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THE SIMPLIFIED GUIDE TO FRACTIONAL DISTILLATION

A Complete Review on the Processes and Applications of Fraction Distillation

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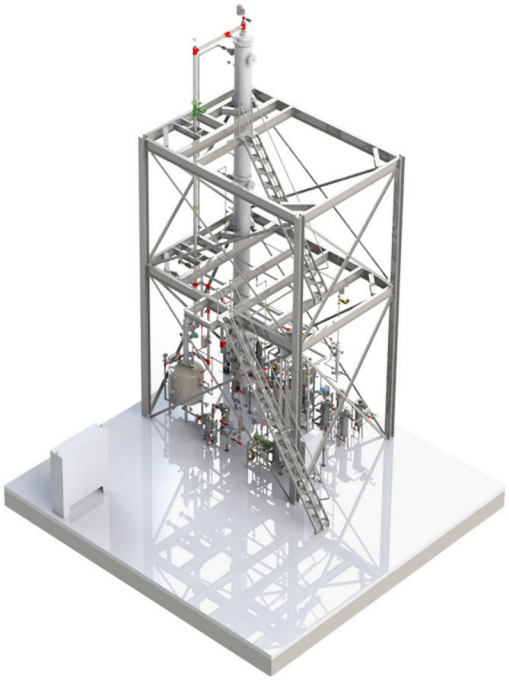
CONTENTS

INTRODUCTION



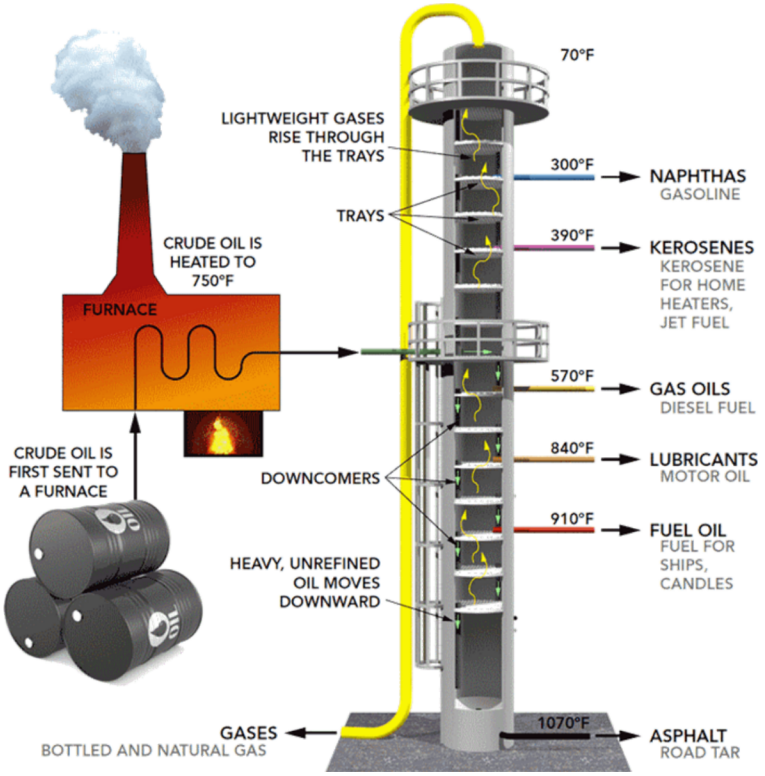
Removing and Improving

Purifying and reusing solvents from liquid waste may seem like a relatively straightforward process, right? However, when you have tens of thousands of gallons of spent solvent, it can be a tedious and time-consuming process.

Recycling solvents is an efficient and cost-effective option in comparison to constantly spending money on brand-new solvents. The disposal of large quantities of spent solvents is not only expensive but also bad for the environment. Solvent recycling is beneficial because it can be done on-site, reduces solvent purchasing, and is environmentally friendly.

In this e-book, we will discuss what fractional distillation is, the benefits of fractional distillation over simple distillation, the pros and cons of using fractional distillation, and how it can benefit you.

WHAT IS FRACTIONAL DISTILLATION?

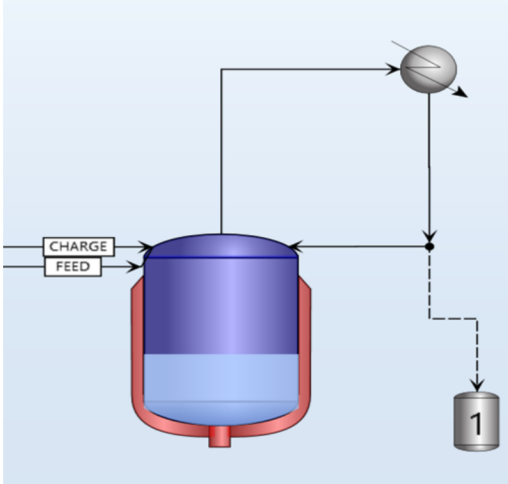


(Graphic courtesy of Bismarck State College National Energy Center of Excellence)

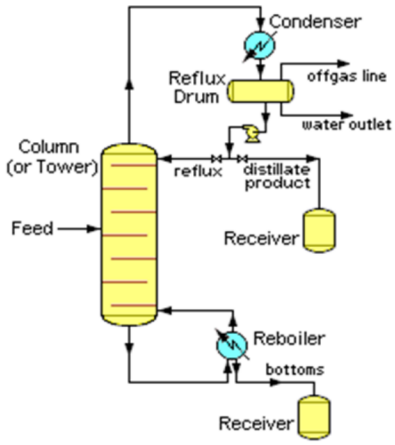
Fractional distillation has been used for decades to separate crude petroleum oil into different hydrocarbon products (fractions) based on their relative volatility, vapor pressure, or boiling points in a distillation tower. The same tower can be used to recover the solvents from the spent solvent mixtures, a solvent recycling process for purifying as well as refining. It is a separation process that removes one component, usually a solvent, from a waste product.

Distillation works on the principle of difference in boiling points of the components being separated from each other. The light component with a lower boiling point will boil out first and be recovered as a distillate from the top whereas the heavy component (higher boiling) will be collected at the bottom. Fractional distillation creates a high-purity product, with a process that requires less effort and turnaround time compared to running simple distillations repeatedly.

THE WORKING PRINCIPLE



Batch Distillation



Continuous Distillation Column

(Ref: https://engineering.fandom.com/wiki/fractional_distillation)

Working Principle: Distillation works on the principle of separating different boiling liquids using heat to boil out light components from top leaving heavy components on the bottom. Both batch and continuous distillation columns have the following functional steps:

01

Introduction of Feed Mixture

Feed is introduced in the batch (as charge) or column (on the feed plate). It can enter in any of the following conditions – saturated liquid (%vapor = 0), saturated vapor (Liquid% = 0), or mixed feed containing partial vapor and liquid mixture.

02

Heating Process

The batch vessel is heated either by steam in jacket or heating coils, whereas the feed mixture in continuous column needs to be heated in the reboiler.

03

Vapor-Liquid Equilibrium

Once the boiling point of the light component is attained in the column, the solvent vapors from the top are condensed in the condenser and recovered. Whereas the heavy component stays in the bottom, a fraction is removed, and the remaining is re-boiled and fed back.

VAPOR-LIQUID EQUILIBRIUM

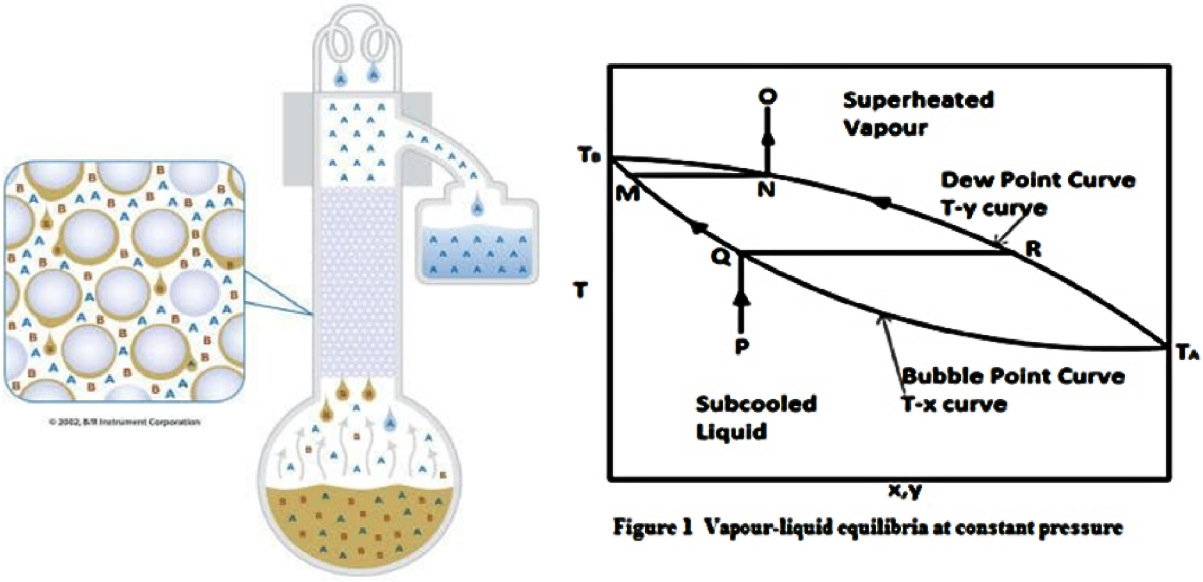


Figure 1 Vapour-liquid equilibria at constant pressure

(Ref: http://vlabs.iitkgp.ernet.in/vlabs/vlab14/flashdrum/)

Heat is supplied to the column through a constant external heat source, usually hot oil, or pressurized steam. By connecting the heat source to one side of a heat exchanger, the fluid from the bottom of the column can be pumped through the other side to increase the temperature and boil the column contents.

The contents, now at their boiling point, are returned to the column and the vapors begin rising through column internals. As the vapor rises, a portion of it will cool and condense and the liquid will fall back to the reboiler. This liquid will mix with the rising vapor and improve separation efficiency.

This rising and falling of vapor and liquid will continue in a closed loop inside the column until specified purities are reached and vapor-liquid equilibrium is attained.

As per the molecular theory, the distance between the molecules is increased by adding energy in the form of heat. During condensation, this energy is removed, and the intermolecular distance is restored. This process will not alter the physical-chemical properties of the solvent involved and therefore it is theoretically possible to repeat the distillation process an indefinite number of times.

PRINCIPLE OF SEPARATION

Distillation exploits the difference in relative volatility of feed mixture components at a given temperature and pressure. Under this condition, there will be a marked difference in the vapor and liquid composition at equilibrium due to the component's partial pressure which promotes the desired separation.

At equilibrium conditions, the component with lower volatility also known as the heavy key component (higher boiling point) will move into the liquid phase and more the volatile components aka light key components move into the vapor phase.

Sections of the Distillation Column

The distillation column is separated into three sections:

1) Rectification or Enrichment Section
2) Stripping or Exhausting Section
3) Feed or flashing Section

Rectification Section

The collection of trays above the feed stage is called rectifying section, also called the enrichment section. The more volatile component is removed by contacting the rising vapor with down-flowing liquid and is recovered from the top section; condensed and collected in a distillate drum.

Stripping or Exhausting Section

The trays below the feed stage comprise the stripping section also known as the exhausting section. Here the down-flowing liquid is stripped of the more volatile component by the rising vapor.

Feed or Flashing Section

The middle section is where the feed enters the tower and part of the feed is vaporized. This vaporization is commonly known as flashing, so this section of the tower is often called the flash zone.

OPERATING PARAMETERS

Temperature Profile

The basic temperature profile of a fractional distillation column is hotter at the bottom and cooler at the top. For simple binary distillation, the temperature at the bottom is just lower than the boiling point of the heavier component. The temperature at the top of the column is just above the boiling point of the lighter component to keep in the vapor phase.

Bottoms have heavy components in the liquid phase whereas the top of the column is saturated with light components in the vapor phase. The temperature of the column is maintained by adding heat using a heat exchanger called a reboiler at the bottom of the column and it is easy to maintain by adjusting the rate of heating (Heat Duty).

The temperature at the top of the column is maintained by adjusting the reflux rate, ie. the flow rate of the liquid sent back after condensing the overhead vapor (Condenser Duty). A higher reflux rate means cooler liquid falling down the column against the rising warmer gas resulting in lower temperatures at the top. So, overall heat is added at the bottom of the column and extracted at the top. The temperature balance inside the column is maintained by hot gas rising and cooler liquid falling down the column.

Heat Duty (Sensible Heat – No Phase Change)

$$Q = M * Cp * \Delta T$$

Heat Duty (Latent Heat – Phase Change)

$$Q = M * \lambda$$

Heat Duty for Multiphase Streams

$$Q = Qg + Qo + Qw$$

M = Mass (Kg)
Cp = Specific heat of the liquid (J/(kg K)
ΔT = Temperature difference (K)
λ = Latent heat of vaporization (J/kg)

OPERATING PARAMETERS

Pressure Profile

A pressure gradient is developed across the column due to higher pressure at the bottom than at the top, which occurs as the liquid coming down the column impedes the flow of vapor up the column and imposes a pressure loss on the flow. In steady-state distillation, the pressure in the column is held constant, and the temperature is adjusted to control the composition of the product streams.

Vacuum and Atmospheric Distillation Principles

The solvent(s) boiling temperatures are normally the boiling points evaluated at atmospheric pressure (1 atm or 760 mm Hg). Once the pressure is reduced, the boiling point of the liquid is reduced according to the ideal gas law PV = nRT. Therefore, when a vacuum is created inside the distillation chamber, the boiling point of the liquid(s) to be distilled is considerably reduced.

Atmospheric Distillation

The distillation process can be performed at atmospheric pressure. This is not recommended for a solvent with a very high boiling point. It is advised for solvents with boiling points between 70 °C and 170 °C.

Vacuum Distillation

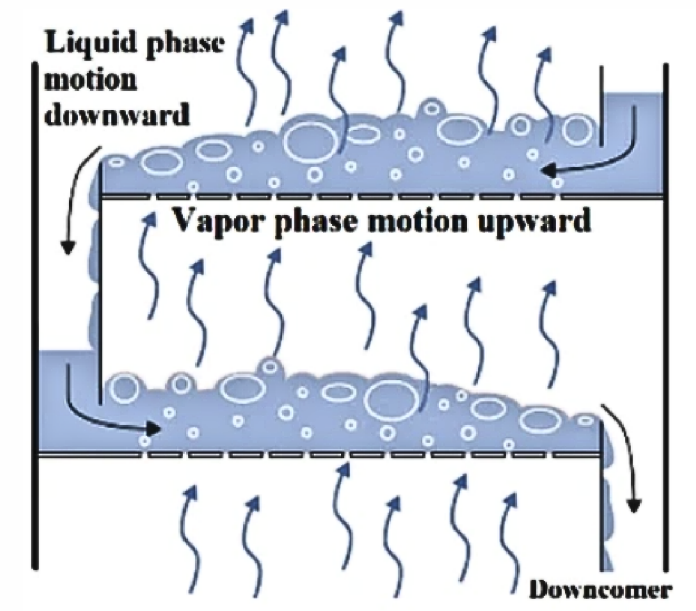
Lowering the pressure lowers a solvent's boiling point. The lower the pressure, the lower the boiling point of the solvents. By creating a vacuum inside the distillation vessel, reaching a pressure of about 150 Torr to 200 Torr, the boiling point will be lowered by about 30 to 40%. In certain cases, it is necessary to use vacuum distillation.

Selection Criteria for Vacuum Distillation

- When processing solvents with high boiling points.
- When processing a solvent with an auto-ignition point close to the boiling point.
- Contaminants that disintegrate at high temperatures.

COLUMN INTERNALS

TRAY COLUMN



(Ref: https://link.springer.com/article/10.1007/s42452-020-03470-y/figures/7)

In a distillation column installed with trays, the gas-liquid interaction takes place on each tray. An equilibrium is attained at each tray and the phase compositions change as they both enter and exit a single tray.

The liquid entering the tray will contact the gas exiting the tray. The hotter vapor phase will heat the incoming liquid phase as it bubbles through the tray above, evaporating the light key components which further leave the tray with the vapor phase.

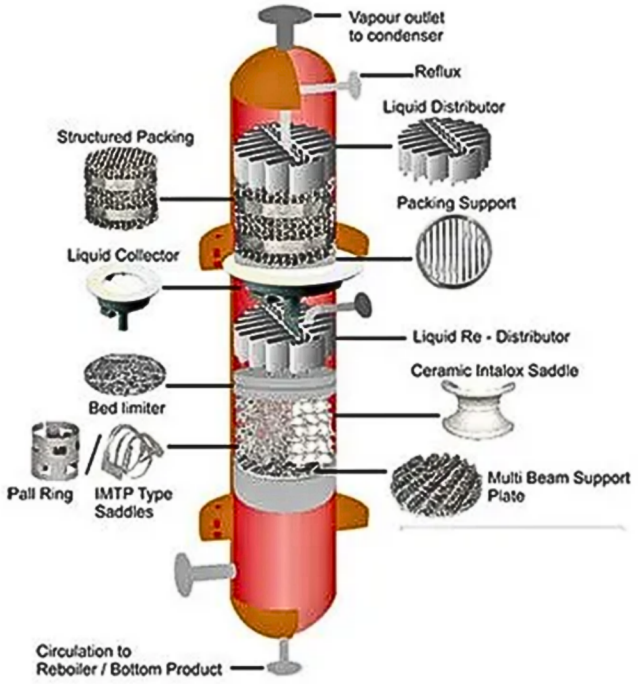
Conversely, the cooling of the vapor phase by the downcoming liquid phase will cause the heavy key components of the vapor phase to condense and exit the tray with the liquid phase downwards.

Type of Trays

- Sieve trays
- Bubble cap trays
- Valve trays
- Proprietary trays

COLUMN INTERNALS

PACKED COLUMN



(Ref: https://www.theengineersperspectives.com/packed-distillation-column/)

A packed distillation column is comprised of a cylindrical vessel filled with packing material that enhances continuous mass transfer between two fluids (liquid-liquid) and (gas-liquid) by effectively increasing the contact surface area. One fluid should preferentially wet the packings, flowing as a film over its surface while the second fluid flows over the remaining volume of the vessel. Fluids are passed through the column in countercurrent flow and the type and material of packing are selected which will offer a large surface area for mass transfer.

Type of Packing

Random packing uses a random distribution of small packing materials to assist the separation process and is made of plastic or metal. Raschig ring, Lessing ring, Pall ring, Berl saddle, and Intalox saddle are a few to name.

Structured packing is a type of organized packing used to channel liquid material into a specific shape. It uses discs composed of materials such as metal, plastic, or porcelain with their internal structures arranged into different types of honeycomb shapes.

PURITY OF PRODUCTS

Reflux

The liquid that is obtained by condensing the overhead vapor of the light key component in the condenser is called condensate. When a fraction of condensate is returned to the top of the column to improve the %purity is called reflux.

Reflux Ratio

The ratio of the liquid fraction returned to column (L) and one recovered out as distillate (D) is called reflux ratio (R_d).

$$R_d = L/D$$

Total Reflux

Total reflux is an operating condition where the overhead vapors are condensed and returned to the column without any distillate recovered. This allows the vapor and liquid interaction on each column tray to occur with no products removed.

$$(D = 0, R_d = L/D = \infty)$$

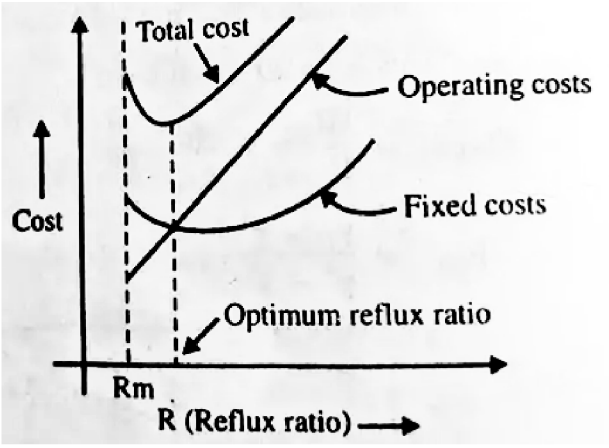
When the column is running under total reflux conditions, the minimum number of trays is required. Generally, all the distillation columns run under total reflux conditions initially to attain equilibrium. The OpEx will be higher due to increased heat duties.

Minimum Reflux Ratio

The minimum reflux ratio (R_{min}) is the lowest ratio at which separation can be achieved, however, it requires an infinite number of theoretical plates to get this separation done. As the reflux ratio increases, the number of theoretical plates required decreases. Higher CapEx due to a greater number of trays and lower OpEx due to lower reboiler and condenser heat duties.

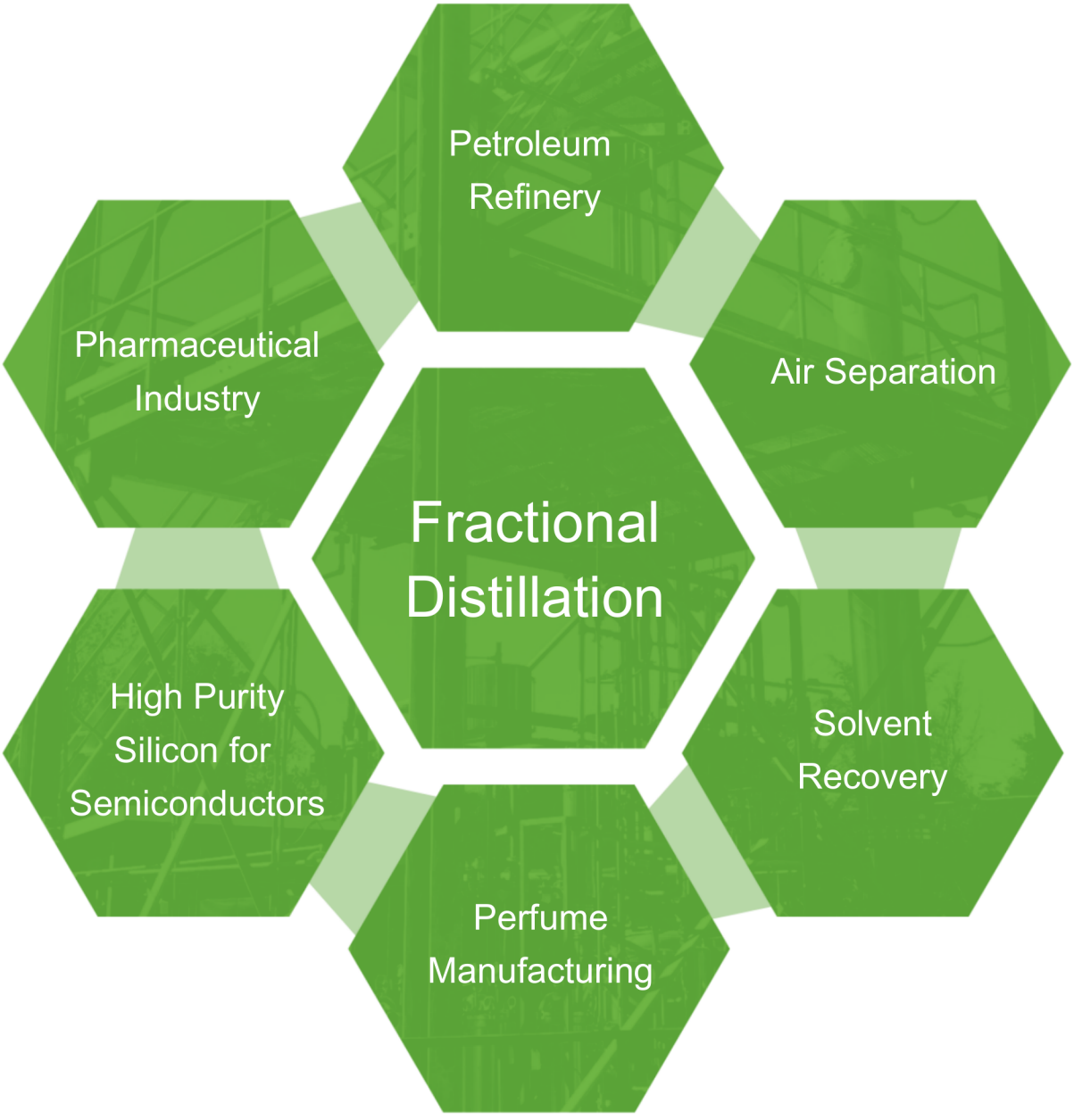
Optimum Reflux Ratio

The Optimum Reflux Ratio (Ro) is calculated based on the total capital cost and operating cost of the distillation column. At this ratio, the total cost of the distillation is minimum as the capital cost of the column (which depends on the number of the theoretical plates) and operating cost (depends on the reflux ratio, heat duties) are optimized or minimized.



Optimum Reflux Ration for Distillation

INDUSTRIAL APPLICATIONS



INDUSTRIAL APPLICATIONS

Petroleum Refining

Oil refineries use the most technologically advanced fractionating columns to distill out different petroleum cuts. The complex mixture of hydrocarbon in crude oil can be separated into fractions by the technique of fractional distillation. The crude oil is fed at temperatures around 650 K in a 25-100 m high fractionating column where different fractions condense at certain temperature ranges and separate out as petroleum cuts. The topmost comes natural gas, gasoline, naphtha, kerosene, diesel, fuel oil, wax, and bottom-most bitumen.

Solvent Recovery

Waste spent solvents like hexane, ethanol, gun wash, MEK, IPA, acetone, lacquer thinners, and line wash are often discharged without recovering, and fresh solvents are used every time. With environmental waste being a hot issue, the benefits of recovering and recycling these solvents outweigh burning them for fuel or discharging them. It also reduces hazardous waste disposal and associated costs. Solvent recovery with fractional distillation is not only environmentally friendly, but it is also cost efficient. The ability to separate solvent mixtures that have been previously used, then clean and purify them to produce a solution that is able to be used over and over is a win-win situation for industries.

Cryogenic Fractional Distillation for Air Separation

Cryogenic distillation (also known as low-temperature rectification) is a process of separation of a gaseous mixture, using simple distillation, at high pressure and low temperature. It is a separation process that works by liquifying the gas mixture at very low temperatures and then selectively distilling the specific gas component at its boiling point. This process is used for large industrial-scale manufacturing of high-purity oxygen and nitrogen products with high yields however the process is energy-intensive due to refrigeration requirements.

GLOSSARY

Fractionation - Another term for distillation, or fractional distillation.

Feed - The liquid and/or gas feed into the distillation column. The tray below the inlet nozzle is called the feed tray.

Heavy Key Component - The component with the lower relative volatility, low vapor pressure, and higher molecular weight. Found in higher concentration in the bottom product of the column.

Light Key Component - The component with the higher relative volatility, higher vapor pressure, and lower molecular weight. Found in higher concentration at the top of the column.

Light Key Component - The trays between the bottom of the column and the feed tray. In the stripping section heavier component is concentrated in the liquid phase.

Rectifying Section - The trays between the feed tray and the top of the column. In the rectifying section, the lighter component is concentrated in the vapor phase.

Top Product - The product which leaves the top of the column, passes through a condenser and condensate is collected as distillate.

Bottom Product - The product which leaves through the bottom of the column.

Reflux - A portion of vapor from the top of the column which has been condensed to a liquid and returned to the column as a liquid above the top tray.

Reboiler - A heat exchanger at the bottom of the column which boils some of the liquid leaving the column. The vapor generated returns to the column at the bottom of the stripping section.

Vapor-Liquid Equilibrium (VLE) Curve - A plot of the actual composition of the lighter component in the vapor phase for a given composition in the liquid phase and is derived from thermodynamic data.

CONTACT

About Maratek

Maratek is a global market leader in professionally engineered solvent recycling technologies, simulation and optimization of fractionation, and cannabis/hemp extraction technologies.

As a Canadian-based, award-winning, industry leader– Maratek has proudly served industrial manufacturers globally for over 50 years. Maratek manufactures eco-friendly and state-of-the-art equipment that recycles waste for reuse from printing, coatings, pharmaceuticals, automotive, aerospace, paint, and many other related manufacturers. Helping them stay competitive in the marketplace by cutting costs and saving money.

Maratek is a company that focuses on development efforts to reduce, reuse, and recycle solvents and other liquid wastes in various industries. Maratek develops the latest technologies utilizing our vast experience, supported by ASPEN simulation and optimization helping clients worldwide achieve the best ROI possible.

Visit www.maratek.com to learn more about our solvent recycling services.

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