Troubleshooting Distillation Systems

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Distillation columns often do not perform as planned, for various reasons. These could be basic design errors or wrong inputs. The equipment or piping may not be sized properly, feed composition may not be as per design or utilities may not be available at the required pressure or flowrates.

If the actual feed composition is different from that specified at design stage, column performance will be affected. In fact, if the feed contains any impurity that was not specified earlier, the column could fail entirely. One such case is discussed in this lecture. It is commonplace to have fluctuations in steam pressure – which causes fluctuations in vaporisation rate. This may result in problems like inadequate reflux, flooding etc.

However, such problems are easily diagnosed and addressed. This lecture concentrates on case studies where problems were less obvious. They arose due to operational, equipment or detailing reasons.

Case-1

This case is regarding system holdup. This is the volume of liquid remaining or 'held-up' in the system, during operation. It includes liquid present in reflux drum, piping, reboiler, trays and packing distributors.

Holdup does have some advantages. It has a dampening effect on performance; hence it smoothens out fluctuations in the output because of perturbations in the inputs. In some cases, such as reactive distillation, it also provides required residence time. However, its disadvantages are that holdup must be fully filled first - at the required concentration profile - before any distillate can be taken. This increases the time cycle. Furthermore, some good product always remains within the holdup, which reduces overall recovery.

In the case under consideration, a large diameter batch packed column, with six distributors, was being used to separate isomers. The separation was difficult, and required high reflux ratio. The plant could not produce any product of required quality.

It was found that diameter of liquid distribution holes on the distributor was too small. Therefore, liquid level (hence, the holdup) on each distributor was too high. Total holdup became so high, that almost all the low-boiling component accumulated in the holdup itself, without anything coming out as distillate. Furthermore, it took 48 hours of total reflux to stabilise the concentration profile. This problem became worse because of difficulty of separation.

The problem was solved by increasing diameter of distributor holes. This reduced the liquid height on the distributors, thereby reducing holdup as well.

Figure (1): Packed column distributor

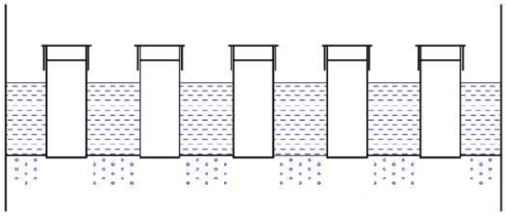


Figure (2): Level with small distributor hole

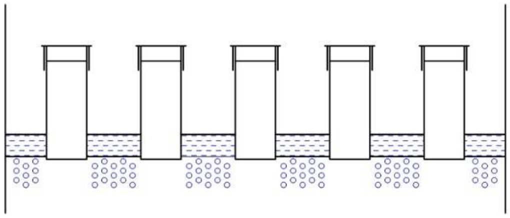


Figure (3): Level with large distributor hole

Case-2

Tube-to-tubesheet weld joints were cracking regularly. This was a fixed-tubesheet exchanger, and shell and tubes were both welded to common tube sheets.

In operation, shell was much hotter than the tubes and expanded more than the tubes. Thus, shell tended to push away the tube-sheets much more, compared to the tubes. This created thermal stresses which damaged the tube-tubesheet weld joints.

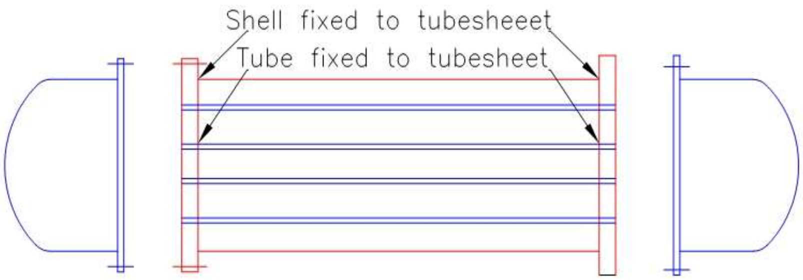


Figure (4): Shell-Tube-Tubesheet joints

The fixed tubesheet heat exchanger was replaced by a U-tube heat exchanger. The shell and tubes of a U-tube heat exchanger are welded to only one tubesheet, while their other ends are free to expand. Thus, there is no thermal stress generated in this case.

Another way of eliminating thermal stresses is by using shell-side bellows. These are a sort of cylindrical springs welded on shell side. Thus, any differential expansion between shell and tubes gets absorbed in the bellows, and does not show up as thermal stress.

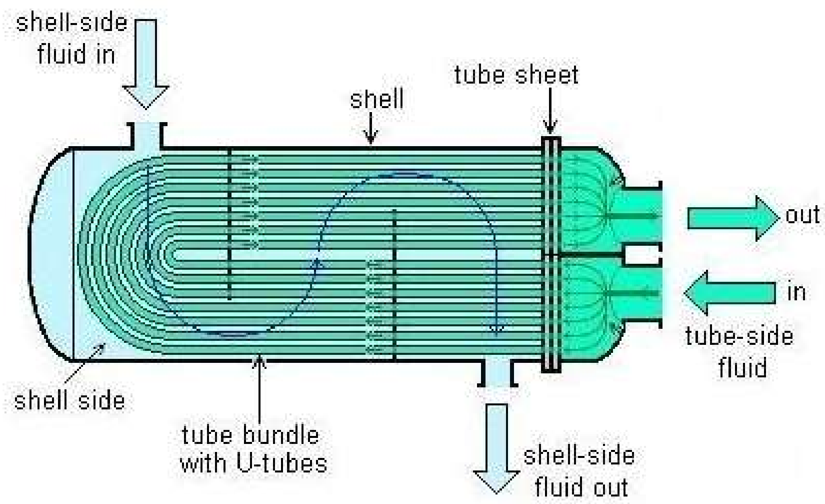


Figure (5): U-tube heat exchanger

Figure (6): Shell-and-Tube heat exchanger with shell-side bellows.

Figure (7): Bellows detail

Case-3

Reflux to a column was flowing in sudden spurts. Distillate was not of acceptable purity, even though relative volatility was high and separation was technically very easy.

The column was provided with a large vertical condenser, which was not vented. The reflux drum was, however, vented as shown. When vapour condensed, a partial vacuum was generated in the condenser. This vacuum did not allow condensate to flow to column. Instead, it backed up into the condenser. After it reached a certain level, hydrostatic head overcame the pressure differential, and reflux rushed into the column. Because reflux was not smooth, it was not possible to obtain required distillate concentration.

Pressure equalisation line was provided to the reflux pot, which eliminated this problem.

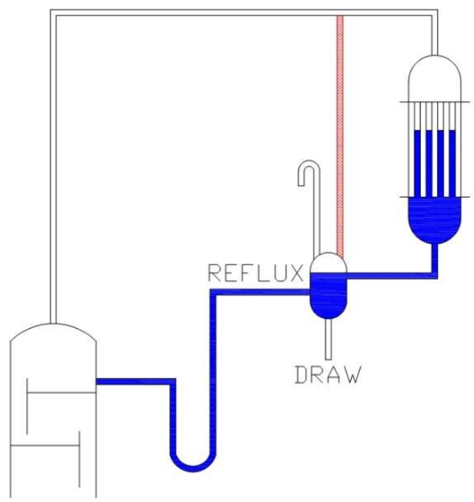


Figure (8): Vacuum effect in unvented condenser

Case-4

A distillation column was working satisfactorily under normal design conditions. However, if capacity was lowered to around 30% of design value, reflux flow stopped completely and distillate purity became unacceptable.

Nozzle diameters of condenser and column reflux did not match. The piping contractor had used an eccentric, in such a way that the draw line was tapped from lower part of pipe, while the upper part continued to reflux. When the flows were at normal design capacity, the reflux line was full of condensate, and reflux flowed. However, when flows were turned down, liquid levels decreased and upper part of the pipe became empty. Condensate flowed to draw, but there was no reflux, and distillate purity was affected. The eccentric reducer was eliminated, and sizes of nozzles and condensate piping were made the same.

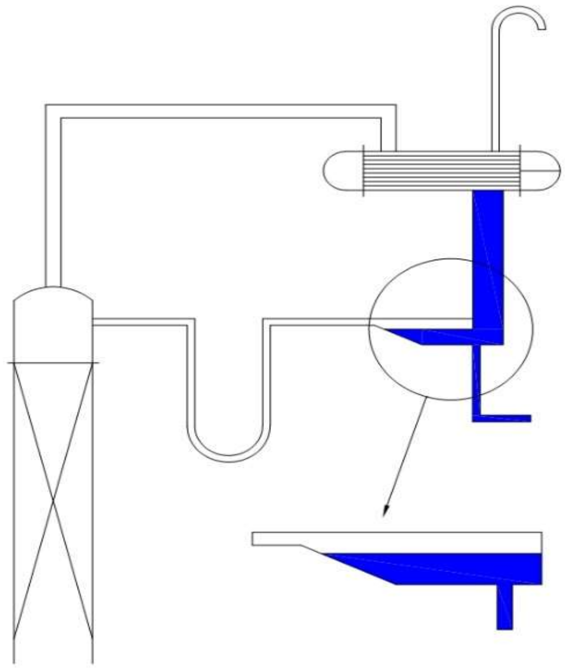


Figure (9): Illustration of eccentric reducer case

Case-5

In a batch distillation, structured packings in bottom section of a column were getting corroded frequently. Removing and replacing damaged packings was a tedious process, since it required dismantling the column, removing the packings and replacing them from the body flange opening.

The feed contained some inorganic acids. The acid would not ordinarily vaporise, but it was found that the vapour going from the still to the column was entraining liquid droplets - which carried some inorganic acids. The acid corroded the thin packings much more rapidly, than the much thicker still.

The vapour was taken to a cyclone separator that removed much of the entrained liquid, and returned it to the still. The liquid-free vapour then went on to the column. Further a small section of random packings was introduced at column bottom. Damaged random packings could now be removed and replaced in-situ easily, through external nozzles and without dismantling the column.

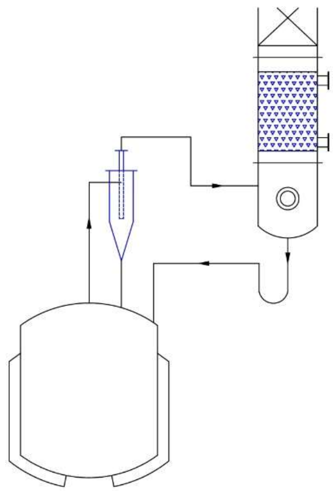


Figure (10): Illustration of corrosion of column bottom packings

Case-6

In a batch distillation, vaporisation rate decreased as cycle progressed, resulting in sub-optimal performance. As liquid level in the still decreased, less jacket area was exposed to heat transfer. Heat transfer could only take place from condensing steam to liquid inside the vessel. Lower effective heat transfer area resulted in lower vaporisation.

An external forced circulation reboiler was provided. The entire area of the reboiler could now be utilised - even when still level had dropped.

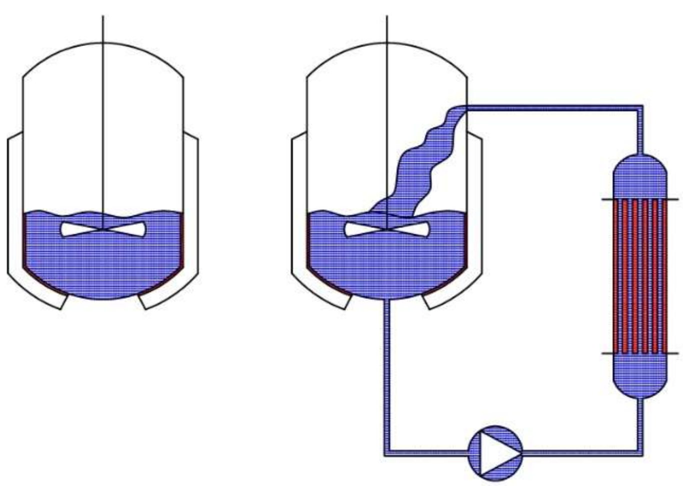


Figure (11): Illustration of jacket heat transfer area case

Case-7

A column-mounted condenser had adequate heat transfer area, but was not performing as per design.

This condenser was mounted on top of a column, and its vapour entry nozzle was on the bottom centre of the shell. Furthermore, its vent nozzle on the condenser was located on top centre of the shell - right above the vapour inlet nozzle. Vapour was bypassing most of the tube area and going to vent.

The condenser was replaced by one with a horizontal baffle, that redirected vapour so as to flow over the entire heat transfer area.

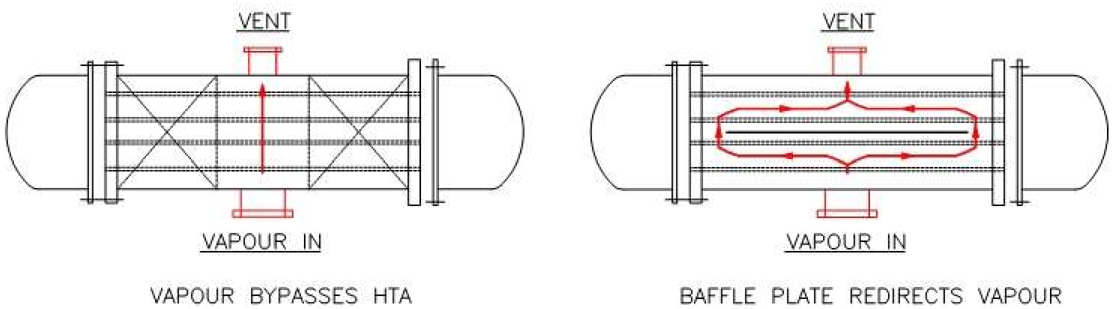


Figure (12): Illustration of vapour bypassing in condenser

Case-8

A simple vacuum distillation system was not giving adequate condensation rate. To augment the heat transfer area, several condensors were added in series. However, recovery rates actually decreased as more condensors were added in series.

It was realised that adding condensers, in fact, increased the overall pressure drop, hence still pressure. This elevated the boiling point at the still, reducing ΔT, and resulting in lesser vaporisation. The condensers were replaced by a single condenser with larger heat transfer area, but much lower overall pressure drop.

No. of condensors	Total ΔP, mm Hg	Still Pressure, mm Hg	Still Temp, C	Still ΔT, C
1	25	75	144	36
2	50	100	152	28
3	75	125	159	21
4	100	150	165	15

Case-9

In a batch azeotropic distillation of isopropanol, with cyclohexane entrainer, water layer did not form in the phase separator; hence water could not be removed. As a result, most of the water remained in the feed and isopropanol could not be dehydrated.

Laboratory analysis showed that the feed contained small amounts of chloroform. This has a low boiling point and is also hydrophobic. Therefore, it rose to column top and accumulated in the organic layer. Normal density of organic layer is lower; hence it forms an upper layer in the separator. But, now, since its density was higher, overall density of the organic layer increased until it became almost the same as water layer. Separation of the two layers depends on density difference between them. Since this had become negligible, the two layers could not separate, and remained intermixed.

The feed was pre-distilled to remove chloroform. Then, chloroform-free feed was distilled with cyclohexane entrainer to remove water.

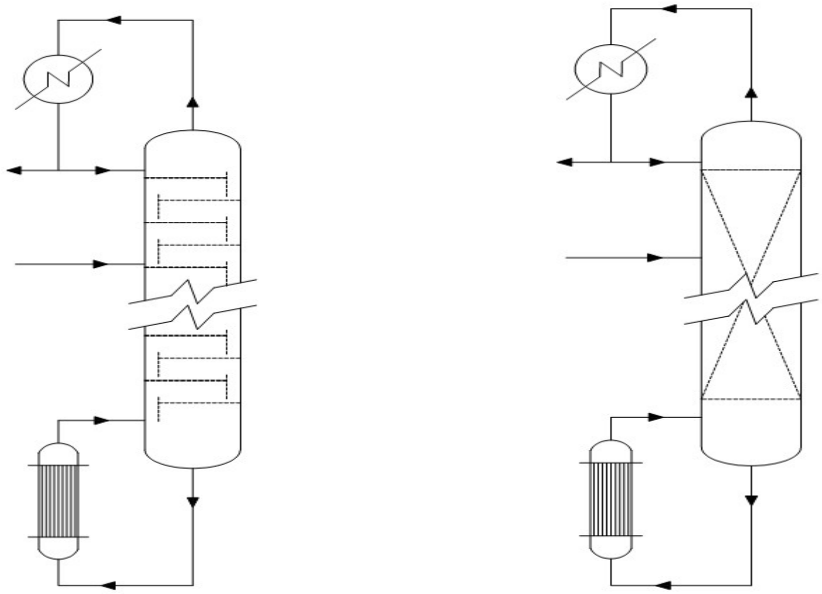
Figure (13): Illustration of interference in phase separation

Case-10

A bubble-cap tray column was being used for vacuum distillation of isomers. Vaporisation was inadequate and separation could not be achieved.

Bubble-cap tray column had high pressure drop. This resulted in a high column bottom pressure and, hence, high temperature. The lower temperature difference in the reboiler resulted in lower vaporisation.

The tray column was replaced with a structured packing column, which has a much lower pressure drop. Column bottom pressure and temperature was substantially decreased, which permitted proper vaporisation.



No. of trays: 40	Packed height: 8 mts
Pressure drop per tray: 3 mm Hg	Pressure drop per meter: 1 mm Hg
Total pressure drop: 120 mm Hg	Total pressure drop: 8 mm Hg
Column top pressure: 50 mm Hg	Column top pressure: 50 mm Hg
Column bottom pressure: 170 mm Hg	Column bottom pressure: 58 mm Hg
Column bottom temperature: 155 deg-C	**Column bottom temperature: 122 deg-C**

Figure (14): Illustration of effect of pressure drop

Case-11

A multicomponent feed was being distilled in a column heated by a thermosiphon reboiler. Vaporisation rate kept oscillating from a very high value to zero.

Because of certain design errors, recirculation rate in the reboiler was very low. In the beginning, when the reboiler tubes were full, low-boiling component evaporated away and left high boilers behind. As a result, reboiler fluid temperature increased, delta-T decreased, and evaporation rate came down to zero. At this stage, in the absence of any vapour, feed (containing low-boilers) dropped down to reboiler. The replenishment of low-boiler component in the reboiler, reduced reboiler fluid temperature, increased delta-T and evaporation started again.

Thermosiphon reboiler was replaced by a forced circulation reboiler that ensured circulation and homogeneity of reboiler fluid.

Case-12

In a Dimethyl Sulfoxide (DMSO)-Water batch distillation, vent condenser was getting choked towards the end of batch cycle. It was noted that the vent condenser was on chilled water, whose temperature was less than the melting point of DMSO (19 C), causing it to solidify.

Vent condenser was changed over to cooling water with higher temperature.

Case-13

There was an explosion while distilling a THF (Tetrahydrofuran)-water mixture, which had been lying around for some time. Further testing showed that THF had formed peroxides during storage, which had exploded.

The mixture was treated with ferrous sulphate to remove peroxides, and then stabilised with butylated Hydroxytoluene

Case-14

A continuous column was flooding, starting at a point just above the feed.

Column was designed for saturated liquid feed, but actual feed was superheated. The extra superheat in feed was converted to vapor by flashing, after feed entered the column. This increased the vapour load beyond design value, resulting in flooding.