

## Distillation Technologies

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Distillation is one of the most commonly used separation techniques in the chemical industry. It relies on differences in the volatility of chemicals.

To illustrate roughly, consider a mixture of two components - a low-boiler (LB), which boils more easily, and a high boiling component (HB), which boils less easily. Furthermore, assume that LB vapour concentration is always 2 times its liquid concentration, and that we start with a liquid with 0.1 mole fraction LB. If the liquid is evaporated, the initial vapour will have 0.2 mole fraction LB (i.e. two times the concentration in liquid). If this vapour is completely condensed, the liquid so produced will also contain 0.2 mole fraction LB. Now, if this liquid is again evaporated, its initial vapour will now have 0.4 mole fraction LB (i.e. two times the concentration in liquid). Thus, by vaporizing the liquid and condensing the vapour, we can increase the concentration of LB.

In actual practice, vapour concentration is never a fixed multiple of liquid concentration, and, in any case, cannot increase beyond 1.0. It can be determined more accurately by using 'relative volatility', which depends on the vapour pressure of components and molecular interactions between them. It is given as  $\alpha = [y / (1-y)] \div [x / (1-x)]$ , where  $x$  = concentration in liquid,  $y$  = concentration in vapour.

A distillation column consists of a number of stages. Liquid from an upper stage mixes with vapour from a lower stage. The mixture so formed splits into another liquid and vapour, which are now in equilibrium. The new vapour has higher concentration of more volatile material. The process is repeated to separate the components. An example with relative volatility of 2.0 is given below.

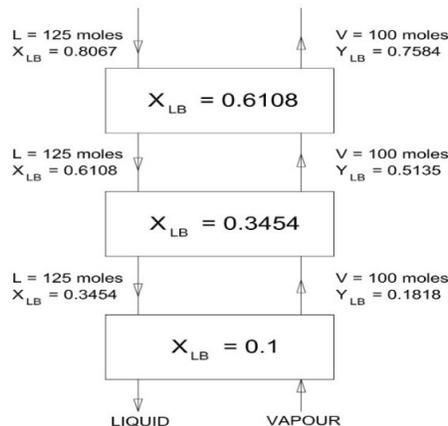


Figure (1): Stage-by-stage equilibrium



## Types of distillation

In a simple binary distillation, the low-boiling (or more volatile) component is produced at column top, and high-boiling (or less volatile) component is produced at column bottom.

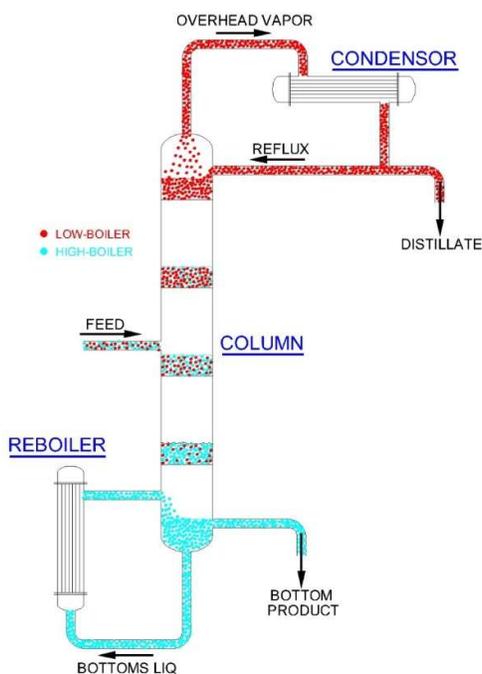


Figure (2): Binary Distillation

For some mixtures, liquid concentration is exactly equal to its vapour concentration. When this liquid is vaporised, it produces a vapour of the same concentration as the liquid. It is not possible to achieve further separation beyond this point. This is called the azeotropic point and such mixtures are called azeotropes. One well-known azeotropes are of ethanol and water, which forms at 95.5 % w/w ethanol and boils at 78.4 centigrade, at 1 atmosphere.

Azeotropes can be separated (or 'broken') by various techniques. Some such methods are vacuum distillation, azeotropic distillation with/without entrainer, extractive distillation etc.

Some azeotropes 'disappear' under vacuum. For example, ethanol-water mixture becomes non-azeotropic at 75 mm Hg absolute pressure. If it is distilled below this pressure, it is theoretically possible to obtain dry ethanol.

Some azeotropes could be 'broken' by adding another component. For example, ethanol-water azeotrope can be broken by adding cyclohexane. This produces a three-component azeotrope of ethanol, water and cyclohexane - boiling at around 64 C. It moves to the top of column, and splits into an organic layer and a water layer after condensation. Organic layer is refluxed back to column and water layer drained out from a phase separator. Thus,



although water's boiling point is higher (100 C), it is removed from column top. Cyclohexane, increases the volatility of water and 'entrains' i.e. carries water to column top, so it is also called an 'entrainer'. This process is called azeotropic distillation.

In extractive distillation, another component is added that reduces the volatility of one of the components. For example, ethylene glycol may be added to ethanol-water azeotrope. It has a high boiling point and greater affinity for water. Water is carried to bottoms along with ethylene glycol and dry ethanol is obtained from column top. Ethylene glycol and water mixture, produced at the bottom of column, is separately fractionated to recover and reuse ethylene glycol. Ethylene glycol is called the extraction solvent, because it "extracts" water out of the ethanol-water azeotrope.

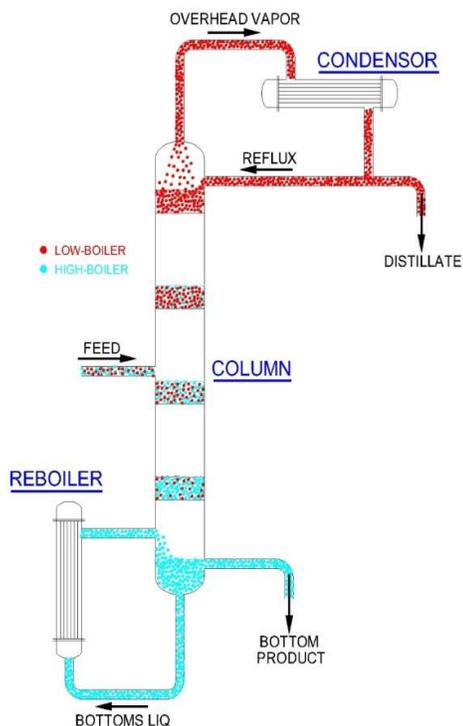


Figure (3): Azeotropic Distillation

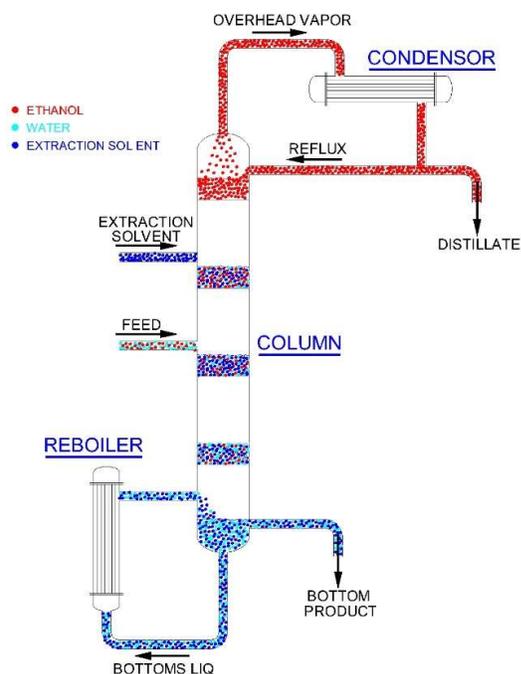


Figure (4): Extractive Distillation

### Batch v/s Continuous distillation

Distillation can be carried out in batch or continuous modes.

In batch distillation, entire feed is taken to a distillation still at the beginning. The distillation column is mounted on the still, or connected to it. Vapour from the still goes to the column bottom and rises to the top. Here it is condensed, partly refluxed and partly taken to draw.



In batch distillation, multiple products can be obtained in one column. For example, if the mixture contains a low-boiler (LB), a medium-boiler (MB) and a high-boiler (HB), then LB fraction can be taken first, followed by MB fraction and lastly by HB fraction from the same column and one after another. The highest boiling component can also be vaporized and condensed. Thus, even this fraction can be obtained free of solids and color.

In continuous distillation, feed is taken to the distillation column at a fixed continuous rate. It is sent to the feed nozzle on the column itself, which produces an overhead and a bottoms product. A number of columns are required for multiple products. For example, in the mixture of LB, MB and HB, first column will produce LB at column top and a (MB+HB) mixture at bottom. This bottom mixture is taken to a second column that produces MB at top and HB at its bottom. Thus, two columns are required to achieve separation. Also, high-boiling components are produced in liquid form at bottom, so they will contain solids and color originally present in feed.

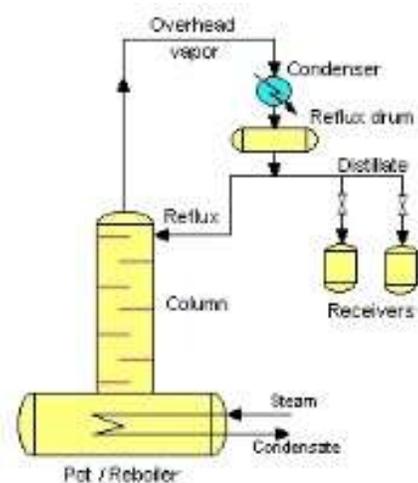


Figure (5): Batch Distillation

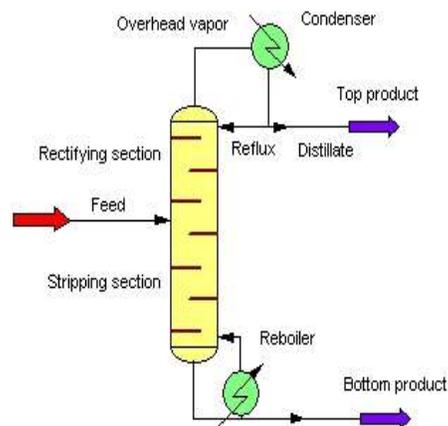


Figure (6): Continuous Distillation

## Design Techniques

Design of a distillation system, involves process design followed by detailed design and engineering. Process design has been traditionally done by techniques such as McCabe-Thiele diagram, Ponchon-Savarit diagram, Fenske-Underwood-Gilliland correlations etc.

These days, sophisticated computer software is available for design and engineering. Some of them are as follows.

- Aspen Plus by Aspen Technologies
- Chemcad by Chemstations
- Batchcolumn, ProSimPlus etc by ProSim
- gPROMs by PSE Ltd
- CADSIM Plus by Aurel Systems



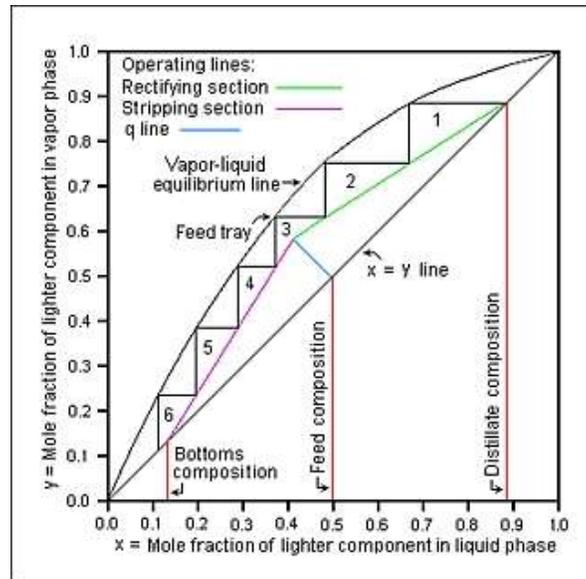


Figure (7): McCabe Thiele Diagram

## Tray columns

Each distillation stage requires thorough contacting of the liquid and vapour phase – as well as separation of the mixture into a new liquid and a new vapour phase. This is achieved by trays or packings.

Trays involve physical mixing of vapour and liquid - similar to sparging. Some common types of trays are sieve tray, bubble-cap tray and valve-tray. Packings involve forming of a liquid film on their surface. Vapour flows over the liquid film and mass transfer takes place at the liquid-vapour surface. Packings can be random or structured packings. They are available in different materials of construction.

For all trays, liquid comes in from the upper tray via a downcomer. It flows across the tray into another downcomer - which takes it to the tray below. Vapour rises up through holes or riser pipes on the tray surface. Vapour-liquid mixing takes place on the tray.

Sieve trays have small holes drilled on them. Vapour rising through the holes ensures that liquid does not drip down through the holes itself. However, if vapour flow rate is too small, it may not be enough to hold up the liquid and then the liquid can drip down. This is called “weeping” and reduces efficiency.

Bubble-cap trays have a number of small pipes called risers, welded to holes on the tray. A slotted cap is mounted on each riser. The slots are immersed in the liquid. Vapour rises through the riser, turns around inside the cap, and bubbles into the liquid through the slots. There are no exposed holes on the tray surface, so liquid cannot drip down to the bottom tray even at low vapour flow rates.



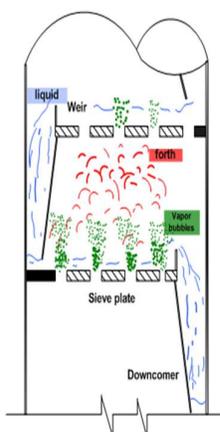


Figure (8): Sieve Trays

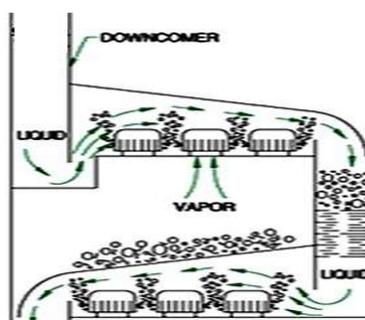


Figure (9): Bubble-cap trays

Valve trays also have holes punched on the tray and vapour rises through these holes. However, these holes are “covered” by valves that can move up and down. When vapour flow rate is high, valves open more and when flow rate is low valves open to a lesser extent. If vapour flow rate is too small, valves close and reduce the quantity of liquid that can drip down. This gives some protection from weeping.

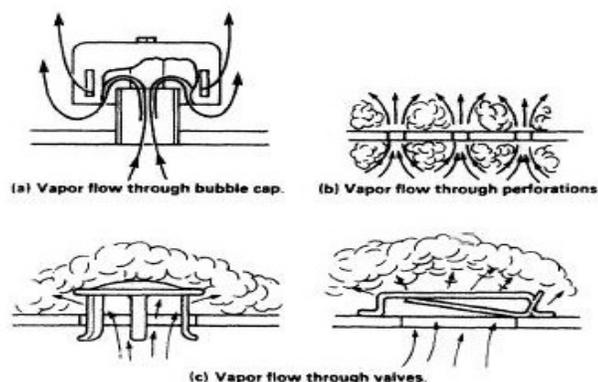


Figure (10): Mechanisms of tray operation

Bubble-cap Tray	Valve Tray	Sieve tray
No possibility of weeping	Some possibility of weeping	Very susceptible to weeping
Resistant to choking	Susceptible to choking	Susceptible to choking
Expensive	Relatively cheaper	Least expensive
Not easy to clean physically	Easier to clean physically	Very easy to clean physically
Suitable for high turndown ratio	Less suitable for high turndown ratio	Not suitable for high turndown ratio



## Packed columns

In the case of packings, a liquid film forms on the surface. Vapour flows over the surface and mass-transfer takes place at the vapour-liquid interface.

Thus, if higher surface area is available for mass transfer, efficiency is higher. Packings are available in areas from 75 m<sup>2</sup> to 1500 m<sup>2</sup> per m<sup>3</sup> of packed volume. Furthermore, packings are more effective if liquid forms a film over the entire available surface. This generally happens for organic liquids of low surface tension. However, for liquids with high surface tension such as water, the liquid forms tiny rivulets on the packing surface instead of a film. In such cases, efficiency is lower.

Packings can be random or structured. Random packings are small pieces of metal alloy or ceramic, formed to specific shapes. The most common random packings are raschig rings, lessig rings, splined rings, pall rings, berl saddles and intalox saddles. Random packings are randomly filled in distillation columns.

Structured packings are manufactured to fit into column shell ID. For small columns, they are made in one piece. For larger columns they are made in several pieces and put together inside the column. Structured packings have higher surface area than random packings.

Packed columns are subject to “channelling”. This means the liquid will tend to flow along the inside surface of the column rather than distribute equally over the entire cross-section. To minimise this effect, redistributors are placed every few meters inside the column. These collect the liquid that has strayed towards the periphery and distribute it uniformly again.

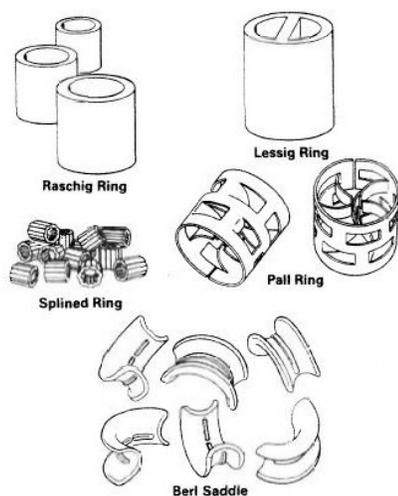


Figure (11): Random packings



Figure (12): Structured packings



Packings and trays can be compared as follows.

Parameter	Trays	Random Packings	Structured Packings
Column height	Tallest	Medium height	Shortest
Effect of column pressure drop	Not suitable for vacuum service	May be acceptable for vacuum service	Recommended for vacuum service
Liquid holdup	Very high	Low	Very low
Liquid surface tension	Does not matter significantly	Matters to some extent	Matters to significant extent
Corrosion resistance	Moderate	Low	Very low
Fouling liquids	Can handle	Cannot handle	Cannot handle

### **Erection**

Plant erection starts after the civil and structural work is completed and all fabricated / bought-out items are procured. It includes -

- 1) Erection of equipment on foundations / structure.
- 2) Fabrication of process piping as per piping diagrams.
- 3) Fabrication of utility piping, which connect utility equipment to plant.
- 4) Electrification of pumps, compressors, agitators, lighting etc.
- 5) Installation of control and indicating instruments.
- 6) Hot and cold insulation of equipment.

Some important considerations in designing the layout and erecting the plant are as follows.

- 1) Provide adequate space on each floor for personnel movement.
- 2) Provide access to equipment, piping and instruments for operation and maintenance.
- 3) Utility headers and local instruments should be on a designated operating floor.
- 4) Piping should unobtrusive and not impede personnel movement.
- 5) Drain points should be at the lowest local points.
- 6) Provide sufficient gravity head for flow between equipment, drains, NPSH requirement of pumps, space for u-seals etc.
- 7) Vents, relief valves etc. should open to a safe region, away from personnel.





Figure (13): Typical Plant layout

## **Commissioning**

Commissioning is carried out after a plant is fully erected. It is the procedure of establishing operating procedures and parameters. It consists of four main steps. These are given below along with some of their features.

- 1) Pre-commissioning. These activities are done before taking actual feed to the column.
  - Confirm that equipment, piping and instruments are erected / installed as per design specifications.
  - Test the erected plant for leakages, by hydrotest or pneumatic test.
  - Calibrate / tune indicating and control instruments.
  - Establish the fluid flows. This is often done by 'water boiling', where the plant is run using plain water as feed.



2) Start up and normal operation.

- Start vacuum, cooling water, chilled water, chilled brine, feed, steam etc. in the proper sequence.
- Start product draw after concentration profile is established in the column.
- Initial product cut may not match specifications. Such off-spec product may be recycled for re-distillation.

3) Shutdown.

- Shut vacuum, cooling water, chilled water, chilled brine, feed, steam etc. in the proper sequence.
- Extract residual product present in the column at high reflux.
- Drain the holdup and Flush the system by water-boiling.

4) Troubleshooting. During commissioning, column may have teething troubles. Some common problems are as follows.

- Column flooding due to excess vapour flow, blockages etc.
- Unacceptable purity of product due to inadequate reflux, insufficient utilities etc.
- Losses of valuable material through effluent due to insufficient utilities etc.
- Special conditions resulting in non-performance, which will be considered in a separate lecture.

