

## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1 Background**

Distillation is a commonly used process equipment in industries such as oil refineries. The distillation process is a separation process of liquid-liquid mixtures containing two or more components based on the difference in volatility of each component. An ethanol-water solution is a mutually soluble liquid-liquid mixture where both have sufficiently high boiling points, allowing their separation to be performed via distillation. In a laboratory setting, the distillation separation process can be carried out in a tray column operated in batch mode.

To enhance the efficiency of distillation separation, a reflux system can be employed, where the condensed liquid from the top of the column is returned to the column to re-contact the vapour phase.

Using the same apparatus, improved efficiency can be observed through increased ethanol purity in the distillate. Based on this, a batch distillation experiment was conducted to determine the effect of the reflux ratio on the ethanol composition in the distillate.

#### **1.2 Problem Statement**

Ethanol-water solutions can be separated by batch distillation using a reflux system. When compared to existing equipment, the reflux ratio will affect the increase in separation efficiency, thereby increasing the ethanol composition in the distillate.

#### **1.3 Practicum Objectives**

1. Able to carry out batch distillation experiments with a reflux system.
2. Able to assess the effect of heating energy on the boil-up rate obtained during one minute of operation.
3. Able to assess the effect of reflux ratio ( $R$ ) on the ethanol composition in the distillate during one minute of operation.

#### **1.4 Manfaat Hasil Percobaan**

By using the same equipment and control variables, it is possible to separate the desired product and ethanol composition by operating the equipment



at a specific reflux ratio, which can serve as a guide for practitioners to perform batch distillation operations with a reflux system.



## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Definition of Distillation

Distillation is a separation process for a homogeneous mixture (liquids that are mutually soluble) based on differences in boiling points or vapour pressures (of each component in the mixture) using heat as the separating agent or Energy Separating Agent (ESA).

Distillation is a separation process based on the principle of diffusion. Through diffusion, the separation process occurs due to the mass transfer in opposite directions between the vapour phase and the liquid phase, or vice versa, as a result of the potential difference between the two phases in contact. At a certain temperature and pressure, the system reaches equilibrium. Simply put, the distillation process can be illustrated as follows:

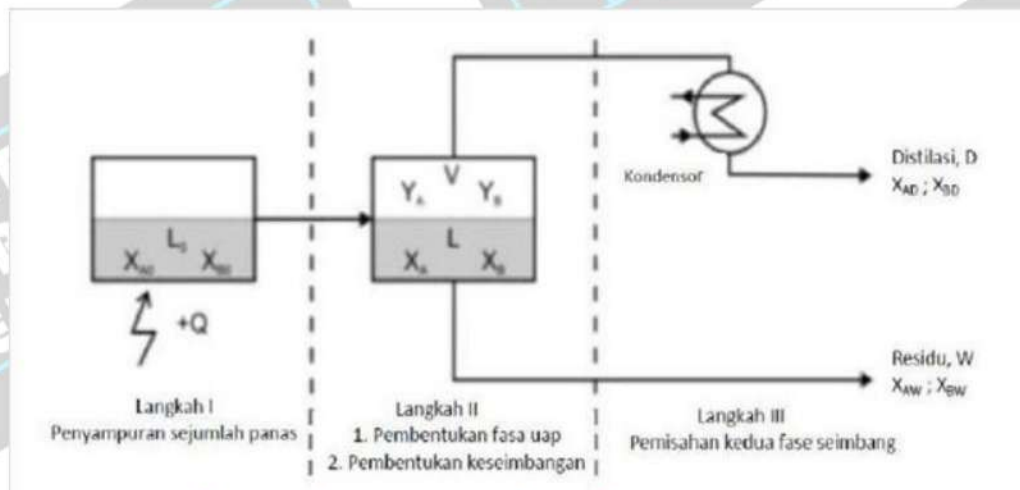


Figure 2.1 Steps in the distillation separation process

In another form, distillation is defined as follows:

$$[X_A]_D > [X_A]_W \text{ dan } [X_B]_D < [X_B]_W$$

Where:  $X_A, X_B$  = Composition of Components A and B

A, B = Components with high vapour pressure, low

D = Peak yield (distillate)

W = Bottom result (residue)

A simple diagram in Figure 2.1 shows that the distillation process consists of three basic steps, namely:

1. Addition of a certain amount of heat (ESA) to the solution to be separated.
2. Formation of a vapour phase, which may be followed by equilibrium.
3. Separation step.

In distillation separation operations, the vapour phase will immediately form after the mixture is heated. Heat is obtained from the reboiler to produce



vapour. The vapour and the remaining liquid are allowed to come into contact with each other so that at some point all components form a phase equilibrium. Once equilibrium is reached, the vapour phase leaves the top of the column and is condensed into liquid, which is collected in a vessel. The liquid phase or bottom liquid exits the bottom of the column and is heated in the reboiler. Some of the formed liquid is returned to the column, while the remainder is discharged as residue (Geankoplis, 1993).

In equilibrium, the composition of the distillate is not the same as the composition of the residue, where:

1. Components with high pure vapour pressure are more abundant in the distillate.
  2. Components with low pure vapour pressure are mostly found in the residue.
- Pure vapour pressure is defined as the pressure exerted by vapour in thermodynamic equilibrium with its condensed phase (liquid or solid) at a certain temperature in a closed system. This pressure indicates the rate of evaporation of a liquid and the tendency of particles to escape into the vapour phase. Vapour pressure is a measure of a compound's ability to bond with itself. Compounds with molecules that bond well with each other will have low vapour pressure (a smaller tendency to escape into the vapour phase), while compounds with poor bonding will have high vapour pressure.

## **2.2 Types of Distillation Processes**

### **1. Batch Distillation**

In some chemical industries, especially when the feed quantity is small, distillation is carried out in batches. The same applies when distillates with varying compositions are desired. Batch distillation is typically carried out in a distillation column with a specific number of plates, and the feed is introduced only once per batch operation. The distillate is continuously removed, but the bottom product (residue) is only removed after the batch operation is complete.

In batch distillation, the composition of the distillate is highly dependent on the composition of the residue, the number of stages in the column, and the reflux ratio of the operation. Immediately after the column begins operation, the distillate will have a very high concentration of the more volatile components. On the other hand, the residue concentration will decrease due to the absence of incoming feed. As a result, the concentration



of subsequent distillates will also decrease. Based on this, batch distillation can operate under two possibilities:

- a) Dengan kadar distilat konstan, rasio refluks berubah
- b) Dengan rasio refluks konstan, kadar distilat berubah

## 2. Continuous Distillation

Continuous distillation using reflux is usually carried out in a distillation column that has trays adjusted to the requirements. The calculation method in the distillation process developed by McCabe and Thiele is based on the mass balance in the enriching section, the mass balance in the stripping section, and equilibrium data.

The McCabe-Thiele calculation assumption is constant molar overflow (equimolar overflow), meaning that the number of moles between the feed entering the top tray and the bottom tray is the same.

Total mass balance equation:

$$V_{n+1} + L_{n+1} = V_n + L_n$$

Component mass balance equation:

$$V_{n+1} Y_{n+1} + L_{n-1} X_{n-1} = V_n Y_n + L_n X_n$$

dimana :

$V_{n+1}$  = Flow rate of vapour from tray n+1

$Y_{n+1}$  = Fraction of vapour molecules in  $V_{n+1}$

$L_{n-1}$  = Flow rate liquid from tray n-1

$X_{n-1}$  = Fraction of liquid molecules in  $L_{n-1}$

$V_n$  = Flow rate of vapour from tray n

$Y_n$  = Fraction of vapour molecules in  $V_n$

$L_n$  = Flow rate liquid dari tray n

$X_n$  = Fraction of liquid molecules in  $L_n$

### 2.3 Characteristics of Batch Distillation

In batch distillation operations, a certain amount of solution is placed in a boiling flask and then heated. In batch distillation, the feed is only added once per batch operation. During the process, the solution evaporates and the vapour that forms continuously leaves the boiling flask to be condensed. This results in a distillate with a high concentration of highly volatile components. Meanwhile, the concentration of the residue decreases because no feed is flowing in. One characteristic of separation by batch distillation is that the flow rate and composition of the feed and product change over time during the separation process.



Batch distillation can be viewed as a column composed of an enriching section consisting of vapour feed continuously entering through the bottom of the column. Batch distillation also has low capacity. In the chemical industry, batch distillation is chosen when the feed is in small quantities and the desired distillate has varying composition.

#### 2.4 Batch Distillation with Reflux System

To improve separation efficiency, distillation can be operated with a reflux system. The reflux system is intended to recontact some of the condensed liquid that exits the top of the column with its vapour phase along the column.

Thus:

1. Overall, the contact time between phases is getting longer.
2. Mass transfer and heat transfer are occurring again.
3. The distribution of temperature, pressure and concentration in each phase is becoming more uniform.
4. Equilibrium is getting closer.

Reflux comparison function:

1. For columns to be constructed

That to achieve the same purity, the greater the reflux ratio used, the fewer ideal plates are required.

1. For existing columns

That for the same number of plates, the greater the reflux ratio used, the higher the purity of the product produced.

#### 2.5 Effect of Reflux Ratio on Distillate Composition

The reflux ratio is one of the operating variables that determines the success of the distillation separation process. In practice, the reflux ratio used is above the minimum reflux ratio ( $R_{min}$ ) and below the total reflux ratio. Thus, the correlation between the reflux ratio and the composition of light components present in the distillate of an ethanol-water mixture can be shown as in Figure 2.2.



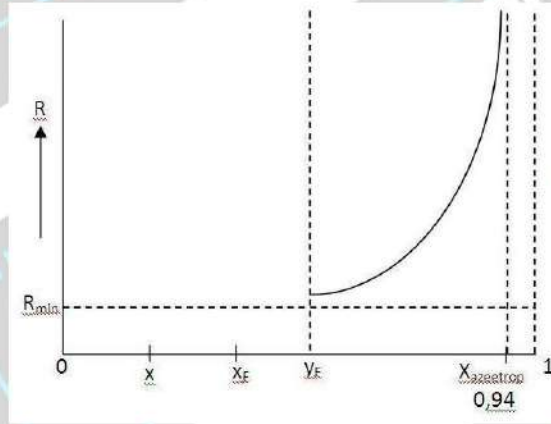


Figure 2.2 The effect of reflux ratio on distillate composition in ethanol-water mixtures

For existing columns, the composition of light components in the distillate increases as the reflux ratio increases. In distillation separation operations, the increase in the composition of light components in distillation never reaches one. Specifically for ethanol-water mixtures, the ethanol component in the distillate will not reach its azeotropic composition, while the composition of light components is above the feed composition.

In batch distillation, the feed is in the form of vapour that continuously enters through the bottom of the column. The composition of the feed entering the column can be estimated using Figure 2.3 below:

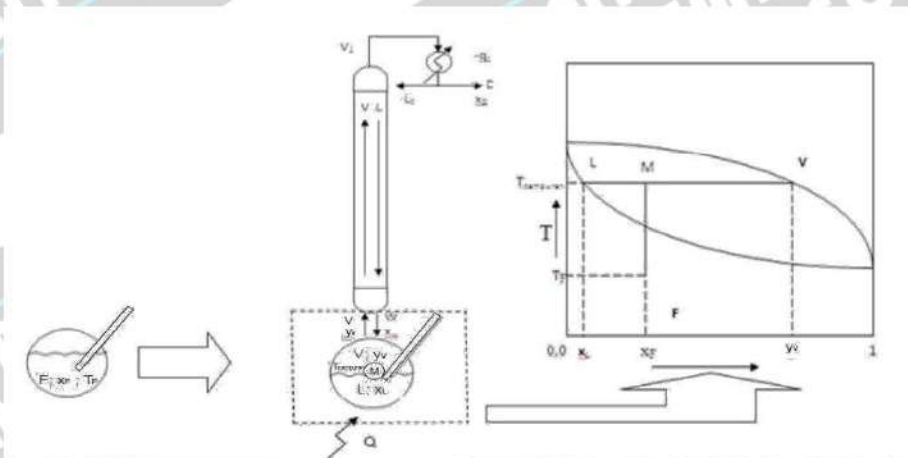


Figure 2.3 T-x,y diagram as a tool for determining the composition of column feed

## 2.6 Vapor-liquid equilibrium

Vapour-liquid equilibrium is a condition in which no macroscopic phase change occurs. The relationship between the equilibrium composition in the vapour phase and the composition of the liquid phase can be expressed by the term volatility (relative volatility). This condition occurs thermodynamically when the temperature, pressure, and fraction of each phase are constant. The



components are distributed between the phases depending on their relative volatility. The vapour-liquid equilibrium ratio for component A is expressed as:

$$K_A = \frac{y_A}{x_B}$$

For a mixture of two components A and B, the relative volatility value is expressed as  $\alpha$ , which is defined as:

$$\alpha = \frac{K_A}{K_B} = \frac{y_A x_A}{x_A y_B} = \frac{y_i(1 - x_i)}{x_i(1 - y_i)}$$

Relative volatility is a direct measurement of the distillation process. If  $\alpha = 1$ , separation of components is impossible because the vapour and liquid phases are identical. The distillation process becomes easier as the value of  $\alpha$  increases.

In the theoretical separation of ethanol-water, it is not possible to obtain a substance with 100% purity, but with reflux, a substance with higher purity can be obtained. This is due to the azeotropic point, where a mixture has the same composition in both the liquid and vapour phases at the equilibrium point, making it impossible to separate the mixture using conventional distillation.

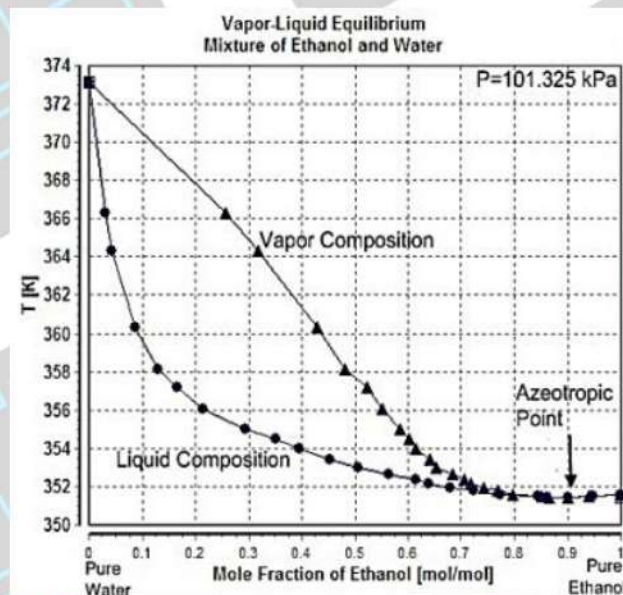


Figure 2.4 Ethanol-ir vapour-liquid equilibrium diagram (including azeotrope)

The product obtained from the separation of the ethanol-water mixture by distillation never reaches its azeotropic composition (0.95-0.96). Nevertheless, the composition of the distillate will not be less than the composition of the feed entering the column ( $y_f$ ). The ease or difficulty of separation by distillation depends on the extent of the difference in the properties of substances that are similar to one another; separation by distillation is difficult to achieve.



## CHAPTER III METHODOLOGY

### 3.1 Experimental Design

To answer the objective of the experiment, which is to examine the effect of the reflux ratio ( $R$ ) on the ethanol composition in the distillate during a four-minute operation. This practicum was carried out in two stages, namely:

#### a) Preparation stage

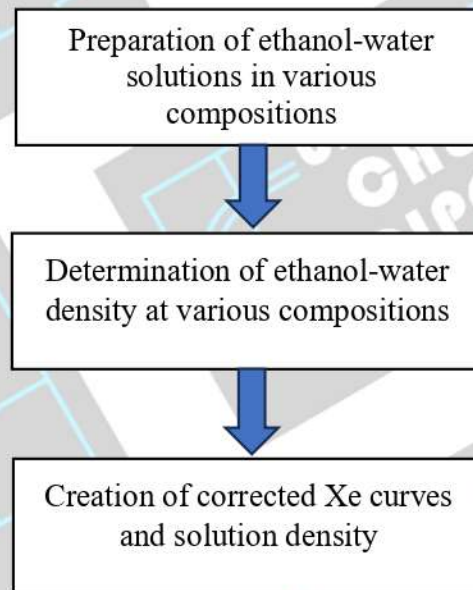


Figure 3.1 Preparation stage design

#### b) Operation stage

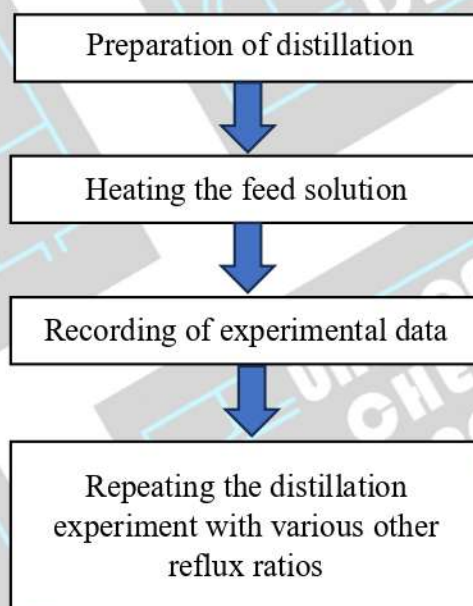


Figure 3.2 Preparation operation design



3.1.1 Preparation Stage

The preparation stage is intended to create a standard curve of the relationship between density ( $\rho$ ) and the concentration of the Xe solution, with the following steps:

- 1. Prepare ethanol-water solutions of various compositions.
- 2. Determine the density of ethanol-water solutions of various compositions.
- 3. Plot the corrected Xe and  $\rho$  of the solution on the x and y axes for the standard curve. (Appendix)

3.1.2 Operation Stage

To examine the effect of reflux ratio on the ethanol composition in the distillate, the experiment was conducted under constant conditions.

- a. Column feed composition : 0.7
- b. Operating time : 1 minute
- c. Feed volume : 10 L

Meanwhile, the reflux ratio was varied. At the end of each experiment, the ethanol composition (% by weight) was tested. Another form of the experimental design at the operational stage can be seen in Table 3.1.

Table 3.1 Distillate volume data, distillate density, and distillate composition of various reflux ratios

Heating Energy (Watt)	C (Product to Column)	D (Product Received)	L (Reflux Ratio)	V Product	W Product	Dens ity	Xe
√	√	√	√	√	√	√	√
	√	√	√	√	√	√	√
	√	√	√	√	√	√	√
	√	√	√	√	√	√	√
	√	√	√	√	√	√	√
√	√	√	√	√	√	√	√
	√	√	√	√	√	√	√
	√	√	√	√	√	√	√
	√	√	√	√	√	√	√
	√	√	√	√	√	√	√



### 3.2 Materials and Tools Used

#### a. Materials Used

- *Absolute ethanol 0.980 (Merck, Germany)*
- *Technical ethanol*
- *Distilled water*

#### b. Tools Used

- One batch distillation unit with reflux system.
- Picnometer and analytical balance
- Stopwatch
- 10 mL measuring glass
- 50 mL beaker glass

### 3.3 Gambar Alat Utama

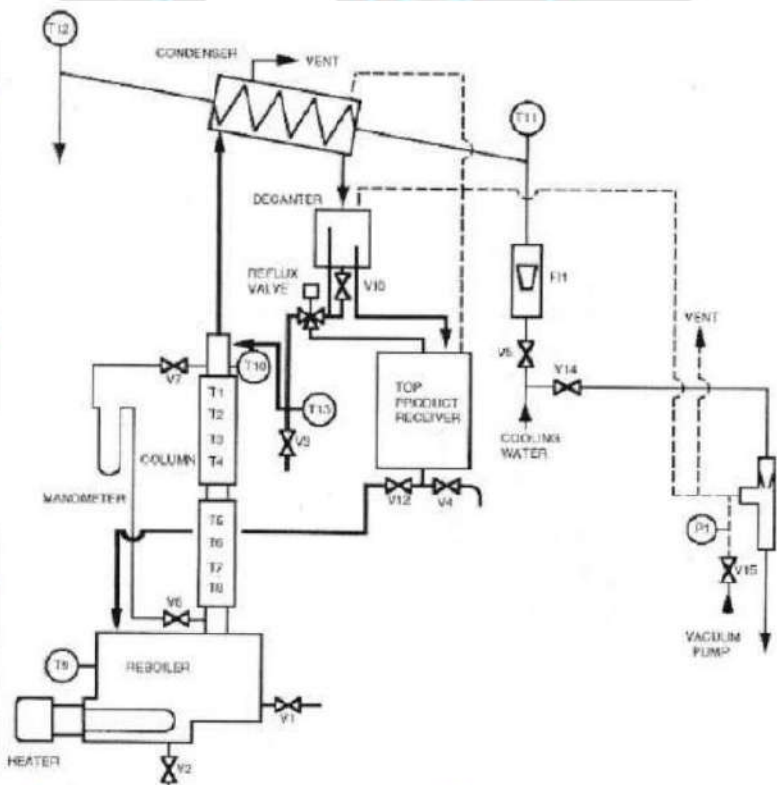


Figure 3.3 Main batch distillation equipment series



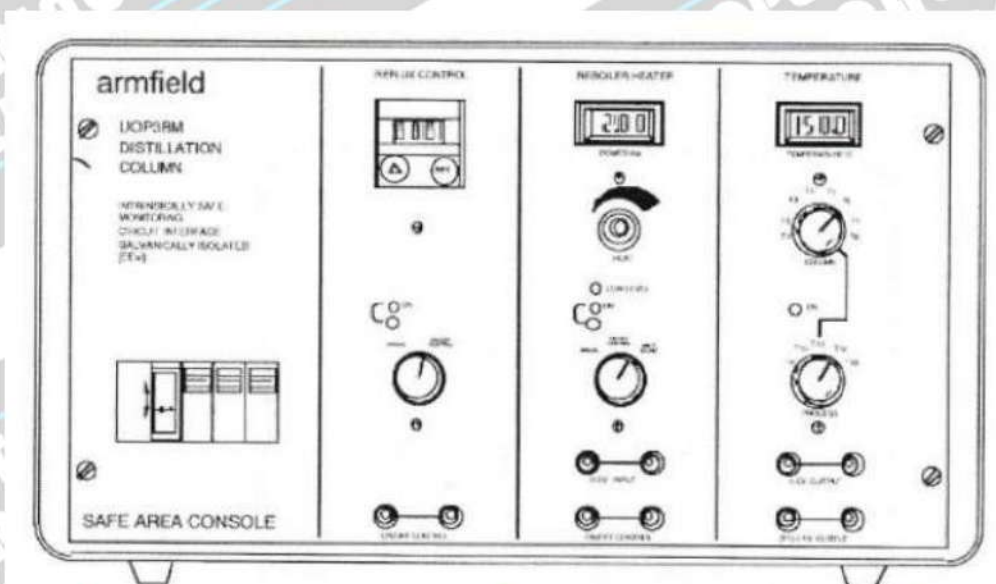


Figure 3.4 Control panel for batch distillation equipment

### 3.4 Experimental Procedures During the Operational Phase

1. Prepare the equipment so that it is ready for operation.
  - Check several pieces of equipment, including the equipment connections, heater, cooling water, thermometer, and tap.
  - Close all valves except valve 10.
2. Put the prepared feed into the boiler.
3. Circulate the cooling water through the condenser and set the flow rate to 4000 cc/min.
4. Connect the console to the electrical contact and set the heat control button to 1.5 Watts until bubbling occurs, then reduce it to the specified variable.
5. Wait until the system reaches a steady state, i.e., until the steam temperature and liquid temperature are relatively constant.
6. Wait until the steam condenses and the liquid returns to the column.
7. Adjust the control panel so that the reflux value = 0 to calculate the amount of product obtained at a specific heating energy variable, record the data obtained.
8. Adjust the reflux controller on the control panel according to the variable.
9. Perform the distillation operation for 1 minute.
10. Turn off the reflux system on the control panel.
11. Open the distillate outlet valve, collect the distillate that comes out, and close the distillate collection valve.
12. Measure the volume and weight of the distillate obtained to determine the density value.
13. Repeat steps 8–12 for other reflux comparisons.



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