# System Identification for a Pilot Scale Acetone and Isopropyl Alcohol Distillation Column

by

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Dissertation submitted in partial fulfilment of the requirement for the Bachelor of Engineering (Hons) (Chemical Engineering)

# MAY 2014

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# CERTIFICATION OF APPROVAL

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A project dissertation submitted to the

**Chemical Engineering Programme** 

Universiti Teknologi PETRONAS

In partial fulfilment of the requirement for the

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Approved by,

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# MAY 2014

# CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and the original work contained herein have not been undertaken or done by unspecified sources or person.

NOR ADIERA AMIZ BT ABDUL MUTALIB

### ABSTRACT

System identification is a technique to build an accurate and precise model of complex system from noisy data. It has been used to represent the relationship between output and input data and also to develop understanding on the internal working system. System identification can be applied in various fields of applications.

In this project the system identification is basically focused on the pilot scale of Acetone – Isopropyl alcohol distillation column by relate the control variables which are acetone top and bottom composition with the manipulated variables that are reflux flow rate and steam flow rate. This distillation process is to separate the mixture of acetone and isopropyl alcohol into individual components with certain composition. Basically this project involves both experimental work and simulation work.

System identification requires data from experimental work from Distribution Control System (DCS) in order to simulate the relation between input and output of the system by using System Identification Toolbox. In this report, the low order transfer function model is developed and represents the acetone – isopropyl alcohol distillation column. Model validation has been done to compare performance between the mathematical models with the actual plant. From this project the modelling of system identification of the pilot scale distillation column will be further used in the advanced process control application to improve the performance of the process control in the system.

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# TABLE OF CONTENTS

CHAPTER	CONTENTS	PAGES
	CERTIFICATION OF APPROVAL	i
	<b>CERTIFICATION OF ORIGINALITY</b>	ii
	ABSTRACT	iii
	ACKNOWLEDGEMENT	iv
	Table of Contents	v
	List of Figures	vii
	List of Tables	viii
CHAPTER 1	INTRODUCTION	
	1.1 Background Of Study	1
	1.2 Problem Statement	3
	1.3 Objectives	3
	1.4 Scope Of Study	4
	1.5 Relevancy of the project	4
	1.6 Feasibility of the project	4
CHAPTER 2	LITERATURE REVIEW	
	2.1 System Identification	5
	2.1.1 Input signal design	7
	2.1.2 Model structure of System Identification	9
	2.1.3 Identification method	12
	2.2 Distillation process	13
CHAPTER 3	METHODOLOGY	
	3.1 General Flow Chart	15
	3.2 Experimental Procedure	17
	3.3 Simulation (System Identification)	20
	3.4 Gantt Chart and Milestone	24

<b>CHAPTER 4</b>	<b>RESULTS AND DISCUSSIONS</b>			
	4.1 Experiment Step Testing	26		
	4.2 Simulation (System Identification)	28		
	4.2.1 Reflux Flow Rate	29		
	4.2.2 Steam Flow Rate	32		
	4.3 Model Validation	35		
	4.3.1 Reflux Flow Rate	36		
	4.3.2 Steam Flow rate	38		
	4.4 Problems Encountered	40		
CHAPTER 5	CONCLUSION AND RECOMMENDATION			
	5.1 Conclusion	41		
	5.2 Recommendations	42		
CHAPTER 6	REFERENCES	43		
CHAPTER 7	APPENDICES			
	7.1 Gantt Chart	46		
	7.2 Distribution Control System (DCS) Results	47		
	7.3 Model Validation	48		

# LIST OF FIGURES

Figure 2.1: u as the input to the system and produce output y	5
Figure 2.2: Noises are constraints in measurements	5
Figure 2.3: Input-output data (noisy case) (Hugues Garnier, 2008)	6
Figure 2.4: The ARX model structure	11
Figure 2.5: The NARX model structure	12
Figure 3.1: General flow chart of the project	15
Figure 3.2: Experimental procedures Figure 3.3: Schematic flow chart of system identification	17 20
Figure 3.4: Import data	21
Figure 3.5: Selecting ranges- Data estimation (green) and data validation (red)	22
Figure 4.1: Reflux flow rate (input) vs. Acetone composition at top and bottom	27
Figure 4.2: Steam flow rate (input) vs. Acetone composition at top and bottom	27
Figure 4.3: Time plot of u1 and y1. $(G_{11})$	30
Figure 4.4: Time plot of u1 and y2 ( $G_{21}$ )	30
Figure 4.5: Step response for $G_{11}$	31
Figure 4.6: Step response for $G_{21}$	31
Figure 4.7: Time plot of $u_2$ and $y_1$ (G <sub>12</sub> )	32
Figure 4.8: Time plot of $u_2$ and $y_2$ (G <sub>22</sub> )	33
Figure 4.9: Step response for $G_{12}$	34
Figure 4.10: Step response for G <sub>22</sub>	34
Figure 4.11: Comparison between the experiment and the model for $G_{11}$	36
Figure 4.12: Comparison between the experiment and the model for $G_{21}$	37
Figure 3.13: Comparison between the experiment and the model for $G_{12}$	38
Figure 4.14: Comparison between the experiment and the model for $G_{22}$	39

# LIST OF TABLES

Table 3.1: Description on Acetone-Isopropyl alcohol distillation column	16
Table 3.2: Key Milestone for FYP 2	24
Table 3.3: Key Milestone for System Identification of Acetone-Isopropyl           Alcohol Distillation Column	25
Table 4.1: Experimental data	26
Table 4.2: Input and Output Parameters for Experiment Step Testing	27
Table 4.3: Model validation for $G_{11}$	36
Table 4.4: Model validation for G21	37
Table 4.5: Model validation for $G_{12}$	38
Table 4.6: Model validation for $G_{22}$	39

### **CHAPTER 1**

### **INTRODUCTION**

### **1.1 Background Study**

System identification in a broad sense deals with many restraints coming up when designing, conducting and interpreting result from such an experiment. In other words, system identification could be describe as design, conduct, process and interpret the results from the experiment applied to the system to get an accurate model of its internal working.(Ljung, 2001)

The purpose of this system identification is to derive models from available input and output data of the system. System identification includes dealing with various tasks of parameter to estimate based on the observations that originating from a dynamical system.(Halizamri Md. Shariff, 2014) It also used to determine the dynamic behaviour of physical object or process based on the mathematical relation between the inputs and outputs. (Schoukens, 2012) Generally mathematical equation is closely related with this system identification.

System is the overall behaviour which people could study and understand. The techniques for this system identification can be applied in various system and applications. For example in industrial plant which the feed stocks are undergoing some complicated and many processes in order to produce the desired yield. This internal mechanism of the industrial plant processes is focused on the detail. System identification could be applied in this condition as it will makes simpler model by relating the input and output which is easier and can be directly tuned based on our need. (Ragnar Wallin, 2010) This also gives advantages as the system identification might be better in any adjustment and handle any unforeseen disturbances.

Apart from relating the input and output of the process, one of the important characteristic in system identification is the input signal design. System identification is one of the modelling method which deals with problem on how to estimate the model of a system from a measured input and output signals. The system can be described as linear or non-linear are depending on the estimation of the model itself and the type of system used in the process. It is crucial for the perturbation signal that

used as input to the system to have an adequate excitation and enough fluctuation of desired effect in order to estimate and measure property of the dynamic system of the process.(Halizamri Md. Shariff, 2014)

In the case of non-linearity identification system, perturbation signal should have sufficient fluctuations to ensure the full range of non-linearity process dynamics can be identified. (Mark. L. Darby, 2014). Linear system usually used deterministic input such as Pseudo Binary Random Sequence (PRBS), however due to some constraint; the input design may appear not suitable with some processes as it will produce bias and erroneous to the estimated model. Thus study of comparison between the types of input signal design will be done in this project and does it as a new research other than generating the response of model from system identification method.

System identification could be beneficial in many ranges of fields and mechanisms in life. It could be used in various applications such as petrochemical plant, multimedia system, signal system and even human body system. For this project generally will be focused on the industrial plant process which is pilot scale for acetone-isopropyl alcohol distillation column

Distillation basically is the physical separation process and commonly used method for purifying liquids and separating mixtures of liquids into their individual components. Most of the process industry used distillation column for the separation operation. For this binary distillation column, acetone and isopropyl alcohol are to be separated in a fractionation column operating at certain pressure.(Thanm, 1997)

In this project the main point basically to describe the identification of a pilot scale of acetone-isopropyl alcohol distillation process and determine the transfer function for the working model. Transfer function is a compact description of the input and output relation for a linear system and these are used to represent the system.(Levine, 1999) It is as a representation of a linear time invariant dynamical system. The transfer function mathematically is a function with the complex variables. For this binary distillation column, the main control variables are the overhead and bottom compositions. These simultaneous controls of both bottom and overhead composition are depending on the manipulated variable. Overhead composition is controlled by the reflux flow rate whereas the bottom composition is controlled by the steam flow rate.

### **1.2 Problem Statement**

Based from the model that has developed by Wood-Berry on the terminal composition control of the binary distillation column, it has significantly different from the existing pilot scale as the distillation column has more number of trays compared to the Wood-Berry model. There some issue on the non-linearity of the pilot scale as it is half bigger than the model.

Therefore, system identification needs to be developed to determine the relation of input and output of the pilot scale and to identify its transfer function. The control variable is remaining the same which is the composition of the overhead and bottom product by using reflux and steam flow rate as the manipulated variable.

Furthermore, experiment on the step testing for the pilot scale acetone- isopropyl alcohol distillation column need to be re-done. This is because some issue on the previous experiment data which are not precise. The new step testing will be done by using refractometer in determining the composition at the top and bottom of the distillation column and also to analyse and identify the compound.

Thus in this project the relationship between inputs and output which represent the system can be done by using system identification method.

# 1.3 Objective

The objective of the project is to construct a mathematical model of dynamic system in order to describe and relate the relationship between output and input of the system. The aim is to get the mathematical model of dynamic system from the experimental data. This system identification can be done by determining the transfer function First Order Process Time Delay (FOPTD) or Second Order Process Time Delay (SOPTD) to represent the pilot scale of acetone-isopropyl alcohol distillation column.

Other than that, this project also to identify and predict the behaviour of this dynamic system. The transient response of the dynamic model could be recognising through this system identification method. Besides that, this project will develop the understanding and knowledge on the Advanced Process Control (APC) application and the internal working model of the system.

### 1.4 Scope of Study

This project focuses on determining the modelling of the pilot scale acetoneisopropyl alcohol distillation column by system identification. The control variables used in this project are the composition of the overhead and the bottom product whereas the manipulated or process variables are the reflux flow rate and the steam flow rate. The pattern of the input output relation can be identified throughout the system identification process.

# **1.5 Relevancy of the Project**

Basically this project is relevant as in this final year degree of Chemical Engineering student require to develop knowledge on the process industry. On the other hand, students can gain more understanding how does the control system working in the distillation column in order to get the desired composition of product.

# 1.6 Feasibility of the Project

This project is feasible as the time given to complete it are two semesters which are approximately eight months. The Gantt chart has been developed to ensure the student keep track on the dateline. Two semesters are adequately enough to complete on the experimental work for step testing and modelling using system identification toolbox from Mat Lab.

### **CHAPTER 2**

### LITERATURE REVIEW

### 2.1 System Identification

Process of developing or improving the mathematical representation of a physical system using experimental data is known as system identification. (Nagarajaiah, 2009)



Figure 2.1: u as the input to the system and produce output y

From the figure above, the system identification procedure generally used u as certain input that applied to the system and result in y which is the system response. From the relationship, the transfer function as well as other mathematical equations that represent the system can be determined. The output can be optimized based on the adjustment of input which is u. However, there are some constraints in order to generate a suitable model such as the corruption of noise which could disturb the measurement.(R.Smith, 2011) Therefore, several iterations need to be done in order to improve and generating a more precise model based on actual pilot plant.



Figure 2.2: Noises are constraints in measurements



Figure 2.3: Input-output data (noisy case) (Hugues Garnier, 2008)

Figure above shows the anticipated result due to noises in the system. The inputs are from the experimental data that imported in the System Identification Toolbox in the Mat Lab.(Hugues Garnier, 2008) In order to compute the coefficient of the finite difference model from input and output data the formulations are derived. Based from the coefficient the output prediction model is derived which later giving the relationship between the future input-output data and past output-input data.(Juang & Phan, 2001)

System identification is only useful when there are available information regarding the input and output data. There are three components in determining the dynamic system from the observed input-output data that are the model structure, input-output data and the identification method which means some criterion to select a particular model in the set with the available information in the data.(Activemedia, 2012)

Generally there are two options in obtaining the model for system identification. Firstly is the grey box identification which requires deriving from the first principles such as physical laws and then identifying parameters by running the experiments. Experiments are needed in this method in order to identify and determine all the unknown parameters. Secondly is the black box identification which this case has very inadequate and limited information regarding the corresponding system. Basically this method only has measurements (u and y) and certain working assumptions about the system and some noise. (Pelckmans, 2012) Thus the goal from the method is derive a system model out of these assumptions and measurement. Therefore in this project, the system identification are based on the grey box identification which require to run the experiments to get more precise data in order to derive an accurate model that represent the system.

There are several terms in the mathematical model for system identification such as linear and non-linear, time-invariant, time-varying, static and dynamic system.(Keesman, 2011) These used to categorize what type of the system that used in the system identification.

Linearity is a function or relationship which is directly to each other. For example under zero conditions  $u_1(t)$  and  $u_2(t)$  are the inputs to system with the corresponding outputs  $y_1(t)$  and  $y_2(t)$ . This system is considered as linear when input equation  $Au_1(t) + Bu_2(t)$  and the result from the corresponding response is  $Ay_1(t) + By_2(t)$ , where A and B are constants. If the systems response appeared to be different this means that the model structure appeared to be non-linear. For time-invariance, assume  $u_1(t)$ is the input to a system with the corresponding output of  $y_1(t)$ . A system is timeinvariant if the response to the  $u_1(t + r)$  is  $y_1(t + r)$ , with r as the time shift.(Katayama, 2006)

Dynamic system is when a system output is just not depending on the present input but depends on the history. Dynamic system in this modelling usually is represent by the terms of differential equation whereas static system is basically depending on the present input and designate by algebraic equations. (Goodwin, 1977)

#### 2.1.1 Input Signal Design

Performance of a system can be evaluated based on test signals input used. In this project of system identification of acetone-isopropyl alcohol distillation column, various ways to measure the performance of the system can be done. Based on this step input design the transient response and design of control system can be identified. The data used for system identification of the model is generated from a well design plant test. Generally most of the industries are depending on the

uncorrelated input signals although various studies and researchers have developed correlated input signal which brings more advantages in system identification.(Mark. L. Darby, 2014)

Multivariable process identification usually only rely on uncorrelated input signals such as PRBS. Uncorrelated test signal of PRBS is applied to the process to gain the data on the system. It can be either in closed or open loop and the magnitude is set on the basic of the constraint on inputs and outputs.

Basically the study of the input design for a multivariable identification has been developed and generating input data via a well-designed experiment is essential to ensure the identification is done precisely. There are two types of data generation that are single open loop Multiple Input Multiple Output (MIMO) and open loop Single Input Multiple Output (SIMO). MIMO means that all the inputs are executed simultaneously whereas SIMO means only one input is executed at one time. There are several challenges of multivariable system for SISO and MIMO. For SISO, the accurate estimation of the individual transfer function elements in a transfer matrix may not be sufficient to guarantee robust closed loop stability and for MIMO, the most crucial concern is on the distribution of model errors among individual transfer function. (Ali Nooraii, 1999)

Both PRBS and Schroeder-phased input are periodic inputs and based are from the design of dominant open loop time constant and desired loop speed of response. According to Chien and Ogunnaike, conventional input design of PRBS may lead to ambiguous as well as inaccurate for the system thus the input design are proposed to be a modelling scheme by using integrator which represents each element of variable in the transfer matrix. (Chien, 1992)

From Li and Lee research journal in 1994, the input design is preferable in a combination of open loop and closed loop identification experiment as the closed loop identification can estimate accurate plant inverse which can obtain the gains directly from the information. (Li. W, 1994). However according to Gaikwad and Rivera the single loop is adequate to obtain model that can gives a good information on the closed loop by testing two open loop input design that are PRBS and Schroeder-phased input. This can be further review in the case of the design of input due to the system identification. For this project it focused on MIMO as it required

obtaining the transient response between both manipulated variables data that are reflux flow rate and steam flow rate that corresponds to the control variables that are distillate composition and bottom composition. Thus the design on input data must be correlated with both variables to get the transfer function precisely. (S.V. Gaikwad 1997)

In addition sinusoidal input design Multi-Sine (M-Sine) signal also can be applied to generate data for identification process. M-Sine input signals are easy to implement in a real time setting and it is versatile periodic signals. By using this type of input signal only one cycle can be designed to include all the frequency content needed for the consistent model identification. It can be identified that this type of input signal is plant friendly by referring to the research that has been conducted as it allows users to simultaneously specify important frequency and time domain properties of the signals. (Lee, 2006). This type of input signal also promoting the presence of low gain directionality in the data as well as give benefits for an optimization-based problem in a system.

System identification basically is to develop a precise model and usually at the start of identification testing step the good model may not be available.(Michael Deflorian, 2011). However preliminary models developed in the course of identification testing may be useful in order to determine the suitable identification test monitoring or test input design that can predict model accurately.

### 2.1.2 Model Structure of System Identification

A study has been done to develop a control relevant constrained design of experiment for system identification of the dynamic multivariable model. In order to identify the dynamic models of the system, plenty of model structures are used such as low order transfer function models, finite impulse response (FIR), high-order auto regressive with exogenous input (ARX) and subspace.

These model structures are developed in the Mat Lab System Identification Toolbox in order to predict the models of the system. Closed loop identification method of multivariable system is very useful and consuming less time compared to step testing for generating the data. It also can reduce the cost of model identification. For ARX models that used as model structure for system identification, there are model parameters that are used to represent and describe the function of system. The transfer function G(s) converted to ARX models with no approximations except zero order hold. The model parameters of parameters can be described as below:

• First Order Process Time Delay (FOPTD):

$$G(s) = \frac{K}{\tau s + 1} e^{-Ds}$$
(Eq 2.1)

• Second Order Process Time Delay (SOPTD) system (time constant):

$$G(s) = \frac{K(\tau_0 s + 1)}{(\tau_1 s + 1)(\tau_2 s + 1)} e^{-Ds}$$
(Eq 2.2)

• Second Order Process Time Delay (SOPTD) system (damping ratio):

$$G(s) = \frac{K(\tau_0 s + 1)\omega^2}{s^2 + 2\xi\omega s + \omega^2} e^{-Ds}$$
(Eq 2.3)

• Ramp system

$$G(s) = \frac{K(\tau_0 s + 1)}{(\tau s + 1)s} e^{-Ds}$$
(Eq 2.4)

Where,

K= process gain,

 $\boldsymbol{\tau}$  = time constant,

 $\boldsymbol{\tau}_0 = \text{lead time},$ 

D= dead time,

 $\boldsymbol{\omega}$  = fixed angular frequency,

 $\boldsymbol{\xi}$  = damping coefficient

ARX model structure is a model that comprising of past output exogenous input variable is represented as past input data. It is one of simplest linear model in the system identification. Apart from that, there are nonlinear auto regressive with exogenous input (NARX) which represent the non-linear system. NARX are able to describe global behaviour of system over the whole operating range and it is different from the linear model as the linear model are only able to estimate the system around at certain operating point only.(Suleyman Karacan, 2007).

ARX model can be written as:

$$y(t) = q^{-nk} \frac{B(q)}{A(q)} u(t) + \frac{1}{A(q)} e(t)$$
 (Eq. 2. 5)

Where the polynomial A(q) and B(q) are defined by,

$$A(q) = 1 + a_1 q^{-1} + \dots + a_{na} q^{-na}$$
 (Eq. 2.6)

 $B(q) = b_0 + b_1 q^{-1} + \dots + b_{na} q^{-nb}$  (Eq. 2. 7)



Figure 2.4: The ARX model structure

Equation of NARX can be written as:

$$y(t) = L^{T}(u-r) + g((u-r)Q) + d$$
 (Eq. 2.8)

Where, y(t) is output, r are the regressors, u is input and L is ARX linear function, D is scalar offset and g((u-r)Q) is describing the output of non linear function and Q is the projection matrix that ensure the calculations is well designed.



Figure 2.5: The NARX model structure

A study has been done by comparing the PRBS and Multi-Sine (M-Sine) perturbation signal which applied to the non-linear system and from the research both ARX and NARX model structure is investigated. Thus it has been clarified that NARX model developed based on M-Sine signal is the most convenient and accurate compared to model developed by PRBS.(Junichiro Kon, 2013). The actual non-linear system behaviour is failed to revealed by using the PRBS signal since at certain levels PRBS input may insufficient to identify the non-linear behaviour by not providing enough information regarding the system. For the binary distillation column case usually the model will be non-linear which is much complex in terms of its algorithm but it also can represented a reunion of linear models one linear model for each operating point and process channel. Thus by using PRBS this model can be identified as well but the probability of getting the bias is quite higher. (Baeisyu, 2011)

#### 2.1.3 Identification Method

One of the methods for system identification is Linear Parameter-Varying (LPV) modelling. In LPV modelling approach the comparison various identification method is used to generate good model. (H. Hapoglu, 2001). Basically there are two approach in the LPV modelling that are local approach and global approach.

Local approach is relies on the identification of multiple Linear Time Invariant (LTI) models at several operating points of the process. All the interpolation of the data set will result in model estimation for the entire operating range. Basically in simplified way, local approach is the interpolation of LTI at different steady state of operating points of the system. Global approach is parameterized LPV at global data set and

has varying operating points. Both approaches have been reviewed in a research and the comparison of the data has been done. It is justified that global approach is suggested if the transient behaviour of the system need to be modelled which require generating data with variations of operating conditions. However if the operating conditions is varies slowly the local approach is recommended as it can generate data around steady state conditions. The combination of both approaches is not really advised as both involve different method as well as the result would be erroneous and require higher costs.(A.A. Bachnas, 2014)

### **2.2 Distillation Process**

Process of heating liquid at its boiling point, condensing it and collecting vapours are known as distillation. Distillation is a method which used to separating mixtures into each individual components and purifying liquids. It is the most common separation technique which consumes high amount of energy for the heating and cooling processes. (Luyben & Yu, 2009) This distillation technology is useful because of its thermodynamic efficiency which is at 10% but other processes are also inefficient and it has high mass transfer rates. However distillation also has the limitations as it is not suitable for a compound that is thermally unstable even under vacuum conditions and for the mixture that is extremely corrosive.

Distillation is highly depending on the boiling point and concentrations of the components. The mixtures separate by distillation also depending on the volatility between the components.(Steinberger, 1982) The greater the relative volatility, the easier the separation between the components can be done. Basically volatility is determined by the components boiling point and the lower the boiling point, the greater the volatility.

Separation can be improved by increasing the contact on the trays within the distillation column tower. As the number of trays increases, there will be higher contact thus providing a better fractionation inside the column. Internal reflux is one of the important operations in the distillation.(Zekieni R. Yelebe, 2014) Internal reflux is the vapours that rise up through the tower and contacting liquid that are dropping back down through the tower. Similar with the number of trays, more reflux will result in more contact and thus higher separation can occur. However reflux rate need to be optimized as too much reflux can be costly as it need more

heat. Apart from this, there are many other variables that can affect the rate of separation and the composition of the products in the distillation column. (Luyben, 2013)

Therefore in this project the control variable are the top and bottom composition. In order to control the both composition, the steam and reflux flow rate is manipulated. Based from Wood-Berry concept on the terminal composition of the binary distillation column, the acetone-isopropyl alcohol distillation has evaluated two control systems that are non-interacting control system and ratio control system. (Berry, 1973) This non-interacting control system is determined by the transfer function that was generated by the modelling of the distillation column dynamics.

# **CHAPTER 3**

# METHODOLOGY

# **3.1 General Flowchart**



Figure 3.1: General flow chart of the project

The methodology of this project covers experimental and simulation work. System identification requires series of basic steps. By following the steps the result gain will be more accurate and precise .Figure above shows the proposed general flowchart for the system identification of the pilot scale of Acetone-Isopropyl alcohol distillation column. In case of any failure during the model validation, where the model validated is not precise compared to the actual plant, several steps need to be repeated to ensure good model is produced. Below are the information and descriptions on the distillation column that located in Block 3, UTP.

Height	5.5 m
Diameter	150 mm
Number of trays	15
Type of tray	Bubble cap
Tray spacing	350 mm
Feed tray location	Tray 7
Feed flow rate	0.5 L/min
Reflux flow rate	0.7 L/min
Distillate flow rate	0.3 L/min
Bottom product flow rate	0.2 L/min
Steam flow rate	20 kg/hr
Top temperature	72.7°C
Bottom temperature	80.5℃
Column pressure	1.013 bar

Table 3.1: Description on Acetone-Isopropyl alcohol distillation column

### **3.2 Experimental Procedures**



Figure 3.2: Experimental procedures

# 3.2.1 Feed mixture preparation

The feed mixture is prepared according to the design. The amount of feed is calculated to ensure that it would be sufficient for the whole experiment. In this experiment the composition used as feed is 70% acetone and 30% isopropyl alcohol. The feed tank (V-104) is filled up to 75% which is 225L in order to maintain continuous feed flow throughout the experiment. To ensure that the feed is sufficient,

during the experiment the products tank from (V-106) and (V-107) is pumped back to the feed tank.

### 3.2.2 Start-up distillation column

The start-up procedure are by checking the boiler, initial valves position, liquid and steam leakages and tune the PID controller. The cooling water flow rate is charged into the condenser. The reboiler is started and the steam flow rate is pumped to the distillation column until the steady temperature profile is achieved.

### **3.2.3** Switching to steady continuous operation

The feed mixture of acetone-isopropyl alcohol is introduced into the distillation column. The control valves and steam flow rate is adjusted until steady state is achieved. The trending variable can be seen through the DCS to determine the stability of the system. It took around 4 to 5 hours for the distillation column to achieve its steady state from the start up. The variables need to be checked frequently to ensure smooth operation.

### **3.2.4 Step change input**

After the column achieved its steady state, the step change input could take place. The manipulated variables that are steam flow rate and reflux flow rate is adjusted to obtain the response from the system. For each step change, it required around 15 minutes to ensure that the system was stable then only another step input could take place. There were 10 step changes that have been done throughout the experiment.

Steam flow is adjusted by controlling valve FCV 331 and the normal opening of this valve are from 10% to 40%. The steam flow exhibited 'spike' about every one to two hours, thus it is crucial to ensure the valve is controlled slowly to ensure the value become stable. Reflux flow rate is adjusted by controlling FCV 323a. Reflux flow rate was smoother and easier to control however it was disturbed by column's pressure. Thus the column's pressure is checked frequently throughout the operation.

### 3.2.5 Samples collection

For each step change, the samples from each tray, top product and bottom product was collected. The composition of acetone and isopropyl alcohol in the samples then was analysed using refractometer. Three readings were taken from each samples and the average is calculated to ensure precise results. These data is then used in the simulation procedure to obtain the system identification model.

### 3.2.6 Data extraction

The experimental data was retrieved from the APC server. All the trending of the process variables system is recorded and extracted to Microsoft Excel to make simulation easier.

# 3.2.7 Shutting down the distillation unit

The distillation unit is shut down carefully. All the pumps were stopped and control valves were closed. Ensure all the liquid mixture is transferred to the feed tank (V-104) to avoid any overflow. Once fully transferred, close compressed air as well as cooling water supply. This is to prevent any damage to the instruments and injuries to the operators.

# 3.3 Simulation (System Identification)



Figure 3.3: Schematic flow chart of system identification

Figure above shows the schematic flow chart of system identification. This project requires using the system identification toolbox from Mat lab. By using the system identification toolbox, model of the observed system in the system can be determined. The noise model in the system is because of the ambiguity information data and this knowledge on the noise will allow the programmer to compensate it. Robust controller can be created from plant uncertainty information and this robust controller can reduce the plant uncertainty thus improving the performance of the application.(Activemedia, 2012)

# 3.3.1 Perform experiment-step testing and collect data

The first procedures that need to be done are the experiment of acetone-isopropyl alcohol distillation process by using Distribution Control System (DCS). The procedure for the experiment can be referred in Figure.

### 3.3.2 Import and preparing data for System Identification

The data collected from the DCS were imported into the System Identification Toolbox as time domain data. Time domain data is choosing as it represents the data with respect to time as well as the data is recorded in a continuous time. The data imported were labelled as  $u_1$ ,  $u_2$ ,  $y_1$  and  $y_2$ .  $u_1$  and  $u_2$ , represent the step input in reflux flow rate and steam flow rate whereas  $y_1$  and  $y_2$  represent the compositions top and bottom of acetone.



Figure 3.4: Import data

# 3.3.3 Pre-processing data

The data need to be pre-processing when:

- Missing or faulty values
- Offsets and drifts in signal levels/ low frequency disturbances
- High frequency disturbances above the frequency interval of interest for the system dynamics
- Nonlinearities in the data

Pre-processing requires several steps in order to provide a good model:

i. Removing means:

In order to improve the system identification, the mean values of the input and output data were removed.

ii. Selecting ranges

The data then were divided into data estimation and data validation. The estimation data is the working data which used to estimate the model whereas the validation data is the data used for testing the accuracy of the estimated model. The data must be separated and the estimation data must be in a wider range compared to the validation data. This is to ensure that the data can predict the model smoothly.



Figure 3.5: Selecting ranges- Data estimation (green) and data validation (red)

### **3.3.4** Choosing a model structure

System identification toolbox can estimate model parameters however it cannot predict the entire models. Thus structures of the model need to be determined. The model needs to be correctly selected as it represents the entire system. Model structures include in the system identification tools box such as process models, state-space models and polynomial models.

For this project, process models low order transfer function is used to estimate the model. This is because the step testing method used in this project is step input which is useful for determining low order (slow) dynamics and for calculating delays. A process model is the simplest model which can easily verify the data and it is a good rule to start with simple model to get a feel for the system and then move towards complex models if necessary.

# 3.3.5 Validating the estimates

To perform validation, the model output can be shown in the toolbox. It will compare between the measured and simulated model output and showed the best fit of the data. If the best fit is more than 80% then, the model can be described as very good model and if below than 80% then it may due to noise or non-linearity in the data.

### 3.4 Gantt Chart and Milestone

All projects have a work plan that lays out the specific steps and actions that are necessary to complete the project. This helps to keep track the progress towards completion. It is also as guidance to identify which actions are required before the next step is possible. All of the activities are expected to be completed within the time to ensure the punctuality as well as producing a valuable research work. However some of the activities were lag behind due to the equipment errors which affect the accuracy of the results. Some maintenance has been done and frequent checking is needed to ensure the outcomes of the experiment were precisely measured.

The Key milestone for General Final Year Project 2 and System Identification of Acetone-Isopropyl Alcohol Distillation Column are shown below. In the Appendix 7.1, the details Gantt chart on this project is presented.

Activities	Time Completion
Experiment step testing	Week 2 – Week 6
Simulation work	Week 7
Submission of Progress Report	Week 8
Project work and modifications continues	Week 9 – Week 10
Pre Engineering Design Exhibition	Week 11
Submission of Draft Report	Week 12
Submission of Dissertation	Week 13
Submission of Technical Paper	Week 13
Oral Presentation	Week 14

 Table 3.3: Key Milestone for System Identification of Acetone-Isopropyl Alcohol

 Distillation Column

Activities	Time Completion
Experiment step testing (Start-up the distillation column	Week 2
and prepare the feed mixture)	
Distillation columns achieve steady state and step input	Week 2 – Week 4
take place.	
Samples gathering and testing using refractometer	Week 5 – Week 6
Simulation work using Matlab System Identification	Week 7
Toolbox	
Project work and modifications continues in case of any	Week 8 – Week 10
non-linearities	
Complete the poster for Pre-SEDEX	Week 11
Validation of model (Simulated model vs. Actual model)	Week 12
Complete dissertation and technical report	Week 13

# **CHAPTER 4**

### **RESULTS AND DISCUSSIONS**

# **4.1 Experiment Step Testing**

# **Reflux and Steam Flow Rate**

The step change for the reflux flow rate has been done for the pilot scale distillation column of acetone and isopropyl alcohol by using the Distribution Control System (DCS). I managed to complete 8 step changes for the reflux flow rate for each 15 minutes and samples for each tray, distillate and bottom has been taken. By using refractometer the compositions for the samples can be identified and tabulated.

Similar with the reflux flow rate, 8 step changes also has been done to the process input steam flow rate to identify the top and bottom composition as the steam flow rate increases. Appendix 7.2.2 and Appendix 7.2.1 showed the several data that has been recorded by using DCS.

Reflux flow rate (L/min)	Steam rate (kg/hr)	Acetone composition (top),X <sub>d</sub>	Acetone composition (bottom),X <sub>b</sub>
0.2	13	0.5485	0.6621
0.3	11.84	0.5802	0.5688
0.4	11.55	0.6055	0.5611
0.5	9.48	0.6207	0.5501
0.6	7.41	0.6561	0.4735
0.7	7.03	0.6858	0.4722
0.8	6.4	0.7133	0.4658
0.9	6.15	0.7384	0.4545

Table 4.1: Experimental data

Where,

	Variables	Symbol
Input	Reflux flow rate	u <sub>1</sub>
Input	Steam Flow rate	u <sub>2</sub>
Output	Acetone composition top,X <sub>d</sub>	y1
Output	Acetone composition bottom, X <sub>b</sub>	<b>y</b> <sub>2</sub>

Table 4.2: Input and Output Parameters for Experiment Step Testing



Figure 4.4: Reflux flow rate (input) vs. Acetone composition at top and bottom



Figure 4.5: Steam flow rate (input) vs. Acetone composition at top and bottom

From Figure 4.1 and Figure 4.2 it can be seen the relationship between both manipulated input that are reflux flow rate and steam flow rate with the corresponding output that are acetone composition at the top and bottom of the distillation column. As the reflux flow rate increases in Figure 4.1, the acetone composition at the top also increases whereas the acetone composition at the bottom decreasing. Based on Figure 4.2, the response between the steam flow rate with acetone composition at the top and bottom are different. It can be seen from the graph; the higher the steam flow rate will result in increasing purity of acetone at the bottom whereas the concentration decreases at the top of the distillation column.

It is desirable to maximise the purity of the top and bottom products. The composition of acetone increases as the reflux flow rate is increases. This is because when the reflux flow rate higher, the more portion of the overhead liquid product from the distillation column is returned to the upper part of the column. Hence this will increase the efficiency of the separation as it can separate the lower boiling materials from higher boiling materials.

For steam flow rate, as the amount of steam into the column increases, the composition of acetone in bottom and top product decreases. This happens because acetone which has lower boiling point than isopropyl alcohol vaporizes more at low temperatures. When the steam flow rate higher, all liquid pushed towards the top of the column thus result in low acetone concentration in condenser compared to isopropyl alcohol.

### 4.2 Simulation (System Identification)

The data from the experiment were used for the modelling step by construct the mathematical model that represents the pilot scale distillation column.

The expected result from the system identification is shown in the form of equation below (Berry, 1973) :

$$\begin{array}{l} X_D(s) \\ X_B(s) \end{array} = \begin{vmatrix} G_{11} & G_{12} \\ G_{21} & G_{22} \end{vmatrix} \begin{pmatrix} R(s) \\ S(s) \end{matrix} \quad (\text{Eq. 4.1}) \end{array}$$

Where,

 $X_D$  = distillate composition

 $X_B$  = bottom composition

$$R = reflux flow rate$$

S= steam flow rate

The relationship between the manipulated variables with control variable can be described as:

$$X_D(s) = G_{11}R(s) + G_{21}S(s)$$
 (Eq. 4.2)  
 $X_B(s) = G_{21}R(s) + G_{22}S(s)$  (Eq. 4.3)

### 4.2.1 Reflux Flow Rate

General equation below used to estimate the result of reflux flow rate step change:

$$G_{11} = \frac{X_D(s)}{R(s)}$$
 (Eq 4.4)  
 $G_{21} = \frac{X_B(s)}{R(s)}$  (Eq 4.5)

 $G_{11}$  represent the relationship between process variable  $u_1$ , reflux flow rate with the  $y_1$ , distillate composition (acetone) whereas  $G_{21}$  represent the relationship between process variable  $u_1$ , reflux flow rate with  $y_2$ , bottom composition (acetone). Figure below showed the time plots which are the output due to step input measured against the time.



Figure 4.3: Time plot of u1 and y1.  $(G_{11})$ 



Figure 4.4: Time plot of u1 and y2 (G<sub>21</sub>)

Figure 4.3 showed as the step input of reflux flow rate,  $u_1$  increases, the step response,  $y_1$  increases as well .Figure 4.4., showed the time plot between the reflux flow rates steps input with the output signals from acetone composition at the bottom. The responses are different as the increasing step input the bottom concentration is dropped. These data has been processed by removing all the noises and disturbance to increases accuracy and to provide a better configuration in describing the relationship between the step inputs with the process output.

Below shown the step response plotted against the time which used to describe the relationship between the input and output in term of transfer function. For this cases the transfer function that obtained is in the low order and linear form which means that this process is a simple process and it can be easily predicted the output response form the input.



Figure 4.5: Step response for G<sub>11</sub>

The transfer function for the step response from  $y_1$  is described as:



$$G_{11} = \frac{0.187}{1.29s + 1} \qquad (\text{Eq } 4.6)$$

Figure 4.6: Step response for G<sub>21</sub>

The transfer function for the step response from  $y_2$  is described as:

$$G_{21} = \frac{0.0031}{0.96s + 1} \qquad (Eq \ 4.7)$$

# 4.2.2 Steam Flow Rate

General equation below used to estimate the step change on the steam flow rate:

$$G_{21} = \frac{X_D(s)}{S(s)}$$
 (Eq 4.8)  
 $G_{22} = \frac{X_B(s)}{S(s)}$  (Eq 4.9)

 $G_{21}$  represent the relationship between the process variable, u2, steam flow rate with y1, distillate composition (acetone) whereas  $G_{22}$  represent the relation between the process variable u2 with y2, bottom composition (acetone).



Figure 4.7: Time plot of  $u_2$  and  $y_1$  (G<sub>12</sub>)



Figure 4.8: Time plot of  $u_2$  and  $y_2$  (G<sub>22</sub>)

From Figure 4.7 and Figure 4.8, the input and output signals are shown. Figure 4.7 presented as the steam flow rate input is decreases, the composition of acetone at the top increases whereas in Figure 4.8 the input signal which is steam flow rate is directly proportional to the acetone composition at the bottom. Both are in negative gradient.

From the graph it can be seen that the step change for the steam flow rate is not uniformly made as there were fluctuations occurred. Steam flow signal is not smoothly changed as the new step input take place. The normal opening for the steam valve is from 10% to 40% however in this experiment the valve is opened wider. This is because when the valve is opened within the range there were absolute no response from the output signal. There was also inconsistency in the composition response as seen from the graph of time plot at certain point the concentration is seem to be maintained and only changes around 0.01. This can be assuming that steam flow rate is not highly affecting the composition in this distillation column as it is hardly to control during the experiment.

Below is shown the step response plotted against time for representing the relationship between steam flow rate with acetone composition at the top and bottom of the distillation column.



Figure 4.9: Step response for  $G_{12}$ 

The transfer function from step response from  $y_1$  is described as:

$$G_{12} = \frac{-0.0086}{0.73s + 1}$$
 (Eq 4.10)



Figure 4.10: Step response for G<sub>22</sub>

The transfer function from step response from  $y_2$  is described as:

$$G_{22} = \frac{-0.00024}{0.84s + 1} \qquad (Eq \ 4.11)$$

The overall mathematical model:

Based on the result gained it can be observed that the transfer function for  $G_{11}$  and  $G_{21}$  have positive gain whereas the transfer function for  $G_{12}$  and  $G_{22}$  are having negative gain. This difference in terms of gain is happened because the different respond towards the process variables. It can be seen that for the positive change in process variables which is reflux flow rate, the distillate composition as well as the bottom composition are also positive. However as the steam flow rate increases the process output which are the distillate and bottom composition are decrease which result in negative gain. This mathematical model can be used to predict the outcome of the distillate and bottom product as the process variables is manipulated.

Time constant is to measure how quickly or how fast a system respond to the change of the input. Based on the time constant of the transfer function that obtained from the simulation, it can be observed that  $G_{11}$  had the largest time constant which is 1.29 whereas  $G_{12}$  had the smallest time constant which is 073. This showed that the respond between the process inputs, reflux flow rate with the distillate composition was slower. As shown in step response figure above, the steepest slope means the faster the process response. Thus for the transfer function  $G_{11}$  which relates  $u_1$  with  $y_1$ , the slope is the least steeper and take longer time to response. As the step input for the reflux flow rate changes, the system requires a longer time correspond.

#### 4.3 Model Validation

Model validation is the most important steps in the model building sequence. Thus in this project model validation is used to determine the accuracy of the model by comparing the simulated model from System Identification Toolbox with the actual data from the experiment. Based from the comparison, the error is calculated to know how accurately the models to represent the system of the distillation column. Error is calculated as follows:

$$Error(\%) = \frac{Experimental data - Model}{Experimental data} \times 100$$

It can be said that assuming the model is very accurate if the percentage of error is less than 10%. In order to do the model validation, the output is calculated by using the transfer function model obtained from the System Identification and using the

similar input with the experiment. Thus, the transfer functions need to be derived using Laplace Transform.

The example of calculation of derivation is shown in the Appendix 7.3. Each of the transfer function is validated and tabulated as follows:

### 4.3.1 Reflux Flow Rate

Following is the model validation for  $G_{11}$  and  $G_{21}$  which the transfer function are describing relationship between the reflux flow rate and the acetone composition for top and bottom of distillation column.

Time				
(min)	Reflux flow rate	X <sub>d</sub> experiment	X <sub>d</sub> model	Error (%)
15	0.2	0.5485	0.3740	31.8146
30	0.3	0.5802	0.5610	3.3092
45	0.4	0.6055	0.5797	4.2609
60	0.5	0.6207	0.5984	3.5927
75	0.6	0.6561	0.6171	5.9442
90	0.7	0.6858	0.6358	7.2908
105	0.8	0.7133	0.6545	8.2434
120	0.9	0.7384	0.6732	8.8299

Table 4.3: Model validation for G<sub>11</sub>



Figure 4.11: Comparison between the experiment and the model for G<sub>11</sub>

Time				
(min)	Reflux flow rate	X <sub>b</sub> experiment	X <sub>b</sub> model	Error (%)
15	0.2	0.6621	0.6200	6.3586
30	0.3	0.5688	0.5704	-0.2813
45	0.4	0.5611	0.5270	6.0773
60	0.5	0.5501	0.5115	7.0169
75	0.6	0.4735	0.4960	-4.7518
90	0.7	0.4722	0.4650	1.5248
105	0.8	0.4658	0.4495	3.4994
120	0.9	0.4545	0.4340	4.5105

Table 4.4: Model validation for G<sub>21</sub>



Figure 4.12: Comparison between the experiment and the model for  $G_{21}$ 

Based from the model validation, it can be seen that reflux flow rate error are mostly less than 10% which is smaller and the model for  $G_{11}$  and  $G_{21}$  are acceptable. However for  $G_{11}$ , as seen in Figure 4.11, the first point of the step change has high error, which is at 32%, this probably because the distillation column has not yet achieved its steady state and disturbances occurred in the reading of the step input. In this case, it is better to do some repeating measurements to increases the accuracy of the model.

# 4.3.2 Steam Flow rate

For the steam flow rate step input, the model represented this response are transfer function  $G_{12}$  and  $G_{22}$ . The model validation for  $G_{12}$  and  $G_{22}$  are as follows:

Time				
(min)	Steam flow rate	X <sub>d</sub> experiment	X <sub>d</sub> model	Error (%)
15	13	0.5485	0.5074	7.4932
30	11.84	0.5802	0.5332	8.1007
45	11.55	0.6055	0.5418	10.5202
60	9.48	0.6207	0.5504	11.3259
75	7.41	0.6561	0.5590	14.7996
90	7.03	0.6858	0.5676	17.2353
105	6.4	0.7133	0.5762	19.2205
120	6.15	0.7384	0.6708	9.1549

Table 4.5: Model validation for  $G_{12}$ 



Figure 6.13: Comparison between the experiment and the model for  $G_{12}$ 

Time				
(min)	Steam flow rate	X <sub>b</sub> experiment	X <sub>b</sub> model	Error (%)
15	13	0.6621	0.5832	11.9166
30	11.84	0.5688	0.4800	15.6118
45	11.55	0.5611	0.4795	14.5393
60	9.48	0.5501	0.4778	13.1358
75	7.41	0.4735	0.4320	8.7645
90	7.03	0.4722	0.4080	13.5959
105	6.4	0.4658	0.4294	7.8231
120	6.15	0.4545	0.4246	6.5875

Table 4.6: Model validation for G<sub>22</sub>



Figure 4.14: Comparison between the experiment and the model for G<sub>22</sub>

Based from the above model validation, the error between the experiment and model for  $G_{12}$  and  $G_{22}$  are mostly more than 10% which means that the model is not very accurate. This is because reflux flow rate signal is smoother compared to steam flow signal and it is easier to control. Other than that the difference between the model and the experiment is due to the equipment error as there were some inconsistency occurred during the experiments which are stated in the problems encountered in the next section. The steam valve also was not very sensitive to the input in the DCS which require opening the valve wider and leading to aggressive behaviour of the distillation column.

# 4.4 Problems Encountered

There are several problems encountered in conducting the experiment and simulation. These problems might affect the accuracy of the results for this project.

# **Equipment setting**

The biggest challenge in conducting the step testing experiment is to attain the process's steady state. It took more than 5 hours to ensure that the process is ready for doing the step change. The temperature of each tray in the distillation column need to be stable to ensure that there is reflux. There are also some difficulties in controlling the steam flow rate and the level for the reflux tank is not matching between the distillation column and the Distribution Control System (DCS). It is observed that when the distillation column showed the reflux drum level is at its maximum but the level at the DCS is at 0 m. The distillation column need to undergo services to ensure that the parameter and data between DCS and distillation column is accurate and precise. Hence it is difficult to ensure that the data correlated as we need to frequently check on the distillation column itself and compared it with the data in the DCS manually.

Other than that, the step input is not an easy way to measure a step response for a complex system since the pump and valve cannot be change instantaneously as it requires more amount of time for the system to stable.

### **Parameter evaluations**

The parameter that I used in this model prediction showed the relation between reflux flow rate and the composition of the samples for bottom and distillate. The samples were taken once in 15 minutes for every step change. Due to the limited workforce, the total samples for each process variables were only 8 which were not correspond with the data from the DCS. Thus the numbers of data used for the simulation were very small and thus it affects the accuracy of the model predicted. During the simulation, I got the best fit data less than 80% which shown that the data of compositions that I gained from the experiment were not really good to represent the model. Thus I need to re-evaluate the parameter that I choose in doing the system identification as the compositions of samples is hardly measure as well as the equipment used to determine the composition was less precise.

40

#### **CHAPTER 5**

### CONCLUSION AND RECOMMENDATION

#### 5.1 Conclusion

As conclusion, this project is important to develop knowledge and understanding on the advanced process control of the system. It also help student in familiarizing with the software as well as understand how the software works. This skill will be very helpful in the student's future career. Identification process is a complex task which requires dealing in numerous interacting parameters. It is very important to do proper interpretations during the model building process and a depth understanding on the application should be gained in order to set up a good and precise model. The process control in the distillation column is essential for product optimization as well as prepared the system whenever any disturbances occur. System identification not only defines the relationship between input and output but also important in making critical decisions to improve the performances of the system.

It is mentioned earlier there were some problems need to be encountered to ensure that an accurate result can be gained. The parameters need to be evaluated correctly so that it can precisely represent the pilot scale distillation column. As a recommendation, a future work need to be done to analyse the suitable interacting parameters to reflect the relationship between the reflux flow rate and steam flow rate with the composition. The other parameters such as temperature and pressure of the distillation column also can be used in relating the mathematical model.

It can be said that this project is showing a good progress and expected result are obtained from the experiment and simulation conducted. It can be conclude that the model  $G_{11}$  and  $G_{21}$  can represent the acetone pilot plant distillation column well enough whereas for model of  $G_{12}$  and  $G_{22}$  require further improvement. The overall mathematical model to represent the acetone-isopropyl alcohol distillation column is obtained and can be used for further research study. The objective is achieved and it is hoped that this project will be beneficial to others.

### **5.2 Recommendations**

#### Obtain good measurement data

The model will be a good model if more samples are taken for each step change as in the modelling the best and accurate configuration can be gained using a huge number of data. Estimating a model appropriately requires that the data collected excites the model in the frequency range of interest. Therefore, wider range of data will provide a higher accuracy model. For more reliable results, it is best to get more data sets since the data can be divided into two that are estimation and validation. 75% of data used for estimation whereas 25% of data is used for validation.

Several improvisations can be done by comparing the step input by using HYSIS model with the actual model or using other type of input signal design such as Pseudo binary Random Sequences (PRBS) and ramp input .Other than that, the refractometer is used in this project to measure the composition of the samples, thus it is preferred if other methods such as Gas Chromatography (GC), High Performance Liquid Chromatography (HPLC) or Spectrophotometer can be used since refractometer is not very accurate compared to other measurement method.

### Variables for System Identification

In this project, the output variables used are composition of acetone at the top and bottom of distillation column. Concentration is the parameters which hardly to measure therefore other type of variables such as pressure and temperature can be used to describe the model very well. Further research need to be done to get the best parameters which can estimate the system perfectly. In addition, it is also good to estimate as many models as possible and this data can simulate into higher order transfer function if necessary to see the difference in terms of the error between experiment and model.

#### **CHAPTER 6**

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# **CHAPTER 7**

# APPENDICES

# 7.1 Gantt Chart

Months		М	ay		June				July				August		
Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	
Experimental work (Reflux Flow Rate)															
Experimental work (Steam Flow Rate)															
Simulation work (Data validation)															
Simulation work (System Identification)															
Submission progress report															
Consultation with supervisor for any changes in result															
Project work continues (Changing parameter)															
Pre-EDX															
Submission Draft Report															
Submission of Dissertation															
Submission of Technical Paper															
Oral Presentation															
Submission of Final Report															

# 7.2 Distribution Control System (DCS) Results

A	В	С	D	E	F	G	Н	I	J
		utpepkssvr	utpepkssvr						
		AI_DACA_LT	AI_DACA_T300	AI_DACA_T300	AI_DACA_T300	AI_DACA_T300	AI_DACA_T300	AI_DACA_T300	AI_DACA_T30
		DACA_005_LT405.PV	DACA_080_TT127.PV	DACA_082_TT129.PV	DACA_084_TT131.PV	DACA_086_TT133.PV	DACA_088_TT135.PV	DACA_090_TT137.PV	DACA_092_T
19/6/2014	2:35:00 PM	503.7559204	73.66134644	65.1521225	64.98138428	65.45092773	61.26776123	58.62126923	56.
19/6/2014	2:34:55 PM	503.6589661	73.60469055	65.12367249	64.95292664	65.42247009	61.26776123	58.63549042	56.
19/6/2014	2:34:50 PM	503.4651489	73.56217957	65.09521484	64.92446899	65.42247009	61.25352859	58.64971924	56
19/6/2014	2:34:45 PM	503.3682251	73.63301086	65.12367249	64.89602661	65.30864716	61.23930359	58.64971924	56.
19/6/2014	2:34:40 PM	503.1743469	73.64718628	65.16635895	64.85333252	65.35132599	61.25352859	58.64971924	56.
19/6/2014	2:34:35 PM	503.0773926	73.7321701	65.2232666	64.8391037	65.37978363	61.26776123	58.63549042	56.
19/6/2014	2:34:30 PM	502.8835144	73.74634552	65.25172424	64.89602661	65.46516418	61.31044769	58.62126923	56.
19/6/2014	2:34:25 PM	502.7866211	73.77467346	65.29441833	64.95292664	65.76395416	61.43849945	58.74932098	56.
19/6/2014	2:34:20 PM	502.6896973	73.8030014	65.32286072	64.98138428	65.89200592	61.48118591	58.80623245	56.
19/6/2014	2:34:15 PM	502.4958191	73.84549713	65.32286072	65.00984192	65.86355591	61.48118591	58.79200745	56.
19/6/2014	2:34:10 PM	502.3988953	73.9163208	65.33709717	65.03829956	65.84931946	61.49541473	58.79200745	56.
19/6/2014	2:34:05 PM	502.2049866	73.88800049	65.33709717	65.08098602	65.86355591	61.48118591	58.7635498	56.
19/6/2014	2:34:00 PM	502.0111389	73.83133698	65.35132599	65.13790131	65.77818298	61.49541473	58.74932098	56.
19/6/2014	2:33:55 PM	501.9142456	73.77467346	65.33709717	65.1521225	65.80664063	61.49541473	58.7635498	56.
19/6/2014	2:33:50 PM	501.8173218	73.76050568	65.32286072	65.13790131	65.87778473	61.48118591	58.74932098	56.
19/6/2014	2:33:45 PM	501.6234436	73.68968201	65.29441833	65.09521484	65.80664063	61.45272446	58.73509598	56.
19/6/2014	2:33:40 PM	501.5265198	73.70384216	65.32286072	65.08098602	65.66435242	61.42427444	58.72087097	56.
19/6/2014	2:33:35 PM	501.3326111	73.71800995	65.32286072	65.08098602	65.6501236	61.42427444	58.74932098	56.
19/6/2014	2:33:30 PM	501.2356873	73.77467346	65.32286072	65.08098602	65.69281769	61.41004181	58.77777863	56.
19/6/2014	2:33:25 PM	501.1387939	73.8030014	65.35132599	65.06676483	65.6501236	61.38158417	58.74932098	56.
19/6/2014	2:33:20 PM	500.9449158	73.88800049	65.39402008	65.09521484	65.6501236	61.42427444	58.70663452	56.
19/6/2014	2:33:15 PM	500.8479919	73.90216064	65.45092773	65.1521225	65.82086182	61.50964355	58.77777863	56.
19/6/2014	2:33:10 PM	500.6540833	73.9871521	65.4936142	65.2232666	65.87778473	61.55232239	58.84891891	56.
19/6/2014	2:33:05 PM	500.5571594	74.07215118	65.47938538	65.28018188	65.9489212	61.59501648	58.86315155	56.
19/6/2014	2:33:00 PM	500.3632813	73.95883179	65.46516418	65.29441833	65.87778473	61.58078384	58.84891891	56.
19/6/2014	2:32:55 PM	500.2663879	73.83133698	65.43669891	65.32286072	65.80664063	61.58078384	58.83469391	56.
19/6/2014	2:32:50 PM	500.1694641	73.83133698	65.40824127	65.30864716	65.84931946	61.56655884	58.82045746	56.
19/6/2014	2:32:45 PM	499.9756165	73.77467346	65.37978363	65.29441833	65.83509827	61.5381012	58.83469391	56.
19/6/2014	2:32:40 PM	499.8786316	73.77467346	65.36555481	65.25172424	65.77818298	61.49541473	58.82045746	56.

Appendix 7.2.1: Several data that has been recorded using DCS for the reflux flow rate step change

Α	В	C	D	F	F	G	Н	1		K		M	N	0	P	0	R		S
		utnenkssv	utnenkssv	utnenkssv	utnenkssv	utnenkssv	utnenkssv	, utnenkssvi	utnenkssv	utnenkssy	rutnenkssvr		utnenkssvrutne						
		AL DACA	AL DACA	AL DACA	AL DACA	AI DACA	AL DACA	AI DACA	AL DACA	AI DACA	AL DACA	AI DACA	C10 FIC331	-	C12 LIC40 C18				
		DACA 005	DACA 080	DACA 082	DACA 084	DACA 086	DACA 088	DACA 090	DACA 092	DACA 09	DACA 095	DACA 096	DACA 09	DACA 098	DACA 10	DACA 10	2 pida.PV	[	DATAACQ pida
30/6/201	4 1:18:40 PN	454.1267	79.20011	76.32147	73,70344	70.11786	71.27037	69.50604	66.95914	66.07698	60,1864	29.56675	23.8075	30,59403	77.63556	75.08351		0	23.55779
30/6/201	4 1:18:35 PN	453.5451	79.20011	76.34993	73.76035	70.1321	71.29883	69.49181	66.94492	66.04852	60.22433	29.5952	23.79805	30.57513	77.63556	75.08351		0	23.79777
30/6/201	4 1:18:30 PN	452.9635	79.18594	76.39262	73.81726	70.1321	71.34151	69.49181	66.91646	66.02007	60.27176	29.62366	23.8075	30.56567	77.66391	75.05517		0	24.03774
30/6/201	4 1:18:25 PN	452.382	79.20011	76.42107	73.87418	70.14632	71.36996	69.52026	66.91646	65.97738	60.30971	29.63315	23.79805	30.55622	77.67809	75.05517		0	24.27772
30/6/201	4 1:18:20 PN	451.8004	79.20011	76.46375	73.91686	70.17477	71.38419	69.5345	66.93069	65.93469	60.35714	29.64263	23.79805	30.57513	77.70645	75.06934		0	24.51769
30/6/201	4 1:18:15 PN	451.2187	79.18594	76.50644	74.00224	70.21746	71.39843	69.5345	66.93069	65.89201	60.41405	29.6616	23.79805	30.56567	77.72062	75.11188		0	24.71767
30/6/201	4 1:18:10 PN	450.6371	79.20011	76.5349	74.04492	70.24592	71.39843	69.5345	66.90224	65.8351	60.46148	29.6616	23.79805	30.54677	77.7348	75.14023		0	24.99765
30/6/201	4 1:18:05 PN	450.0556	79.18594	76.54913	74.10184	70.27438	71.41266	69.54872	66.888	65.77818	60.51839	29.68057	23.79805	30.53731	77.72062	75.1544		0	25.23763
30/6/201	4 1:18:00 PN	449.474	79.14345	76.57758	74.1872	70.33129	71.42688	69.57718	66.87377	65.72126	60.58479	29.69955	23.79805	30.52787	77.72062	75.18277		0	25.47761
30/6/201	4 1:17:55 PN	448.8924	79.14345	76.62027	74.22989	70.37398	71.44111	69.59141	66.85954	65.65012	60.65119	29.70903	23.79805	30.50896	77.7348	75.21111		0	25.67759
30/6/201	4 1:17:50 PN	448.3108	79.25677	76.64873	74.31526	70.3882	71.44111	69.59141	66.84532	65.56476	60.71759	29.71852	23.79805	30.49951	77.72062	75.2253		0	25.91756
30/6/201	4 1:17:45 PN	447.7292	79.29928	76.69141	74.35794	70.43089	71.44111	69.57718	66.83109	65.47939	60.80296	29.728	23.8075	30.49006	77.72062	75.23948		0	26.15754
30/6/201	4 1:17:40 PN	447.1476	79.20011	76.71987	74.44332	70.44512	71.46957	69.56296	66.81686	65.37978	60.87885	29.728	23.79805	30.47115	77.72062	75.2253		0	26.39751
30/6/201	4 1:17:35 PN	446.566	79.14345	76.73409	74.45753	70.4878	71.49803	69.56296	66.7884	65.28018	60.95473	29.71852	23.79805	30.45225	77.7348	75.23948		0	26.6375
30/6/201	4 1:17:30 PN	445.9845	79.14345	76.77679	74.486	70.50203	71.51225	69.57718	66.77418	65.16636	61.0401	29.70903	23.79805	30.39554	77.7348	75.25366		0	26.87748
30/6/201	4 1:17:25 PN	445.4028	79.18594	76.80524	74.54292	70.51626	71.54071	69.59141	66.74571	65.02407	61.12548	29.71852	23.8075	30.41444	77.76316	75.26782		0	27.11745
30/6/201	4 1:17:20 PN	444.8212	79.18594	76.80524	74.59984	70.50203	71.55493	69.59141	66.71725	64.86756	61.20136	29.71852	23.79805	30.37663	77.77734	75.26782		0	27.35743
30/6/201	4 1:17:15 PN	444.2397	79.24261	76.81947	74.64252	70.50203	71.55493	69.59141	66.70303	64.71104	61.26776	29.71852	23.79805	30.37663	77.76316	75.25366		0	27.5974
30/6/201	4 1:17:10 PN	443.6581	79.34177	76.84792	74.74211	70.51626	71.56917	69.59141	66.67458	64.52608	61.34365	29.70903	23.79805	30.35773	77.79151	75.25366		0	27.83738
30/6/201	4 1:17:05 PN	443.0764	79.38426	76.86215	74.75634	70.53049	71.5834	69.59141	66.64612	64.32689	61.41953	29.70903	23.79805	30.35773	77.81987	75.28201		0	28.07736
30/6/201	4 1:17:00 PN	442.4949	79.39843	76.87639	74.77058	70.53049	71.59763	69.57718	66.61766	64.08499	61.48593	29.69955	23.79805	30.34828	77.83405	75.29619		0	28.31734
30/6/201	4 1:16:55 PN	441.9133	79.39843	76.90484	74.75634	70.54472	71.61185	69.59141	66.60344	63.82888	61.56181	29.69955	23.79805	30.32937	77.8624	75.29619		0	28.55732
30/6/201	4 1:16:50 PN	441.3317	79.44093	76.89061	74.79903	70.5874	71.61185	69.57718	66.57497	63.51585	61.62821	29.69955	23.79805	30.32937	77.87658	75.29619		0	28.79729
30/6/201	4 1:16:45 PN	440.7501	79.52592	76.87639	74.87017	70.71545	71.61185	69.59141	66.56075	63.17437	61.71358	29.69006	23.79805	30.31047	77.87658	75.29619		0	29.03727
30/6/201	4 1:16:40 PN	440.1685	79.61092	76.86215	74.87017	70.85774	71.62608	69.59141	66.54652	62.80443	61.80844	29.69006	23.79805	30.30102	77.8624	75.29619		0	29.27725
30/6/201	4 1:16:35 PN	439.5869	79.62508	76.91907	74.95554	70.8862	71.65454	69.60564	66.51806	62.37758	61.89381	29.69955	23.79805	30.27266	77.8624	75.31037		0	29.51722
30/6/201	4 1:16:30 PN	439.0053	79.65342	76.97598	75.02668	70.87197	71.68299	69.60564	66.50384	61.89381	61.94123	29.6616	23.8075	30.25376	77.87658	75.33871		0	29.7572
30/6/201	4 1:16:25 PN	438.4237	79.66758	76.9902	75.0836	70.87197	71.69722	69.61987	66.4896	61.41004	61.97918	29.63315	23.8075	30.2443	77.87658	75.3529		0	29.99717
30/6/201	4 1:16:20 PN	437.8422	79.66758	76.97598	75.15474	70.90043	71.71145	69.61987	66.47537	60.96896	62.05507	29.5952	23.79805	30.2065	77.90494	75.3529		0	30.23716

Appendix 7.2.2: Several data that has been recorded using DCS for the steam flow rate step

changes

# 7.3 Model Validation

Example calculation:

Derive transfer function into Laplace Transform for G<sub>11</sub>:

$$G_{11} = \frac{X_D(s)}{R(s)}$$
 (Eq 8.1)  
 $X_D(s) = R(s).G_{11}$  (Eq8.2)

Where,

$$G_{11} = \frac{0.187}{1.29s + 1} \quad (Eq 8.3)$$
$$X_D'(s) = \frac{0.187}{1.29s + 1} R'(s) \quad (Eq 8.4)$$

Where, R'(s) is the magnitude of step change for the reflux flow rate and for the first step change is:

$$R' = \frac{0.2}{s}$$
 (Eq 8.5)

Thus,

$$X_{D}' = \frac{0.187}{1.29s+1} \cdot \frac{0.2}{s} = \frac{0.0374}{s(1.29s+1)}$$
 (Eq 8.6)

Deriving in the Laplace Transform:

y = KM 
$$\left(1 - e^{-\frac{t}{\tau}}\right)$$
 (Eq 8.7)  
X<sub>D</sub>'(t) = 0.0374  $\left(1 - e^{-\frac{t}{1.29}}\right)$  (Eq 8.8)

Where t is the respective time at the step change is take place. Then by using this equation the model predictive top composition is estimated.