

Chapter 18

Ethanol distillation: the fundamentals

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Fundamentals of a distilling system

Certain fundamental principles are common to all distilling systems. Modern distillation systems are multi-stage, continuous, countercurrent, vapor-liquid contacting systems that operate within the physical laws that state that different materials boil at different temperatures.

Represented in Figure 1 is a typical distillation tower that could be employed to separate an ideal mixture. Such a system would contain the following elements:

- a. a feed composed of the two components to be separated,
- b. a source of energy to drive the process (in most cases, this energy source is steam, either directly entering the base of the tower or transferring its energy to the tower contents through an indirect heat exchanger called a reboiler),
- c. an overhead, purified product consisting primarily of the feed component with the lower boiling point,
- d. a bottoms product containing the component of the feed possessing the higher boiling point,
- e. an overhead heat exchanger (condenser), normally water-cooled, to condense the vapor

resulting from the boiling created by the energy input. The overhead vapor, after condensation, is split into two streams. One stream is the overhead product; the other is the reflux which is returned to the top of the tower to supply the liquid downflow required in the upper portion of the tower.

The portion of the tower above the feed entry point is defined as the 'rectifying section' of the tower. The part of the tower below the feed entry point is referred to as the 'stripping section' of the tower.

The system shown in Figure 1 is typical for the separation of a two component feed consisting of ideal, or nearly ideal, components into a relatively pure, overhead product containing the lower boiling component and a bottoms product containing primarily the higher boiling component of the original feed.

If energy was cheap and the ethanol-water system was ideal, then this rather simple distillation system would suffice for the separation of the beer feed into a relatively pure ethanol overhead product and a bottoms product of stillage, cleanly stripped of its ethanol content. Unfortunately, the ethanol-water (beer) mixture is not an ideal system. The balance of

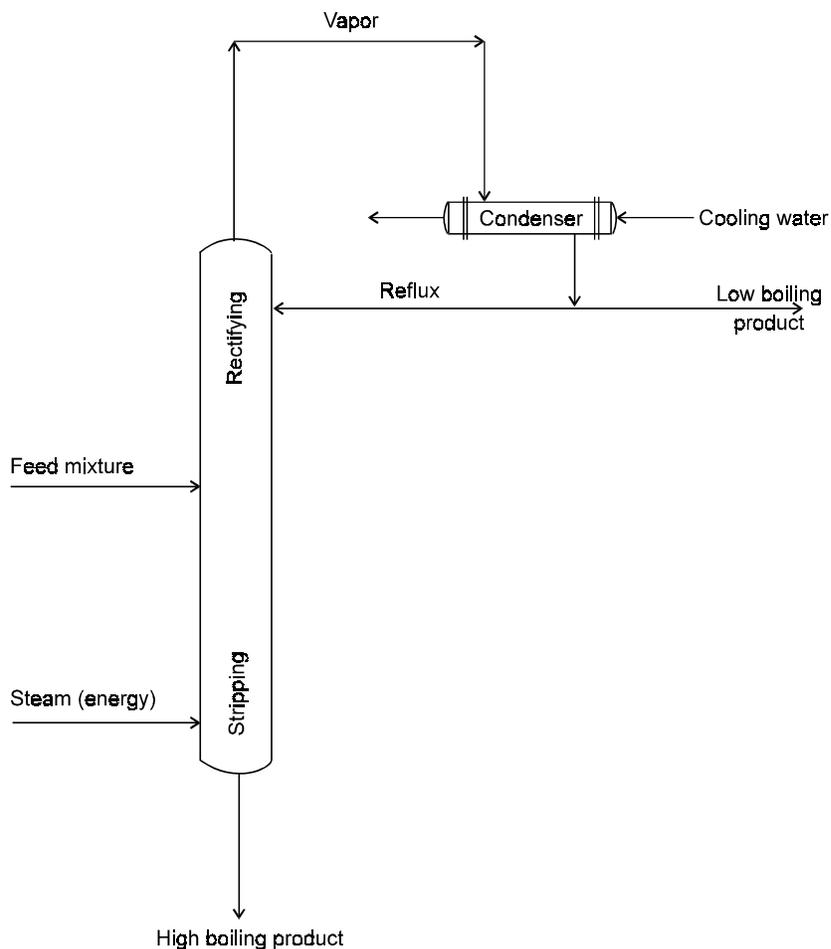


Figure 1. Ideal distillation system.

this chapter will be devoted to a description of the modifications required of the simple distillation system in order to make it effective for the separation of a very pure ethanol product, essentially free of its water content.

Figure 2 expands on Figure 1 by showing some additional features of a distillation tower. These are:

- The highest temperature in the tower will occur at the base.
- The temperature in the tower will regularly and progressively decrease from the bottom to the top of the tower.
- The tower will have a number of similar, individual, internal components referred to as

'trays' (these may also be described as stages or contactors).

- Vapor will rise up the tower and liquid will flow down the tower. The purpose of the tower internals (trays) is to allow intimate contact between rising vapors and descending liquids correlated separation of vapor and liquid.

Figure 3 shows a vapor-liquid equilibrium diagram for the ethanol-water system at atmospheric pressure. The diagram shows mole percent ethanol in the liquid (X axis) vs mole percent ethanol in the vapor (Y axis). The plot could also be made for volume percent in the liquid vs volume percent in the vapor and the equilibrium

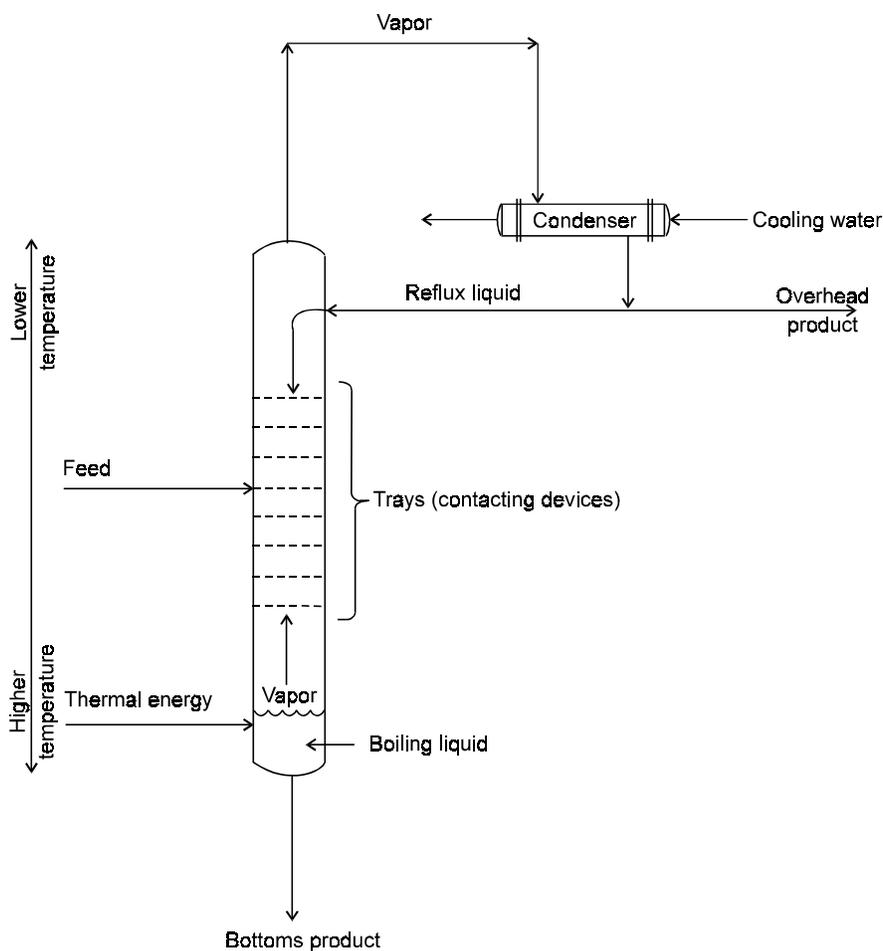


Figure 2. Typical distillation relationships.

curve would only be slightly displaced from that shown in Figure 3. Mole percent is generally used by engineers to analyze vapor/liquid separation systems because it relates directly to molecular interactions, which more closely describe the process occurring in a distillation system.

Analysis of the ethanol-water distillation system is mathematically straightforward when using molar quantities rather than the more common measurements of volume or weight. This is because of an energy balance principle called 'constant molal overflow'. Essentially, this principle states that the heat (energy) required to vaporize or condense a mole of ethanol is approximately equal to the heat (energy) required to vaporize or condense a mole of

water; and is approximately equal to the heat (energy) required to vaporize or condense any mixture of the two. This relationship allows the tower to be analyzed by graphic techniques using straight lines. If constant molal overflow did not occur, then the tower analysis would become quite complex and would not lend itself easily to graphic analysis.

Referring to Figure 3, a 45° line is drawn from the compositions of the 0, 0-100% and 100%. This 45° line is useful for determining ranges of compositions that can be separated by distillation. Since the 45° line represents the potential points at which the concentration in the vapor equals the concentration in the liquid; it indicates those conditions under which distillation is

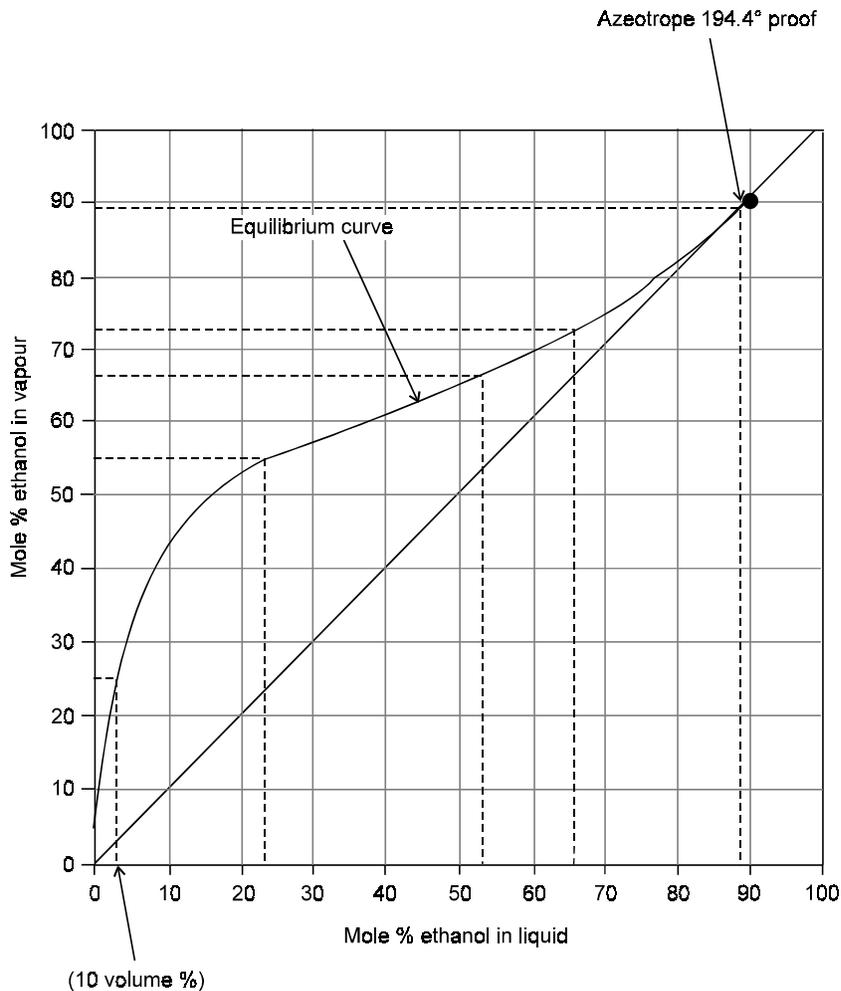


Figure 3. Vapor/liquid equilibrium for the ethanol/water system at atmospheric pressure.

impossible for performing the separation. If the equilibrium curve contacts the 45° line, an infinitely large distillation tower would be required to distill to that composition of vapor and liquid. Further, if the equilibrium curve crosses the 45° line, the mixture has formed an azeotrope. This means that even if the tower were infinitely large with an infinite amount of energy, it would be impossible to distill past that point by simple rectification.

Consider a very simple system consisting of a pot filled with a mixture of ethanol in water (a beer) containing 10 % by volume ethanol (3.3 mole %). This composition is identified in the

lower left portion of Figure 3. A fire could be kindled under the pot, which would add thermal energy to the system. The pot would begin to boil and generate some vapor. If we gathered a small portion of the vapor initially generated and measured its ethanol content; we would find about 24 mole % ethanol (53 volume %). If we condense this vapor (note: there will be only a small amount of this vapor), boil it in a second pot and again collect a small amount of the first vapor generated, this second vapor would contain about 55 mole % (83 volume %) of ethanol (see Figure 3). If we should continue this simplified process to a third and fourth

collection of small amounts of vapor; analysis would reveal that each successive portion of vapor would become richer in ethanol.

Thus we have created a series of steps by which we kept increasing the ethanol content of the analyzed sample, both liquid and vapor. Unfortunately, this oversimplified process is idealized; and practically speaking, is impossible. However if we had supplied our original pot with a continuous supply of ethanol-water feed and vapor generated in the first pot was continuously condensed and supplied to the second pot, etc. then the process becomes similar to the industrial distillation tower operation shown in Figure 2.

How far can this process be extended? Could we produce pure ethanol by continuously extending our process of boiling and reboiling? The answer is, no! We would finally reach a point in one of the downstream pots, where the vapor boiling off of the liquid was of the same composition as the liquid from which it was being generated. This unfortunate consequence limits our ability to produce anhydrous ethanol from a dilute ethanol-water feed. What we finally encounter in our simplified process is the formation of an 'azeotrope'. This is a concentrated solution of ethanol and water that when boiled produces a vapor with a composition identical to the composition of the liquid solution from which it originated.

In summary then, we are limited in ethanol-water purification in any single multistage distillation tower to the production of azeotropic ethanol-water mixtures. These azeotropic solutions of ethanol and water are also known as constant boiling mixtures (CBM) since the azeotropic liquid will have the same temperature as the azeotropic equilibrium vapor being boiled from itself. Without some sort of drastic process intervention, further ethanol purification becomes impossible. The question then becomes: What can we do to make it possible to produce anhydrous ethanol? Methods of doing so will be covered later in this chapter.

Figure 4 depicts the structure of the distillation process by dividing the vapor/liquid equilibrium information into three distinct zones of process and equipment requirements: stripping, rectifying

and dehydration. This division is the basis for the design of equipment and systems to perform the distillation tasks.

Considerations in preliminary design

The engineer, given the assignment of designing a distillation tower, is faced with a number of fundamental considerations. These include:

- What sort of contacting device should be employed? (e.g. trays or packing). If trays are chosen, what type will give the most intimate contact of vapor and liquid?
- How much vapor is needed? How much liquid reflux is required? (What ratio of liquid: vapor is required?)
- How much steam (energy) will be required?
- What are the general dimensions of the distillation tower?

Distillation contactors

Trays are the most common contactor in use. What are the functions expected of tray contactors in the tower? Figure 5 depicts a single tray contactor in a distillation tower and shows the primary functions desired:

- Mixing rising vapor with a falling fluid.
- Allow for separation after mixing.
- Provide path for liquid to proceed down the tower.
- Provide path for liquid to proceed up the tower.

Figure 6 depicts a perforated tray contactor with certain accoutrements required to control the flow of liquid and vapor and to assure their intimate contact. Another type of tray contacting device, the disc-and-donut or baffle tray is shown in Figure 7. The characteristics of this type of contactor make it especially useful for distilling materials such as dry-milled grain beer, which would foul ordinary trays.

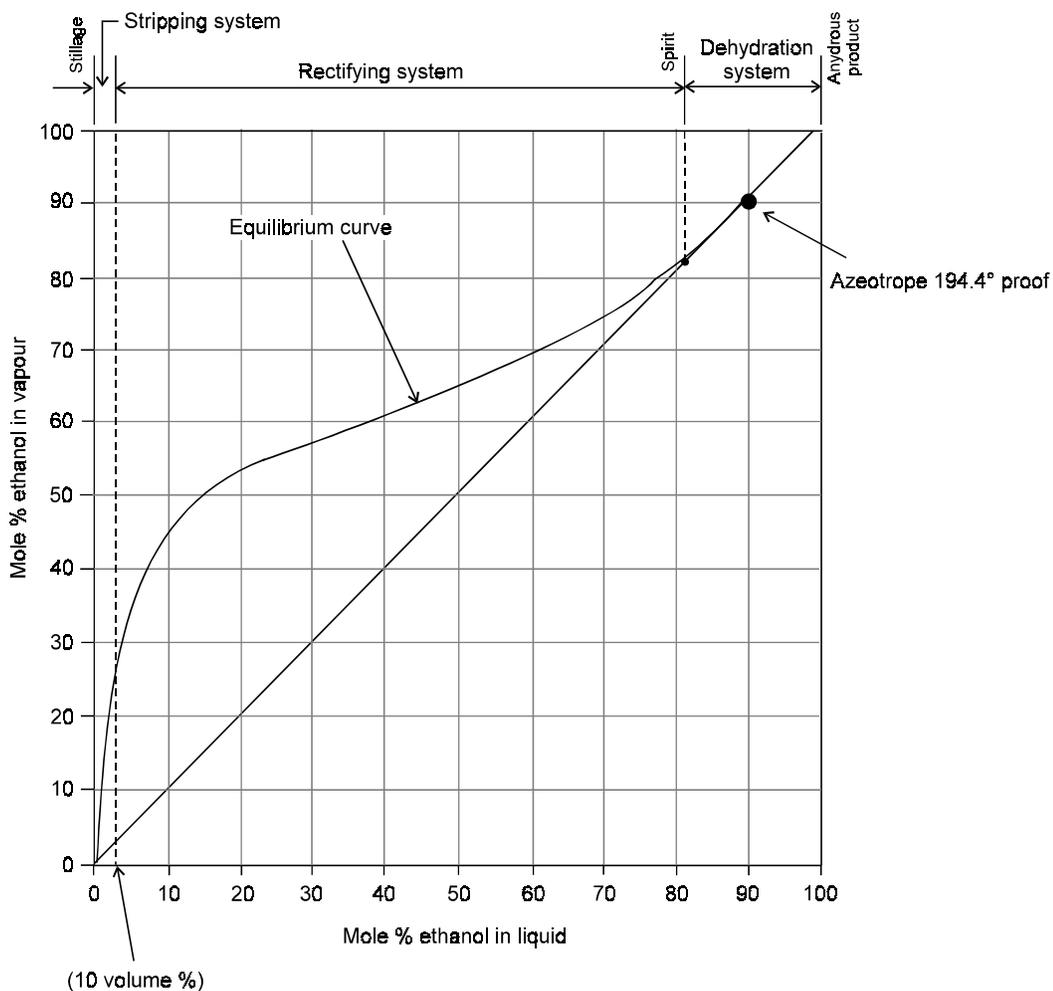


Figure 4. Structuring the distillation system strategy.

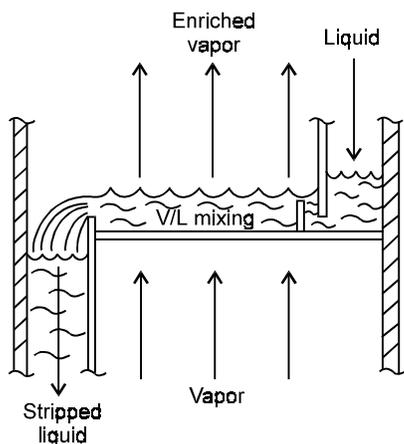


Figure 5. Distillation tray functions.

Energy analysis

In addition to the selection of the basic contacting device, the energy requirement must be established. This is accomplished by analyzing the vapor/liquid equilibrium data from Figure 4, for the liquid:vapor ratio to perform a continuous series of steps within the limits of the equilibrium curve. Table 1 demonstrates a simplified procedure to calculate the approximate energy requirement from the liquid:vapor ratio that will be employed in the tower design. Repetition of this type of calculation for different conditions produces a design chart like that shown in Figure 8 for the ethanol-water system. Such a graph is

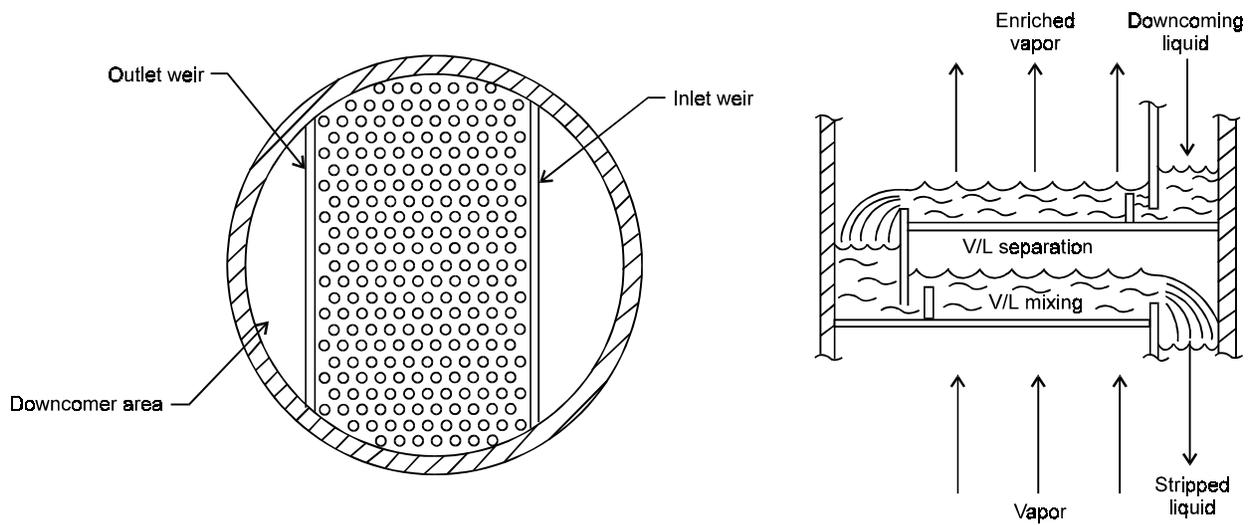


Figure 6. Perforated trays.

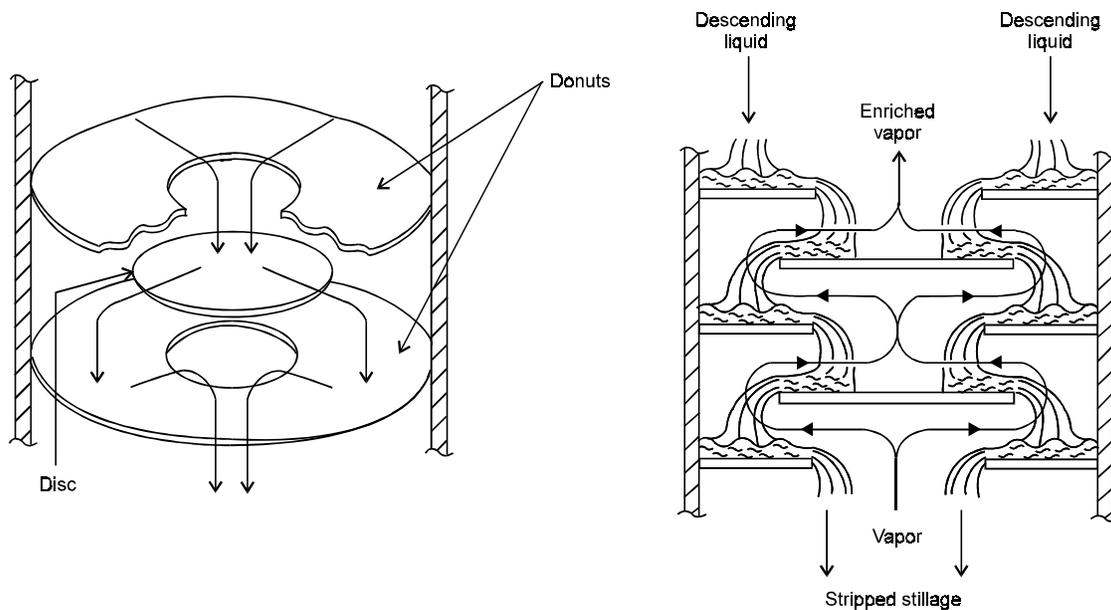


Figure 7. Disc-and-donut trays.

useful when calculations are needed to ascertain technical and economic feasibility and preliminary conditions for the design.

Figure 9 demonstrates how the liquid:vapor ratio, in connection with the number of stages (theoretically ideal trays) required for a specified separation between ethanol and water, is graphically determined. Note that the stages are constructed by drawing straight lines vertically

and horizontally between the equilibrium curve (previously determined experimentally) and the operating lines. For an ethanol stripper/rectifier, there are two operating lines: one for the rectification section and one for the stripping section. The operating lines represent the locus of concentrations within the distillation tower of the passing liquid and vapor streams. The operating lines for a given tower are based on

Table 1. Simplified calculations for steam requirements for ethanol distillation.

Example 1. Calculate the steam requirement (lbs/gallon of product) for a 10% volume beer at 100 gpm (90 gpm water/10 gpm ethanol).

$$L/V^* = 5.0 \text{ (typical for a 10\% volume beer) or } L = 5 \times V$$

$$L = 90 \text{ gpm} \times \frac{500 \text{ lbs/hr}}{\text{gpm}} = 45,000 \text{ lbs/hr} = 5 \times V$$

Therefore, $V = 9,000 \text{ lbs/hr}$ (steam)

$$\text{And: } \frac{9,000 \text{ lbs/hr (steam)}}{10 \text{ gpm}} \times \frac{\text{hr}}{60 \text{ min}} = 15 \text{ lbs steam/gallon of product}$$

Example 2. Calculate the steam required for a 5% volume beer at 100 gpm (95 gpm water/5 gpm ethanol).

$$L/V = 6.33 \text{ (typical for a 5\% volume beer) or } L = 6.33 \times V$$

$$L = 95 \text{ gpm} \times \frac{500 \text{ lbs/hr}}{\text{gpm}} = 47,500 \text{ lbs/hr} = 6.33 \times V$$

Therefore, $V = 7,500 \text{ lbs/hr}$ (steam)

$$\text{And: } \frac{7,500 \text{ lbs/hr (steam)}}{5 \text{ gpm}} \times \frac{\text{hr}}{60 \text{ min}} = 25 \text{ lbs steam/gallon of product}$$

*L and V are liquid and vapor flow rates, respectively, expressed in lb-mole per hr.

Note: at base of column use simplifying assumption of water/steam. Therefore: $\frac{L \text{ (lb-mole/hr)}}{V \text{ (lb-mole/hr)}} = \frac{L \text{ (lbs/hr)}}{V \text{ (lbs/hr)}}$

the energy input, as calculated and represented in Figure 8. Because of the principle of constant molal overflow, the operating lines can be represented as straight lines. If constant molal overflow was not valid for the ethanol/water distillation, then these lines would be curved to represent the changing ratio of liquid flow to vapor flow (in molar quantities) throughout the tower. The slope of the operating line (the ratio of liquid flow to vapor flow) is also called the internal reflux ratio. If the energy input to a tower is increased while the beer flow remains constant, the operating lines will move toward the 45° line, thus requiring fewer stages to conduct the distillation. Likewise if the energy input is reduced (lowering the internal reflux ratio), the operating lines will move toward the equilibrium curve, reducing the degree of separation achievable in each stage and therefore requiring more stages to conduct the distillation.

The calculations underlying the preparation of Figure 9 go beyond the scope and intent of this text, but have been included for continuity. The dashed lines represent the graphical solution to the design calculations for the number of theoretical stages required to accomplish a desired degree of separation of the feed components. Figure 9 is referred to as a McCabe-Thiele diagram. For further pursuit of this subject, refer to the classical distillation textbook by Robinson and Gilliland (1950).

Tower sizing

The goal of the design effort is to establish the size of the distillation tower required. Table 2 shows the basic procedure to determine the diameter required for the given distillation tower. Since all of the distillation 'work' is done by the

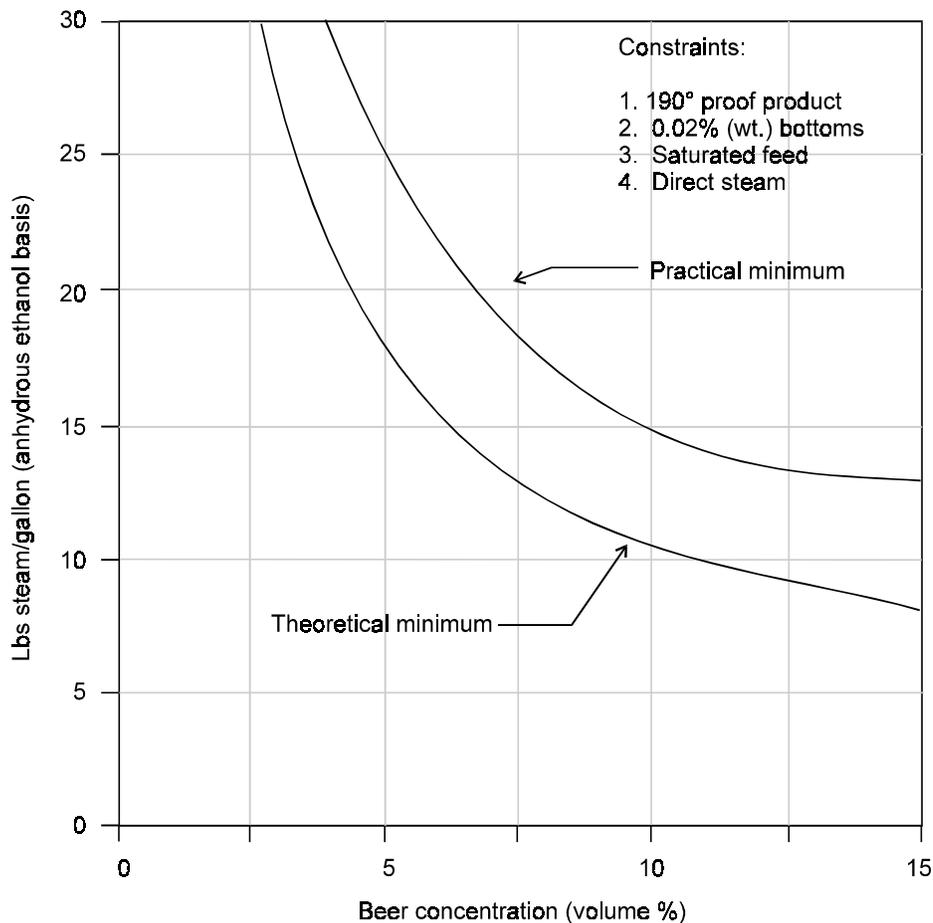


Figure 8. Steam requirements ethanol stripper/rectifier

trays, the tower is actually the 'container' to surround the vapor and liquid activity that is 'managed' by the trays. Tower diameter design is, therefore, actually the design of the necessary tray diameter for proper vapor/liquid interaction and movement.

The f factor (vapor loading) is an empirically determined factor that depends primarily upon tray type and spacing, fluid physical properties, froth stability and surface tension at the operating conditions of the system. The proper values for f are determined by field observations. In summary then, the f factor can be described as an adjusted velocity term (units are ft/sec) that when multiplied by the square root of the density ratio of liquid to vapor, will give the allowable vapor velocity in the empty tower shell, such that

liquid entrainment and/or vapor phase pressure drop in the tower will not be excessive.

Excessive vapor velocity will first manifest itself by causing excessive liquid entrainment rising up the tower, causing loss of separation efficiency. Ultimately the excessive entrainment and pressure drop will cause tower flooding.

To achieve a well-balanced tower design, the foregoing analysis must be performed at each stage of the tower, from bottom to top. Composition changes, feed points, draws, etc., each can cause a different requirement. The tower must be examined to locate the limiting point.

Similar analyses, with empirically-observed performance coefficients, are applied to vapor passing through the trays and liquid, and to the

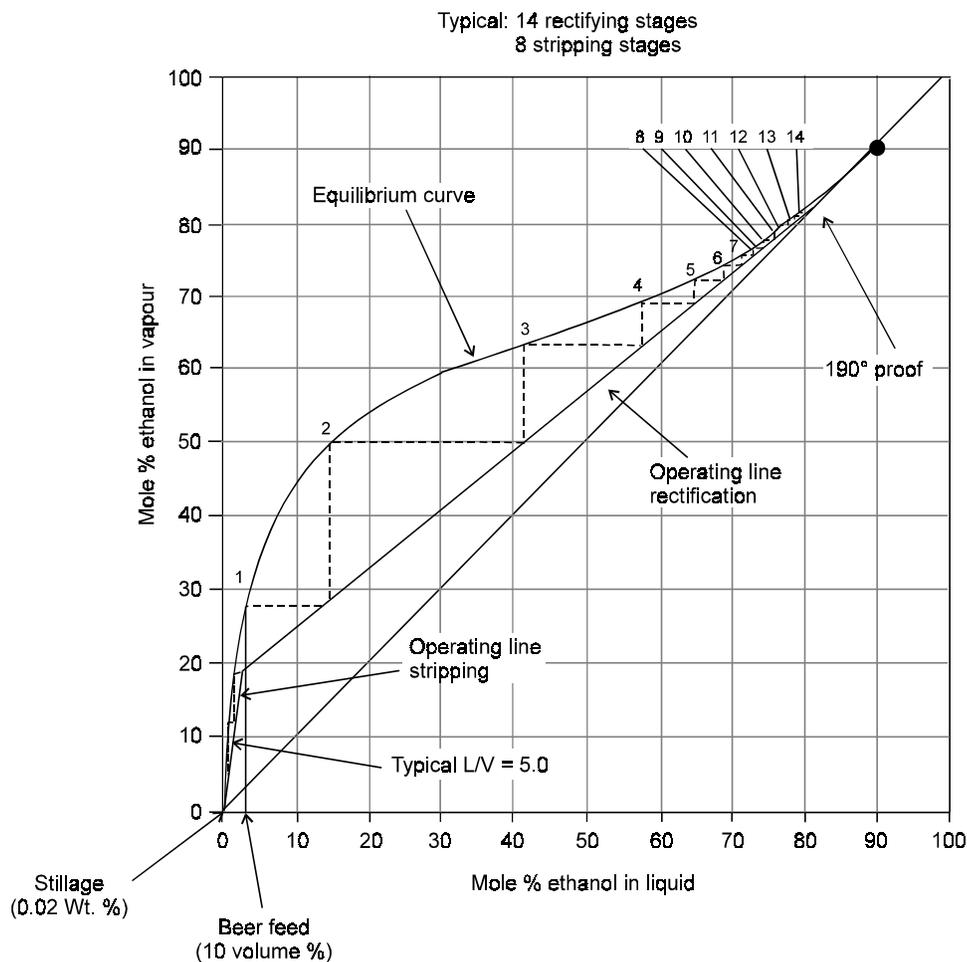


Figure 9. Vapor/liquid equilibrium stage analysis.

movement and control of liquid passing through downcomers and across the trays. These analytical procedures are beyond the scope of this text. Reference should be made to the aforementioned text by Robinson and Gilliland for further information.

Considerations in optimizing distillation system design

Optimizing the technical and economic design of distillation equipment and similar gas and vapor/liquid mass transfer systems involves a number of interrelated parameters. The positive/negative balance of a variety of contacting devices with different capacities and efficiencies for promoting vapor/liquid mass transfer must be taken into consideration. Along with the

technical issues considered in such designs, economical operation is essential not only in the reduction of energy and other direct costs, but also in relation to investment and return on investment from the operation being considered. In this respect, distillation towers are not independent process-wise, as consideration must also be given to other auxiliaries such as reboilers, condensers, pumps, controls and related equipment.

Sizing towers

In determining optimum diameter and height of towers for distillation, absorption, stripping and similar mass transfer operations, design factors are affected by whether the installations will be

Table 2. Calculations for tower sizing (base of stripper).**Example 1. Calculate tower diameter required for a 10% volume beer at 100 gpm (15 lbs steam/gallon of product)**

W (Vapor flow rate)	= 9,000 lbs/hr (steam)
P (Operating pressure at base)	= 1.34 ATM
M _{AVG} (Average MW of vapor)	= 18 lbs/lb-mole
ρ _L (Liquid mixture density)	= 59.5 lbs/ft ³ (227 °F)
T (Absolute operating temperature)	= 687 °R
D (Tower inside diameter (inches))	
f (Vapor loading factor)	= 0.05-0.3

$$\text{Sizing equation: } D = 0.2085 \cdot \sqrt{\frac{W}{f \cdot \sqrt{\frac{P \cdot M_{AVG} \cdot \rho_L}{T}}}} = 0.2085 \cdot \sqrt{\frac{9000}{f \cdot \sqrt{\frac{1.34 \cdot 18 \cdot 59.5}{687}}}} = \frac{16.45}{\sqrt{f}}$$

Assuming $f = 0.16$ (specific to tray design and spacing), the tower diameter is:

$$D = \frac{16.45}{\sqrt{0.16}} = 41.125 \text{ inches}$$

Example 2 utilizes the following equation. Values for the terms are indicated in Example 1.

$$D = 0.2085 \cdot \sqrt{\frac{W}{f \cdot \sqrt{\frac{P \cdot M_{AVG} \cdot \rho_L}{T}}}} \quad (\text{Eq. 1})$$

The final design equation can be derived beginning with the fundamental equation:

$$u = f \cdot \sqrt{\frac{\rho_L - \rho_V}{\rho_V}} \quad \text{Where } u = \text{average vapor velocity in empty tower shell (ft/sec)} \quad (\text{Eq. 2})$$

ρ_L = liquid density (lbs/ft³)
 ρ_V = vapor density (lbs/ft³)
 f = tower vapor loading factor (ft/sec)

(Note: For most cases ρ_L is much greater than ρ_V , so that $\rho_L - \rho_V \approx \rho_L$. For example, water (steam) at 212°F and atmospheric pressure: $\rho_L = 59.8$ lbs/ft³ and $\rho_V = 0.0373$ lbs/ft³. Then $\rho_L - \rho_V = 59.7627$ lbs/ft³ ≈ 59.8 lbs/ft³, which results in a negligible 0.06% error.)

$$\text{Consequently, } u \approx f \cdot \sqrt{\frac{\rho_L}{\rho_V}} \quad (\text{Eq. 3})$$

Imposing the equation of continuity: $W = A \cdot \rho_V \cdot u$ or $u = W/A \cdot \rho_V$

Where A = column cross-section area (ft²)
 ρ_V = vapor density (lbs/ft³)
 u = average vapor velocity in empty tower shell (ft/sec)
 and W = vapor mass flow (lbs/sec)

Substituting for u in the equation above:

$$\frac{W}{A \cdot \rho_V} \approx f \cdot \sqrt{\frac{\rho_L}{\rho_V}} \quad \text{then} \quad \frac{W}{A} \approx f \cdot \sqrt{\rho_L \cdot \rho_V} \quad \text{or} \quad A \approx f \cdot \sqrt{\frac{W}{\rho_L \cdot \rho_V}}$$

Use the Ideal Gas Law to express the vapor density.

$$\rho_v = \frac{P \cdot M_{AVG}}{R \cdot T} \quad \text{Then by substitution one obtains} \quad A = \frac{W}{f \cdot \sqrt{\frac{P \cdot M_{AVG} \cdot \rho_L}{R \cdot T}}}$$

Using the Universal Gas Constant $R = 0.73 \text{ (ft}^3\text{)(atm)/((lb-mole)(}^\circ\text{R)}$ the equation becomes:

$$A = \frac{W}{1.17 \cdot f \cdot \sqrt{\frac{P \cdot M_{AVG} \cdot \rho_L}{T}}} \quad \text{Now} \quad A = \frac{\pi \cdot D^2}{4} = 0.7854 \cdot D^2$$

$$\text{and} \quad D^2 = \frac{W}{0.9192 \cdot f \cdot \sqrt{\frac{P \cdot M_{AVG} \cdot \rho_L}{T}}} = \frac{1.0879 \cdot W}{f \cdot \sqrt{\frac{P \cdot M_{AVG} \cdot \rho_L}{T}}}$$

$$\text{Adjusting units: } D = \frac{1.043 \cdot 12}{60} \cdot \sqrt{\frac{W}{f \cdot \sqrt{\frac{P \cdot M_{AVG} \cdot \rho_L}{T}}}} \quad \text{Therefore, } D(\text{inches}) = 0.2085 \cdot \sqrt{\frac{W}{f \cdot \sqrt{\frac{P \cdot M_{AVG} \cdot \rho_L}{T}}}}$$

indoors or outdoors. With indoor installations, building height limitations, as well as floor level accessibility, are an important factor in the design. Where there are height limitations, towers must be increased in diameter to provide for reduced tray spacing, which in turn will require lower vapor velocities. With outdoor installations, the 'sky is literally the limit', and refinery and petrochemical towers of 200 feet in height are not uncommon.

In either case, indoors or outdoors, the interrelated tower diameter and tray spacing are limited by allowable entrainment factors (f factors) (Katzen, 1955). If outdoors, tower heights and diameters must be related to maximum wind loading factors in the specific plant location and may be complicated by allowance for earthquake factors.

Tray and packing selection

Vapor/liquid contacting devices may be of two distinct types, namely packed or tray (staged) towers. In packed towers, the transfer of material between phases occurs continuously and differentially between vapor and liquid throughout the packed section height. By contrast, in

tray towers, the vapor/liquid contact occurs on the individual trays by purposely interrupting down-flowing liquid using downcomers to conduct vapor-disengaged liquid from tray to tray and causing the vapor/liquid contact to occur between cross-flowing liquid on the tray with vapor flowing up through the tray. In other words, the vapor/liquid contact is intermittent from tray to tray, and is therefore referred to as being stagewise. Thus, for any given separation system, the degree of vapor/liquid contact will be greater with a greater height of the packed section, or in the case of tray towers, a greater number of trays used.

It is generally considered that packing-type internals may be used with relatively clean vapor and liquid systems where fouling is not a problem. Economics indicate that packing is applicable in small and modest sized towers. As the towers become larger, packing becomes complicated by the need for multiple liquid redistribution points to avoid potential vapor/liquid bypassing and reduction in efficiency. Structured packings (Fair *et al.*, 1990; Bravo *et al.*, 1985) are designed to minimize these problems by reducing the height requirement and controlling, to some extent, the distribution of liquid. However, high fabrication and specialized installation costs

would indicate that these are applicable only for relatively low volume, high value product processing.

Trays of various types are predominant in vapor/liquid contacting operations, particularly on the very large scale encountered in the petroleum and petrochemical industries, in large scale operations of the chemical process industries and in the large scale plants of the motor fuel grade ethanol industry.

The venerable bubble cap tray, with a wide variety of cap sizes, designs and arrangements to maximize contact efficiency, has fallen out of favor during the past few decades because of the relatively high cost of manufacture and assembly. Valve trays of several types have taken over in operations requiring a relatively wide vapor handling capacity range (turndown). This has been extended by use of different weights of valves on the same tray. Specialty trays such as the Ripple, Turbogrid, tunnel cap and others designed to improve contact under certain specific circumstances have been used to a limited extent.

The long established perforated tray is a contacting tray into which a large number of regularly oriented and spaced small circular openings have been drilled or punched. These trays are commonly referred to as 'sieve trays' because of the original practice of putting the maximum number of holes in any given tray area. This original design produced a fairly inefficient operation at normal loading, and a very inefficient operation with decreased vapor loading. About 50 years ago, engineers began to suspect that the design approach had been in error, and that the hole area in the trays should be limited by the hole velocity loading factor to obtain maximum contact by frothing, as indicated in Figure 10.

The hole velocity loading factor (or perforation factor) is defined as the vapor velocity through the perforations adjusted by the square root of the vapor density at the specific tower location of a given perforated tray. With the parallel development of separation processes in the petroleum refining, chemical and ethanol industries, the modern approach has developed

to what is now called 'perforated tray' design. The Fractionation Research Institute of the American Institute of Chemical Engineers diverted its efforts from bubble cap studies to perforated tray testing; and have established a basis for the design of perforated trays with high efficiency and wide capacity range (Raphael Katzen Associates, 1978).

Where foaming or tray fouling (caused by deposition of solid materials in the tower feed) can be an operational problem, novel designs such as the baffle tray may permit extended operating time between cleanings. Baffle trays may take a number of different forms. They can be as simple as appropriately spaced, unperforated, horizontal metal sheets covering as much as 50-70% of the tower cross sectional area; or they may take the form of a series of vertically spaced, alternating, solid disc-and-donut rings (see Figure 7). Towers up to 13ft in diameter are in operation using this simple disc-and-donut design concept.

Although system-specific data have been developed for each type of tray, it is difficult to correlate tray loading and efficiency data for a wide variety of trays on a quantitative basis. Each system must be evaluated based upon empirically-derived loading factors for vapor and liquid operations within the tower.

Auxiliaries

Energy input is of prime importance in tower design, particularly in ethanol stripping and rectification units. In aqueous and azeotrope-forming systems, direct steam injection has been common practice to maintain simplicity. However, current requirements to reduce the volume of waste going to pollution remediation facilities have minimized use of this simple steam injection technology to avoid the dilution effect of the steam being condensed and added to the stillage. Direct steam injection transfers both the energy and the water into the process. By imposing a heat exchanger (reboiler) between the steam and the process, only the energy is transferred into the tower. The condensate water is returned

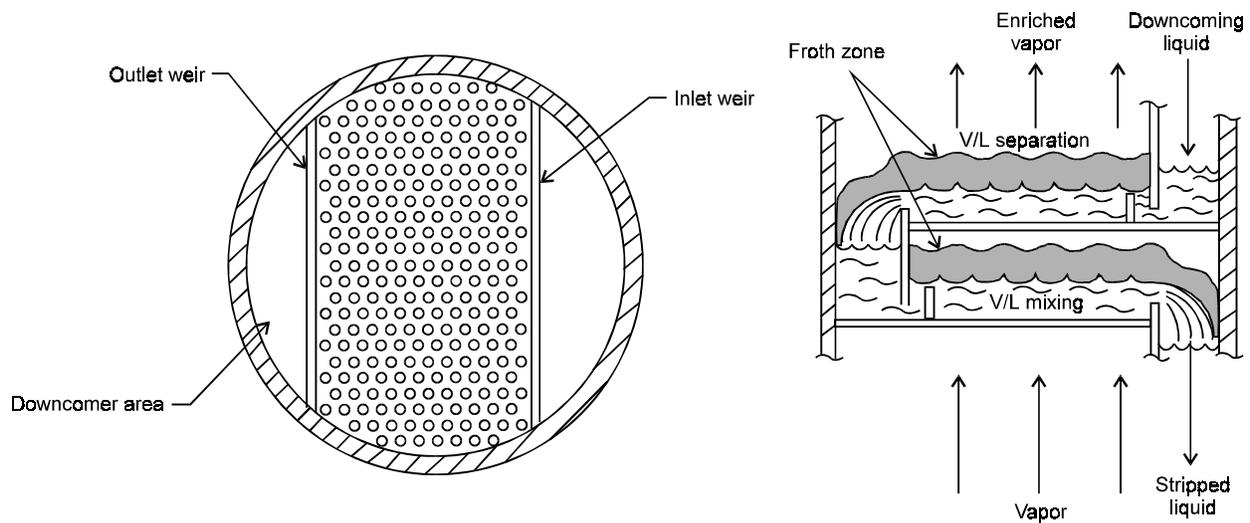


Figure 10. Perforated tray frothing.

in a closed-loop to the boiler, thus reducing the bottoms outflow from the process. Reboilers are thus growing in acceptance, and several types may be employed. Kettle and thermosyphon reboilers are preferred where fouling is not a problem. Where fouling can occur, high velocity, forced-circulation, flash heating reboilers are preferred. Figure 11 depicts the reboiler energy transfer by a forced-circulation reboiler as compared to Figure 1 which depicts direct steam injection.

Thermocompression injection of steam has also been utilized where low pressure vapors are produced from flash heat recovery installations and where higher pressure motive steam is also available.

Condenser design would appear to be simple. However, in many cases, water limitations require adapting condenser designs to the use of cooling tower water with limited temperature rise and minimal scale-forming tendencies. On the other hand, where water is extremely scarce, air-cooled condensers are used.

Energy conservation

The increasing cost of thermal energy, whether provided by natural gas, fuel oil, coal or biomass,

is fostering an increased emphasis on heat recovery and a reduction in primary thermal energy usage (Fair, 1977; Petterson *et al.*, 1977; Mix *et al.*, 1978). Conventional bottoms-to-feed heat exchangers are now being supplemented with recovery of overhead vapor latent heat by preheating feed streams and other intermediate process streams. Techniques of multistage distillation (similar to multiple effect evaporation) are also practiced. Pressure-to-atmospheric, atmospheric-to-vacuum, or pressure-to-vacuum tower stages are utilized, with the thermal energy passing overhead from one tower to provide the reboiler heat for the next one. Two such stages are quite common and three stage systems have also been utilized (Katzen, 1980; Lynn *et al.*, 1986).

Furthermore, the modern technique of vapor recompression, commonly used in evaporation systems, is also being applied to distillation systems. Such a system can provide for compression of overhead vapors to a pressure and temperature suitable for use in reboiling a lower pressure stripping tower. However, the compression ratios required for such heat recovery may consume almost as much electrical energy as would be saved in thermal input. Alternative systems, using vapor recompression as an intermediate stage device in the distillation system, have also been proposed.

Control systems

Control systems can vary from manual control, through simple pneumatic control loops to fully automated distributed control (Martin *et al.*, 1970). High level computer control has facilitated the application of sophisticated control algorithms, providing more flexibility, reduced labor and higher efficiency with lower capital investment. Such systems, when properly adapted to a good process design, have proven more user-friendly than the control techniques utilized in the past.

Economic design

In integrating the technology discussed, the final analysis must be economic. Alternative systems must be compared on the basis of investment requirements, recovery efficiency and relative costs of operation. Thus, any heat exchangers installed for heat recovery must show a satisfactory return on the investment involved in their purchase and installation. In comparing alternative separation systems, the overall equipment costs must be compared against energy and other operating costs to determine

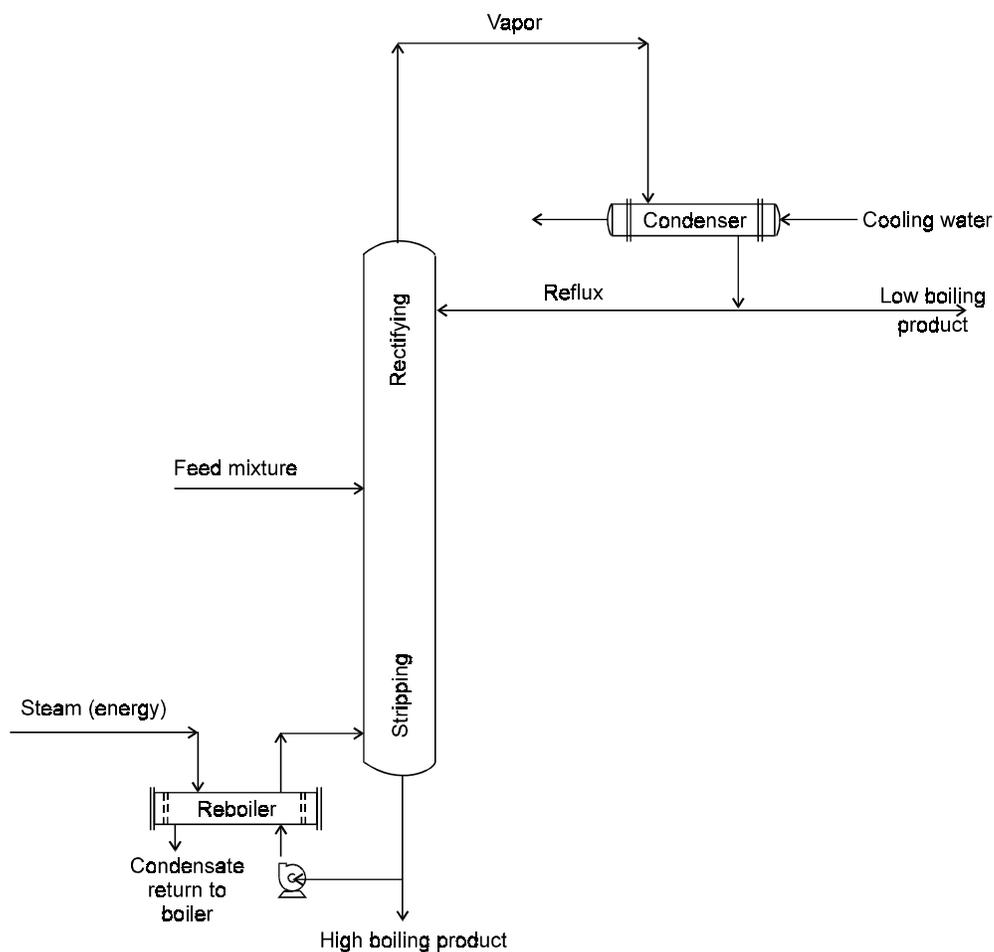


Figure 11. Energy transfer by a forced-circulation reboiler (See Figure 1 to compare with direct steam injection).

which system offers the best return. Modern computer-assisted designs incorporate economic evaluation factors so economic optimization can be determined rapidly.

Ethanol distillation/dehydration: specific systems technology

Proven industrial technologies are available for distillation of various grades of ethanol from grain, sugarcane, molasses and other feedstocks. Improvements have been made over the years, particularly during development of the motor fuel grade ethanol industry. In such installations, a key requirement is the mini-mization of total energy usage.

The operation that has been most subject to critical comment is the distillation process. Many relatively new 'authorities' in the field have based their criticism on technologies that go back 50-60 years, and have created an unwarranted condemnation of distillation as a viable process for low energy motor fuel grade ethanol production. Systems developed over the years will be described to show that much of such criticism is unwarranted and unjustified.

Production of industrial ethanol

Prior to the recent emphasis on motor fuel grade ethanol, the major ethanol product utilized worldwide was high purity, hydrous industrial ethanol, which is generally produced at a strength of 96° GL (192° US proof) (°GL = degree Gay Lussac = % by volume ethanol; US proof = 2 x % by volume ethanol). Efficient systems have been in commercial operation for many years for the production of such high grade ethanol from ethylene, grain, molasses and sulfite waste liquor. The basic distillation system is shown in Figure 12.

In the case of synthetic ethanol (outside the scope of this publication), the beer stripping tower is not required and the refining system is a simple three tower unit, which achieves 98% recovery of the ethanol in the crude feed as a

first grade product. The final product may contain less than 30 ppm total impurities and has a 'permanganate time' of more than 60 minutes.

For the production of industrial or beverage spirit products made by fermentation of grain, molasses or sulfite liquor, the system utilizes the full complement of equipment shown in Figure 12. The beer feed is preheated from the normal fermentation temperature in several stages, recovering low level and intermediate level heat from effluent streams and vapors in the process. This preheated beer is degassed and fed to the beer stripper, which has stripping trays below the beer feed point and several rectifying trays above it. The condensed high wines from the top of this tower are then fed to the extractive distillation tower, which may operate at a pressure in the order of 6-7 bars (87-101.5 psi). In this tower, most of the impurities are removed and carried overhead to be condensed as a low grade ethanol stream, from which a small purge of heads (acetaldehyde and other low boiling impurities) may be taken while the primary condensate flow is fed to the concentrating tower. The purified, diluted ethanol from the bottom of the extractive distillation tower is fed to the rectifying tower, which has an integral stripping section. In this tower, the high grade ethanol product, whether industrial or potable, is taken as a side draw from one of the upper trays. A small heads cut is removed from the overhead condensate. Fusel oils (mixtures of higher alcohols such as propyl, butyl, and amyl alcohols and their isomers, which are fermentation by-products or 'congeners') are drawn off at two points above the feed tray but below the product draw tray to avoid a buildup of fusel oil impurities in the rectifying tower. The overhead heads cut and the fusel oil draws are also sent to the concentrating tower.

It should be noted that the rectifying tower is heated by vapors from both the pressurized extractive distillation tower and the pressurized concentrating tower.

In the concentrating tower, the various streams of congener-containing draws are concentrated. A small heads draw is taken from

grade ethanol (99.5° GL) in four installations in Cuba (prior to the advent of the Castro regime). The dehydrating tower and the entrainer-recovery tower are operated at atmospheric pressure. Thus, they may utilize either low pressure steam, hot condensate or hot waste streams from other parts of the ethanol process to minimize steam usage. To simplify equipment and minimize investment, a common condensing-decating system is used for the two towers.

The entrainer used to remove water as a ternary (three component) azeotrope may be benzene, heptane (C₆-C₈ cut), cyclohexane, n-pentane, diethyl ether or other suitable azeotropic agents. The entrainer serves to create a three component azeotrope that boils at a temperature lower than any of the three individual components and lower than the ethanol/water binary (two component) azeotrope. Therefore the ternary mixture will pass overhead from the tower, carrying the water upward. Upon condensing, the mixture

separates in a decanter into an entrainer-rich layer and a water-rich layer.

The hydrous ethanol feed enters the dehydrating tower near the top. The feed contacts the entrainer in the upper section of the tower. The three component mixture in this section of the tower seeks to form its azeotrope, but is deficient in water and contains more ethanol than the azeotrope composition. Therefore, the ethanol is rejected downward in the liquid and is withdrawn as an anhydrous product from the bottom of the tower. The water joins the entrainer, passing upward as vapor to form a mixture that is near the azeotrope composition for the three components. The condensed mixture separates into two layers in the decanter and the entrainer-rich layer is refluxed from the decanter back to the top of the tower. The aqueous layer is pumped from the decanter to the entrainer-recovery tower, in which the entrainer and ethanol are concentrated overhead in the condenser-decanter system. The

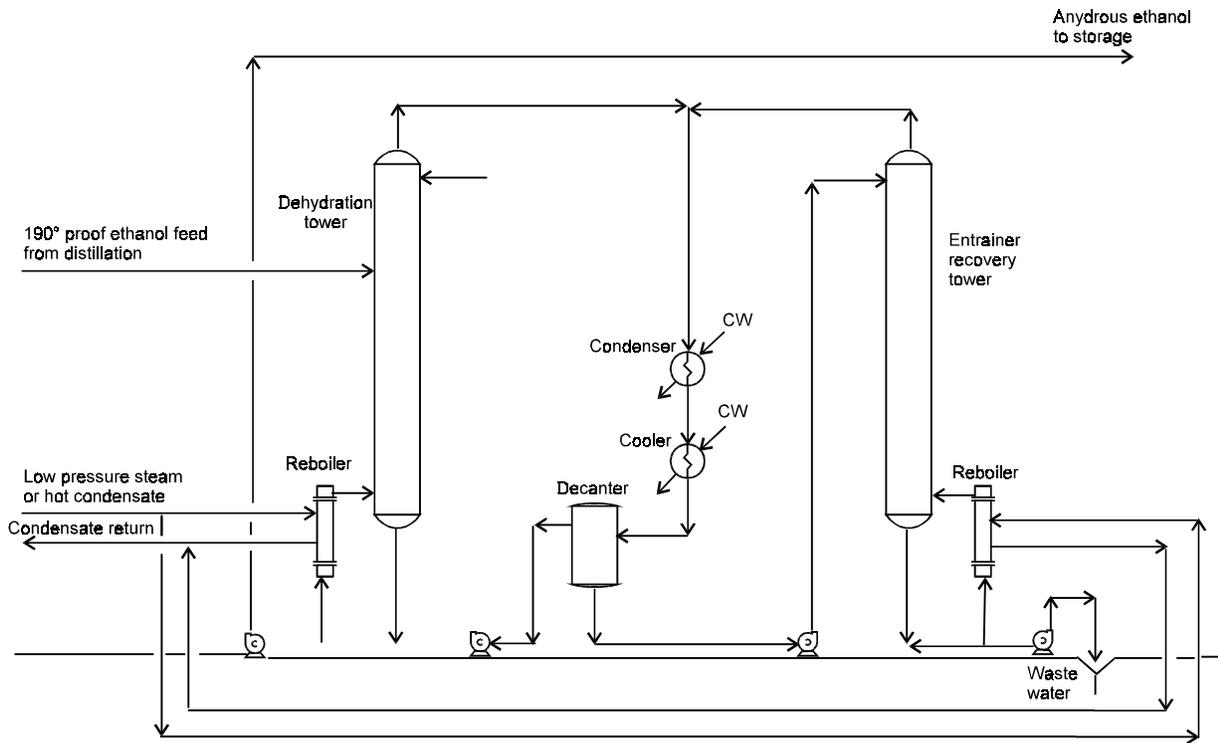


Figure 13. High grade anhydrous ethanol system.

stripped water, emerging from the base of the tower, may go to waste. If it has substantial ethanol content, it may be recycled to the beer well feeding the spirit unit, but this introduces the risk of traces of the entrainer in the hydrous ethanol which may not all be sent to the dehydration system. This system operates with a steam consumption of 1-1.5 kg/liter (8.3-12.5 lb/gallon) of anhydrous ethanol depending on the quality of product required. As indicated above, a major part of the equivalent steam energy can be provided by hot condensate and hot waste streams from the spirit unit.

Production of anhydrous motor fuel grade ethanol

In view of the growing demand for motor fuel grade ethanol (MFGE) in the US and other countries, a combination of key features in the improved systems described in Figures 12 and 13 has been used to maximize recovery of MFGE from fermented beer, while minimizing

energy consumption. This system is shown in Figure 14 (US patent 4,217,178; Canadian Patent 876,620, 1980). Fermented beer feed is preheated in a multistage heat exchange sequence, varying somewhat in complexity with the size of the commercial facility. In effect, beer is preheated in a 'boot strapping' operation, which takes the lower level heat from the azeotrope vapors in the dehydrating system and then picks up heat from the excess of overhead vapors from the pressurized beer stripping and rectifying tower. Finally the beer is preheated in exchange with hot stillage from the same tower. This preheated beer, essentially at its saturation temperature, is de-gassed to remove residual CO_2 before it enters the pressurized beer stripper. This tower operates at a pressure of approximately 4 bars with heat provided by steam through a forced circulation reboiler. This is the only use of steam in this distillation system. The stillage leaving the base of this tower is partially cooled to the atmospheric boiling point via heat exchange with the preheated beer feed. This provides a stillage feed for vapor recompression

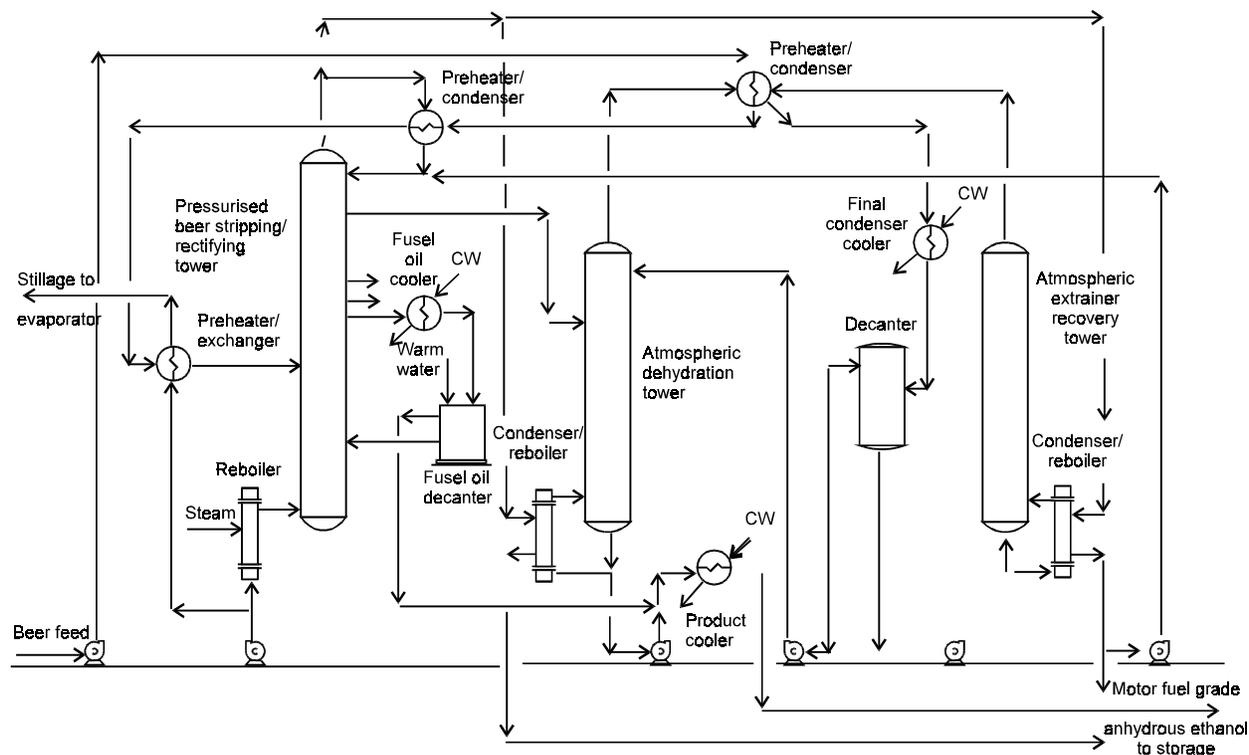


Figure 14. Motor fuel grade anhydrous ethanol system.

evaporation at an ideal temperature, requiring neither preheat nor flashing in the evaporator.

The ethanol stripped from the beer in the lower part of the beer tower is rectified to approximately 95° GL and taken as a side draw a few trays below the top of the rectifying section. The overhead vapors, under pressure, are used to boil up the atmospheric dehydration tower and the atmospheric entrainer recovery tower, as well as to provide preheat to the beer feed. The condensed overhead vapors are refluxed to the top of the pressurized beer tower, with a small draw of heads taken to avoid accumulations of the more volatile congeners such as acetaldehyde. The heads stream, amounting to less than 1% of ethanol production, can be burned as fuel in the plant boiler or sent directly into the MFGE final product, thus bypassing the dehydration system.

Side stream fusel oil draws are also taken from the rectifying section of the pressurized tower to a fusel oil decanter. The aqueous washings are returned to the beer stripping section of this tower; while the decanted, washed fusel oil is combined with the anhydrous ethanol plant. Fusel oil not only has a higher fuel value than ethanol, but serves as a blending agent between the ethanol and gasoline.

The 95° GL ethanol entering the atmospheric dehydration tower is dehydrated in the manner previously described. Steam consumption in this system, varying somewhat with the percentage of ethanol in the beer, is in the range of 1.8-2.5 kg/liter (15-21 lb/gallon).

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