

“Resolving Process Distillation Equipment Problems”

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Introduction

In most chemical processing systems two main unit operations dominate; chemical reaction followed by separation. The chemical reaction step is normally completed in a reactor. The reactor can be in numerous forms, from a plug flow reactor, to a CSTR (Continuously Stirred Tank Reactor), which can be in the form of a batch reactor, to a fixed or a fluidized catalytic bed reactor. From the reactor the reactants are then sent to a separation unit. The reactants are separated into desired products, unreacted products for recycle, and unwanted or by products.

Most Separation Units contain distillation equipment. Distillation Equipment was developed to separate ethanol from the by-products of fermentation. From the original batch stills, distillation equipment has progress to the type of trayed and packed columns used today. Today columns range from absorbers, extractors, strippers, and rectifying towers. They include vapor / liquid columns, liquid / liquid columns, and extractive distillation and reactive distillation columns. Vapor / liquid columns are designed to separate products by boiling point differences. Liquid / Liquid columns are designed to separate products by a physical property difference such as polarity. Extractive and reactive distillation columns shift equilibrium by removing one of the products to improve the equilibrium distribution.

General Distillation Equipment Design

The first step in resolving any distillation problem is to understand the operating and technical fundamentals of the column. Knowledge of how a column functions, hydraulic constraints, thermodynamic and equilibrium limits, and heat and material balances are required. This knowledge needs to be accumulated in advance of formulating any resolution of a problem.

At least three types of distillation equipment problems exist. The first problem is inappropriate design, the second problem is inappropriate operation, and the third is potential damage to internal equipment. Before a process is shut down for repairs the inappropriate design and damage to internal equipment should be determined, and inappropriate operation should be eliminated.

Appropriate Stage Design

The design of stage operations has progressed from a trial and error basis to a computer-modeled system. The computer-modeled system came become an error-based system if operational feedback is not utilized. The computer model should match existing field data if the tower is operating properly, and if not, field data should be re-verified. If field data is accurate, the model should be adjusted to match existing data. The computer model can be verified by developing a McCabe-Thiele plot to verify the number of separation stages required. If you assume that the model is correct in all cases, you will soon have opportunities for new employment.

Trayed Columns utilize a pressure and temperature differential, to create a mass transfer gradient, to separate the products. Packed Columns generate a mass transfer area by providing a large surface area over which the liquid can transfer heat and mass to the vapor.

Pressure is a very important constraint in stage design. In low-pressure systems the vapor is considered the continuous phase and in high-pressure systems the liquid is considered the continuous phase. In low-pressure systems packing can be successfully utilized. In high-pressure systems packing can fail, due to a back mixing mechanism, therefore trays are the preferred system.

The temperature at which the reflux can be condensed usually determines the tower pressure. Normally the preferred temperature is that of cooling water. If cooling water cannot be used, the pressure is set using the best combination of variables. The first variable is that hydrocarbons separate easier at lower pressures, because the differences in their relative volatilities are larger at lower pressures. Therefore lower pressure equates to less reflux, less stages and smaller columns.

The second variable is the increased cost of cooling at lower pressures. Often at lower pressure, refrigeration is needed equating to increased operating cost. A balance has to be constructed between capital cost and operating cost. This balance is also utilized in the determination of the amount of reflux versus the number of stages. This balance is between the energy requirement versus the cost of the additional stages. Computer based models has made this balance optimal.

Minimum Reflux Ratio and Minimum Number of Stages by use of Simulation

To determine the minimum reflux ratio and the minimum number of stages, one develops a reflux-stage plot and extrapolates from it. To develop this plot, simulation runs are performed at different number of stages while keeping the material balance, product compositions, and the ratio of the feed stage to the number of stages constant. The reflux ratio is allowed to vary. Then a plot of the number of stages versus reflux or reflux ratio is plotted. The curve is extrapolated asymptotically to an infinite number of stages to obtain the minimum reflux ratio and asymptotically to an infinite reflux ratio to obtain the minimum number of stages

Optimization of Feed Stage by Simulation

To determine the optimum feed stage, simulation runs can be performed at several different feed positions. In the simulation runs, the material balance, reflux ratio, and total number of stages need to be kept constant. Then two main plots can be created. One plot is the McCabe-Thiele diagram and the other is a concentration versus feed stage diagram. The McCabe-Thiele diagram is plotted using the mole fraction data calculated for each stage by the simulation. The equilibrium data and the operating lines are determined from this data. The McCabe-Thiele diagram then shows how an optimum feed stage versus a non-optimum feed stage looks when using the simulation data.

Pressure Choices

Many times when designing a distillation tower, the controlling factor in choosing the column pressure is the heating requirements of the reboiler or the condensing requirements of the overhead condenser. For example, a new column was being installed to separate benzene and toluene. For this design case the controlling factor in choosing the column pressure was the ability to use low-pressure boiler feed water as a condensation medium to produce low-pressure steam. This choice would mean that the column was going to operate under pressure. Performing this separation under pressure had a couple of advantages.

1. Increasing the column pressure would increase the vapor density and therefore the vapor handling capacity. This would lead to a reduction in the diameter of the column, which would reduce the overall cost of the project.
2. It would allow the possibility of having the benzene-toluene splitter share a condenser with a tower used to remove benzene from vent gas. Both columns' overhead products would go to the same location. The cost of installing a complete condenser system for this column would be considerably reduced.

However, in raising the column's operating pressure there are some unfavorable effects.

1. Raising the pressure lowers the relative volatility and increases the separation difficulty.
2. Raising the pressure also raises the reboiler temperature, thereby requiring a more expensive heating medium. A reboiler with a larger heat transfer area would be required.
3. Above 100 psig pressure, the columns shell thickness increases in order to handle the higher pressures. This will constitute an increase in capital costs.

Reboiler Design and Selection

Several types of reboilers can be selected based upon operational needs and reboiler duty requirements. For larger duty requirements forced circulating reboilers are required. The lowest in cost are the once through thermosyphon reboilers and they are preferred if the required duty can be delivered. If fouling due to low velocities or a higher duty is required a circulating thermosyphon reboilers or forced circulation reboilers may be preferred.

Most types of reboilers use condensing steam as a heating medium. Steam condensation may occur on the shell side or the tube side depending of the type of reboiler used. The steam flow may be horizontal or vertical. As with all condensing systems, it is very important to see that the system is regularly vented to prevent the

build up of non-condensable gas in the system. Non-condensable gas in condensers is the most common reason why the condensation heat transfer coefficient is less than expected.

In industry many different types of reboilers are used. Overviews of three types used are given below.

1 Vertical Thermosyphon Reboiler

A vertical thermosyphon reboiler is very similar to a long tube evaporator and a climbing film evaporator. Liquid from the column sump flows through the inlet leg of the reboiler, enters the bottom channel, and is distributed uniformly to the tubes. A shell-side fluid, often condensing steam heats the tubes. Condensate flow in this type of reboiler is vertical. The process fluid entering the tubes is below its boiling point due to static head effects and must undergo sensible heating; for vacuum systems, such heating may consume a significant portion of the tube length.

Heat is transferred by both nucleate boiling and two-phase convective mechanisms. The two-phase mixture exiting from the heated zone returns to the column for disengagement of the phases, with the net vapor representing the needed boil-up for the distillation process and the liquid representing a recycle. Good design calls for vaporization per pass in the range of 10% to 30%; thus, there is a significant recycle flow.

The advantages of the vertical thermosyphon reboiler are the low residence time of the process liquid, the low liquid inventory of process fluid and, the low floor area required. Another advantage of this type of reboiler is the high heat transfer coefficients that are obtained. Vertical thermosyphon reboilers are usually the best value for the heat supplied. This type of reboiler can be used in fouling services because these exchangers are easy to clean.

The disadvantage of this type of exchanger is that they require extensive amounts of headroom. A distillation column may have to be raised off the ground in order to accommodate the reboiler. This may cause a mechanical design problem with the column. Stability of the column may become an issue.

2 Horizontal Thermosyphon Reboiler

This is perhaps the most common type of reboiler. A horizontal thermosyphon reboiler consists of a horizontal shell and tube exchanger with a single horizontal baffle. The process fluid flows along the shell-side along the length of the tube bundle from its point of entry midway along the shell to the ends. The fluid then turns 180° and flows back to the midpoint of the shell along the upper part of the shell. Boiling takes place over most of this flow path. The heating medium, usually steam, flows inside the tubes usually in two paths. The steam enters along the upper pass and leaves along the lower pass, allowing the condensate to drain naturally out of the bundle. The flow of process fluid through the reboiler is governed by thermosyphon action, although a pump could be installed in the inlet pipe if necessary. The flow rate through this type of reboiler is controlled by density differences.

The main advantage of the horizontal thermosyphon reboiler is the ease of removing the tube bundle for cleaning. Also, the horizontal arrangement permits a lower elevation of the return line. This allows for a lower column elevation in relation to the reboiler elevation.

3 Kettle Type Reboiler

The kettle reboiler is similar to a shell-side evaporator. The heating fluid is usually condensing steam flows inside the tubes, which are commonly U-Tubes. The U-Tube bundle occupies the lower part of the K-Type shell. Liquid boiling is outside the tubes and the eccentric bundle arrangement makes available space for vapor-liquid disengagement. An internal weir controls the liquid level in the shell. The liquid level is such that the top of the bundle is only just submerged. The liquid enters the reboiler by gravity feed. A valve usually controls the feed. The overflow from the weir is the bottom product of the distillation column. If necessary a pump can be installed in the pipe between the distillation column and the reboiler. A properly designed kettle can produce a near-equilibrium vapor mixture and thus can provide an extra theoretical stage for the separation. It has an advantage of convenient bundle removal for tube inspection, and is relatively insensitive to varying loads of vapor production. It is comparatively expensive, however due to the type of shell design used for this type of reboiler.

Trayed Columns Design

One of the very first trays to be developed was the sieve tray. It is essentially a plate with holes punched into the plate. The number and size of the holes is based on the vapor flow up the tower. The liquid flow is transported down the tower by downcomers, a dam and overflow device on the side of the plate, which maintains a set liquid level on the tray. To maintain the liquid level on the tray a minimum amount of vapor traffic up the tower must be maintained, or the liquid level on the tray will weep down to the next tray through the holes punched on the plate. Typically sieve deck trays have a minimum capacity, or downturn, of approximately 70%.

One of the next developments was to add a variable valve opening to the tray deck. This valve would open in relation to the vapor flow. The advantage to this design was the ability to maintain the liquid level on the tray deck. Typically valve deck trays have a minimum capacity, or downturn, of approximately 60%.

Some of the latest developments in tray design include changes to the downcomer and changes in the valve design. The downcomer requires a disengaging area to separate the liquid from the vapor. This area requires a minimum distance that normally sets the tray spacing. To use multiple downcomers reduces this distance and the total height of the tower. The liquid is required to travel across the deck to the next downcomer. If the valves are designed to help direct the liquid flow across the deck, by directing the vapor, the total time on the deck will be reduced leading to increased capacity. Trays are the most commonly selected type of tower internal. Generally trays perform well at high liquid and vapor loadings. At low flow parameters the capacity and efficiency of trays can be reduced.

Some other items to consider when deciding to use trays in a tower.

1. Trays have downcomer capacity problems in heavy foaming services.
2. Trays have a high resistance to corrosion.
3. Trays have higher pressure drop than structured packing or random packing.
4. Entrainment is an issue with trays. Trays usually have more entrainment than packings. Excessive entrainment can lead to efficiency loss.
5. Excessive vapor and liquid mal-distribution can lead to a loss of efficiency in a tray tower.

Mal-distribution can be caused by the feed and reflux inlet design. A good feed and reflux design will affect the equilibrium on the feed tray and the adjacent trays slightly. A poor inlet design can affect several trays above and below the feed point. If a tower has 20 trays and a poor inlet feed design disrupts the equilibrium on 4 of the trays, the capacity and efficiency of the tower can be reduced by 20%. Installing better inlet designs is an efficient way to improve separation.

Trayed Columns Trouble Shooting

Trayed Columns utilize a pressure and temperature differential to separate the products. The weir holds a liquid level of each tray. The vapor must overcome this liquid head to move up the column. On the tray the vapor and liquid are contacted and then above the tray they are separated. Any deviation that develops that restricts the vapor and liquid from contacting and then separating will deteriorate the column's ability to meet design specifications.

Deviations that will restrict the ability of a column to contact include, but are not restricted to;

1. Corroded, fouled, or eroded tray valves and feed devices,
2. restrictions in downcomers,
3. physical damage.

Deviations that will restrict the ability of a column to separate include, but are not restricted to;

1. excessive rates,
2. contaminants that cause foaming,
3. improperly sized downcomer openings,
4. liquid entrainment
5. foaming
6. excessive liquid back-up in the downcomers
7. physical damage.

Because a trayed column uses a pressure and temperature differential to separate the products, the pressure and temperature profile of a column is a key indicator of how the column is performing. The column temperature and pressure should gradually increase as one surveys down the column.

A calculation can be developed for what the column pressure drop should be, based on the number of stages and the height of the weir, or downcomer dam. If a column has twenty trays and a weir height of three inches the vapor has to overcome a liquid height of sixty total inches. The equivalent height of one pound of water is 2.768 inches. Therefore sixty inches of water equates to 21.6 pounds of pressure drop. This calculation will need to be corrected by the specific gravity of the actual liquid on the tray.

This pressure drop calculation is an essential tool in tower trouble shooting. If the pressure drop is low, tower may be weeping, internal tray man ways may be dislodged, or reflux flow may be inaccurate on the low side. If the pressure drop is high downcomers may be restricted, whole trays may be dislodged, or reflux flow rate may be inaccurate on the high side.

After a calculation of what the pressure drop should be, a pressure survey should be performed. It should be performed with the same pre-calibrated gauge, if not by the engineer, under the supervision of the engineer. Gauges can be damaged in installation and care should be used in the installation of the gauge. Field mounted pressure gauges contain a high level of uncertainty and should not be used for trouble shooting.

A temperature profile will also provide valuable information as to the operations of the tower. If thermo-wells and thermocouples are not available at the desired points throughout the tower, an IR temperature scan gun can be used at the inspection ports through the insulation. If the temperature profile is not consistent several causes are possible, hydraulic tray flooding or weeping, potential tray damage and fouled or corroded trays or downcomers.

In distillation towers there are actually two accumulators. The first is normally obvious, the over head receiver, the second is the bottom section of the tower. These accumulators are used to stabilize the operation of the tower and down stream operations. This internal surge drum creates an inventory to act as a buffer. If this internal level is allowed to rise above the reboiler return, stripping inlet, or feed inlet flooding can occur.

There is an inherent error built into sight glass and level instrumentation. The sight glass and level instrumentation contain non aerated liquid, called clear liquid, which is not a true indication of the condition of the liquid within the tower. The liquid within the tower will have two levels, a clear liquid level below the aerated liquid level. Because the aerated level will have lower specific gravity than the clear liquid within the instrumentation, the tower level will be higher than the instrumentation indicates. If the level in the tower is higher than the feed or reboiler return entrained liquid can be carried to the next stage causing flooding.

Synopsis of Tray Troubleshooting

Do simple checks first.

1. Assure that levels are accurate. Have operations move levels and view changes in the field.
2. Calculate column pressure drop and then measure pressure drop. Review survey pressure reading to operation's readings.
3. Survey column temperature profile. Review survey temperature reading to operation's readings.

Verify Tower Operations

Perform tower simulation to verify Tower Stage efficiency. Sometimes the feed compositions changes and tower is no longer able to meet desired specifications due to thermodynamic or equilibrium constraints. Needed to perform the simulation will be;

1. Accurate tower feed, Overhead, and Bottoms laboratory analysis
2. Accurate tower mass balance, within 2%.
3. Heating and cooling medium temperatures.

If the tower simulation confirms the limits are not beyond thermodynamic or equilibrium constraints and additional check may be to have the tower scanned to look for tray damage. This type of troubleshooting method can determine internal damage, vapor liquid mal-distribution, and packed and trayed tower fouling. Because of economic constraints, scanning should be chosen only after the simple checks and the limits are confirmed. Scanning can sometimes confirm the problem that was identified by the other checks.

Random and Structured Packed Columns Design

Random and Structured Packed Columns generate a mass transfer area by providing a large surface area over which the liquid can transfer heat and mass to the vapor. The packed column has several distinct advantages and some disadvantages.

A major advantage to packed columns is the reduction in pressure across the column. In high-pressure systems this is not important, but in low-pressure systems this can reduce the temperature, if polymerization is a concern as in a Styrene Monomer plant, or reduce the upstream pressure to help improve furnace yields, as in an ethylene plant.

Typically the column pressure drop for a packed column is less than that of a trayed column because of the percent open area. Typical percent open area of a trayed column is 8 to 15%, whereas a packed column can approach 50%. Liquid accumulation for a packed column is lower than that of a trayed column. This is important when degradation of products can occur at higher residence times.

Another advantage of packed column is reduced foaming. Packing generates thin films instead of fine droplets for mass and heat transfer, reducing entrainment when

foaming agents are present. An additional advantage is that residence time within a packed column is shorter leading to less polymerization potential. Because the residence time is short, control systems may need to be modified to account for this difference. For example, the control of a tower bottoms may be based on temperature or boil-up rate versus level.

Here are some points when trying to decide if structured packing should be the application of choice. The vapor and liquid rates, or loadings, of the tower are important when considering structured packing. Generally structure packing performs well at low liquid and vapor loadings. At high flow parameters the capacity and efficiency of structured packings can be significantly reduced. Structured packing is generally most efficient in low-pressure distillation applications.

Some other items to consider when trying to decide to use packing in a tower.

1. Structured packing does not perform well in heavy fouling applications.
2. Structure packing has a low resistance to corrosion.
3. Structured packing is a low-pressure drop device that provides high efficiency. This is why structured packing is extremely successful in low-pressure distillation applications.
4. Structured packing performs extremely well in foaming applications.

The design of a packed column includes the packing, liquid distributors, and liquid collectors. After the packing the liquid distributor is the most important part of the tower internals. It can determine the success or failure of the column. Packed towers are more sensitive to liquid and vapor mal-distribution than trayed towers. Therefore, it is critical that vapor and liquid enter packing evenly distributed. The performance of the packing depends heavily on the initial vapor and liquid distribution entering the packing. Poor vapor and liquid distribution to a packed bed can result in a loss of efficiency.

The liquid inlet distributor is the device that diffuses the liquid across the mass transfer area. This device must be designed properly or the liquid will not create the surface area required for separation. Many times the liquid inlet distributor is the main problem area for random and structured packed column problems. The liquid distributor types include V-notched channel distributor and the pan or orifice distributor.

The V-notched channel distributor is mainly used in towers of greater than three feet in diameter and is the preferred choice. The V-notches allow high liquid turndown and can handle liquids that contain solids or that have fouling potential. The pan or orifice distributor is similar to a sieve tray in operation and is normally used in towers of less than three feet in diameter with clean services.

To avoid the loss of efficiency due to channeling, the liquid should be collected and re-distributed every 15 to 20 feet. Above twenty feet the channeling becomes high and the efficiency is greatly reduced. The liquid should be collected in a chevron type

collector to evenly distribute the vapor to the next bed. The liquid should be taken and re-distributed with a V-notched type distributor.

Bed limiters or hold down grids are used to prevent expansion of the bed at high flow rates. They are attached to the tower wall by means of a support ring. Bed limiters or hold down grids should not be designed to produce a restriction in vapor flow and increase the tower pressure drop.

Tower internals must be installed taking particular care to insure levelness of parting boxes, troughs and similar equipment. In small columns moderate misalignments may be tolerated, but in large towers tolerances must be held to no more than +/- 1/8 inch (3 mm).

One of the great disadvantages of packing is the inability to properly inspect the installation. If the installers crush the packing during the installation the tower can flood because of reduced open area. A trayed tower is more controllable and the intermediate sections are inspectable.

Random and Structured Packed Columns Trouble shooting

Random and Structured Packed Columns generate a mass transfer area by providing a large surface area over which the liquid can transfer heat and mass to the vapor. Any deviation that develops that restricts the liquid from forming this large surface area will deteriorate the column's ability to meet design specifications.

Deviations that will restrict the ability of a column to generate this area include, but are not restricted to;

1. Packing damaged during installation,
2. incorrect distributor design or installation.
3. fouled packing,
4. packing flooding
5. contaminants that cause foaming,
6. liquid entrainment into a packed bed.
7. physical damage.

The pressure and temperature profile of a column is a key indicator of how the column is performing. The column temperature and pressure should gradually increase as one surveys down the column.

A calculation can be developed for what the column pressure drop should be, based on the height to the bed of the packing. For most packed beds the pressure drop should be between 0.1 to 0.8 inches of water per foot of packing. Below this rate the liquid may not be evenly spread across the packing and or the liquid rate may be inaccurate on the low side. Above this rate the packing may be fouled or damaged not allowing the liquid to exit from the packing and / or the liquid rate may be inaccurate on the high side.

After a calculation of what the pressure drop should be, a pressure survey should be performed. It should be performed with the same pre-calibrated gauge, if not by the

engineer, under the supervision of the engineer. Gauges can be damaged in installation and care should be used in the installation of the gauge. Field mounted pressure gauges contain a high level of uncertainty and should not be used for trouble shooting.

A temperature profile will also provide valuable information as to the operations of the tower. If thermo wells and thermocouples are not available at the desired points throughout the tower, an IR temperature scan gun can be used at the inspection ports through the insulation. If the temperature profile is not consistent several causes are possible, hydraulic flooding, potential damage and fouled or corroded packing.

Synopsis of Packing Troubleshooting

Do simple checks first.

1. Assure that levels are accurate. Have operations move levels and view changes in the field.
2. Calculate column pressure drop and then measure pressure drop. Review survey pressure reading to operation's readings.
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2. Accurate tower mass balance, within 2%.
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If the tower simulation confirms the limits are not beyond thermodynamic or equilibrium constraints and additional check may be to have the tower scanned to look for tray damage. Because of economic constraints, scanning should be chosen only after the simple checks and the limits are confirmed. Scanning can sometimes confirm the problem that was identified by the other checks.

Strategies for Field Work

1. Do not trip over the same obstacle twice.
2. Look out for other obstacles and prevent from tripping over them.
3. Arrange and time the obstacles to suit our purposes.
4. Monitor critical obstacles closely and clear them on time.
5. Do not assume there is only one critical obstacle.
6. Be wary of new obstacles and new untested techniques.

Conclusions

In column troubleshooting it is important to understand the fundamentals of distillation, the choice of tower internals and externals and how they interact. Several recommendations have been presented and should be evaluated for the best application. Keep in mind to always do the simple checks first, and use the available tools to verify that the separation that is desired is achievable.

REFERENCES

- 1 Zygula, T. M., Darbonne, T. "THE DESIGN AND PERFORMANCE OF A PACKED BENZENE TOLUENE SPLITTER.", The AIChE 1999 Spring National Meeting March 14-18, 1999 *Distillation: Horizons for the New Millennium - Session I*.
- 2 Zygula, T. M., Dautenhahn, P. Ph.D., "Use of Process Simulation for Distillation Design", The AIChE 2000 Spring National Meeting March 5-9, 2000.
- 3 Zygula, T. M., Barkat, O. Ph.D., "Troubleshooting Vertical Thermosiphon Reboilers", "The AIChE 1999 Spring National Meeting March 14-18, 1999.
- 4 Andrew W Sloley, Karl Kolmetz, "Texas Refiner Expands Aromatics Capacity", Oil and Gas Journal, March 24, 1994
- 5 Chen, Gilbert "Packed Column Internals", Chemical Engineering , March 5, 1984
- 6 Daryl W Hanson, Norm Liberman, "Refinery Distillation Troubleshooting", The AIChE Fall National Meeting November 12, 1995.
- 7 John J. France, "Trouble Shooting Distillation Columns" Sulzer Chemtech, March 1993