



Distillation Technology and Modelling Techniques

Part 1: Concepts in Distillation

In this review of distillation technology and modelling techniques, Konrad Miller and Katherine Shing offer a broad survey of the science of distillation, including a conceptual framework of distillation; distillation column components and, in part two, shortcut distillation design methods such as the McCabe Thiele method and the Fenske Underwood Gilliland algorithm. In part three, Konrad Miller will apply these principles to the production processes of a modern brandy distillery.

What is distillation? Think back to your childhood, when you first observed a pan of boiling water: you watched water vapour rise from the liquid surface at a temperature of approximately 100°C (212°F). If you were really curious, you might have turned the power up and noticed the liquid boil more vigorously with a resulting increase in vapour flow. Now consider a pan containing a mixture of two fluids.

Throughout this discussion, we will be exploring the separation of ethanol and water, so imagine your parents were boiling wine for a sauce. Water boils at 100°C, but ethyl alcohol (the alcohol in the wine) boils at 78.4°C. The liquid no longer boiled at 100°C, but now somewhere in between these two limiting values. As the liquid boils, ethanol, with the lower boiling point, preferentially vaporises over water. The vapour leaving the pan will be richer in ethanol and the liquid remaining will be richer in water. This is the basis of distillation – and indeed how it was discovered.

Commercial distillations

Instead of the single-stage batch pot still described above, commercial distillations are typically performed in a continuous manner, with multiple pot still equivalents (or 'stages') used to increase separation power. Consider the set-up in Figure 1. Three pot stills are connected in a series of stages; where the vapour from one stage rises and intermingles with the liquid in the stage above it, and liquid from a stage drains down to intermingle with the vapour in the stage below it.

Since a series of connected pot stills is an inefficient and needlessly complex design, distillation columns are used industrially to achieve the same results. The internals of a distillation column are comprised of a series of 'plates' or 'trays', each of which allows for the thorough mixing of vapour rising from a stage below and liquid falling from a stage above. This continuous contact promotes mass transfer between the phases and the equilibrium between the liquid and vapour leaving each stage acts in exactly the same manner as the series of pot stills idea shown in Figure 1. Figure 2

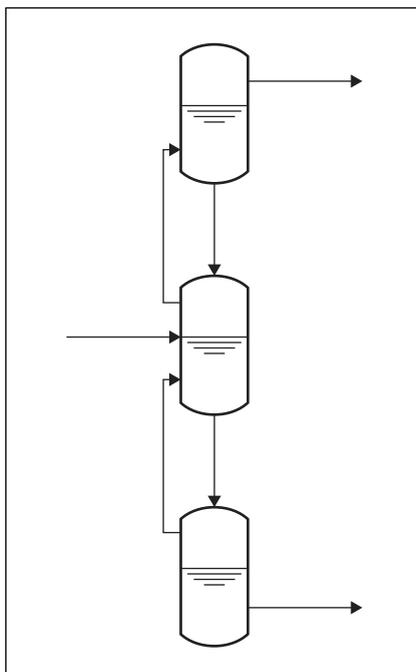


Figure 1: Multiple pot stills operating together as a distillation column. Note the feed and product lines, distillation columns are a continuous, steady-state process

illustrates the vapour and liquid paths along the trays in a distillation column:

Relative volatility

Before we approach the internal workings of a distillation column in more detail, let us flesh out our notion of distillation and discuss the motivation for learning more. Distillation can be applied to separate liquid mixtures whenever the 'relative volatility' of the species to be separated is greater than 1.

Relative volatility (α_{ij}) of species i (more volatile) and j (less volatile) is defined as the ratio of the volatility (k_i) of species i and j , where $k_i \equiv y_i/x_i$, where y_i and x_i are the mole fractions of species i in the vapour and liquid phases, respectively. These values are calculated from thermodynamic equations outside the scope of this paper, but are strong functions of temperature, pressure, and composition.

The relative volatility is an expression of how well two species split when vaporised: a high relative volatility indicates that there is a significant enrichment upon vaporisation, meaning the vapour phase contains more of component i than the liquid phase. A relative volatility of 1 indicates that no separation is possible; the volatility of each component is the same. Pure component boiling points are often used as a proxy for relative volatility, i.e., low boiling species are more volatile than high boiling point species. While this is often true (and a handy

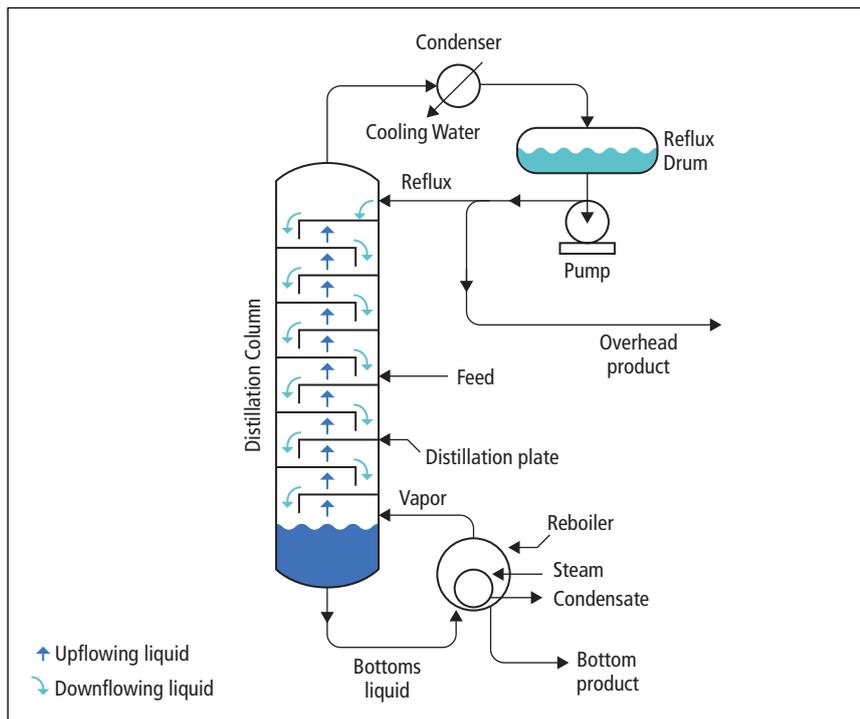


Figure 2: Vapour and liquid paths along trays in a distillation column. Note the column itself is divided into two sections: Rectifying (above feed) and Stripping (below)

conceptual shortcut for thermodynamically well behaved mixtures), there are many instances where a species with a higher boiling point is more volatile than a lower boiling point species.

In binary distillation (such as ethanol and water for spirits making), the 'split' of components is obvious: the lighter species (ethanol) partitions into the vapour top product known as distillate, and the heavier species (water) partitions into the liquid bottom product known as bottoms. The situation is more complex for multicomponent distillation. Separations are usually specified between two 'key' components: a 'Light Key' (LK), and a 'Heavy Key' (HK). The LK is the least volatile component that is enriched in the distillate, or the 'heaviest of the lights'; the HK is the most volatile component that is enriched in the bottoms, or the 'lightest of the heavies'. This conceptualisation allows us to think of a multicomponent distillation as a pseudo-binary distillation between the LK and the HK. Table 1 provides an example of relative volatility values versus N-Butane for the alkane feed to a distillation column:

Robust and well understood

So why is distillation useful, and where is it applied? Distillation is one of the most robust and well understood separations technologies, with application to petroleum refining, commod-

Alkane Species	$\alpha_{i,n-C4}$
C2	250
C3	57
i-C4	2.4
n-C4	1
i-C5	0.4
n-C5	0.009

Table 1: Relative volatility for each species in an alkane feed: n-C4 as HK and i-C4 as LK. Note that relative volatility compared to n-C4 decreases with increasing carbon count

ity chemical purification, food/flavour production and of course distilled spirits. The completeness and rigor with which distillation is understood allows for the design and fabrication of full-scale distillation columns without the need to pilot separations on smaller columns first, as is often required for less-understood unit operations such as membrane separations, resin separations, crystallisation, etc.

Let us return to the components of a distillation apparatus. In broad terms, a distillation column is comprised of three major components:

1. The column

The column offers surface area for mass transfer between the boiling (bubble point) liquid streams falling down the column and the saturated (dew point) vapour traveling up the column. As the vapour and liquid make contact, the more volatile components are enriched in the vapour and the less volatile components are enriched

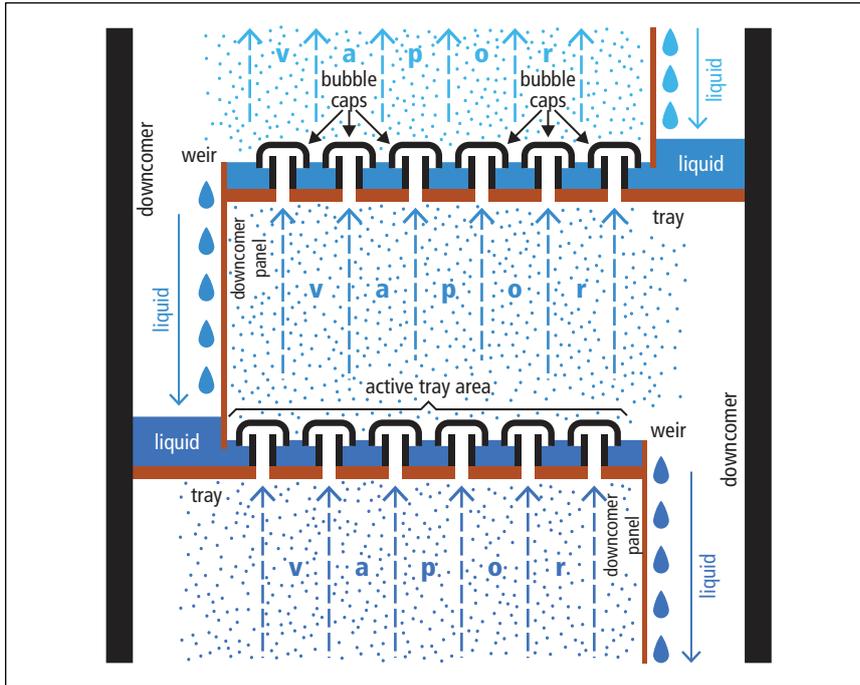


Figure 3: Bubble Cap plates in a column. Note the liquid and vapour paths along the column

in the liquid. Column internals can be divided into two categories:

Plates: A distillation plate (also known as a tray) is a flat disk which supports a liquid level. Vapour paths in the tray allow the vapour from the tray below to rise and bubble through the standing liquid. Trays come in three major types: Bubble Cap trays have very high efficiency, very high turndown, high pressure drop, and are expensive; Valve trays have good efficiency, good turndown, moderate pressure drop, and are moderately priced; and Sieve trays have low efficiency, low turndown, low pressure drop, are good for high solids, and are inexpensive.

All of the above trays are classified as 'crossflow' trays, where liquid falls

down from above to one side of the tray, then travels across the tray to fall onto the tray below. Weirs are installed next to the downcomers to ensure a minimum liquid level on the tray.

Packing: Packing is a more recent alternative to plates. The liquid/vapour contacting surface area is not broken into discrete trays but rather is comprised of a continuous geometry to promote constant exchange between the phases. Packing is used due to its low pressure drop and ability for high mass transfer efficiency vs. plates. Additionally, packing can be applied to newer technologies such as reactive distillation. Unlike a column, there are no 'stages' that act akin to a pot still. A distillation column with plates is typically specified by a number of trays, whereas packed columns are usually specified by 'HETP' (Height Equivalent to a Theoretical Plate) or 'HTU' (Height of a Transfer Unit) and 'NTU' (Number of Theoretical Units), e.g. a distillation column requiring 55 trays might require 55 NTUs with an HETP of 2 meters, for a total packing depth of 110 meters. Packing can be broadly divided into two categories: random packing and structured packing. An example of structured packing is shown in Figure 4.

2. The reboiler

The reboiler is analogous to the stovetop in our boiling pot example; it provides the heat to drive the separation in the column. The typical

reboiler is a large shell and tube heat exchanger with liquid bottoms on the shell side and steam on the tube side. Energy drawn from the steam to the bottoms liquid drives vaporisation, which provides the vapour upflow and heat to drive the separation. The most common reboiler is a 'thermosiphon' reboiler; fed from the standing pool of liquid at the bottom of the column. The reboiler is on the same level or below as the liquid reservoir at the bottom of the column, so head pressure pushes liquid from the bottom of the column to the reboiler. As the material in the reboiler flashes off, the level in the reboiler drops, and more liquid surges in, hence the siphoning action of this system.

Figure 5 depicts a typical thermosiphon reboiler. Other reboiler types include kettle reboilers where liquid is pumped into the heat exchanger, and direct fired reboilers where the reboiler is effectively a furnace and combustion drives the distillation. Material leaving the reboiler is typically known as the 'boilup', which enables the vapour-liquid equilibrium along the height of the column.

3. The condenser

The condenser is the mirror image of the reboiler. The reboiler drives vapour up the column while the condenser drives liquid down the column. If the feed to the column is liquid (as is typical), then liquid falls down the trays below the feed point to enable vapour-liquid contact, but no liquid is available above the feed point. The condenser addresses this by returning an often substantial portion of the condensed vapours as 'reflux'. Reflux is as critical to the operation of the distillation column as boil-up. The condenser itself is typically a shell and tube heat-exchanger with cooling water (or some other coolant) in the tubes and condensing vapours in the shell. The condenser may be above the column with gravity draining reflux back onto the trays, or it may be below the column with reflux pumped back to the top. Figure 6 shows a typical condenser.

Putting all these pieces together, Figure 7 describes the overall action of a distillation column. With a conceptual understanding of the mechanics of distillation we can, in part 2 of this article, review methods for modelling the behaviour of a distillation column.



Figure 4: Structured packing segments prior to installation. Note the complex packing pattern, which maximises contact area

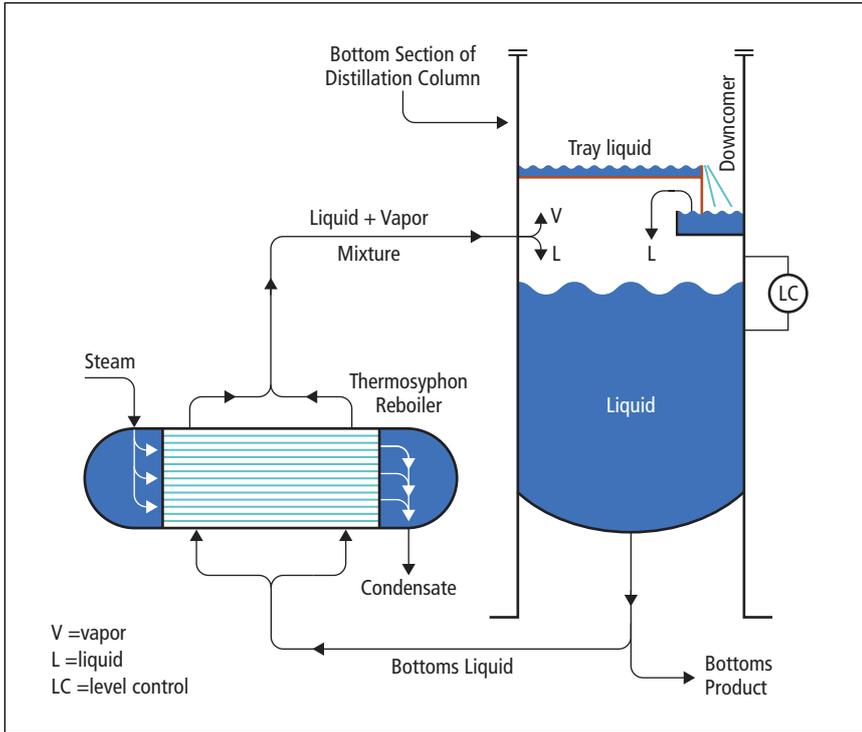


Figure 5: Sketch of a thermosiphon reboiler. Note the liquid inlet at the bottom of the column, and the vapour is above the column liquid level. The vapour is discharged below the bottom stage, and provides the upflow which drives separation

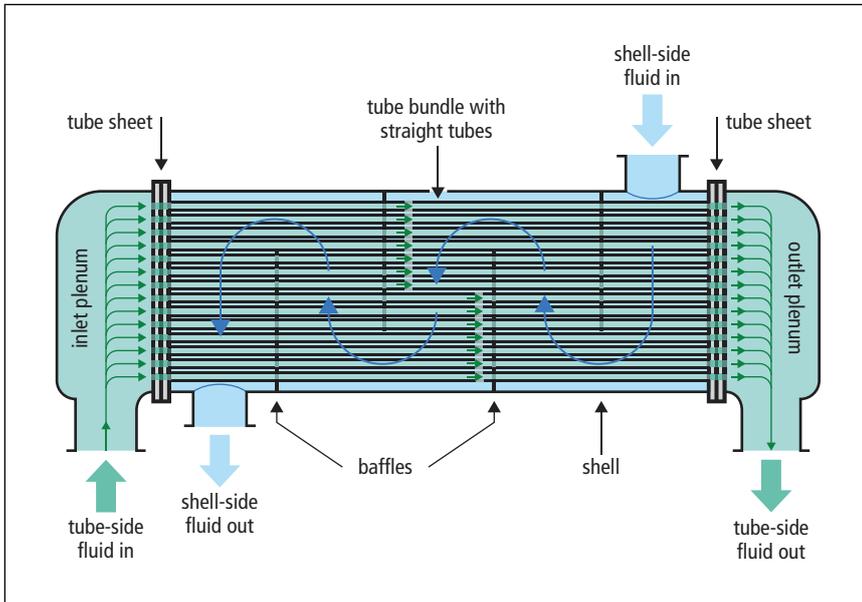


Figure 6: A shell and tube exchanger. Most distillation condensers are in this configuration

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Konrad Miller is a process engineer from the United States. A licensed professional engineer, he graduated from the University of California, Berkeley with a BS in Chemical Engineering and from the University of Southern California with an MS in Chemical Engineering. His work as a process engineer in the alcoholic beverage industry has included process design, plant simulation, fermentation, and distillation. Katherine Shing is a Professor of Chemical Engineering at the University

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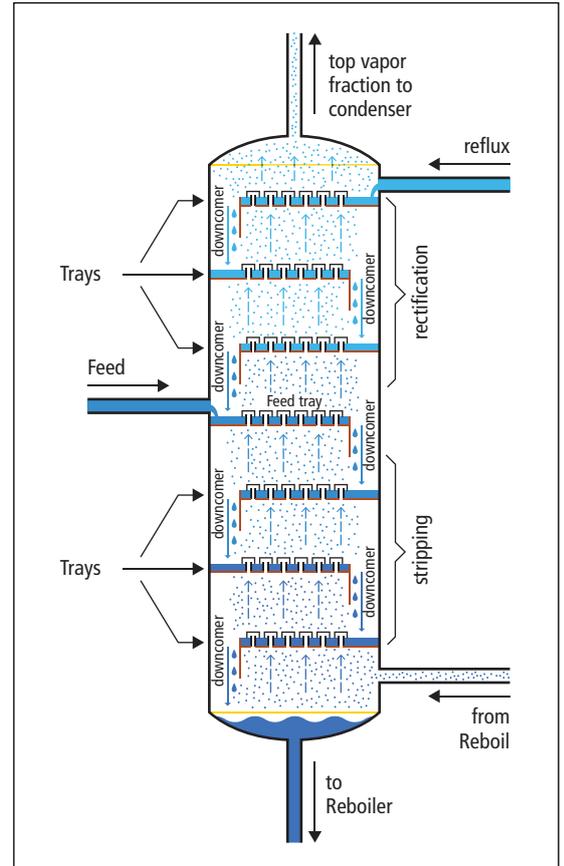


Figure 7: Major flows and components of a tray column

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