International Association of Certified Practicing Engineers	IACPE INTERNATIONAL ASSOCIATION OF CERTIFIED PRACTICING ENGINEERS www.iacpe.com Knowledge, Certification, Networking	October 2017
IACPE No 19, Jalan Bilal Mahmood 80100 Johor Bahru Malaysia	**DISTILLATION** **CERTIFIED PROCESS TECHNICIAN TRAINING MODULE**	

The International Association of Certified Practicing Engineers is providing the introduction to the Training Module for your review.

We believe you should consider joining our Association and becoming a Certified Process Technician. This would be a great option for engineering improvement, certification and networking.

This would help your career by:

1. **Providing a standard of professional competence in the practicing engineering and management field**
2. **Identify and recognize those individuals who, by studying and passing an examination, meets the standards of the organization**
3. **Encourage practicing engineers and management professionals to participate in a continuing program of personal and professional development**

www.iacpe.com

TABLE OF CONTENT

INTRODUCTION	**4**
Scope	**4**
General Consideration	**5**
History	**5**
Mode of Operation	**6**
Column Internals	**6**
Schematic of Distillation	**7**
Demisters	**16**
DEFINITION	**17**
THEORY	**19**
A. Vapor Pressure	**19**
B. Volatility	**21**
C. Partial Pressure	**21**
D. Unsteady State Distillation	**23**
E. Steady State Distillation	**25**
F. Multiple Stills	**26**
G. The Distillation Column	**27**
H. Fractionation	**32**
I. The Tower	**35**
i. Tray Tower	**37**
ii. Packed Column	**44**
J. The Selection of Column Internals	**48**
K. Temperature and Pressure	**49**

LIST OF FIGURE

Figure 1: Batch Still Distillation Process	6
Figure 2: Schematic Diagram of Distillation Column/ Fractionator	7
Figure 3 : The overhead product passes around the tube bundle	8

Figure 4 : Condenser position in distillation column 9
Figure 5 : Condenser at ground level 10
Figure 6 : Partial condenser 11
Figure 7 : Total condenser 11
Figure 8 : Hot Vapor By Pass Condenser 12
Figure 9 : Reboiler illustration 13
Figure 10 : thermosiphon reboiler 14
Figure 11 : Kettle reboiler 15
Figure 12 : Tower B has a reflux distributor 16
Figure 13 : Demister 16
Figure 14 : Mixture of two components 23
Figure 15 : Unsteady state distillation 24
Figure 16 : Steady state distillation 25
Figure 17 : Multiple stills 26
Figure 18 : Multiple stills with equipment changes 27
Figure 19 : a simplified close-up drawing of the separating devices 28
Figure 20 : a simplified close-up drawing of deck or tray 29
Figure 21 : Sections in distillation column 31
Figure 22 : fractions of crude oil 32
Figure 23 : Fractionation 33
Figure 24 : Mixtures properties 36
Figure 25 : Schematic description of a tray section 38
Figure 26 : Bubble cap trays 39
Figure 27 : small bubbles change 40
Figure 28 : Description of distributor weir 41
Figure 29 : Sieve tray 42
Figure 30 : Valve Tray 43
Figure 31 : Mechanism of Packed Column 45
Figure 32 : Type of Random Packing 46
Figure 33 : Structured Packed 47
Figure 34 : Towers are randomly packed and packed in layers 47

INTRODUCTION

Scope

Distillation is by far the most important separation process in the petroleum and chemical industries. It is the separation of key components in a mixture by the difference in their relative volatility, or boiling points. Distillation is a physical separation process, and not a chemical reaction. It is also known as fractional distillation or fractionation.

In most cases, distillation is the most economical separating method for liquid mixtures. However, it can be energy intensive. Distillation can consume more than 50% of a plant's operating energy cost. There are alternatives to distillation process such as solvent extraction, membrane separation or adsorption process. On the other hand, these processes often have higher investment costs. Therefore, distillation remains the main choice in the industry, especially in large-scale applications.

This training module provides an overview of the distillation and its parts along with type of plate and the auxiliary equipment. The knowledge and understanding of the basic principles and concepts of distillation, equilibrium and controlling of pressure-temperature are essential to deal with the way these parts function in the total process of making product purity.

This module includes parts of the distillation, variations of plate, auxiliary equipment in distillation, and factors that effect in operating distillation such as relationship of pressure and temperature to the product.

The sciences of distillation in this module provides a foundation in practical distillation which enable the process technician to solve distillation problem in industries. The student who knows the distillation and how it works – whether or not he is actually engaged in distillation operation - is better prepared to understand the reasons for making the most of separation everywhere in the refinery. He is a more understanding workman and more valuable in any job.

This module also has figures and tables to illustrate the equipment and condition cases as example and reference. It is important to assist the students to understand and can be applied in industries practically.

General Consideration

As crude oil comes from the ground, it is a mixture of many kinds of molecules. In this form it is not very useful. However, through refining it is separated into various products , or fractions. Each fraction has a specific use and can be sold for that use. The fraction which is sold for use as car fuel is gas. One method of separating crude oil into fractions is called distillation or fractionation. Since distillation or fractionation does not involve the breaking or forming of molecules, the separation of crude oil into fraction by distillation is not a chemical reaction.

Very large hydrocarbon molecules have a limited use fulness. By means of a chemical reaction called cracking they can be broken down into smaller molecules, which are more useful. Breaking large, complex hydrocarbon molecules into smaller molecules is called cracking.

Hydrocarbon molecules in the nephtha range can be reformed and changed into more useful molecules of roughly the same size. Through a series of chemical reactions, atoms or groups of atoms are added, removed, or rearranged, resulting in different hydrocarbon molecules with higher octane properties. In oil refining, the name for this restructuring of hydrocarbon molecules is reforming.

The term reforming refers to the rearrangement of molecular structure. The term cracking refers to breaking large molecules into smaller molecules. The term fractionation refers to the separation of mixtures of hydrocarbons without affecting their molecules structure. Hydrocarbon molecules may be changed into different, more useful molecules by either cracking or reforming; these changes are chemical reactions.

History

Early distillation consisted of simple batch stills to produce ethanol. Crude ethanol was placed in a still and heated, and the vapor drawn from the still was condensed for consumption. Lamp oil was later produced using the same method, with crude oil heated in batch stills.

The next progression in the history of distillation was to continually feed the still and recover the light product. Further advancements include placing the stills in series and interchanging the vapor and liquid from each still to improve recovery. This was the first type of counter-current distillation column that we have today.

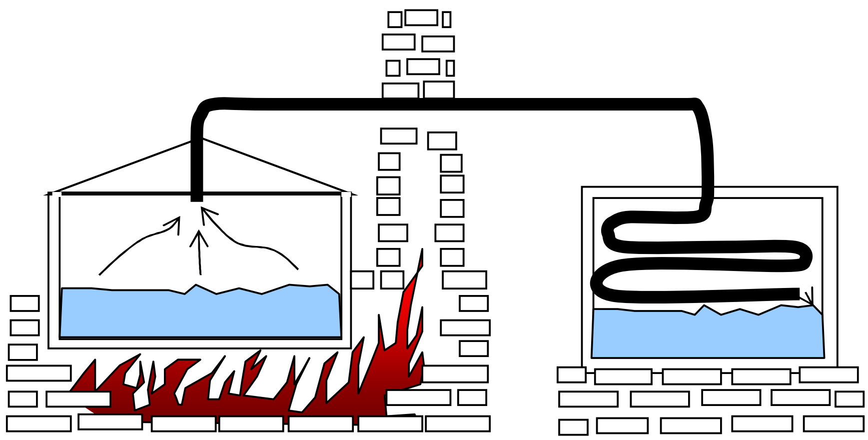

Figure 1: Batch Still Distillation Process

Mode of Operation

Distillation towers can be classified into two main categories, based on their mode of operation. The two classes are batch distillation and continuous distillation.

In batch distillation, the feed to the column is introduced batch-wise. The column is first charged with a ‘batch’ and then the distillation process is carried out. When the desired task is achieved, the next batch of feed is introduced. Batch distillation is usually preferred in the pharmaceutical industries and for the production of seasonal products.

On the other hand, continuous distillation handles a continuous feed stream. No interruption occurs during the operation of a continuous distillation column unless there is a problem with the column or surrounding unit operations. Continuous columns are capable of handling high throughputs. Besides, additional variations can be utilized in a continuous distillation column, such as multiple feed points and multiple product drawing points. Therefore, continuous columns are the more common of the two modes, especially in the petroleum and chemical industries.

Column Internals

Column internals are installed in distillation columns to provide better mass and heat transfers between the liquid and vapor phases in the column. These include trays, packings, distributors and redistributors, baffles and etc. They promote an intimate contact between both phases. The type of internals selected would determine the height and diameter of a column for a specified duty because different designs have various capacities and efficiencies. The two main types of column internals discussed in this guideline are trays and packing.

There are many types of trays or plates, such as sieve, bubble-cap and valve trays. Packing, on the other hand, can be categorized into random and structured packing. In random packing, rings and saddles are dumped into the column randomly while structured packing is stacked in a regular pattern in the column.

Schematic of Distillation

Figure 2 shows a schematic diagram of an example distillation column or fractionator. The feed enters the column as liquid, vapor or a mixture of vapor-liquid. The vapor phase that travels up the column is in contact with the liquid phase that travels down. Column distillation is divided two stages, there are rectifying stages and striping stages.

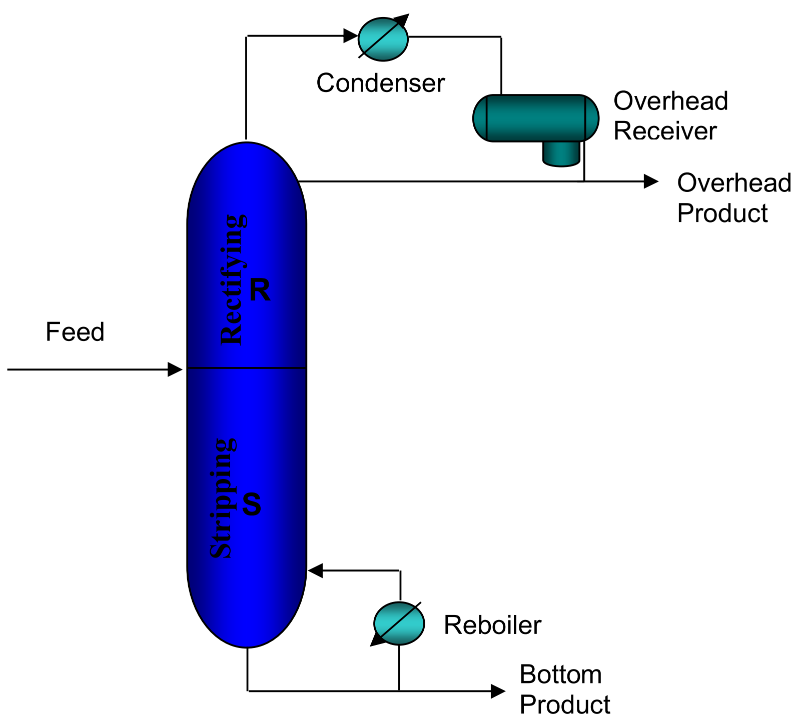

Figure 2: Schematic Diagram of Distillation Column/ Fractionator.

(A) Rectifying Stages

The process above the feed tray is known as rectification (where the vapor phase is continually enriched in the light components which will finally make up the overhead product). A liquid recycle condenses the less volatile components from rising vapor. To generate the liquid recycle, cooling is applied to condense a portion of the overhead vapor its name reflux.

(B) Stripping Stages

The process below the feed tray is known as stripping (as the heavier components are being stripped off and concentrated in the liquid phase to form the bottom product). At the top of the column, vapor enters the condenser where heat is removed. Some liquid is returned to the column as reflux to limit the loss of heavy components overhead.

At each separation stage (each tray or a theoretical stage in the packing), the vapor enters from the stage below at a higher temperature while the liquid stream enters from the stage above at a lower temperature. Heat and mass transfer occur such that the exiting streams (bubble point liquid and dew point vapor at the same temperature and pressure) are in equilibrium with each other.

(C) Condenser

Condensers are used to remove heat from vapor. Condensers are used to liquefy the overhead product. A condenser usually consists of a metal shell containing a tube bundle. It is shown in Figure 3.

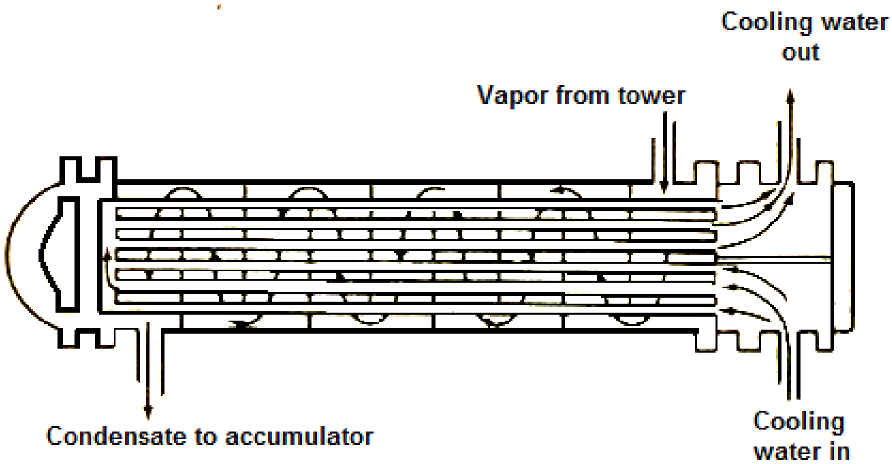

Figure 3 : The overhead product passes around the tube bundle

The overhead vapor continually passes into the shell. Cooling water circulating through the tubes removes heat from the vapor and comes out warmed. The vapor is condensed by cool water passing through the tubes. The water is warmed by the hot vapors passing around the tubes.

The condensate (condensed vapor) passes from the condenser into an accumulator. From the accumulator some of the condensate is removed as product and some is pumped back into the tower as reflux. Reflux enters the tower at the top. Diagrams usually show condensers at the top of the tower. In a small unit the condenser may be locate at the top, but in a large unit the condenser is usually located near the bottom of the tower.

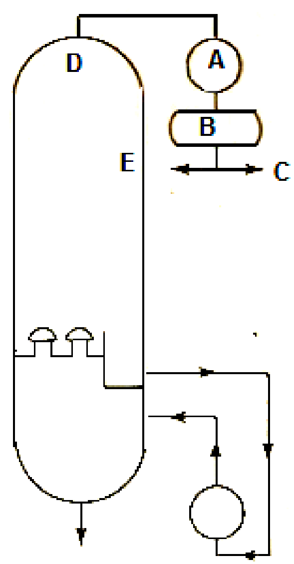

Figure 4 : Condenser position in distillation column

The condenser is at point A. The accumulator is at point B. The overhead product is removed at point C. The overhead vapor is removed at point D. Reflux enters the tower at point E.

In condensers, the shell side of the tubes is often damaged by corrosion. Resulting leakage will let water into the oil or oil into the water. Water sent to the tower with reflux can cause violent pressure surges or poor operation. If condenser leakage is not prevented or repaired, water may re-enter the tower with the reflux. Gas escaping from the condenser with the water can be a financial loss and fire hazard. If there is leakage in the tube bundles, dangerous gases may get out of the condenser into the cooling tower or sewer, with the warmed water. If the water in the condenser is allowed to become too hot, scale froms in the tubes. Scale tubes cause poor cooling.

In a large unit, the condenser would be too large or heavy to be mounted at the top of the tower. With condensers at ground level, it is more convenient to supply the large quantities of cool water that are needed. If the condenser is at ground level, pumps are used to return reflux to the top of the tower

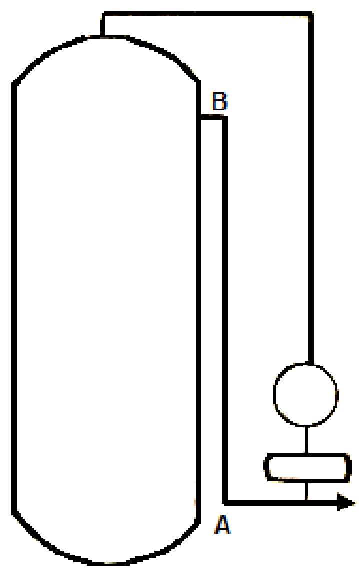

Figure 5 : Condenser at ground level

In a large unit, pumps are used to take reflux from the accumulator at point A, to the top of the tower at point B.

<u>Partial Condenser</u>

The partial condenser is best used when there is a large difference in the overhead vapor compositions. For example when there is a small amount of methane and hydrogen mixed in a propylene stream, like in the propylene towers. The partial condenser condenses the propylene and leaves the methane and hydrogen as a vapor to be vented from the overhead receiver. This type of condenser works well for most applications. The system needs to be reviewed to address the potential build of on non-condensable gases in the heat exchanger that can reduce the cooling potential of the exchanger. Partial condenser is given as shown in figure 6.

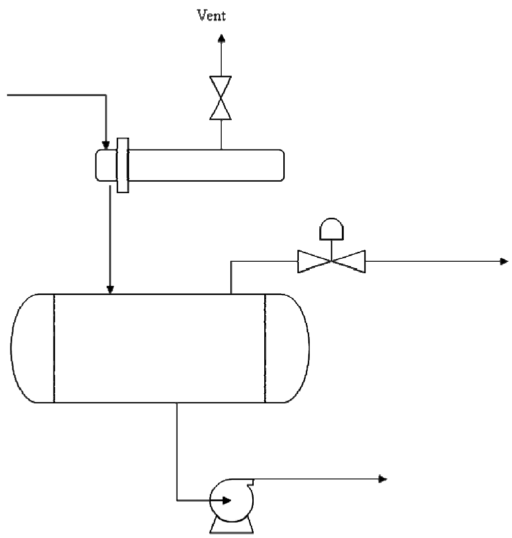

Figure 6 : Partial condenser

Total Condenser

The total condenser is best used when there is a small difference in the overhead vapor compositions. The overhead vapors can be condensed at approximately the same temperature. This system also needs to be reviewed to address the potential build of non-condensable gases in the heat exchanger that can reduce the cooling potential of the exchanger. Total condenser is given as shown in figure 7.

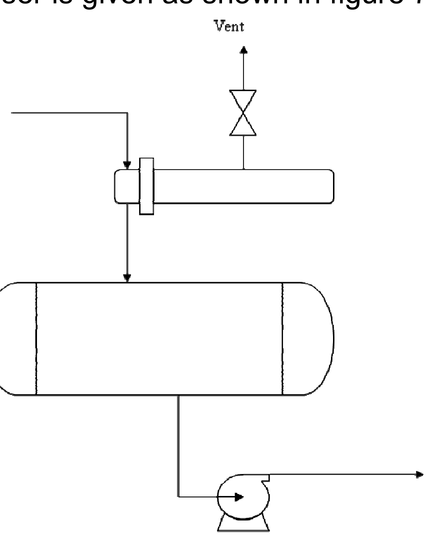

Figure 7 : Total condenser

Hot Vapor By Pass Condenser

The hot vapor by pass condenser is best utilized when there is the potential for large changes of overhead vapor composition. The vapor by pass can be used to maintain the pressure in the tower system when the light components are lower than design. The hot vapor by pass condenser also has a lower installed cost due to the heat exchanger being installed on the ground level.

The negatives of the Hot Vapor By Pass Condenser is that the by pass can be opened too much, increasing the temperature of the reflux. This reduces the tray efficiency in the top of the tower, and raises the tower pressure, which makes hydrocarbons harder to separate. This system, because of the physical location of the exchanger, has even higher potential to build non-condensable gases in the heat exchanger that can reduce the cooling potential of the exchanger. Hot Vapor By Pass condenser is given as shown in figure 8.

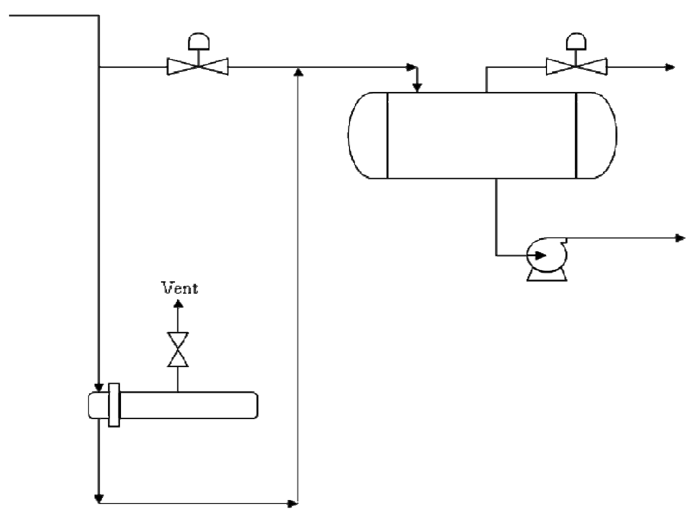

Figure 8 : Hot Vapor By Pass Condenser

(D) Reboiler

Heat for fractionation is normally supplied by a preheater, by a reboiler, or by a combination of preheater and reboiler. A reboiler is a heat exchanger that is used to generate the vapor supplied to the bottom tray of a distillation column. The liquid from the bottom of the column is partially vaporized in the exchanger, which is usually of the shell-and-tube type. The heating medium is most often condensing steam, but commercial heat-transfer fluids and other process streams are also used. Boiling takes place either in the tubes or in the shell, depending on the type of reboiler.

In a reboiler tower, the preheated feed entering the tower contacts vapors rising from the bottom of the tower. The vapors rising from the bottom of the tower are generated in the reboiler. The vapors rising from the reboiler strip the lighter components from the liquid on each tray. At the bottom of the tower, only the heavier fractions remains.

The overhead vapor is removed from the tower and condensed. Part of this condensate returns to the tower as reflux. The reflux drains down the tower and gets progressively heavier. Part of this heavy liquid is drawn off the reboiler where the lightest fractions are vaporized and fed back to the tower. The bottom product can be drawn off from the bottom of the tower. This bottom product is essentially free of the lightest fractions.

The reboiler is usually a separate unit located beside the tower. The reboiler is a heat exchanger that provides heat to the bottom of the tower. The reboiler consists of a metal shell with tube bundles inside. The liquid flows through the shell around the bundle, and is heated by the hotter material in the tube bundle. It can be shown in figure 9.

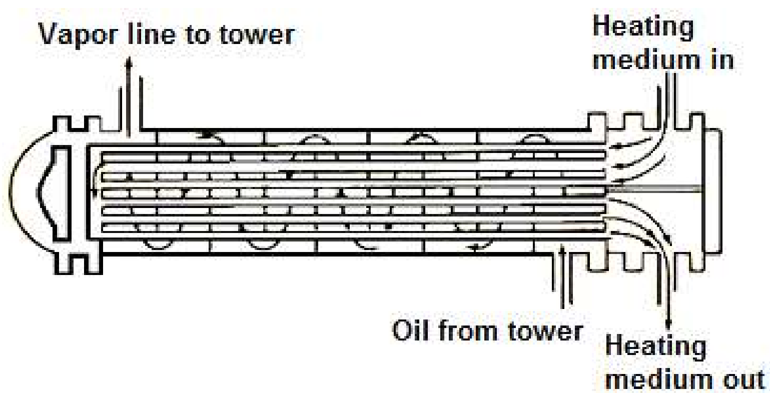

Figure 9 : Reboiler illustration

Thermosyphon Reboiler

This is a very common type of reboiler. Horizontal thermosiphon reboilers are the preferred reboiler type in refining applications. The process side is on the shell side, and the heating medium is on the tube side. The boiling occurs inside shell in horizontal thermosyphon. There is recirculation around the base of the column. A mixture of vapor and liquid leaves the reboiler and enters the base of the column where it separates.

Vertical thermosiphon reboilers are used almost exclusively in chemical applications, while the petrochemical industry is about 70% vertical and 30% horizontal. In vertical thermosyphon reboiler, the liquid circulation occurs due to density difference between vapor-liquid mixture (two phase) in the exchanger from the reboiler and the liquid through the downcomer to the reboiler. It is given as shown in figure 10.

- Very common in refining industry
- Simple design- no rotating equipment
- Mixed phase return to column
- Relatively inexpensive
- Good reliability.

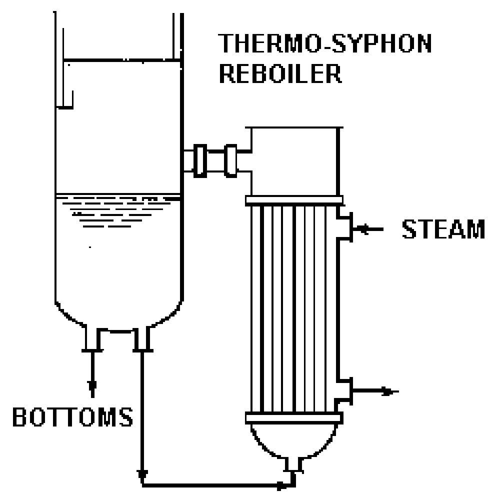

Figure 10 : thermosiphon reboiler

Kettle Reboiler

The kettle reboiler is an exchanger that has a tube bundle immersed in a liquid bath, with substantial vapor disengaging space above the vapor. Vapor and liquid are separated in the reboiler's disengaging space, so the return line carries essentially vapor. Kettle arrangements are once-through systems. It can be shown in figure 11.

- Not as common as thermosyphon
- Simple design – no rotating equipment
- Vapor return to column
- More expensive due to larger exchanger.

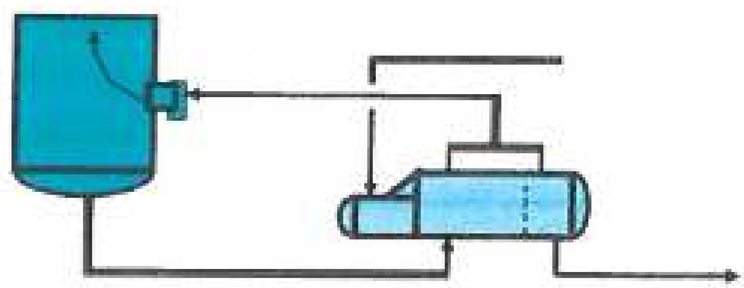

Figure 11 : Kettle reboiler

(E) Reflux section

Towers use reflux to achieve a more complete separation of products . Reflux refers to the portion of the condensed overhead liquid product that is returned to the upper part of the tower as shown above. Inside the tower, the reflux liquid flowing downwards provides the cooling needed to condense the vapors flowing upwards, thereby increasing the effectiveness of the distillation tower.

Reflux represents cooled, condensed top product returned to the tower top and, as such it is being reprocessed. The top product will therefore be purer. In general, the higher the reflux, the fewer the number of trays required for a given separation.

However, too high a reflux may cause flooding in the tower resulting in poor separation and causing ‘off-spec’ products throughout the system. The reflux rate is normally controlled by a temperature controller in the vapor outlet which operates a control valve in the reflux pump discharge. An increase in tower top temperature will cause the valve to open, increasing the reflux rate, and vice versa.

If there is nothing to spread the reflux evenly across the tray, all of the reflux will pour into the tower at the same spot. Some towers have a means to spread the reflux evenly over the whole top tray. Unless liquid spreads evenly across the whole tray vapor and liquid contact will be poor. If vapor-liquid contact is poor on any part of the tray, that tray will be less efficient.

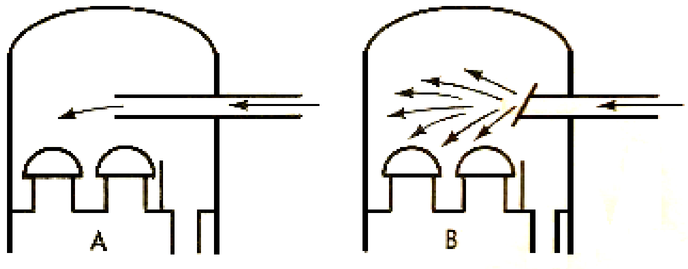

Figure 12 : Tower B has a reflux distributor

Demisters

Entrainment occurs when drops of liquid suspended in vapor are carried up into the next tray, or into the overhead. Entrainment can be serious when the overhead product must be a dry gas. Entrainment can usually be cut down by controlling vapor velocity and by proper tray spacing. Entrainment can also be cut down by “demisters” fitted in the tower where entrainment is most likely to have serious consequences.

Demisters or entrainment eliminators are usually located at the top of the tower. Demisters are constructed of fine gauge wire knitted into mesh. Vapors can pass easily through the mesh, but drops of liquid cannot. Demisters must be kept clean of dirt or foreign matter. Plugged demisters hamper the flow of vapor. A demister would most likely be found in section E as shown in figure 13.

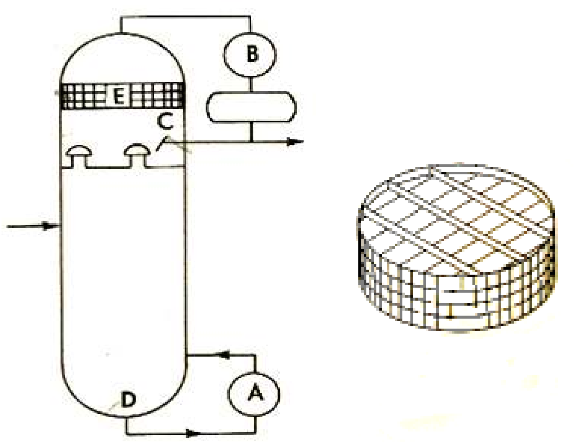

Figure 13 : Demister

DEFINITION

Active Area (or Bubble Area) - the deck area of the tray which may either be perforated or fitted with valves or bubble caps and is the area available for vapor/liquid contacting

Bottoms – The stream of liquid product collected from the reboiler at the bottom of a distillation tower.

Bubble point – The temperature at constant pressure (or the pressure at constant temperature) at which the first vapor bubble forms when a liquid is heated (or decompressed).

Condenser- Is a heat exchanger which condenses a substance from its gaseous to its liquid state.

Dew point – The temperature at constant pressure (or the pressure at constant temperature) at which the first liquid droplet forms when a gas (vapor) is cooled (or compressed).

Distillate – The vapor from the top of a distillation column is usually condensed by a total or partial condenser. Part of the condensed fluid is recycled into the column (reflux) while the remaining fluid collected for further separation or as final product is known as distillate or overhead product.

Downcomer - a vertical channel that connects a tray with the next tray below which carries froth and creates residence time which helps the vapor disengage from the froth.

Downcomer Area - is the area available for the transport of liquid from one tray to the next tray below.

Heavy key – The heavier (less volatile) of the two key components. Heavy key is collected at the bottoms. All non-key components heavier than the heavy key are known as the heavy components.

Light key – The lighter (more volatile) of the two key components. Light key is collected at the distillate. All non-key components lighter than the light key are known as the light components.

Open Area (or Hole Area) - is the aggregate area available for vapor passage through the tray deck via perforations or valve and bubble cap slots. This is a critical factor in the tray operating range since high vapor velocity through the open area (hole velocity) will induce heavy liquid entrainment (as well as high pressure drop), but low hole velocity may allow liquid to "weep" or even "dump" through the tray deck to the tray below. The influence of open area on pressure drop also impacts on the liquid back-up in the downcomer

Packed tower - contains 'Beds' of packing material which are used to bring the rising vapors into intimate contact with falling within the tower

Reboiler –Is a heat exchanger typically used to provide heat to the bottom of industrial distillation columns. They boil the liquid from the bottom of a distillation column to generate vapors which are returned to the column to drive the distillation separation.

Trays - used to bring the rising vapor and falling liquid into intimate contact. Tray towers do the same job as packed towers but they are very much more efficient in the separation process than packed towers and, they are also more costly.

Tray Pressure Drop - may also be a limiting criterion particularly in low pressure services. The operating tray pressure drop is the sum of the dry pressure drop caused by the resistance to vapor flow through the tray open area and the head of clear liquid on the tray deck.

Tray Spacing - is the vertical distance between adjacent tray decks. This effects both the height of spray that may be generated on the tray deck before liquid carryover and also the allowable head of liquid in the downcomers.

Vapor pressure – The pressure exerted by the vapor phase that is in equilibrium with the liquid phase in a closed system. For moderate temperature ranges, the vapor pressure at a given temperature can be estimated using the Antoine equation.