DISTILLATION

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Introduction

Although many people have a fair idea what "distillation" means, the important aspects that seem to be missed from the manufacturing point of view are that:

- distillation is the most common separation technique
- it consumes enormous amounts of energy, both in terms of cooling and heating requirements
- it can contribute to more than 50% of plant operating costs

The best way to reduce operating costs of existing units, is to improve their efficiency and operation via process optimisation and control. To achieve this improvement, a thorough understanding of distillation principles and how distillation systems are designed is essential.

Distillation Definition

- Distillation is an unit operation in which constituents of liquid mixture (solution) are separated using thermal energy.

- Basically, the difference in vapor pressure of different constituents at the same temperature is responsible for such separation.

- Distillation is also termed as fractional distillation or fractionation.

- With this technique it is possible to separate liquid mixture into its components in almost pure form and this fact has made distillation perhaps the most important of all mass transfer operations.

- Separation of petroleum crude into gasoline, kerosene, fuel oil etc., and Separation of ethanol and water mixture for the production of ethyl alcohol are the examples.

Definition

- Distillation is method of separation of components from a liquid mixture which depends on the differences in boiling points of the individual components and the distributions of the components between a liquid and gas phase in the mixture.

Vapor Pressure

- The vaporization process changes liquid to gaseous state.

- The opposite process of this vaporization is called condensation.

- At equilibrium, the rates of these two processes are same.

- The pressure exerted by the vapor at this equilibrium state is termed as the vapor pressure of the liquid.

- It depends on the temperature and the quantity of the liquid and vapor.

Boiling Point

- For any given pressure, a pure liquid when heated will boil or vaporise at a certain single temperature known as boiling point of the liquid.

- Boiling point of the liquid increases with increase in pressure and vice versa.

- Boiling points at a given pressure vary greatly for different liquids.

- In the binary mixture (two component system), the component with lower boiling point or component with higher vapor pressure at a given temperature, is termed as **More Volatile or Lighter component.**

- The component with higher boiling point or component with lower vapor pressure at a given temperature, is termed as **Less Volatile or Heavier component.**

- **Equilibrium:** It is the condition at which transfer of material ceases for a given set of operating conditions that exist for all combinations of phases.

- **Driving force:** The concentration difference is the driving force for mass transfer and boiling point difference is the driving force for Distillation.

- **Raoult's Law:** It states that equilibrium partial pressure of a constituent at a given temperature is equal to the product of its vapor pressure in pure state and its mole fraction in the liquid phase. $p_j = P_j^0 x_j$

- **Relative Volatility:**

- Relative volatility is a measure of the differences in volatility between two components, and hence their boiling points.

- It indicates how easy or difficult a particular separation will be.

- The relative volatility of component 'A' with respect to component 'B' in a binary mixture is defined as

$$\alpha_{AB} = \frac{y_A / x_A}{y_B / x_B}$$

- where, y_A = mole fraction of component 'A' in the vapor,

- x_A = mole fraction of component 'A' in the liquid.

- **Relative Volatility:**

- In general, relative volatility of a mixture changes with the mixture composition.

- For binary mixture, $x_B = 1-x_A$ and $y_B = 1-y_A$

- So above Equation can be rearranged, simplifying and expressed by dropping subscript 'A' for more volatile component as:

$$y = \frac{\alpha_{ave}x}{1-(\alpha_{ave}-1)x}$$

- The above Equation is a non-linear relationships between x and y.

- This Equation can be used to determine the equilibrium relationship (y vs. x) provided the average relative volatility, α_{ave} is known.

- **Relative Volatility:**

- If the system obeys Raoult's law, the relative volatility can be expressed as:

$$\alpha_{AB} = \frac{p_A / x_A}{p_B / x_B}$$

- where p_A is the partial pressure of component A in the vapor,

- p_B is the partial pressure of component B in the vapor

- and P is the total pressure of the system.

- Thus if the relative volatility between two components is equal to one, separation is not possible by distillation.

- The larger the value of Relative volatility, above 1.0, the greater the degree of separability, i.e. the easier the separation.

- Some examples of optimal relative volatility that are used for distillation process design are given in Table

More volatile component (normal boiling point °C)	Less boiling component (normal boiling point, °C)	Optimal Relative volatility
Benzene (80.1)	Toluene (110.6)	2.34
Toluene (110.6)	p-Xylene (138.3)	2.31
Benzene (80.6)	p-Xylene (138.3)	4.82
m-Xylene (139.1)	p-Xylene (138.3)	1.02
Pentane (36.0)	Hexane (68.7)	2.59
Hexane (68.7)	Heptane (98.5)	2.45
Hexane (68.7)	p-Xylene (138.3)	7.0
Ethanol (78.4)	iso-Propanol (82.3)	1.17
iso-Propanol (82.3)	n-Propanol (97.3)	1.78
Ethanol (78.4)	n-Propanol (97.3)	2.10
Methanol (64.6)	Ethanol (78.4)	1.56
Methanol (64.6)	iso-Propanol (82.3)	2.26
Chloroform (61.2)	Acetic acid (118.1)	6.15

TYPES OF DISTILLATION COLUMNS

- There are many types of distillation columns, each designed to perform specific types of separations, and each design differs in terms of complexity.

- One way of classifying distillation column type is to look at how they are operated

1. Batch columns

2. Continuous columns

- Batch columns: In batch operation, the feed to the column is introduced batch-wise. That is, the column is charged with a 'batch' and then the distillation process is carried out. When the desired task is achieved, a next batch of feed is introduced.

- Continuous columns: Continuous columns process a continuous feed stream. No interruptions occur unless there is a problem with the column or surrounding process units. They are capable of handling high throughputs and are the most common of the two types.

Types of Continuous Columns

- **The nature of the feed that they are processing**

1. Binary column - feed contains only two components

2. Multi-component column - feed contains more than two components

- **The number of product streams they have**

1. Multi-product column - column has more than two product streams

- **Where the extra feed exists when it is used to help with the separation**

1. Extractive distillation - where the extra feed appears in the bottom product stream

2. Azeotropic distillation - where the extra feed appears at the top product stream

- **The type of column internals**

1. Tray column - where trays of various designs are used to hold up the liquid to provide better contact between vapor and liquid, hence better separation

2. Packed column - where instead of trays, 'Packings' are used to enhance contact between vapor and liquid

Main Components of Distillation Columns

- Distillation columns are made up of several components, each of which is used either to transfer heat energy or enhance material transfer

1. A vertical shell where the separation of liquid components is carried out

2. Column internals such as trays/plates and/or packings which are used to enhance component separations

3. A reboiler to provide the necessary vaporization for the distillation process

4. A condenser to cool and condense the vapour leaving the top of the column

5. A reflux drum to hold the condensed vapour from the top of the column so that liquid (reflux) can be recycled back to the column

6. The vertical shell houses the column internals and together with the condenser and reboiler, constitute a distillation column

# A schematic of a typical distillation unit
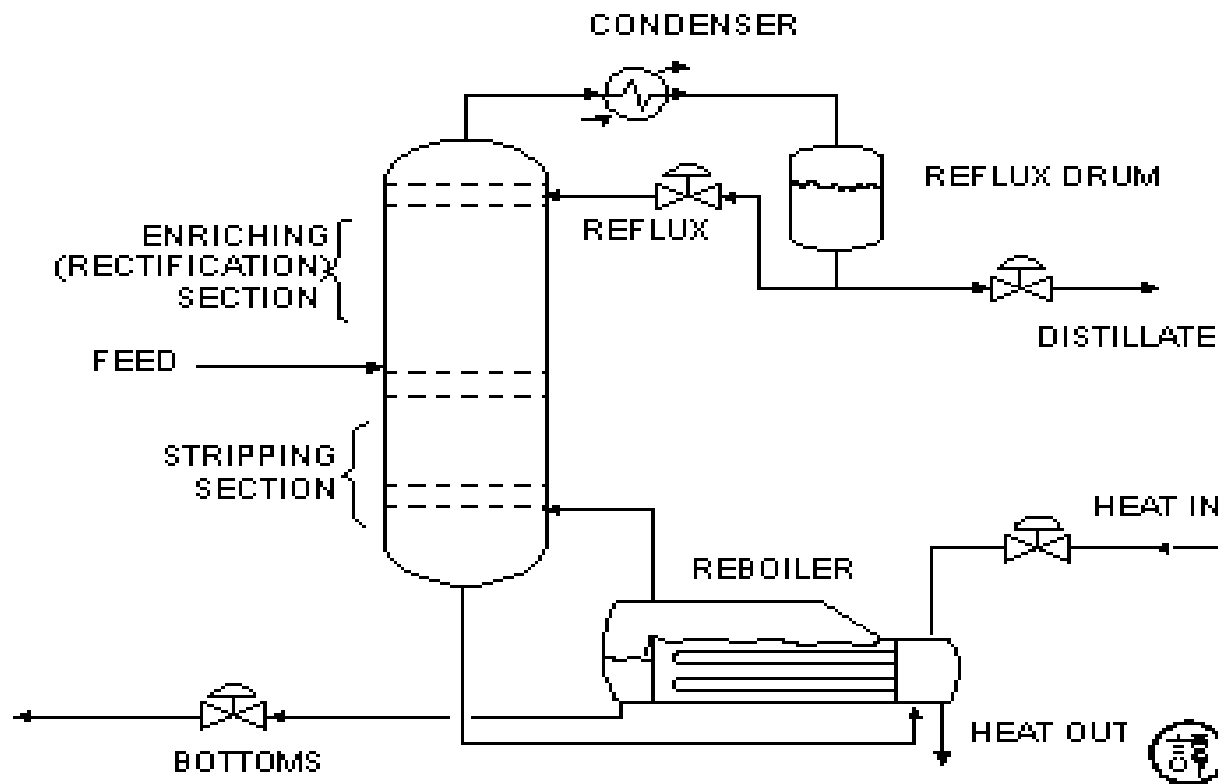
Basic Operation and Terminology

- The liquid mixture that is to be processed is known as the feed and this is introduced usually somewhere near the middle of the column to a tray known as the feed tray

- The feed tray divides the column into a top (enriching or rectification) section and a bottom (stripping) section. The feed flows down the column where it is collected at the bottom in the reboiler

- Heat is supplied to the reboiler to generate vapour. The source of heat input can be any suitable fluid, although in most chemical plants this is normally steam

- The vapour raised in the reboiler is re-introduced into the unit at the bottom of the column

- The liquid removed from the reboiler is known as the bottoms product or simply, bottoms

- The vapour moves up the column, and as it exits the top of the unit, it is cooled by a condenser

- The condensed liquid is stored in a holding vessel known as the reflux drum

- Some of this liquid is recycled back to the top of the column and this is called the reflux

- The condensed liquid that is removed from the system is known as the distillate or top product

Distillation Methods

- In this course will study the following distillation methods. They are

- Batch or Simple or Differential Distillation

- Continuous or Rectification or Fractional Distillation

- Flash or Equilibrium Distillation

- Steam Distillation

- Azeotropic Distillation

- Vacuum Distillation
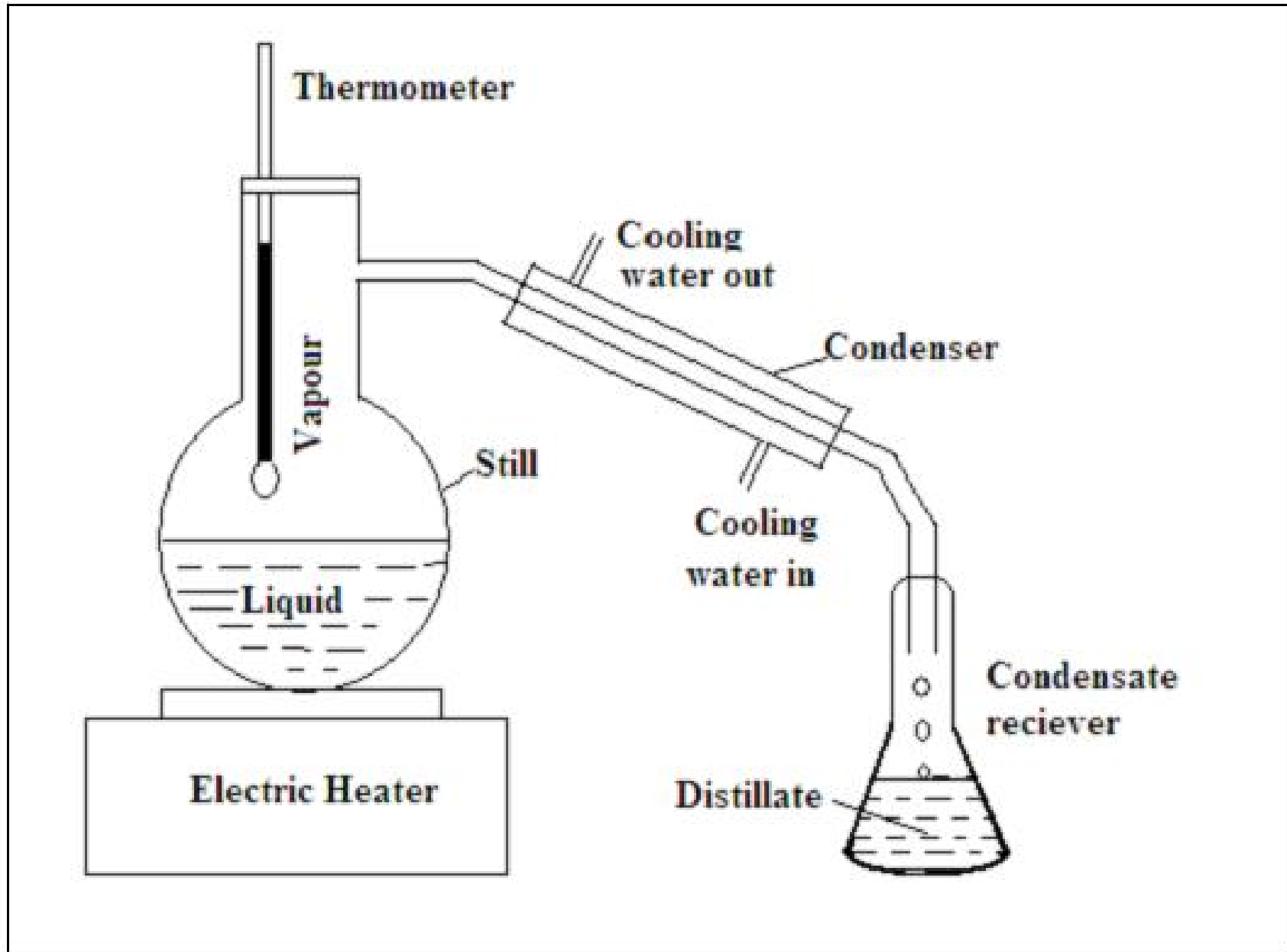
Simple or Batch or Differential Distillation Process

Batch Distillation

- In batch operation, the feed is introduced batch -wise to the column.

- That is, the column is charged with a 'batch' and then the distillation process is carried out.

- When the desired task is achieved, a next batch of feed is introduced.

- Consider a binary mixture of components A (more volatile) and B (less volatile).

- The system consists of a batch of liquid (fixed quantity) inside a kettle (or still) fitted with heating element and a condenser to condense the vapor produced as shown in Figure.

- The condensed vapor is known as the distillate. The distillate is collected in a condensate receiver.

- The liquid remaining in the still is known as the residual.

Batch Distillation

- The process is unsteady state.

- Detailed mathematical calculations carried out using the Rayleigh Equation.

- As the process is unsteady state, the derivation is based on a differential approach to changes in concentration with time.

- Let L_1= initial moles of liquid originally in still,

- L2 = final moles of liquid remained in still,

- x1 = initial liquid composition in still (mole fraction of A),

- x2= final liquid composition in still (mole fraction A).

- D = Distillate

- yD = Mole fraction of A in Distillate

$$\ln\left(\frac{L_1}{L_2}\right) = \int_{x_2}^{x_1} \frac{1}{(y-x)}dx \qquad y_D = \frac{L_1 x_1 - L_2 x_2}{D}$$

Rayleigh Equation
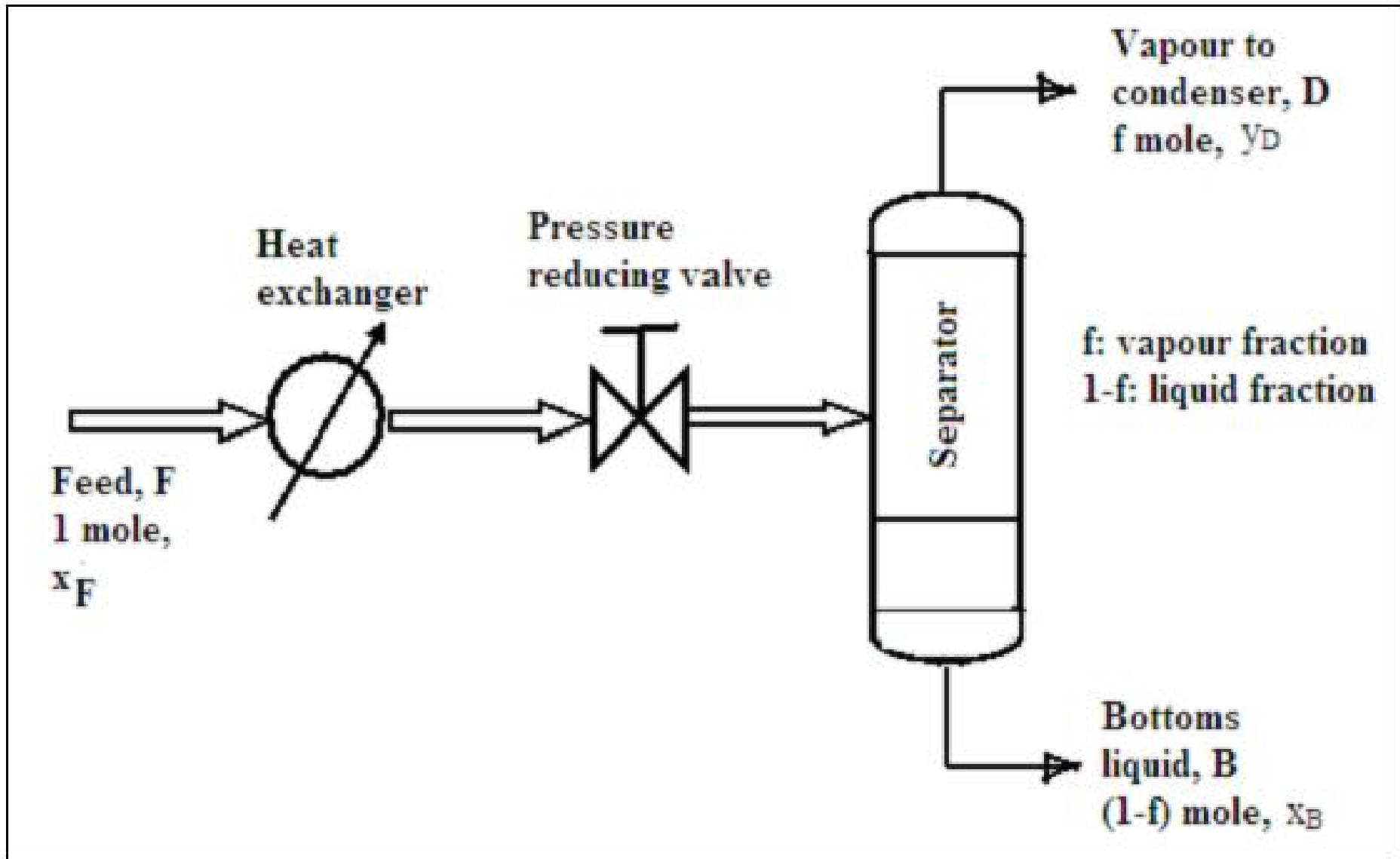
Flash Distillation Process

Flash Distillation

- A single-stage continuous operation occurs where a liquid mixture is partially vaporized.

- The vapor produced and the residual liquids are in equilibrium in the process are separated and removed as shown in Figure.

- Consider a binary mixture of A (more volatile component) and B (less volatile component).

- The feed is preheated before entering the separator.

- As such, part of the feed may be vaporized.

- The heated mixture then flows through a pressure-reducing valve to the separator.

- In the separator, separation between the vapor and liquid takes place.

- The amount of vaporization affects the concentration (distribution) of A in vapor phase and liquid phase.

Flash Distillation

- Define f as molal fraction of the feed that is vaporized and withdrawn continuously as vapor.

- Therefore, for 1 mole of binary feed mixture, (1- f) is the molal fraction of the feed that leaves continuously as liquid.

- Assume, y_D = mole fraction of A in vapor leaving, x_B = mole fraction of A in liquid leaving, x_F = mole fraction of A in feed entering.

- Based on the definition for f, the greater the heating is, the larger the value of f.

- If the feed is completely vaporized, then f = 1.0.

- Thus, the value of f can varies from 0 (no vaporization) to 1 (total vaporization).

Flash Distillation

- Overall Balance:

$$F = D + B$$

More volatile Component (A) Balance:

$$Fx_F = Dy_D + Bx_B$$

$$1 * x_F = f * y_D + (1-f) * x_B \quad [\text{since, } D/F = f \ \& \ B/F = (1-f) \ ]$$

or

$$f * y_D = x_F - (1-f) * x_B$$

$$y_D = -\left(\frac{1-f}{f}\right)x_B + \frac{x_F}{f}$$

Flash Distillation

- The fraction f depends on the enthalpy of the liquid feed, the enthalpies of the vapor and liquid leaving the separator.

- For a given feed condition, and hence the known value of f and x_F, the above Equation is a straight line Equation with slope $-(1-f)/f$ and intercept x_F/f as shown in below Figure of flash distillation.

- It will intersect the equilibrium line at the point (x_B, y_D).

- From this value, the composition of the vapor and liquid leaving the separator can be obtained.

Graphical Presentation of Flash Vaporization