

# Distillation Definitions

## Introduction

There are many separation processes and each one has its best application. They include distillation, crystallization, membrane, and fixed bed adsorption systems. Occasionally the best system may be a combination of these systems.

The choice of the best application should be based on the life cycle cost. The life cycle cost is the initial capital cost of plant along with the first ten years operations and maintenance cost. The life cycle cost should include a reliability factor, which is very important in designing any process plant equipment, reactors or separation equipment. Improved reliability has a very large impact on return on investment (ROI). Many life cycle cost only review energy, but not solvent, adsorbent, or catalyst cost because of accounting rules and this can lead to skewed economic decisions.

Distillation may be the most economical and utilized when possible. Distillation is the separation of key components by the difference in their relative volatility, or boiling points. It can also be called fractional distillation or fractionation. Distillation is favored over other separation techniques such as crystallization, membranes or fixed bed systems when;

1. The relative volatility is greater than 1.2,
2. Products are thermally stable,
3. Large rates are desired,
4. No extreme corrosion, precipitation or sedimentation issues are present,
5. No explosion issues are present

## Trayed Columns

### Tray Capacity

Capacity of perforated trays is often plotted as a function of percent hole area. Actually, the capacity of a perforated tray is not much affected by hole area unless the lack of hole area increases pressure drop and down comer backup to unacceptable values. For example, if a perforated tray has sufficient hole area to limit dry tray pressure drop to a reasonable value (about 2" to 3" liquid at 80% flood) the perforated tray will have the same capacity as a valve tray. A bubble cap tray cannot be designed to have as much hole area as a valve tray and will, therefore, have less capacity.

### Tray flexibility

Tray flexibility is a function of pressure drop and number of flow paths. If it is necessary to reduce the hole area of a perforated tray to meet certain flexibility limits, the perforated tray will have less capacity than a valve tray. If flexibility is not a consideration, the perforated tray is probably the correct choice unless there are conditions such as extremely low liquid rates, which may favor valve or bubble cap trays.

## **Number of flow paths**

The minimum number of flow paths is consistent with the capacity requirements. This will result in highest tray efficiency and flexibility at lowest cost. From a capacity viewpoint, a liquid rate greater than 6 gpm / inch of weir (weir loading), is the rate at which a larger number of flow paths should be considered.

## **Weir Loading**

Weir loading is a measure of the amount of liquid going over the outlet weir. The units are quite strange, area over time, but this term is directly relatable to the clear liquid height on the tray. The Francis weir formula, the basis of most state-of-the-art froth height models, has this term, weir loading, as its basis for determining the liquid depth. A maximum value of 13 gpm/in is a recommended maximum based on experience. Values less than 1.5 should use picket fence outlet weirs to prevent the onset of spray fluidization on the tray.

## **Calculate of tower diameter**

After the tray type has been selected, valve tray diameters may be calculated using the vendor design manuals. Various technical articles have been written on design and sizing of perforated trays. In addition, many companies have established procedures for this type of tray. In the absence of established procedures, the diameter of perforated trays can be calculated using the valve tray sizing procedure. Bubble cap trays may be calculated by the method of Davies or Bolles.

## **Considerations of liquid and vapor distribution**

Consideration must be given to the correct distribution of vapor and liquid regardless of the type of tray selected. Becker and Bolles have studied the effect of mal-distribution on large diameter multi pass trays and methods, which could be used to offset or prevent mal-distribution. One feature of the multi-chordal or sweptback downcomer, which was not stressed, is the improved distribution of liquid to the tray below. This reduces the area of stagnant liquid pools, thereby increasing efficiency and flexibility at reduced rates through decreased leakage.

## **Anti-Jump Baffles**

Anti-Jump Baffles are plates suspended vertically above center and/or off-center downcomers which stops liquid jumping from one deck side over the downcomer onto the opposite flow-path or bridging. It also forces the liquid into the downcomer. Further, the baffle aids the disengaging of vapor from the liquid entering the downcomer. This device is used for large liquid loads and when the width of the center and/or off-center downcomer is small.

## **Top tray feed designs**

For columns where the feed is on the top tray, and when the feed stream contains vapor, special design features are desirable. For example, parts of the tray subjected to abnormally high forces should be strengthened and the feed pipe should be anchored to the

tower wall. Columns such as amine regenerators frequently flow vapor and liquid in slugs to the top or feed tray. Severe hydraulic pounding and tray damage can occur if the feed tray has not been properly designed to withstand this force. For normal fractionators, it is sufficient to feed the reflux behind a 4 - 6" high inlet weir or to use a false down comer. No other precautions are usually necessary.

### **Liquid feed streams vaporization**

Intermediate feed streams should be checked carefully for an estimate of possible vaporization. Feeds normally considered being liquid, frequently contain vapor as a result of pressure and/or temperature changes before entering the column. If vapor can be present or if the feed is at a temperature greatly different from the feed tray, the feed should not be introduced into the down comer. Circulating reflux is an exception here since it can be colder than the liquid in the down comer without fear of vaporization. If vapor is present in the feed, it is recommended that wider tray spacing be used at the feed tray. The increase in tray spacing depends on the quantity of vapor present, but an additional 6-12" is usually sufficient. Generalizations cannot be given for feed streams, which are predominantly vapor.

### **Orientation of seal pans and nozzles**

Orientation of the seal pans below the bottom tray down comer should be set so that the reboiler vapor does not impinge directly on seal pan overflow (this could entrain liquid to the bottom tray). When one must orient the reboiler return nozzle so vapor impinges on the overflow, the nozzle should be extended into the column and a tee or other device added so vapor enters parallel to the edge of the seal pan.

### **Bottom tray Considerations**

If space between the bottom tray and the liquid level is insufficient, vapor might cause excessive liquid to be entrained to the bottom tray and this can damage the trays. Insufficient space below the bottom tray is responsible for 50% of the problems occurring in the lower part of columns. Standard spacing below the bottom tray and seal pan orientations are about three to five times the diameter of the reboiler return nozzle or feed nozzle.

### **Utilizing reboiler baffles**

A preferential reboiler baffle is often used below the bottom tray as a means of directing the liquid from the bottom tray to the reboiler. If one is used, it should be oriented perpendicular to the axis of the seal pan in the case of multi pass trays. Otherwise the vapor must break through a curtain of liquid to enter the bottom tray. This could cause excessive entrainment and possible mal-distribution. If a baffle is used to direct the liquid to the reboiler, it should be oriented parallel to the 90° - 270° axis for single-pass and parallel to the 0° -180° axis for two- and four-pass designs.

### **Product draw sumps**

Product draw sumps are required in several types of columns. These may be for partial or

total draw of the liquid from the down comer above. If the total flow from the down comer is to be withdrawn, a positive seal is recommended to prevent vapor from flowing up the down comer. A total draw sump with a positive down comer seal will generally require additional spacing above the draw tray as well as below.

### **Chimney Tray Utilization**

Chimney trays may be used as draw trays, transition trays and/or protection against leakage. Use a chimney tray if residence time is required for pumping, start-up or other reasons. Since this type of tray frequently maintains a fairly high level of liquid on it (and consequently a tremendous weight), special consideration should be given to its design. One of these is the placement of the draw nozzle. A flat chimney tray can be used with the nozzle located at tray floor level or a portion of the tray floor can be lowered to form a sump and the nozzle located at the sump floor. Both designs require the same liquid head to force the design flow rate through the nozzles. Therefore, locating the nozzle in a sump lowers the liquid level on the chimney tray by an amount equal to the head requirements. This reduces the weight which the tray must support considerably but has little effect on residence time.

Under most conditions, rectangular chimneys are more economical than round ones. If the tray fabricator proposes rectangular chimneys, they should be accepted since this reduces costs. A chimney height of 6-12" is normally adequate for low liquid flow. 12-18" is usual for high liquid flow if the draw nozzle is located in an inlet sump. All chimneys should have hats located a sufficient space above the chimney to give a peripheral area of 1.25 times the chimney area. The hats should extend at least 1" past the chimney on all sides and should be turned down slightly (15 degree angle starting 1" from the edge of the hat, if rectangular) to prevent liquid from running back underneath the hat and down into the chimney. If a leak-free design is required, the inlet sump should be seal-welded and gasketing used on the chimney tray floor.

### **Tray flooding**

Flooding is an expression of the capacity of the fractionating device in a column. Flooding is evidenced by the holdup of liquid in the column. The increased pressure drop denotes this increased liquid holdup across the tray. As the  $\Delta P$  increases with an increase in either liquid or vapor flow rate, the liquid level in the down comer increases. Ultimately, the liquid level will reach the tray above, and if the condition persists, the whole tower fills with liquid. If an attempt is made to increase either the vapor or liquid rate when approaching the flood point, the other rate must be reduced. A flooded column is very unstable and exhibits a reduced efficiency.

Correctly designed fractionating devices flood due to the vapor and liquid rates reaching those rates for which the device was designed. Incorrectly designed trays may flood due to a) insufficient down comer entrance area, and b) excessive pressure drop due to excessive liquid and vapor loadings. Flooding may also be caused by foaming, plugging, mechanical obstructions or a change, usually sudden, in the operating conditions of the tower, for example, loss of vacuum, loss of reboiler, etc.

## **Tray jet flooding**

Jet flood occurs when the vapor velocity through the liquid bed or tray becomes sufficient to aerate the liquid to such a degree that the space between trays is full. This aerated bed causes an increase in the pressure drop and impedes the flow of the liquid into and through the down comers. It is called jet flood because of the "jetting" action of liquid spray from tray to tray.

## **Tray downcomer flooding**

A downcomer may flood due to insufficient entrance area, insufficient downcomer volume, and insufficient escape area from the downcomer or excessive tray pressure drop. The industry generally employs 80% flood as a standard for the maximum operating range for normal design. Most systems show a decline in tray efficiency at rates exceeding 80% flood. This results in extra energy use or a product of lower quality.

## **Tray recessed inlet sumps**

Recessed sumps are used for very high liquid rates as this permits an adequate escape area under the downcomer without resorting to taller overflow weirs. (A taller overflow weir can reduce capacity, by increasing pressure drop and downcomer backup.) Recessed sumps should not be used as a means of maintaining a downcomer seal for low liquid rate services, because of the difficulty in preventing leakage from the sumps. An inlet weir would be more appropriate. Recessed inlet sumps should not be used in those services where fouling and plugging could occur.

## **Mist eliminators**

Mist eliminators have high removal efficiency over a wide range of operating conditions. The efficiency does decrease at low vapor rates, which creates a desire on the part of most designers to reduce the diameter of the pad. Unless the pad is in a service where the mist (entrainment) is independent of the vapor rate (a pad located above a spray header, for example) the diameter should not be reduced normally since the mist is nil at low vapor rates and a decrease in efficiency is not apparent. Some manufacturers show removal of particles 8 microns diameter and larger. Frequently, the particles smaller than 8 microns are equal volumetrically to the particles larger than 8 microns.

## **Tray efficiency**

Calculations are made which indicate the vapor and liquid composition that would result if equilibrium existed at that particular temperature and pressure. One theoretical plate would accomplish these compositions. By repeating these calculations until the desired terminal compositions are reached, the total number of theoretical plates, or trays, can be calculated. The number of theoretical trays used divided by the actual trays results in the tray efficiency for that system and at those operating conditions. In the case of packings, the height of packing to equal a theoretical plate is used (HETP) to express efficiency. If trays on 24" spacing have 50% efficiency, four feet are required for a theoretical plate. If packing has an HETP of four feet, the two devices are equal efficiency-wise.

## **Bubble Cap Tray**

The bubble cap tray is one of the earliest types of trays. It consists of a perforated deck with risers around the holes, and caps in the form of inverted cups over the risers. The vapor leaving the riser is diverted downward by the cap, exits at the cap rim or through slots cut in the cap and then enters the liquid layer. The bubble caps maintain a positive liquid seal with the risers, which allows the tray to operate with minimal weeping at low vapor rates: as a result, this kind of tray is the most leak resistant one. Bubble cap trays have lower capacity and efficiency and higher entrainment and pressure drop. Bubble cap trays are more costly than sieve and valve trays.

## **Dual Flow Tray**

A type of tray that is essentially a sieve tray with no downcomers. During operation, vapor and liquid counter-currently flow through the perforations in the tray deck. This tray has high capacity and fouling resistance due to high open area. It has lower efficiency than a standard cross flow tray and may become unstable in towers with moderate to large diameters. Dual flow trays have very little turndown capability.

## **System Limit**

This is a Stokes Law criterion that predicts a vapor rate limit beyond which entrained liquid droplets cannot settle back into the bulk liquid phase. The System Limit is independent of tray design or spacing. A System Limit restraint usually occurs only at high tower pressures (> 10 atm).

## **Other Issues**

### **Packings verses trays**

A general statement might be made, that packings are chosen at either of the two ends of the design spectrum:

- a) very small towers (less than 24" diameter),
- b) low pressure drop limits and
- c) services where plastic or aluminum materials would be beneficial.

Packings are highly desirable in small towers due to mechanical limitations imposed due to size. In some tower sizes, trays other than cartridge style are impractical, if not impossible to install. Packings are desirable in situations where pressure drop is a primary consideration. An excellent example is the vacuum crude tower, where  $\Delta P$  is held to a few mm Hg over the entire tower.

### **Crude towers special considerations**

Atmospheric and vacuum crude columns need a considerable space in the flash zone to handle the large volume of vapor present and to prevent entrainment to the tray above. It is recommended that a tangential helical baffle or vapor horn be used at the feed nozzle.

## Start-up Considerations

In order to avoid damage to equipment, several of the more usual causes of problems will be noted here. They are well known but frequently overlooked:

### *High liquid level in the column*

If the liquid level is too high, vapor velocity causes massive entrainment to the bottom tray. This entrainment may even be in the form of waves of liquid. This constant buffeting of the tray loosens bolting and can dislodge the tray.

### *Too rapid liquid drainage*

When a column floods, the liquid level may rise to a point several feet above the bottom tray. If the liquid is withdrawn from the bottom of the column at a rate greater than it can flow down through the trays, a vapor gap will be formed below the bottom tray. The presence of a vapor gap below the bottom tray, however small, imposes on that tray the weight of all the liquid above it and could cause immediate failure. This is fairly common during start-up when the column becomes flooded and one's immediate reaction is to lower the level as rapidly as possible.

### *Pressure surges*

All columns should be designed so any change in pressure causes an upward flow of vapor instead of a downward flow. In the case of vacuum columns, the valve used to change from vacuum to atmospheric pressure should ideally be located near the bottom of the column. If the temperature or contents in that zone prohibit location of the valve in that area, the valve may be located at the top of the column. However, its size must be restricted to  $\frac{1}{2}$  -  $\frac{3}{4}$ ". A larger valve may permit too large a quantity of air or inerts to enter the column, if trays have a liquid level on them, the vapor will not be able to flow downward through the tray freely. This can create a high-pressure differential and cause failure. Similar situations have been noted where rupture disks have been located in the lower part of the column at pressures above atmospheric.

### *Steaming out a column*

Many columns are operated on steam-water prior to start-up in order to check out instrumentation. Caution must be exercised to assure that steam is not condensed during this operation. Condensing steam can result in a downward-acting differential pressure across a tray, which may exceed the mechanical strength of the tray and cause failure,

### *Water in a column*

Many trays are installed and tested for tightness and may even be seal-welded to insure against leakage. As a result of the tray leakage tests or from washing out the column during the shutdown procedure, water may remain in the column. If the feed to the column is extremely hot, this water left in the column may be vaporized instantaneously, causing an increase in vapor rate. Liquid may also be lifted into the trays above. The net effect is damage to the trays.

## Foaming Factor

The empirical de-rating factor reduces the predicted capacity of the tray (increases % flooding, downcomer froth backup, and reduces maximum downcomer inlet velocity) to account for a known foaming system. A value of 1.0 is used to represent a non-foaming system while a lower value, 0.85 for example, represents a moderately foaming system. Lists of general foam factors as well as several specific recommendations are included below.

### General Foaming Guidelines

Classification	Foaming Factor
Non-Foaming	1.0
Low	0.9
Moderate	0.75
High	0.6

Specific Service	Guidelines
Absorbers >0 *F	0.85
Absorbers <0 *F	0.80
Amine Contactor	0.73
Vacuum Towers	0.85
Amine Regenerator	0.85
H <sub>2</sub> S Stripper	0.90
Furfural Fractionator	0.85
Top Section DeC <sub>1</sub> , DeC <sub>2</sub>	0.85
Glycol Contactor	0.50
Glycol Still	0.80
CO <sub>2</sub> Absorber	0.80
CO <sub>2</sub> Regenerator	0.85
Caustic Wash	0.65
Caustic Regenerator	0.60
Sour Water Stripper	0.60
Alcohol Synthesis	0.35
Hot Carbonate Contactor	0.85
Hot Carbonate Regenerator	0.90