

**ANALYSIS OF PARTIAL LEAST SQUARE ESTIMATION PROCESS AND
CONTROL OF DISTILLATION COLUMN PROCESS**

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requirements for the award of the degree of
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I declare that this thesis entitled “*Analysis of Partial Least Square estimation process and control of distillation column process*” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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To my beloved mother, father, younger brothers.
Thank you for your supporting.

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ABSTRACT

Distillation columns are the most important separation process in unit operation. The used of distillation column is widely in industries especially in chemical and petroleum industries for separating the substances based on the differences of substances volatility. To incentive economic and control the quantity of the product in the industry, on-line handling the control system is needed and the MATLAB programming is proposed. In this thesis, the distillation column programme in MATLAB is used to analysis of Partial Least Square (PLS) Estimation Process and Control of Distillation Column Process. The analysis worked by generated the data for dynamic response and the base case control in nominal condition. Based on the generated data the estimation product is predicted by using the PLS estimator to maintain the desired product when the disturbance occurs. The estimation result show that the prediction data is nearest to actual data. Based on the result the distillation column control programme is proposed to be the best control process.

ABSTRAK

Kolum penyulingan adalah proses pemisahan yang terpenting di dalam unit operasi. Penggunaan kolum penyulingan ini sangat meluas di dalam industri terutama sekali dalam industri kimia dan petroleum dengan tujuan memisahkan bahan-bahan di dalam campuran berdasarkan kepada perbezaan kebolehan meruap bahan tersebut. Bagi meransangkan pembangunan ekonomi serta mengawal pengeluaran jumlah produk di dalam industri, pengawal system control secara 'on-line' amatlah diperlukan dan penggunaan program MATLAB disarankan. Di dalam tesis ini, program kolum penyulingan di dalam MATLAB digunakan bagi menganalisa "Partial Least Square (PLS) Estimation Process and Control of Distillation Column Process". Analisis dapat dijalankan dengan menghasilkan data untuk respon dinamik dan kawalan kes dasar pada keadaan biasa. Berdasarkan daripada data yang dihasilkan produk yang dihasilkan adalah anggaran daripada PLS estimator dengan mengekalkan produk yang dikehendaki apabila gangguan wujud. Keputusan anggaran menunjukkan bahawa anggaran produk adalah berhampiran dengan produk yang sebenar. Berdasarkan keputusan ini, program pengawalan kolum penyulingan menggunakan system ini adalah system pengawalan yang terbaik.

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LIST OF SYMBOLS

SYMBOLS

a	-	Last dimension in PLS
B	-	Regression coefficient in PLS
b_0	-	Bias weight of neuron model
\hat{b}_{ak}	-	Regression coefficient in inner PLS
c_i	-	Coefficients of polynomial function.
\mathbf{e}	-	Matrix of mismatch between the \mathbf{u} and $\hat{\mathbf{u}}$
\mathbf{E}	-	Residue matrix of \mathbf{X} blocks in PLS
\mathbf{F}	-	Residue matrix of \mathbf{Y} blocks in PLS
\mathbf{f}	-	Residual matrix of inner PLS
\mathbf{g}_{ak}	-	Loading scores of e-block in inner PLS
\mathbf{h}_{ak}	-	Loading scores of Z-block in inner PLS
k	-	Last dimension in the inner PLS
K_c	-	Controller gain
K_p	-	Steady state gain
\mathbf{P}	-	Input loading factors matrix in PLS
\mathbf{Q}	-	Output loading factors matrix in PLS
\mathbf{T}	-	Input latent scores matrix in PLS
t_s	-	Settling time
\mathbf{U}	-	Output latent scores matrix in PLS
$\hat{\mathbf{u}}$	-	Predicted output latent scores matrix in PLS
\mathbf{v}_{ak}	-	Weight in the inner PLS
\mathbf{w}	-	Input weights matrix in PLS
w_i	-	Neuron weight

\mathbf{X}	-	Independent variables matrix in PLS
\mathbf{x}	-	Input variables
x_i	-	Neuron input
\mathbf{Y}	-	Dependent variables matrix in PLS
y	-	Neuron output
\hat{y}_t^c	-	Filtered output
\hat{y}_t	-	Predicted value at current time
$\hat{y}_{t_f}^c$	-	Previous corrected predicted value at time t_f

GREEK SYMBOLS

α	-	Prediction coefficient in PLS
ϵ	-	Residual matrix of inner relation in quadratic PLS
ω	-	Weight of neural network model
β	-	Bias of neural network model
σ	-	Activation function in neural network
τ_D	-	Derivative time constant
τ_I	-	Integral time constant

ABBREVIATIONS

DMC	-	Dynamic matrix control
EKF	-	Extended Kalman Filter
GPC	-	Generalized Predictive Control
HJB	-	Hamilton Jacobi Bellman
FOPDT	-	First order plus dead time
MPC	-	Model predictive control
MSE	-	Mean squared error of prediction

NIPALS	-	Non-linear iterative partial least squares
NLP	-	Non-linear programming
NLMPC	-	Non-linear model predictive control
QDMC	-	Quadratic dynamic matrix control
OCP	-	Optimal control problem
PCA	-	Principal component analysis
PCR	-	Principal component regression
PID	-	Proportional-Integral-Derivative
PLS	-	Partial least squares regression
PMP	-	Pontryagin's minimum principle
QP	-	Quadratic programming
SISO	-	Single input single output
SPC	-	Statistical process control
TPBVP	-	Two-point boundary value problem

CHAPTER 1

INTRODUCTION

1.1 Introduction

Distillation columns are important process unit operation in separation of substances based on differences in their volatility especially in the chemical and petroleum industries by R.A. Abou-Jeyab et al (2001). Therefore, they need to be maintained close optimum operating conditions because of economic incentives. These units have been considered for application of advanced control and optimization. The optimal operating point usually lies at same constraints and the operation of the distillation columns close to the optimum is an important objective. Based on the market conditions, the real-time optimizer updates the optimum periodically. At these updates, the objective of the control system of distillation columns is to move the process to the new optimal operating point. At the same time, the objective of the control system is to cancel the effect of the disturbances on the controlled variables by making the minimal changes in the manipulated variables from their optimal values. In addition, the constraints on the manipulated and other process variables need to be satisfied. Thus, the control system needs to be handling on-line and the using of MATLAB is purposed.

In this study, the used of distillation column is to separate the mixture of methanol (MeOH) and ethanol. The particular column studied in this unit has 27 trays, a reboiler on the bottom tray, and a total condenser on the overhead stream. A 50%-50% mixture of methanol and ethanol is fed at the fourteenth tray (counted from bottom). This column was originally modeled by K. Weischedel and T.J

McAvoy in 1980 by F.J Doyle et al (1995). It represents a benchmark that has been studied by a number of researchers for the purpose of controlled design. The specific control objective is to achieve an 85% methanol stream at top. Although there are many variables that can be considered in this distillation column but in this study, the variables that considered are reflux ration and vapor flow rate for manipulated variables. Meanwhile for controlled variables is overhead MeOH composition.

Methanol also known as methyl alcohol or wood alcohol and the chemical formula is CH_3OH . It is simplest alcohol and is light, volatiles, colorless, flammable, poisonous liquid with distinctive odor. Its boiling point is 64.7°C . Methanol is produced naturally in anaerobic metabolism of many varieties of bacteria. But nowadays the synthesis of gas is most commonly produced from the methane component in natural gas. The mixture of methane and steam at certain condition can produce carbon monoxide and hydrogen. Basically the carbon monoxide then mix with hydrogen then reacts on the second catalyst to produce methanol. Methanol is used on a limited basis to fuel internal combustion engine. The methanol blends are the fuel of choice in open wheel racing circuit. Besides, methanol is required with supercharge engine in a top alcohol dragster and until the end of 2005 seasons, all the vehicle in the Indianapolis 500 had to run methanol.

1.2 Problem Statement

In recent year, although we know the distillation column is needed to be controlled on-line by using control system but most of the industry still using the manual controller. It is because the cost of software for the control system is too expensive. But using the Matlab software the cost is inexpensive if compared to the other software such as Aspen. Besides this is new technology that have to make big changes for the old company or industry which have develop their controlled previously. Thus, for the new company or industry that in going to develop they have been suggested to use the on-line controlled system so that the engineer can manage the problem of the product or feed before it will affect the whole plan.

Besides, while using the control system it has the sensor in the system so that the engineer can know the prediction of the controlled system before the outgoing product ruin the whole plan. But the problem is most of the industry here still lack off soft sensor in their plan because of using the manual controller. Meanwhile, there are some factor the affecting of distillation column such as the feed condition, the internal liquid and fluid flow condition, state of tray and the weather condition. Because of this factor the controlling system by manually is not effective.

Because of this, in this study the using of Matlab software to estimate the product of the methanol has been used so that it will apply in most of the industry which using distillation column in their plan.

1.3 Objective

The aim of this study is to:

- i. To estimate product composition in distillation column.

1.4 Scope of study

To achieve the objective of this research, there are two scopes that have been identified:

- i. To apply simulation studies of distillation column.
- ii. Analysis of dynamic response of the process.
- iii. To develop base-case control for distillation column.
- iv. To study and analysis the simulation of dynamic response for distillation column using Proportional Integral Derivative (PID).
- v. Development of inferential estimator using Partial Least Square (PLS).

CHAPTER 2

LITERATURE REVIEW

2.1 Overview of Distillation Column

Distillation is a method for separating the various components of a liquid solution which depends upon the distribution of these components between a vapor phase and a liquid phase. All components are present in both phases. The vapor phase is created from the liquid phase by vaporization at the boiling point. The basic requirement for the separation of components by distillation is that the composition of the vapor be different from the composition of the liquid with which it is in equilibrium at the boiling point of the liquid. Distillation is concerned with solutions where all components are appreciably volatile.

Distillation was developed into its modern form with the invention of the alembic by Islamic alchemist Jabir ibn Hayyan c. 800; he is also credited with the invention of numerous other chemical apparatus and processes that are still in use today. The design of the alembic has served as inspiration for some modern micro-scale distillation apparatus such as the Hickman still head.



Figure 2.1: Alembic

2.1.1 The Theory of Distillation Column

It is a common misconception that in a solution, each component boils at its normal boiling point - the vapors of each component will collect separately and purely. This does not occur even in an idealized system. Idealized models of distillation are essentially governed by Raoult's law and Dalton's law.

Raoult's law assumes that a component contributes to the total vapor pressure of the mixture in proportion to its fraction of the mixture and its vapor pressure when pure.

For component A,

$$P_A = X_A P_A^\circ \quad (2.1)$$

Where:

X_A denotes the mole fraction of A

P_A° denotes the vapor pressure of pure A.

If a component changes another's vapor pressure, or the volatility of a component is dependent on its fraction, the law will fail. Dalton's law states that the total vapor pressure is the sum of the vapor pressures of each individual component in the mixture.

$$P_{\text{total}} = \sum P_i, \text{ for components } i = A, B, C, \dots \quad (2.2)$$

2.1.1.1 Raoult's Law

Raoult's law states that the vapor pressure of each component in an ideal solution is dependent on the vapor pressure of the individual component and the mole fraction of the component present in the solution. Once the components in the solution have reached chemical equilibrium, the total vapor pressure of the solution is:

$$P_{\text{solution}} = (P_1)_{\text{pure}} X_1 + (P_2)_{\text{pure}} X_2 \dots \quad (2.3)$$

and the individual vapor pressure for each component is

$$P_i = (P_i)_{pure} X_i \quad (2.4)$$

Where;

(P_i) pure is the vapor pressure of the pure component

X_i is the mole fraction of the component in solution

Consequently, as the number of components in a solution increases, the individual vapor pressures decrease, since the mole fraction of each component decreases with each additional component. If a pure solute which has zero vapor pressure is dissolved in a solvent, the vapor pressure of the final solution will be lower than that of the pure solvent.

This law is strictly valid only under the assumption that the chemical bond between the two liquids is equal to the bonding within the liquids, the conditions of an ideal solution. Therefore, comparing actual measured vapor pressures to predicted values from Raoult's law allows information about the relative strength of bonding between liquids to be obtained. If the measured value of vapor pressure is less than the predicted value, fewer molecules have left the solution than expected. This is put down to the strength of bonding between the liquids being greater than the bonding within the individual liquids, so fewer molecules have enough energy to leave the solution. Conversely, if the vapor pressure is greater than the predicted value more molecules have left the solution than expected, due to the bonding between the liquids being less strong than the bonding within each.

2.1.1.2 Dalton's Law

In chemistry and physics, Dalton's law states that the total pressure exerted by a gaseous mixture is equal to the sum of the partial pressures of each individual component in a gas mixture. This empirical law was observed by John Dalton in 1801 and is related to the ideal gas laws. Mathematically, the pressure P of a mixture of n gases can be defined as the summation

$$P_{total} = p_1 + p_2 + \cdots + p_n \quad (2.5)$$

where p_1 , p_2 , p_n represent the partial pressure of each component.

It is assumed that the gases do not react with each other. The combination of Dalton's Law, Boyle's and Avogadro's laws lead to the conclusion that a gas in an ideal gas mixture exerts a partial pressure whose ratio to total pressure equals its mole fraction.

$$P_i = P_{total} m_i \quad (2.6)$$

Where

m_i = The mole fraction of the i component in the total mixture of m components

The relationship below provides a way to determine the volume based concentration of any individual gaseous component.

$$P_i = \frac{P_{total} C_i}{1,000,000} \quad (2.7)$$

where:

C_i = Is the concentration of the i component expressed in ppm.

Dalton's law is not exactly followed by real gases. Those deviations are considerably large at high pressures. In such conditions, the volume occupied by the molecules can become significant compared to the free space between them. Moreover, the short average distances between molecules raise the intensity of intermolecular forces between gas molecules enough to substantially change the pressure exerted by them. Neither of those effects is considered by the ideal gas model.

The theory of the distillation column is the combination of the Raoult's and Dalton's law which is applied in any types of distillation column.

2.1.2 Types of Distillation Column

There are three important types of distillation that occur in a single stage or still and that do not involve rectification. The first of these is equilibrium or flash distillation, the second is simple batch or differential distillation, and the third is simple steam distillation.

2.1.2.1 Equilibrium or Flash Distillation Column

In equilibrium or flash distillation, which occurs in a single stage, a liquid mixture is partially vaporized. The vapor is allowed to come to equilibrium with the liquid, and the vapor and liquid phases are then separated. This can be done batch wise or continuously.

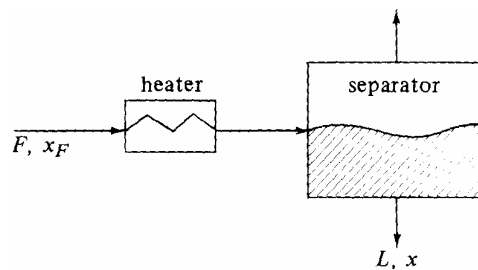


Figure 2.2: Flash distillation

In Figure 2.2 a binary mixture of components A and B flowing at the rate of F mol/h into a heater is partially vaporized. Then the mixture reaches equilibrium and is separated. The composition of F is x_F mole fraction of A. A total material balance on component A is as follows:

$$F x_F = V y + L x \quad (2.8)$$

Since $L = F - V$, becomes

$$F x_F = V y + (F - V) x \quad (2.9)$$

2.1.2.2 Simple Batch or Differential Distillation Column

In simple distillation, all the hot vapors produced are immediately channeled into a condenser which cools and condenses the vapors. Thus, the distillate will not be pure - its composition will be identical to the composition of the vapors at the given temperature and pressure, and can be computed from Raoult's law.

As a result, simple distillation is usually used only to separate liquids whose boiling points differ greatly (rule of thumb is 25 °C), or to separate liquids from involatile solids. For these cases, the vapor pressures of the components are usually sufficiently different that Raoult's law may be neglected due to the insignificant contribution of the less volatile component. In this case, the distillate may be sufficiently pure for its intended purpose.

2.1.2.3 Simple Steam Distillation Column

At atmospheric pressure high-boiling liquids cannot be purified by distillation, since the components of the liquid may decompose at high temperatures required. Often the high-boiling substances are essentially insoluble in water, so a separation at lower temperatures can be obtained by simple steam distillation. This method is often used to separate a high-boiling component from small amounts of nonvolatile impurities.

If a layer of liquid water (A) and an immiscible high-boiling component (B) the phase rule for three phase and two components,

$$F = 2 - 3 + 2 = \text{degree of freedom} \quad (2.10)$$

Hence, if the total pressure is fixed, the system is fixed. Since there are two liquid phases, each will exert its own vapor pressure at prevailing temperature and cannot be influenced by the presence of the other. When the sum of the separate vapor pressure equals the total pressure, the mixture boils and

$$P_A + P_B = P \quad (2.11)$$