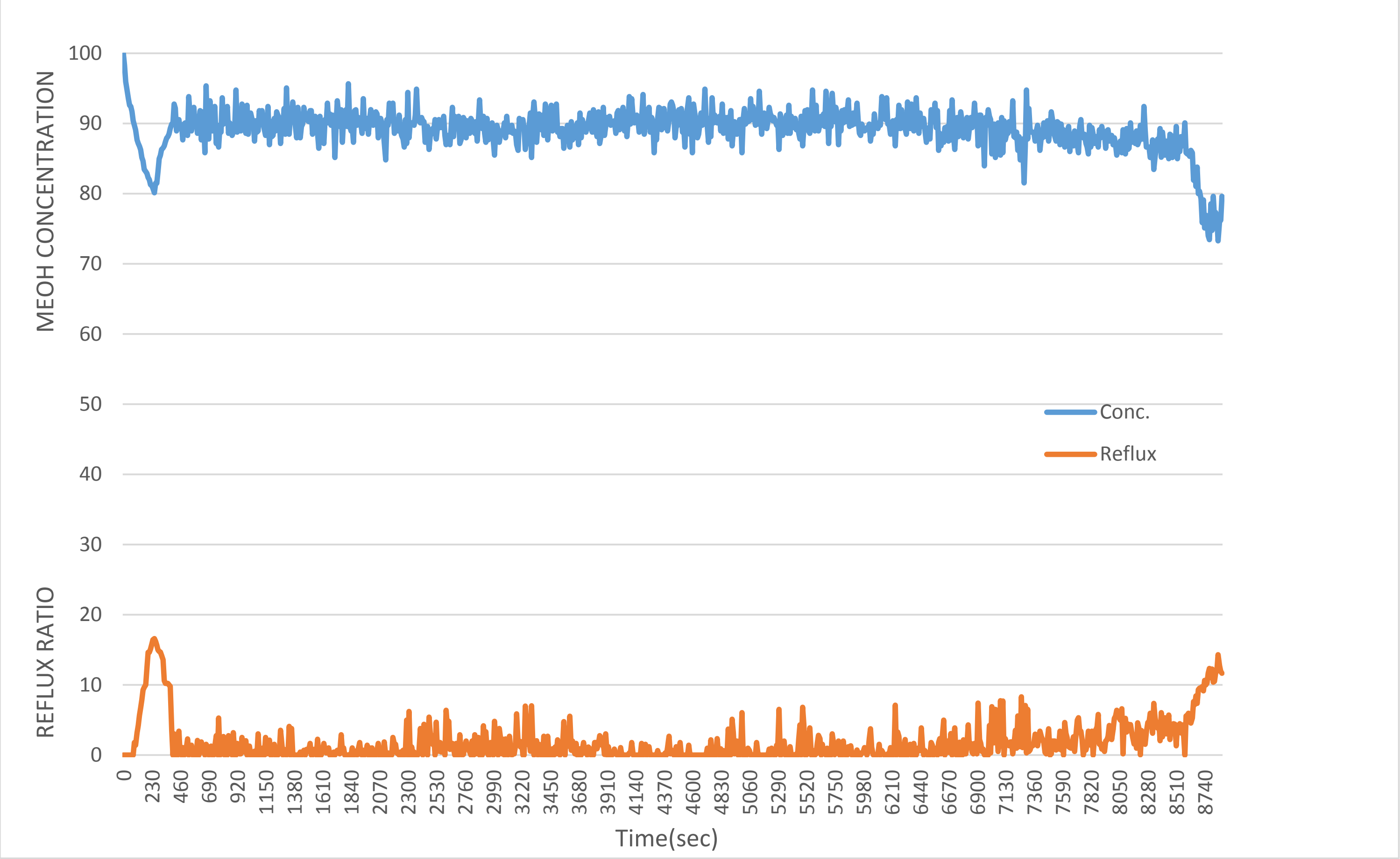


Figure 5.30 concentration profile and reflux ratio profile for experimental results with set point (90%)

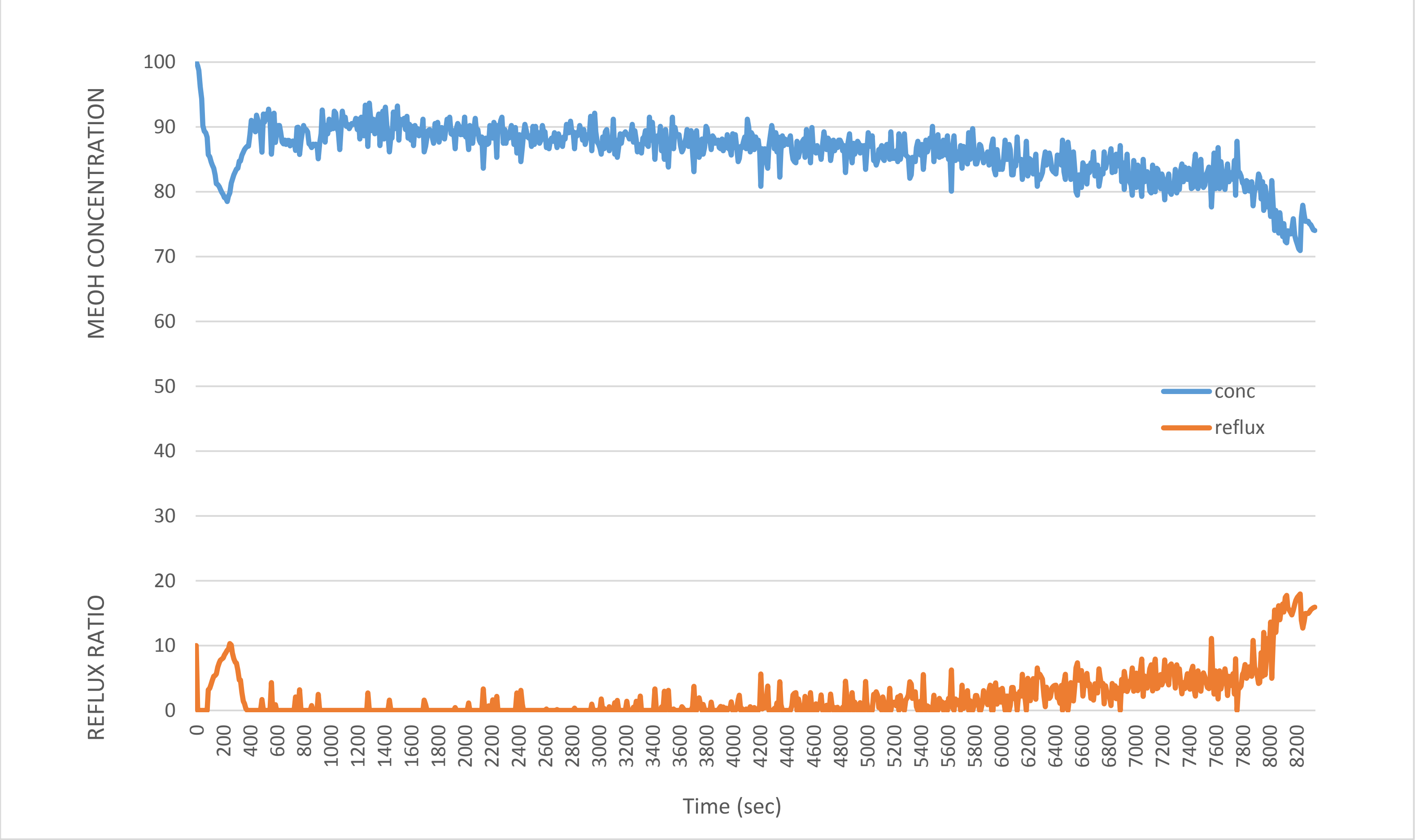


Figure 5.31 concentration profile and reflux ratio profile for experimental results with set point (87%)

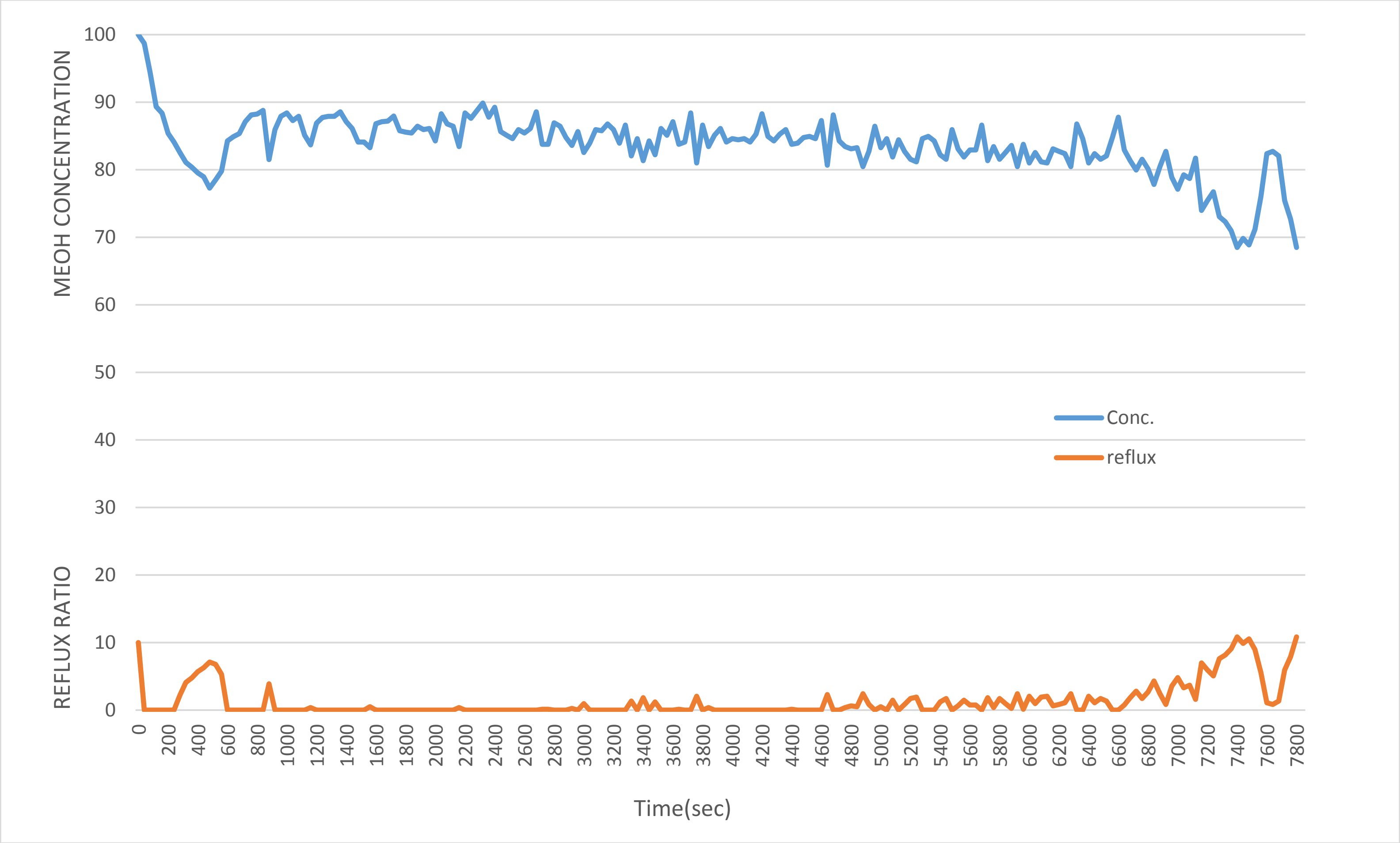


Figure 5.32 concentration profile and reflux ratio profile for experimental results with set point (84%)

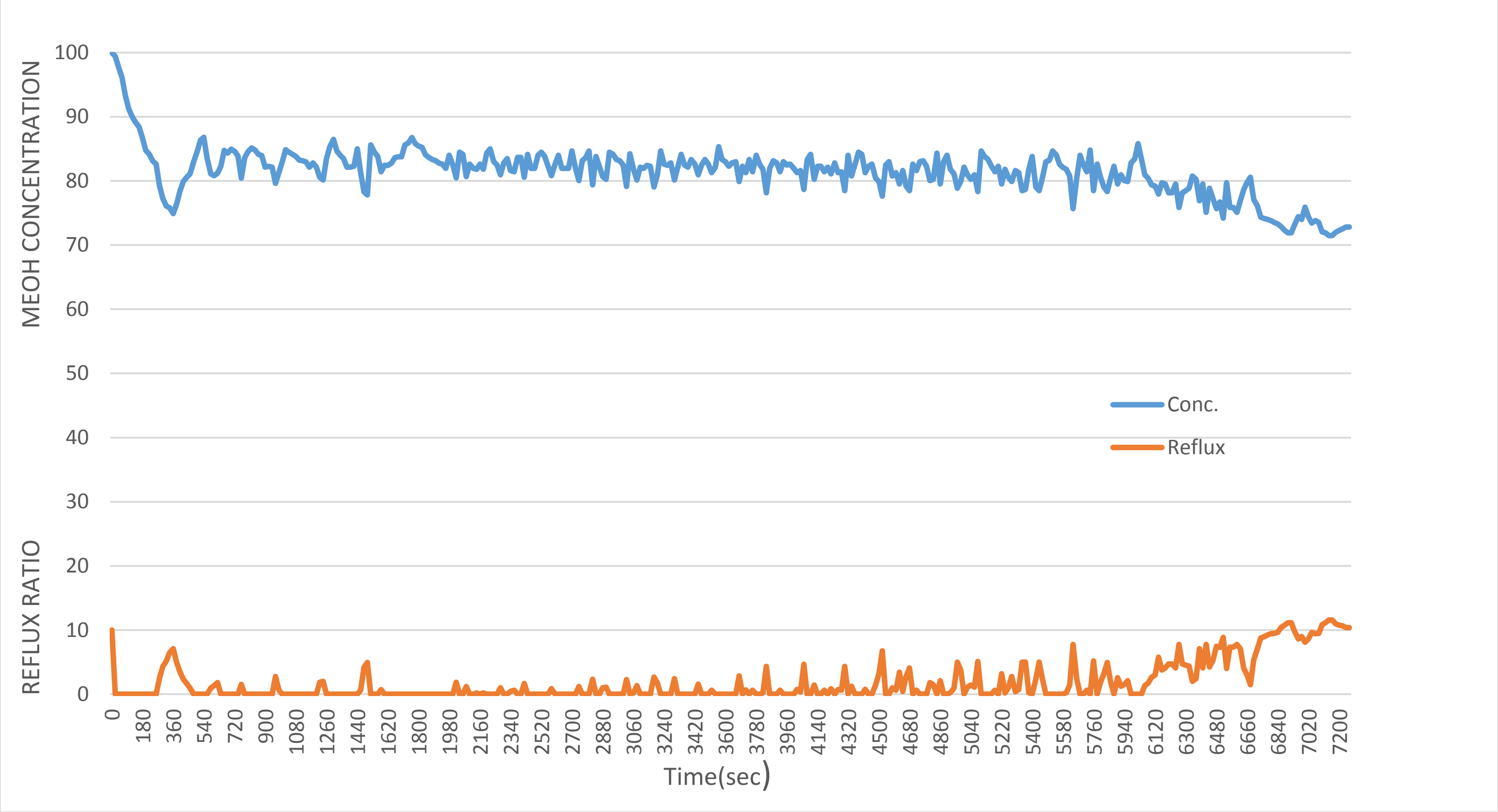


Figure 5.33 concentration profile and reflux ratio profile for experimental results with set point (82%)

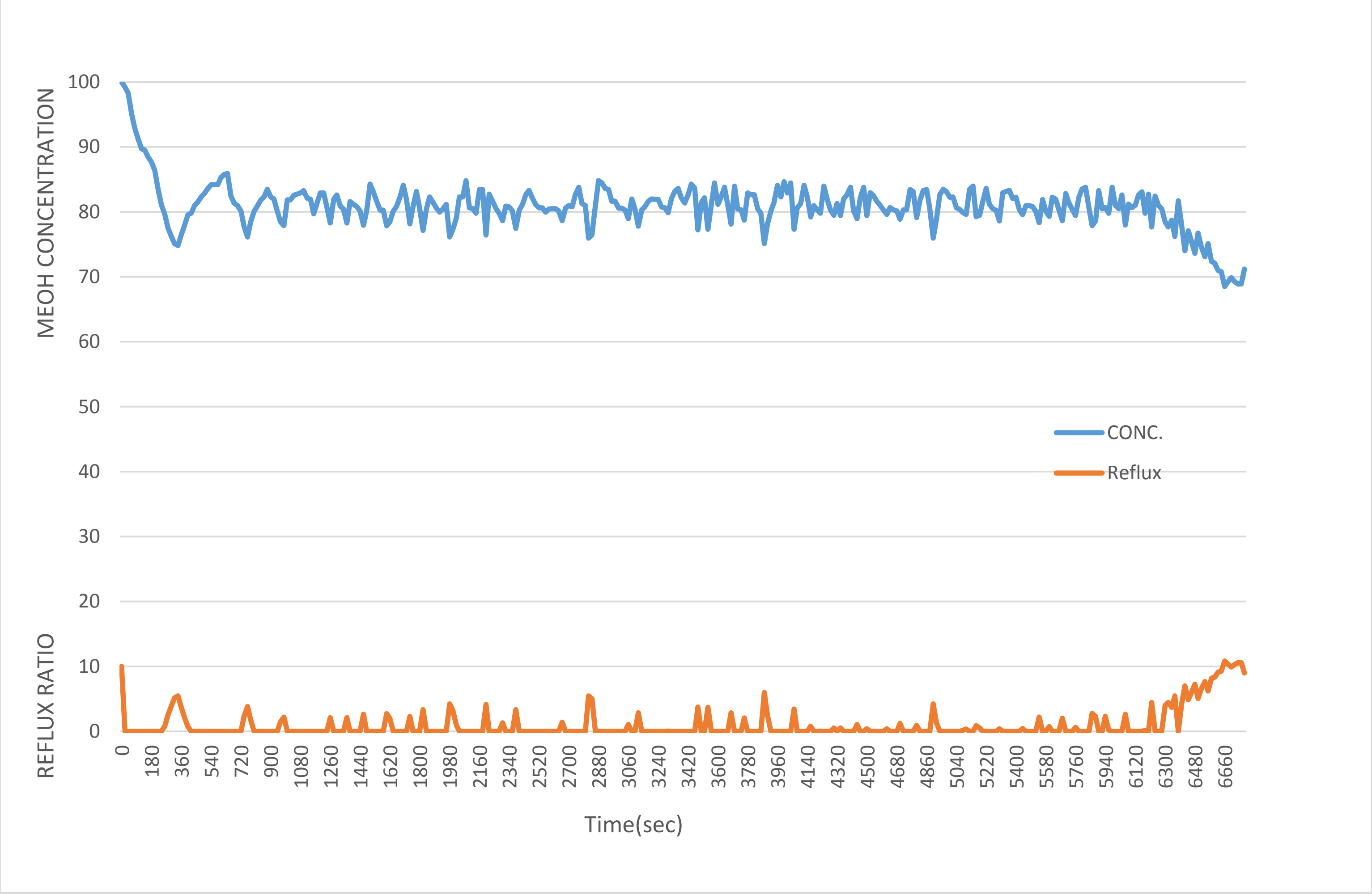


Figure 5.34 concentration profile and reflux ratio profile for experimental results with set point (80%)

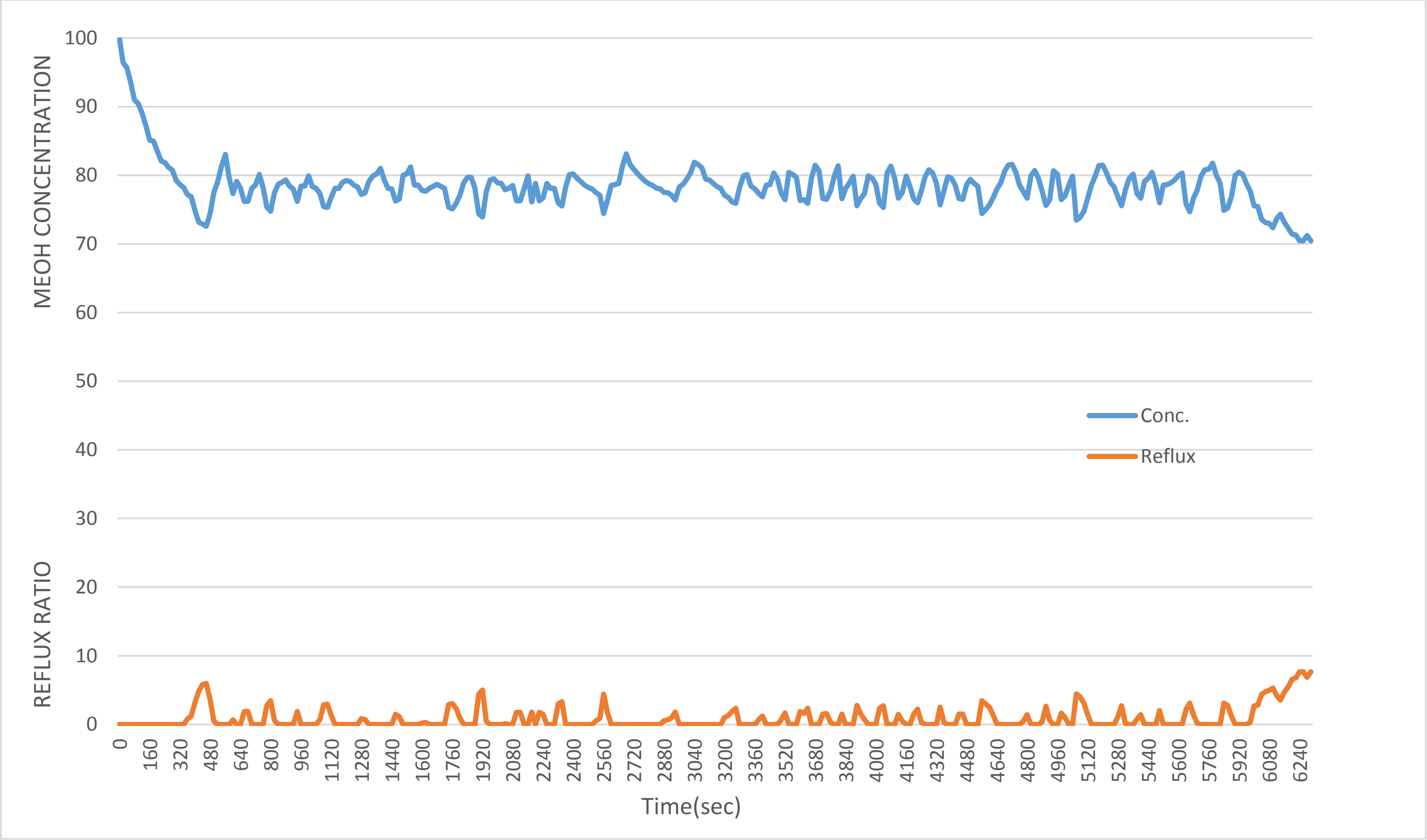


Figure 5.35 concentration profile and reflux ratio profile for experimental results with set point (78%)

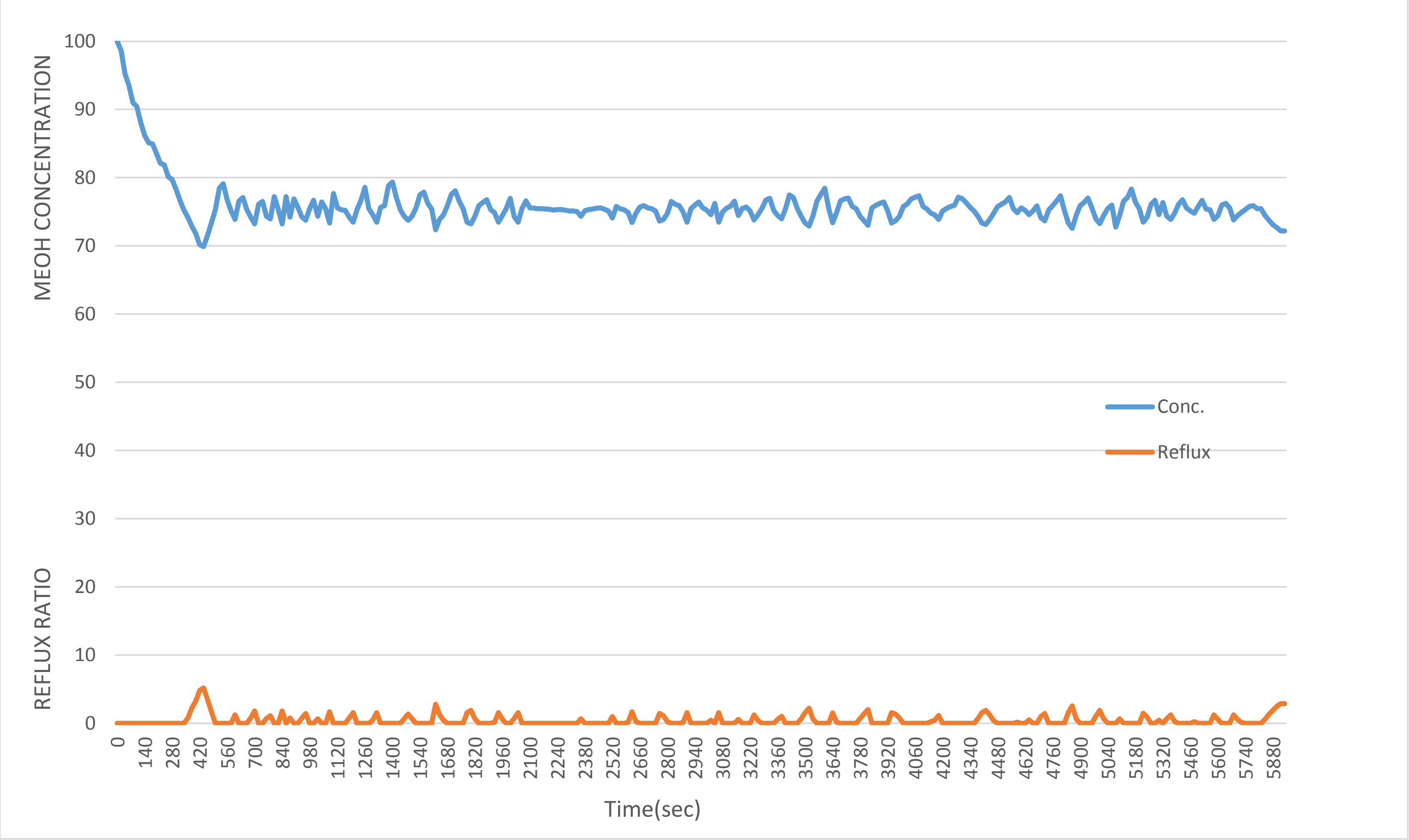


Figure 5.36 concentration profile and reflux ratio profile for experimental results with set point (75%)

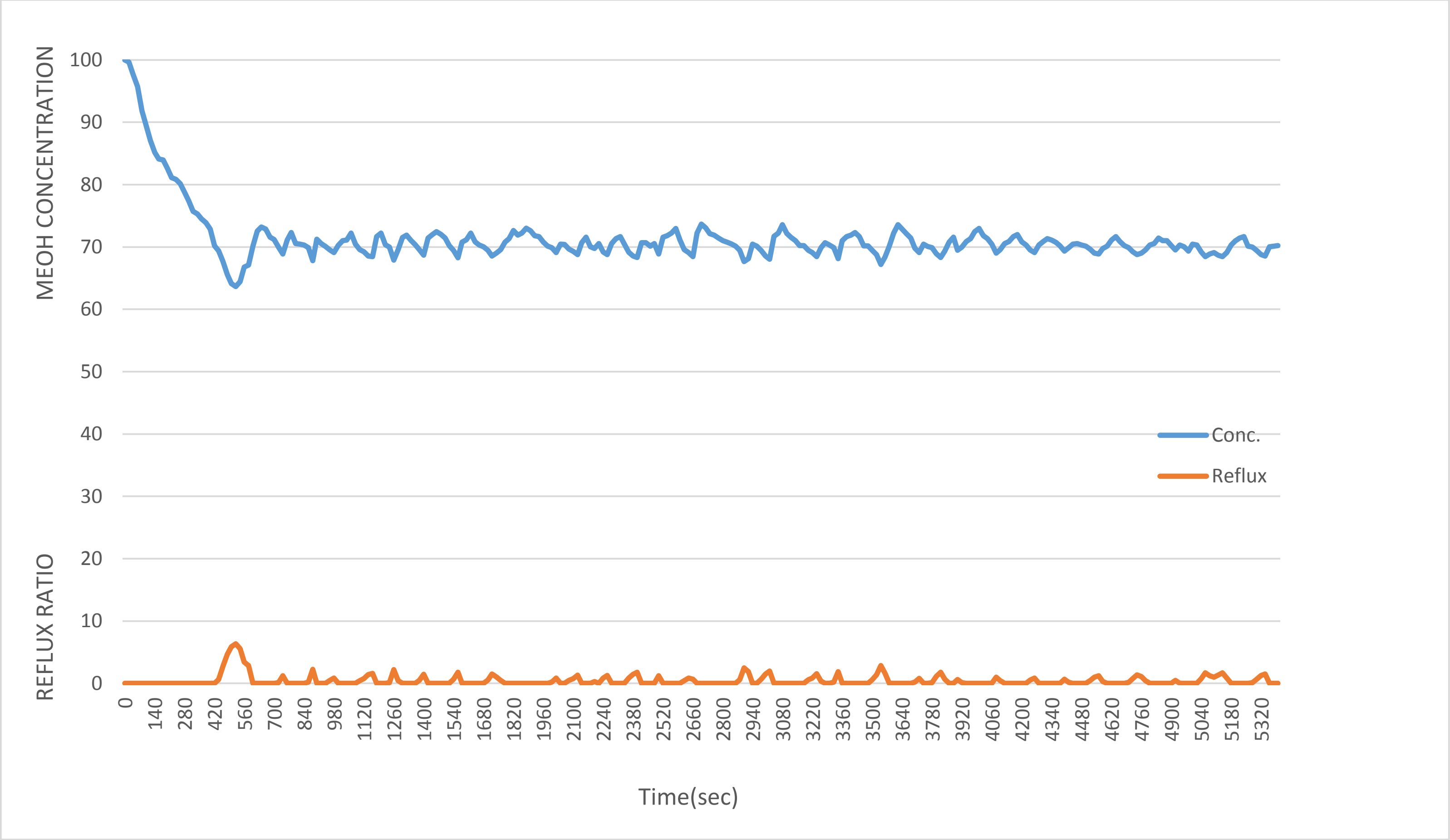


Figure 5.37 concentration profile and reflux ratio profile for experimental results with set point (70%)

All times responses for all figures from 5.28 to 5.37 are represented in table 5.3.

Table 5.3 Times responses from the experiments results

Figure	Rise Time(s)	Peak Time(s)	Settling Time(s)
5.28	70	250	1200
5.29	90	360	1100
5.30	90	250	1000
5.31	130	400	970
5.32	240	460	960
5.33	260	360	660
5.34	260	340	650
5.35	320	460	620
5.36	340	440	550
5.37	420	520	660

Comparing Figures 5.19 to 5.37 indicates that the current simulation results are qualitatively similar to the experimental results. Discrepancies between the two can be attributed to a number of factors including experimental error and uncertainties in estimated parameters for the model. Part of the problem is likely to be the fact that the low composition of methanol in the feed makes the composition of methanol at the lower stages very small and makes the experiment sensitive to small changes in process parameters. Simulation of a column with more methanol would likely be easier. After several attempts to adjust the model so that it would agree with the experiment, it was concluded that an exact match between model and experiment may not be necessary. Conceptual understanding of the process can probably be obtained from a model that is not in complete agreement with the experiment.

The results of simulation that uses LabVIEW and experiments are given in Figures before. It is found that when the concentration set point is 90% or lower values gives better match between model and experiment compared to those higher than 90%, and this happen because the low amount (composition) of methanol in the feed makes difficult to get constant high purity of methanol composition.

5.4. Comparison between two strategies of batch distillation: constant reflux ratio and constant overhead

The purpose of this section is to compare the two strategies of batch distillation: constant reflux ratio and constant overhead product composition and choosing the best strategy to optimal batch distillation. The same existing instillation was considered in both cases. The following input data were constant for the two operation modes: initial amount and composition, the final residue (quantity collected of distillate) and constant heat duty, which mean both operation operate at the same conditions.

Table 5.4 Present comparison between the experimental results for the two strategies of batch distillation: constant reflux ratio and constant overhead product composition.

Constant Reflux Ratio				Constant Overhead product composition.		
Exp.NO.	R	Time (min)	% concentration	EXP.NO	% Concentration	Time (min)
1	10	194	89.6	1	95	183
2	8	182	83.4	2	93	175
3	7	176	81.55	3	90	168
4	6	165	79.6	4	87	159
5	5	158	77.1	5	84	150
6	4	150	75.2	6	82	141
7	3	143	73.8	7	80	133
8	2	135	72	8	78	125
9	1.5	120	70.35	9	75	119
10	0	85	65	10	70	110

The results show reductions in distillation time of up to 20% in comparison with the constant reflux policy. The results indicate that the time saving for the constant overhead composition in comparison with constant reflux of the same condition for the experiments in Table 5.5. The constant overhead composition gives a larger time saving than using the constant reflux ratio.

Based on the experimental result from both cases variables compared are batch time and distillate concentration, which effects on the economic factors. The experimental result are presented in table 5.4. Which shows that the second operation mode (constant overhead product composition) is better than the first operation mode (constant reflux ratio) from economical view because it gives higher purity for methanol concentration in less time than constant reflux ratio so gives larger time saving around 20%,this is lead to save a lot of energy which mean lower cost, so this way gives economical and high purity which is the goal for every separation to produce the product in best quality and economic way.

Table 5.5 Excess time percentage

% X_D	Time(min)		Excess time over constant overhead	Excess time percentage%
	Constant R	Constant x_D		
90	194	168	26	13.4
82	176	141	35	19.8
80	165	133	32	19.4
75	150	119	31	20

CHAPTER SIX

CONCLUSIONS AND RECOMMENDATION FOR FUTURE WORK

6.1 conclusion

From the present work carried out to study the optimal operation for batch distillation in different control strategies the following conclusions are obtained:

1- At constant reflux operation (with constant heat duty), it was found that increasing reflux ratio will increase the distillate concentration but it will be at the expense of the experiment time and power consumption.

2- Studying the dynamic behavior by introducing a step test into the process and recording the response. It has been recognized that a first order may represent process dynamics of batch distillation. The transfer function was found to be:-

$$G_P = \frac{2.5}{46.67\,S + 1}$$

3- By analyzing the open-loop transfer function using Bode diagram, it was found that the relation between reflux ratio and overhead composition is unstable.

4- Zieglar-Nichols tuning rules and Routh's stability criterion were applied to determine the parameters of the implemented PID controller. It was found that $k_p = 3.400536\ \ \tau_I = 11.754, and\ \tau_D = 5.880536\ s$.

5- LabVIEW simulation was used to simulate the system using PID controller. It was found that the changing of the top tray composition (the main control target) was kept within a desired narrow range (4%).

6- The simulation results show good agreement with the experimental results. Discrepancies between the two can be attributed to experimental error and uncertainties in estimated parameters of the model.

7- Using constant overhead strategy can reduce the distillation time of up to 20% in comparison with the constant reflux policy. From economical view, using constant overhead strategy will lead to save energy which is the goal for every separation process.

6.2 Future work

For future work the following recommendations are put forward:

1- Application of the same experimental work on different batch distillation column such as (multi-component distillation, or with more than 10 stage, or with another component).

2- Using the same work but with other controlled and manipulated variables.

3- Using another controller such as cascade control, passive controller and others then comparing the results.

REFRENCES

Bahar, A.; Ozgen, C.; Leblebicioglu, K. & Halici, U. (2004). Artificial neural network estimator design for the inferential model predictive control of an industrial distillation column. Industrial Engineering Chemical Research, Vol. 43 (2004), pp. 6102-6111.

Barolo, M.; Berto, F. An Advanced Strategy for Composition Control in Batch distillation, edited preprints of DYCOPS-5-5th IFAC Symposium on Dynamics and Control of Process Systems; Georgakis, C., Ed.; 1998; pp 430-435.

Bitter, Rick et al. LabVIEW advanced programming techniques, 2nd ed. 2007.

Chipperfield. J Gregory, D Webster A computer-controlled laboratory fractional distillation column Analytica Chimica Acta - Vol. 177 - 1985 - pp. 203-208.

Coward, I., the Time-Optimal Problem in Binary Batch Distillation. Chemical Engineering Science, 22, pp. 503-516 (1967).Block, B. Control of Batch Distillation. *Chem. Eng.* **1967**, Jan. 16, 147.

Hansen, T. T. and Jorgensen, S. B., Optimal Control of Binary Batch Distillation in Tray or Packed Columns. Chemical Engineering Journal, 33, 151-155 (1986).

John E. Edwards, Batch Reactor Systems Technology Symposium, of dynamic modelling, Teesside, October 2001.

Escano, J.M.; Bordons, C.; Vilas, C.; Garcia, M.R. & Alonso, A. A. (2009). Neurofuzzy model based predictive control for thermal batch processes. Journal of Process Control, Vol. 18 (200), pp. 1566-1575.

Fieg, G. (2002). Composition control of distillation columns with a side stream by using gas chromatographs, Chemical Engineering Processing, Vol. 41, (2002), pp. 123-133.

Fileti, A.M.; Antunes, A.J.; Silva, F.V.; Silveira, V. & Pereira. J.A. (2007). Experimental investigations on fuzzy logic for process control. Control Engineering Practice, Vol. 15 (2007), pp. 1149-1160.

Frattini, A.M; Cruz, S.L. & Pereira, J. (2000). Control strategies for a batch distillation column with experimental testing. Chemical Engineering Processing, Vol. 39 (2000), pp. 121-128.

Haykin, S. (2008). Neural networks and learning machines, Prentice Hall. Himmelblau, D.M. (2008). Accounts of experiences in the application of artificial neural networks in chemical engineering. Industrial Engineering Chemical Research, Vol. 47,No. 16 (2008), pp. 5782–5796.

Marchetti, J.L.; Benallou, A.; Seborg D.E. & Mellichamp, D.A. (1985). A pilot-scale distillation facility for digital computer control research. Computers and Chemical Engineering, Vol. 9, No. 3 (1985), pp. 301-309

Norman S. N; “Control system engineering”, 3rd edition, Johan Wiley and sons., Inc, 2000.

Nooraii, A.; Romagnoli, J.A & Figueroa, J. (1999). Process identification, uncertainty characterization and robustness analysis of a pilot scale distillation column. Journal of Process Control, Vol. 9 (1999), pp. 247-264.

Ogata K;" Modern Control Engineering", Prentice Hall, Inc, New Jersey, 2002.

Quintero-Marmol, E.; Luyben, W. L.; Georgakis, C. Application of an Extended Luenberger Observer to the Control of Multicomponent Batch Distillation. *Ind. Eng. Chem. Res.* **1991**, *30*, 1870.

Skogestad, S. (1997). Dynamics and control of distillation columns. A tutorial introduction. Transactions on Industrial Chemical Engineering, Vol. 75(A) (1997), pp. 539-562.

Skogestad, S. (2004). Control structure design for complete chemical plants. Computers and Chemical Engineering, Vol. 28 (2004), pp. 219-234.

Sundaram, S.; Evans, L. B "Shortcut Procedure for Simulating Batch - Distillation Operations", Ind. Eng. Chem. Res. 1993, 32, 511-518.

U.M. Diwekar, Batch Distillation, Simulation, Optimal Design and Control, London: Taylor & Francis, 1995.

Varshney, K. & Panigrahi, P.K. (2005). Artificial neural network control of a heat exchanger in a closed flow air circuit. Applied Soft Computing, Vol. 5 (2005), pp. 441-465.

Venkateswarlu, C. & Avantika, S. (2001). Optimal state estimation of multicomponent batch distillation. Chemical Engineering Science, Vol. 56 (2001), pp. 5771-5786.

Zamprogna, E; Barolo, M. & Seborg, D.E. (2005). Optimal selection of soft sensor inputs for batch distillation columns using principal component analysis. Journal of Process Control, Vol. 15 (2005), pp. 39-52.

Zuniga-Linan,L., Savioli-Lopes, M., 2010 molecular distillation of petroleum resides and physical-chemical characterization of distillate cuts obtained in process.J.Chem.Eng (2010).

Appendix

The Equilibrium data of methanol /water are represented in the figures and tables below:

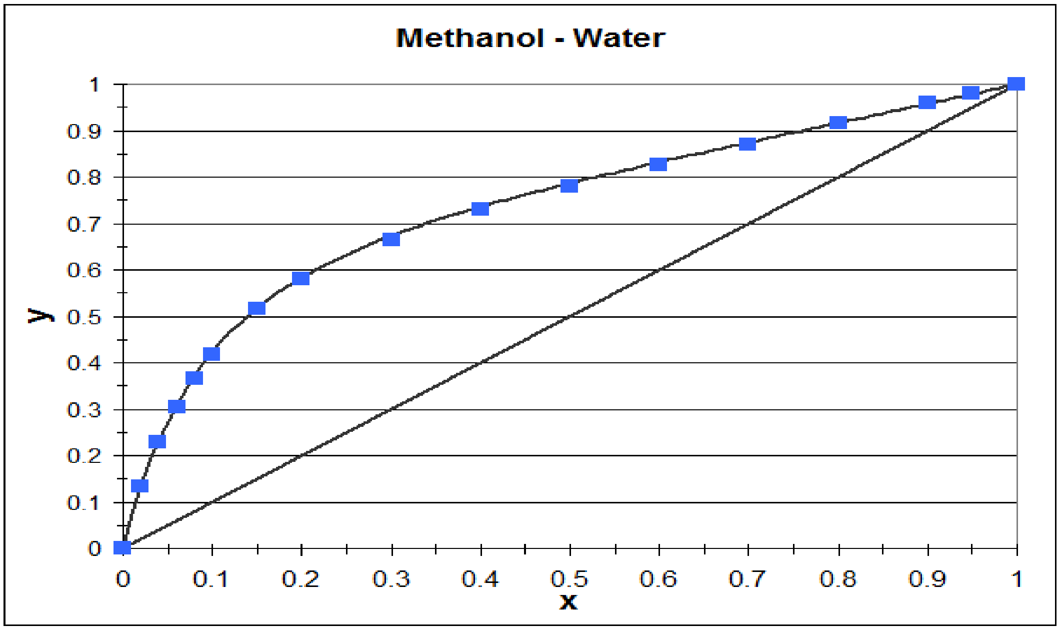


Figure 1 (X Y) vapor-liquid equilibrium of methanol/water [Dechema, vol I, part 1a, p 53].

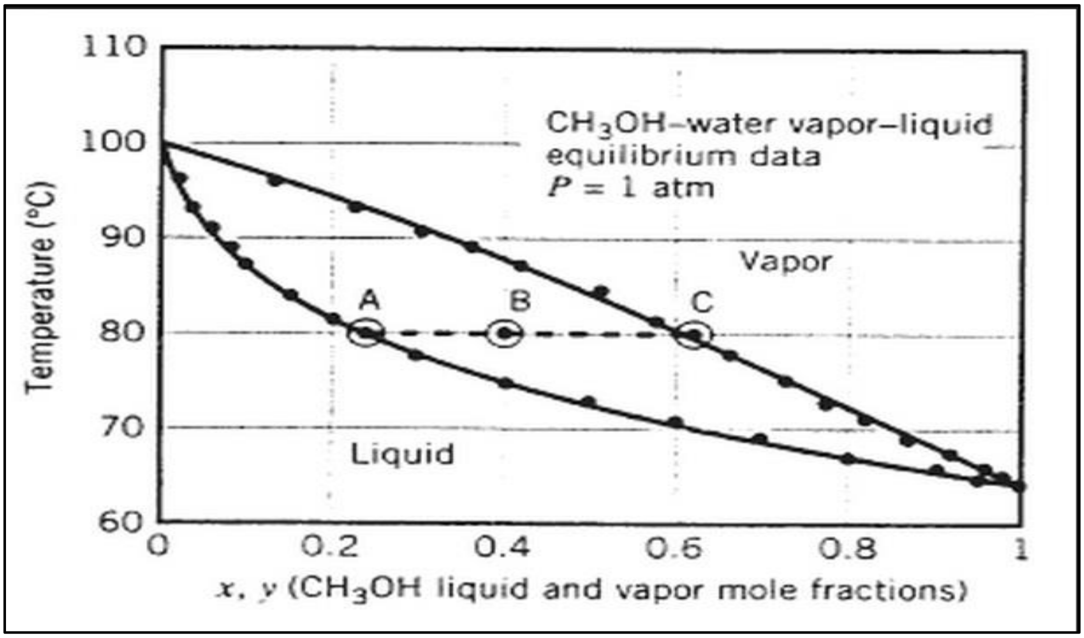


Figure 2 (T X Y) vapor-liquid equilibrium of methanol/water [Dechema, vol I,part 1a, p 55]

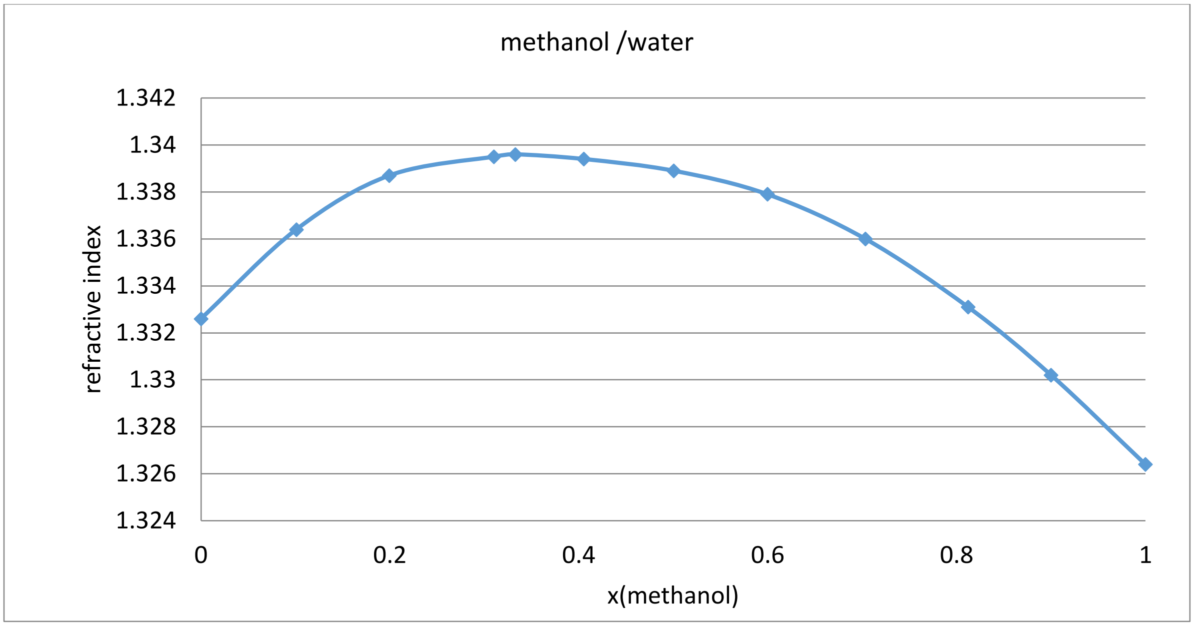


Figure 3 refractive index with concentration of methanol [nD, FPEQ, (1996), vol 126, p 203-223 @ 25 C]

Table 1 density of methanol at different concentration and Refractive index values [nD, FPEQ, (1996), vol 126, p 203-223 @ 25 C].

ρ(kg·m−3)	x	R I
997.02	0	1.3326
971.66	0.101	1.3364
948.15	0.1995	1.3387
922.64	0.3102	1.3395
917.74	0.333	1.3396
901.76	0.4054	1.3394
881.5	0.5006	1.3389
861.17	0.5998	1.3379
840.72	0.7035	1.336
820.14	0.8122	1.3331
804.1	0.9002	1.3302
786.62	1	1.3264

الخلاصة

جهاز التقطير ذو الدفعات أصبح له أهمية متزايدة نتيجة للتوسع في الصناعات الكيميائية والصيدلانية. أصبح ايجاد العملية المثلى لتشغيل التقطير ذو الدفعات متزايده الأهمية. ولذلك تهدف هذه الرسالة إلى إيجاد السبل المثلى لتشغيل التقطير ذو دفعات.

قد تم بناء المنظومة العملية في مختبرات قسم الهندسة الكيميائية والبيولوجية بجامعة كيس ويسترن ريزيرف في الولايات المتحدة الأمريكية ودراسة استراتيجيات التشغيل الأمثل لمثل هذا النوع من التقطير باستخدام برنامج LabVIEW، مع تصميم وحدة تحكم NI LabVIEW والمحاكاة والتي يمكن من خلالها محاكاة النظم الديناميكية، تصميم وحدات التحكم المتطورة، ونشر أنظمة التحكم في الأجهزة في الوقت الحقيقي، وبالتالي يتم التحكم بكل منظومة عمليةعن طريق الكمبيوتر وكان ذلك خلال جهاز اكتساب البيانات (DAQ), NI-ELVIS II وتم استخدامه لتحسس درجات الحرارة وتوليد الإشارات لأغراض المراقبة.

تتناول هذه الرسالة مختلف جوانب العملية والسيطرة على التقطير ذو الدفعات حيث تم اجراءها للحالتين تثبيت نسبة الراجع او تثبيت التركيزللمادة الناتجة خلال التجربة.

وجد ان الدالة التي قد تناسب ديناميكية عملية التقطير ذوالدفعات هي دالة من الدرجة الاولى والتي تم ايجادها عن طريق تجارب عملية تم اجرءها لجهاز التقطير ذو الدفعات ومتغيرات الدالة التي وجدت هي:-

$$k_P = 2.5, \text{ And time constant } \tau_I = 46.67\ s.$$

تم استخدام طريقة Routh's stability criterionوZieglar-Nichols tuning rules لمعرفة المتغيرات التقريبية اللازم تطبيقها للPID controllerوالقيم التي وجدت هي:

$$k_p = 3.400536\ \ \tau_I = 11.754\ s, and\ \tau_D = 5.880536\ s.$$

النتائج التحليلية التي تم الحصول عليها كانت مطابقة للنتائج العملية وتم ملاحظة ان استخدام طريقة تثبيت تركيز المادة الناتجة يمكن من خلالها تقليل وقت التقطير بنسبة ٢٠% بالمقارنة مع الطريقة الاخرى(تثبيت نسبة الراجع من المادة الناتجة).

شكر وتقدير

اتوجه بالشكر والتقديرالى المشرف ورئيس قسم الهندسة الكيمياوية في جامعة النهرين الدكتور نصير الحبوبي لما قدمه لي من توجيهات قيمة ونصائح سديدة طوال فترة انجاز البحث والى الاساتذة الاعزاء في القسم والى عمادة كلية الهندسة وزملائي في الدراسة وموظفي قسم الهندسة الكيمياوية في جامعة النهرين لابداهم المساعدة اللازمة اثناء هذا العمل.

واود ايضا ان اشكر قسم الهندسة الكيمياوية في الجامعة الامريكية (Case Western Reserve University) وبالاخص الدكتور دانيال لاكس والدكتورة هايدي مارتن.لاشرافهم و توجيهاتهم القيمة و دعمهم لي طوال فترة البعثة البحثية في جامعة(Case Western Reserve University).

ولا انسى ان اتقدم بالشكر والامتنان الى من لازمني طوال فترة البحث وخلال اصعب الظروف الى اعز من في الوجود الى عائلتي فلهم جزيل الشكر.

رند قصي كاظم

التقطير ذو الدفعات الامثل القائم على استخدام الحاسوب

رسالة مقدمة الى كلية الهندسة الكيمياوية في جامعة النهرين
وهي جزء من متطلبات نيل درجة الماجستير علوم
في الهندسة الكيمياوية

من قبل

رند قصي كاظم الخفاجي
(بكالوريوس علوم في الهندسة الكيمياوية ٢٠١٢)

شعبان 1436
ايار 2015