7

Mass Transfer Operations

This chapter is concerned with mass transfer operations (MTOs). As with all the chapters in Part II, there are sereval sections: overview, sereval technical sections, illustrative open-ended problems, and open-ended. The purpose of the first section is to introduce the reader to the subject of MTO. As one might suppose, a comprehensive treatment is not provided although sereval technical section are included. The next section contains three open-ended problems; the authors' solutions (there may be other solutions) are also provided. The last section contains 43 problems; *no* solutions are provided here.

7.1   Overview

This overview section is concerned—as can be noted from its title—with mass transfer operations (MTO). As one might suppose, it was not possible to address all topics directly or indirectly related to heat transfer. However, additional details may be obtained from either the references provided at the end of this Overview section and/or at the end of the chapter.

Note: Those readers already familiar with the details associated with MTO may choose to bypass this Overview.

There are host of mass transfer operations. The three that are most encountered in practice are absorption, adsorption, and distillation. As such they receive the bulk of the treatment in this chapter. Operations briefly reviewed include liquid-liquid extraction, leaching, humidification, drying, and membrane processes. Other novel separation processes (not reviewed) include:

1. Freeze crystallization
2. Ion exchange
3. Liquid ion exchange
4. Resin adsorption
5. Evaporation foam fractionation
6. Dissociation extraction
7. Crystallization
8. Electrophoresis
9. Vibrating screens

Details on these operations are available in the literature [1].

The topic of stagewise vs. continuous operation needs to be addressed before leaving this Overview section. Stagewise operation is considered first. If two insoluble phases are allowed to come into contact so that the various diffusing components of the mixture distribute themselves between the phases, and if the phases are then mechanically separated, the entire operation is said to constitute one *stage*. Thus, a stage is the unit in which contacting occurs and where the phases are separated; and, a single-stage process is naturally one where this operation is conducted once. If a series of stages are arranged so that the phases are contacted and separated once in each succeeding stage, the entire *multistage* assemblage is called a *cascade* and the phases may move through the cascade in parallel, counter-current, or cross-flow mode [1–3].

In order to establish a standard for the measure of performance, the *ideal*, or *theoretical*, or *equilibrium* stage is employed and it is defined as one where the effluent phases are in equilibrium, so that (any) longer time of contact will bring about no additional change of composition in either phase. Thus, at equilibrium, no further net change of composition state of the phases is possible for a given set of operating conditions. (In actual equipment in the chemical process industries, it is usually not

practical to allow sufficient time, even with thorough mixing, to attain equilibrium.)

An *actual* stage does not accomplish as large a change in composition as an equilibrium stage. It is for this reason, that the *fraction stage efficiency* is defined by many as the ratio of a composition change in an actual stage to than in an equilibrium stage. Stage efficiencies for equipment in the chemical process industry range between a few percent to that approaching 100 percent. The approach to equilibrium realized in any stage is then defined as the aforementioned fractional stage efficiency [1,2].

In the case of *continuous-contact* operation, the phases in question flow through the equipment in continuous intimate contact throughout the unit *without* repeated physical separation and contacting. The nature of the method requires the operation to be *either* semi batch or steady-state, and the resulting change in compositions may be equivalent to that given by a fraction of an ideal stage or by more than one stage. Thus, and as noted above, equilibrium between two phases at any position in actual equipment is generally never completely established.

The remainder of this chapter addresses the following four topics.

1. Absorption
2. Adsorption
3. Distillation
4. Other mass transfer proccesses

The reader should note that the bulk of the material in this chapter is drawn from J. Barden and L. Theodore, *Mass Transfer Operations*, A Theodore Tutorial, Theodore Tutorials, East Williston, NY, 1997, originally published by the USEPA/APTI, RTP, NC, 1997 [3].

7.2   Absorption

The removal of one or more selected components from a gas mixture by absorption is one of the most important operations in the field of mass transfer. The process of absorption conventionally refers to the intimate contacting of a mixture of gases with a liquid so that part of one or more of the constituents of the gas will dissolve in the liquid. The contact usually occurs in some type of packed column (addressed later in this section), although plate and spray towers are also used. In gas absorption operations the equilibrium of interest is that between a relatively nonvolatile

absorbing liquid (solvent) and a soluble gas (solute). The usual operating data to be determine or estimated for isothermal packed tower systems are the liquid rate(s) and the terminal concentrations or mole fractions. An operating line, that describes operating conditions in the column, is obtained by a mass balance around the column [1–3]. (Details on packing height and diameter follow.)

7.2.1   Packing Height

The height of a packed column is calculated by determining the required number of theoretical separation units and multiplying this number by the packing height. In continuous contact countercurrent operations, the theoretical separation unit is called the transfer unit, and the packing height producing one transfer unit is referred to as the height of a transfer unit. The transfer unit is essentially a measure of the degree of the separation (of the solute out of the gas stream and into the liquid stream) and the average driving force producing this transfer. In actual gas absorption design practice, the number of transfer units N_{OG} can be estimated from Equation 7.2 below One can also show that if Henry's law applies.

The number of transfer units is given by Colburn's equation

$$y = mx \tag{7.1}$$

$$N_{OG} = \ln \frac{\left[\left(\dfrac{y_1 - mx_2}{y_2 - mx_2}\right)\left(1 - \dfrac{1}{A}\right) + \dfrac{1}{A}\right]}{1 - \dfrac{1}{A}} \tag{7.2}$$

where $A = \dfrac{L_m}{mG_m}$

The term A is defined as the *adsorption factor* and is related to the slope of the equilibrium curve where L_m and G_m are the molar liquid and gas flow rates, respectively. If the gas is highly soluble in the liquid and/or reacts with the liquid, Theodore [4,5] has shown that, $N_{OG} = \ln\left(\dfrac{y_1}{y_2}\right)$[4,5] where the y_1 and y_2 refer to the inlet and outlet mole fraction, respectively.

The height of a transfer unit (H_{OG}) is usually determined experimentally for the system under consideration. Information on many different

systems using various types of packings has been compiled by the manu-facturers of gas absorption equipment and should be consulted prior to design. The data may be in the form of graphs depicting, for the specific system and packing, the H_{OG} versus the gas rate (in lb/h·ft²) with the liquid rate (in lb/h·ft²) as a parameter.

The packing height (Z) is then simply the product of H_{OG} and N_{OG}. Although there are many different approached to determine the column height, the $H_{OG} - N_{OG}$ approach is the most commonly use at the present time for approximate calculations, with the H_{OG} usually being obtained from the manufacturer.

7.2.2 Tower Diameter

Regarding tower diameter, consider a packed column operating at a given liquid rate in which the gas rate is gradually increased. After a certain point, the gas rate is so high that the drag on the liquid is sufficient to keep the liquid from flowing freely down the column. Liquid begins to accumulate and tends to block the entire cross – section for flow. This, of course, both increases the pressure drop and prevents the packing from mixing the gas and liquid effectively, and ultimate some liquid is even carried back up the column. This undesirable condition, know as *flooding*, occurs fairly abruptly and the superficial gas velocity at which it occurs is called the flooding velocity. The calculation of column diameter is based on flooding considerations; the usual operating range is 60% to 70% of the flooding rate.

The most commonly used correlation for pressure drop and diameter in the U.S. Stoneware's generalized correlation (see Figure 7.1). This procedure to determine the tower diameter is as follows:

1. Calculate the abscissa, $(L/G)(\rho/\rho_L)^{0.5}$; L,G = mass divided by time·area
2. Proceed to the flood line and read the ordinate.
3. Solve the ordinate equation for G at flooding.
4. Calculate the tower cross – sectional area, S, for the *fraction of flooding velocity chosen for operation, f*, by the equation

$$S = m/fG = (\text{total lb/s})/(\text{lb/s·ft}^2) = \text{ft}^2 \qquad (7.3)$$

The diameter of the tower is then determined by

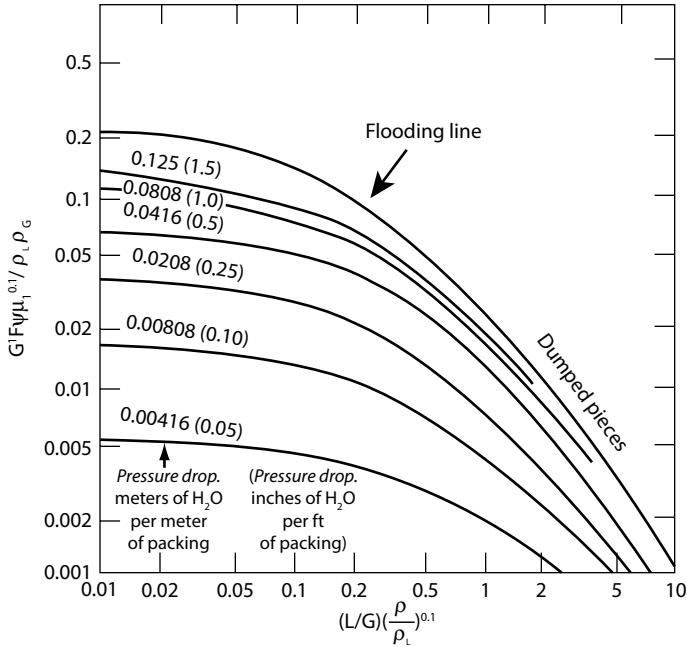$$D = 1.13S^{0.5} = \text{ft} \qquad (7.4)$$

Figure 7.1 Generalized correlation for flooding and pressure drop.

7.2.3   Plate Columns

Plate columns are essentially vertical cylinders in which the liquid and gas are contacted in stepwise fashion (stage operation) on plated or trays [1–3]. The liquid enters at the top and flows downward via gravity. On the way down, it flows across each plate and through a downspout to the plate below. The gas passes upward through openings in the plate, e.g., a sieve tray [1–3], then bubbles through the liquid to form a froth, and passes on to the next plate above. The overall effect is the multiple (repeated) countercurrent contact of a gas and liquid. Each plate of the column is a stage since the fluids on the plate are brought into intimate contact, interphase diffusion occurs, and the fluids are separated. The number of theoretical plates (or stages) is dependent on the difficulty of the separation to be carried out and is determined solely from material balances and equilibrium considerations. Details on plate columns are available in the literature [1-3].

7.2.4   Stripping

Quite often, an absorption column is followed by a liquid absorption process in which the gas solute is removed from the absorbing medium

by contact with an insoluble gas. This reverse operation is called *stripping* and is utilized to generate the solute *rich* solvent so that if may (in many cases) be recycled back to the absorption unit. The rich solution enters the stripping unit and the volatile solute is stripped from solution by either reducing the pressure, increasing the temperature, using a stripping gas to remove the vapor solute dissolved in the solvent, or any combination of these process changes. While the concept of stripping is opposite to that of absorption, it is treated in the same manner [6].

7.2.5   Summary of Key Equations [1,2]

The key equations for absorption and stripping calculations for tower height, including a summary of earlier material, are presented below.

For packed tower absorption:

$$N_{OG} = \frac{\log\{(y_1 - mx_2)/(y_2 - mx_2)\}\left\{1 - \left(\frac{1}{A}\right)\right\} + \{1/A\}}{1 - \frac{1}{A}} \qquad (7.5)$$

For stripping

$$N_{OG} = \frac{\log\{((x_2 - y_1)/m)/((x_1 - y_1)/m)\}\{1 - A\} + A}{1 - A} \qquad (7.6)$$

where the subscripts 1 and 2 refer to bottom and top conditions, respectively. In addition, $A = L_m/mG_m$ and $S = 1.0/A$.

For plate tower absorption

$$N = \frac{\log\{(y_{N+1} - mx_0)/(y_1 - mx_0)\}\left\{1 - \left(\frac{1}{A}\right)\right\} + \{1/A\}}{\log A} \qquad (7.7)$$

Note: the term ln, rather than log, may also be employed in both the numerator and denominator.

If A approaches unity, Equation (7.7) becomes

$$N = \frac{y_{N+1} - y_1}{y_1 - mx_0}$$

or

$$\frac{y_{N+1} - y_1}{y_{N+1} - mx_0} = \frac{N}{N+1} \tag{7.8}$$

Note that the subscripts 1 and N referred to the top and bottom of the column respectively.

For stripping in plate towers

$$N = \frac{\log\left\{\left((x_0 - y)_{N+1}/m\right)/\left((x_N - y_{N+1})/m\right)\right\}\left\{1 - \left(\frac{1}{S}\right)\right\} + S}{\log S} \tag{7.9}$$

or

$$\frac{x_0 - x_N}{x_0 - \left(y_{N+1}/m\right)} = \frac{S^{N+1} - S}{S^{N+1} - 1} \tag{7.10}$$

If S is approximately 1.0, one may use either of the following equations.

$$N = \frac{x_0 - x_N}{x_0 - \left(y_{N+1}/m\right)} \tag{7.11}$$

$$\frac{x_0 - x_N}{x_0 - \left(y_{N+1}/m\right)} = \frac{N}{N+1} \tag{7.12}$$

7.3   Adsorption [3]

The material to follow will primarily address *gas* adsorption. However, the bulk of the material presented may also be applied to liquid adsorption.

Adsorption is a mass transfer process in which a solute is removed from a gas stream because it adheres to the surface of a solid. In an adsorption system, the gas stream is passed through a layer of solid particles referred to as the adsorbent bed. As the gas stream passes through the adsorbent bed, the solute absorbs or "sticks" to the surface of the solid adsorbent particles. Eventually, the adsorbent bed becomes "filled" or saturated with the solute. The adsorbent bed must then be desorbed before the adsorbent bed can be reused.

The process of adsorption is analogous to using a sponge to mop up water. Just as a sponge soaks up water, a porous solid (the adsorbent) is capable of capturing gaseous or liquid solute molecules. The stream carrying the solute must then diffuse into the pores of the adsorbent (internal surface) where they are adsorbed. The majority of the molecules are adsorbed on the internal pore surfaces.

The relationship between the amount of substance adsorbed by the absorbent at constant temperature and the equilibrium pressure or concentration is called the adsorbent *isotherm*. The adsorption isotherm is the most important and by far the most often used of the various equilibria relationships which can be employed. Information on adsorption equilibria was addressed in Chapter 4.

Fixed bed adsorbers are the usual industrial choice when adsorption is the desired method of recovery/control. Consider a binary solution containing a solute at concentration C_0 (see Figure 7.2). The gas stream containing the solute (or adsorbent) is passed continuously down through a relatively deep bed of adsorbent which is initially free of adsorbate. The top layer of adsorbent, in contact with the inlet gas entering, at first absorbs the adsorbate rapidly and effectively. The remaining adsorbate is removed in the lower part of the bed. At this *initial* point in time the effluent concentration from the bottom of the bed is essentially zero. The bulk of the adsorption takes place over a relatively narrow adsorption zone (defined as the mass transfer zone, *MTZ*) in which there is a rapid change in concentration. At some later time, roughly half of the bed is saturated with the adsorbate, but the effluent concentration, C_2, is still substantially zero. Finally, at C_3, the lower portion of the adsorption zone has reached the bottom of the bed, and the concentration of adsorbate in the effluent has steadily risen to an appreciable value for the first time. The system is then said to have reached the *breakpoint*. The adsorbate concentration in the effluent gas stream now rises rapidly as the adsorption zone passes through the bottom of the bed, and at C_4 has essentially reached (approached) the initial value, C_0. At this point the bed is essentially fully saturated with adsorbate. The portion of the curve between C_3 and C_4 is termed the *breakthrough* curve.
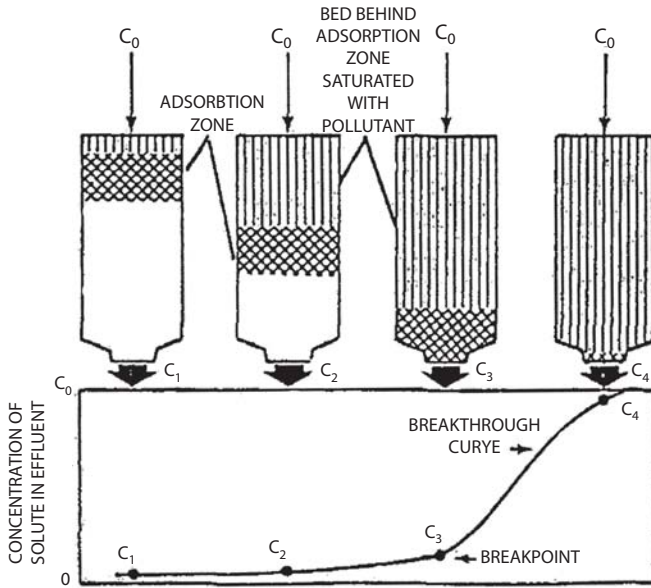
Figure 7.2 Operation Adsorption System.

The *breakthrough capacity* is the bed capacity when traces of adsorbate first begin to appear in the exit gas stream from the adsorption bed. It may be estimated from the following equation.

$$BC = [(0.5)(CAP)(MTZ) + (CAP)(Z\text{-}MTZ)]/Z \qquad (7.13)$$

where BC = breakthrough capacity
    CAP = saturation capacity
    MTZ = mass transfer zone height
    Z = adsorption bed depth.
The usual procedure in practice is to work with a term defined as the *working charge* (or working capacity). It provides a numerical value for the actual adsorbing capacity of the bed of height Z under operating conditions. If experimental data are available, the working charge, WC, may be estimated from

$$WC = CAP\ [(Z - MTZ)/Z] + 0.5\ [MTZ/Z] - HEEL \qquad (7.14)$$

where *HEEL* is the residual adsorbate present following regeneration. Since much of the data requried is rarely available, or just simply ignored,

the working charge may be taken to be some fraction, f, of the saturated (equilibrium) capacity of the adsorbent; i.e., $WC = (f)(CAP)$; $0 \leq f \leq 1.0$.

7.3.1   Adsorption Design

A rather simplified overall design procedure for a system adsorbing an organic with activated carbon that consists of two horizontal units (one on/one off) that are regenerated with steam was developed by one of the authors and is provided below[7]:

1. Select adsorbent type and size.
2. Select cycle time; estimate regeneration time; set adsorption time equal to regeneration time; set cycle time equal to twice the regeneration time; generally, try to minimize regeneration time.
3. Set gas throughput velocity v is it usually in the 80 ft/min range but can increase to 100 ft/min.
4. Set the steam/solvent ratio.
5. Calculate (or obtain) WC for the above.
6. Calculate the amount of solvent adsorbed (M_s) during ½ the cycle time (t_{ads}) using the equation

$$M_s = qC_i t_{ads} \; ; C_i = \text{inlet solvent concentration} \qquad (7.15)$$

7. Calculate the adsorbent required, M_{AC}:

$$M_{AC} = M_s / WC \qquad (7.16)$$

8. Calculate the adsorbent volume requirement.

$$V_{AC} = M_{AC} / \rho_B; \rho_B = \text{carbon bulk density} \qquad (7.17)$$

9. Calculate the face area of the bed.

$$A_{AC} = q/v \qquad (7.18)$$

10. Calculate the bed height, Z.

$$Z = V_{AC} / A_{AC} \qquad (7.19)$$

11. Estimate the pressure drop using any convenient method.

12. Set the L/D (length to diameter) ratio.
13. Calculate L and D, noting that

$$A_{AC} = LD \qquad (7.20)$$

14. Design (structurally) to handle if filled with water.
15. Design vertically if $q < 2500$ actual cubic feet per minute (acfm).

This topic is revisited in Part III, Chapter 28 – *Mass Transfer Operation Term Projects*.

7.3.2   Regeneration

Adsorption processes in practice use various techniques to accomplish regeneration or desorption. The adsorption-desorption cycles are usually classified into four types, used separately or in combination.[8]

1. Thermal swing cycles;
2. Pressure swing cycles use either a low pressure or vacuum to desorb the bed;
3. Purge gas stripping cycles; and,
4. Displacement cycles using an adsorbable purge to displace the previously adsorbed material on the bed.

7.4   Distillation

Distillation is no doubt the most widely used separation process in the chemical and allied industries. Applications range from the rectification of alcohol to the fractionation of crude oil. The separation of liquid mixtures by distillation is based on differences in volatility between the components. The first component to vaporize is considered to be more volatile than the other components in the system. The greater the *relative* volatilities, the easier the separation.

Information on batch and flash distillation is available in the literature. [1–3] This section will primarily review the McCabe-Thiele method [9] and has been primarily adapted from the literature [1–3].

In a distillation column (see Figure 7.3), vapor flows up the column and liquid flows countercurrently down the column. The vapor and liquid are brought into contact on plates as shown in Figure 7.3, or packing. The vapor
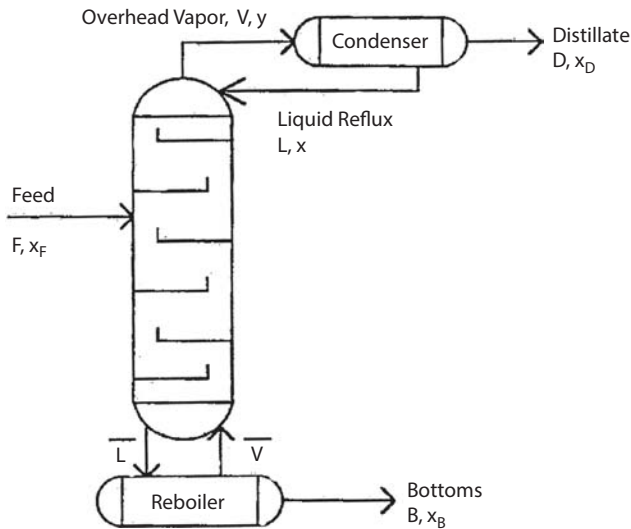
Figure 7.3 Schematic of a trayed distillation column.

from the top of column is sent to a condenser. Part of the condensate from the condenser is returned to the top of the column as reflux to descend counter to the rising vapors. The remainder of the condensed liquid is drawn as product. The ratio of the amount of reflux returned to the column to the distillate product collected is known as the *reflux ratio*. As the liquid stream descends the column it is progressively enriched with low-boiling constituents. The column internals are then used as an apparatus for bringing these streams into intimate contact so that the vapor stream tends to vaporize the low-boiling constituent from the liquid and the liquid stream tends to condense the high-boiling constituent from the vapor. Column-wise distillation involves two sections: an *enriching* (top) and *stripping* (bottom) section, which lies below the feed where the more volatile components are stripped from the liquid; in the enriching or rectifying section above the feed.

A column may consist of one or more feeds and may produce two or more product streams. The product recovered at the top of a column is referred to as the *tops*, while the product at the bottom of the column is referred to as the *bottoms*. Any product(s) drawn at various stages between the top and bottom are referred to as *side streams*. Multiple feeds and product streams do not alter the basic operation of a column, but they do complicate the calculations and analysis of the process to some extent. If the process requirement is to strip a volatile component from a relatively nonvolatile solvent, the rectifying (bottom) section may be omitted, and

the unit is then called a stripping column. Virtually pure top and bottom products can be achieved by using many stages or (occasionally) additional columns; however, this is not usually economically feasible.

The top of the column is cooler than the bottom, so that the liquid stream becomes progressively hotter as it descends and the vapor stream becomes progressively cooler as it rises. This heat transfer is accomplished by actual contact of liquid and vapor; and for this purpvose, effective contacting is desirable. Each plate in the column is assumed to approach equilibrium conditions. This type of plate is defined as a *theoretical plate*, i.e., a plate on which the contact between the vapor and liquid is sufficiently good so that the vapor leaving the plate has the same composition as the vapor in equilibrium with the liquid overflow from the plate. The vapor and liquid leaving are related by the aforementioned equilibrium curve. Distillation columns designed on this basis serve as a standard for comparison to actual columns. By such comparisons, it is possible to determine the number of actual plates that are equivalent to a theoretical plate and then to reapply this factor when designing other columns for similar service.

In some operations where the top product is required as a vapor, the liquid condensed is sufficient only to provide reflux to the column, and the condenser is referred to as a *partial condenser*. In a partial condenser, the reflux will be in equilibrium with the vapor leaving the condenser, and is considered to be an equilibrium stage in the development of the operating line when estimating the column height. When the liquid is totally condensed, the liquid returned to the column will have the same compositions as the top product and is not considered to be in an equilibrium state. A *partial reboiler* is utilized to generate vapor to operate the column and to produce a liquid product if necessary. Since both liquid and vapor are in equilibrium, a partial reboiler is considered to be an equilibrium stage as well.

As noted earlier, an operating line can be developed to describe the equilibrium relation between the liquid and vapor components. However, in staged column design, it is necessary to develop an operating line which relates the passing streams (liquid entering and vapor leaving) on each stage in the column. The following analysis will develop operating lines for the top, or enriching (rectifying) section and the bottom, or stripping, section of a column.

To accomplish the above analysis, an overall material balance is written for the condenser as $V = L + D$, which represents the vapor (V) leaving the top stage, the liquid reflux returning (L) to the column from the condenser (reflux), and the distillate (D) collected. See above Figure 7.3 A material

balance for a component is written as $Vy = Lx + Dx_D$. This can be rearranged in the form of an equation for a straight line straight, $y = mx + b$ as,

$$y = \frac{L}{V}x + \frac{D}{V}x_D \qquad (7.21)$$

From the overall mass balance, $D = V - L$ so that Equation 7.21 can be written as

$$y = \frac{L}{V}x + \left(1 - \frac{L}{V}\right)x_D \qquad (7.22)$$

where L/V = slope. This is the internal reflux ratio (liquid reflux returned to the column/vapor from the top of the column). Even though Equation 7.22 has been developed around the condenser, it represents the equilibrium relationship of passing liquid/vapor streams and can be applied to the top of the column.

The corresponding operating line in the bottom or stripping section can be developed in a similar manner. The overall material balance around the reboiler and the component material balance reduce to Equation 7.23. Note that the terms with the bars over them represent the flow at the bottom of the column. Again, this material balance has been rearranged in the form for a straight line as

$$y = \frac{B}{\overline{V}}x_B + \frac{\overline{L}}{\overline{V}}x \qquad (7.23)$$

But since $B = \overline{L} + \overline{V}$, Equation 7.23 can be rewritten as

$$y = \left(\frac{\overline{L}}{\overline{V}} - 1\right)x_B - \frac{\overline{L}}{\overline{V}}x \qquad (7.24)$$

Equation 7.24 can be rearranged into a form similar to Equation 7.22.

$$y = -\frac{\overline{L}}{\overline{V}}x + \left(\frac{\overline{L}}{\overline{V}} - 1\right)x_B \qquad (7.25)$$

And, as before, the term $\overline{L}/\overline{V}$ is the slope and $\left(\frac{\overline{L}}{\overline{V}} - 1\right)$ represents the y-intercept. The slope can be calculated from the external reflux ratio by Equation 7.26:

$$\frac{\bar{L}}{\bar{V}} = \frac{\dfrac{L}{D}\left(x_f - x_B\right) + q(x_D - x_B)}{\dfrac{L}{D}\left(x_f - x_B\right) + q\left(x_D - x_B\right) - \left(x_D - x_f\right)} \tag{7.26}$$

A procedure for designing a staged distillation column is provided below.

1. Plot the equilibrium data, the top and bottom operating lines and the $y = x$ line on the same graph. This plot is called a McCabe-Thiele diagram as portrayed in Figure 7.4 [9].
2. Step off the number of stages required beginning at the top of the column. The point at the top of the curve represents the composition of the liquid entering (reflux) and gas leaving (distillate) the top of the column. The point at the bottom of the curve represents the composition of liquid leaving (bottoms) and the gas entering (vapor from reboiler) the bottom of the column. All stages are stepped off by drawing alternate vertical and horizontal lines in a *stepwise* manner between the top and bottom operating lines and the equilibrium curve. The number of steps is the number of theoretical stages requires. When a partial reboiler or partial condenser is employed, the number of equilibrium stages stepped off is
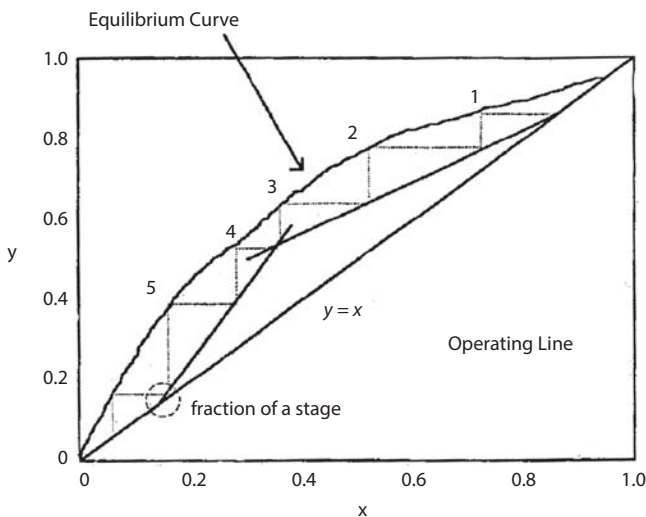

Figure 7.4 McCabe-Thiele Diagram.

$N+1$ and the number of theoretical plates would be N. Thus, if both are to be included, the number of equilibrium stages stepped off would be $N+2$.

3. To determine the actual number of plates required, divide the result in step (2) by the *overall* plate fractional efficiency, typically denoted by E_o. Values can range from 0.4 to 0.8. The actual number of plates can be calculated from

$$N_{act} = N / E_0 \qquad (7.27)$$

4. The column height can be calculated by multiplying the result in step (3) by the tray spacing (9,12,15,18 and 24 inches are typical tray spacings).

5. To determine the *optimum* feed plate location, draw a line from the feed composition on the $y = x$ line, through the intersection of the top/bottom operating lines, to the equilibrium curve. The step straddling the feed line is the correct feed-plate location.

6. Due to the differences in vapor traffic throughout the column, the diameter will vary from the top to the bottom. Typical designs are *swaged* columns, where the diameter is larger at the bottom than the top. A "cone-like" vessel links the bottom of the column with the top of the column. The diameter at the top of a column can be roughly sized by assuming a superficial (column only contains vapor) vapor velocity of 5 ft/s and then using the following equation to calculate the diameter.

$$D = \left(\frac{4q}{\pi v} \right)^{0.5} \qquad (7.28)$$

A somewhat similar procedure is employed to calculate the diameter at the bottom of a column. The interested reader is referred to a mass transfer text which can provide a more detailed analysis [1–3].

As indicated earlier, distillation can be carried out in both trayed and packed columns. The selection of which method to be used depends on the specific application. Packing is frequently comparable in cost with trays and becomes more attractive where a low pressure drop and small liquid hold-up are required. It is generally recommended that packed towers

should be used if the column diameter is less than 1.5 ft. Packed columns also provide continuous contacting between the liquid and gas. This imitate contacting occurs on the packing which promotes the heat transfer required to separate the more volatile component from the process stream.

The Fenske equation calculates the minimum number of equilibrium stages required for *multicomponent* separation at total reflux (100% of the liquid reflux is returned to the column). On the other hand, Underwood developed an equation which estimates the maximum number of equilibrium stages required at minimum reflux, and the actual reflux ratio for a multicomponent system. But in order to estimate the number of actual stages, the Gilliland equation correlates the minimum number of stages at total reflux, the minimum reflux ratio, and the actual reflux rate for a multicomponent system. The application of all three of these equations in the design of a distillation column is referred to as the Fenske-Underwood-Gilliland (FUG) method. Details on each of the above are provided in the literature [1,2,10]

7.5   Other Mass Transfer Processes

7.5.1   Liquid-Liquid Extraction [1,2,11]

Liquid-liquid extraction (or liquid extraction) is a process for separating a solute from a solution employing the concentration driving force between two immiscible (non-dissolving) liquid phases. Thus, liquid extraction involves the transfer of solute from one liquid phase into a second immiscible liquid phase. The simplest example involves the transfer of one component from a binary mixture into a second immiscible liquid phase; such is the case with extraction of an impurity from waste water into an organic solvent. Liquid extraction is usually selected when distillation or stripping is impractical or too costly; this situation occurs when the relative volatility for two components falls between 1.0 and 1.2. Treybal [11] provides extensive details. This topic is also revisited in Part III, Chapter 28 – *Mass Transfer Operation Term Projects*.

7.5.2   Leaching

Leaching is the preferential removal of one or more components from a solid by contact with a liquid solvent. The soluble constituent may be solid or liquid, and it may be chemically or mechanically held in the pore structure of the insoluble solids material. The insoluble solid material is often particulate in nature, porous, cellulite with selectively permeable cell walls, or surface activated. In chemical engineering practice, leaching is also

referred to by several other names such as extraction, liquid-solid extraction, lixiviation, percolation, infusion, washing, and decantation-settling. The simplest example of a leaching process is in the preparation of a cup of tea. Water is the solvent used to "extract", or leach, tannins and other solids from the tea leaf.

7.5.3   Humidification and Drying

In many unit operations, it is necessary to perform calculations involving the properties of mixtures of air and water vapor. Such calculations often require knowledge of: the amount of water vapor carried by air under various conditions, the thermal properties of such mixtures, and the changes in enthalpy content and moisture content as air containing some moisture is brought into contact with water or wet solids and other similar processes. Some mass transfer operations in the chemical process industry involve simultaneous heat and mass transfer. In the *humidification* process, water or another liquid is vaporized, and the required heat of vaporization must be transferred to the liquid. *Dehumidification* is the condensation of water vapor from air, or, in general, the condensation of any vapor from a gas.

Drying involves the *removal* of relatively small amounts of water from solids. In many applications, such as in corn processing, drying equipment follows an evaporation step to provide an ultra-high solids content product stream. Drying, in either a batch or continuous process, removes liquid as a vapor by passing warm gas (usually air) over, or indirectly heating, the solid phase. The drying process is carried out in one of the three basic dryer types. The first is a continuous tunnel dryer. In a continuous dryer, supporting trays with wet solids are move through an enclosed system while warm air blows over the trays. Similar in concept to the continuous tunnel dryer, rotary dryers consist of an inclined rotating hollow cylinder. The wet solids are fed in one side and hot air is usually passed countercurrently over the wet solids. The dried solids then pass out the opposite side of the dryer unit. The final type of dryer is a spray dryer. In spray dryers, a liquid or slurry is sprayed through a nozzle, and fine droplets are dried by a hot gas, passed either concurrently countercurrently, past the falling droplets. This unit has found wide application in air pollution control [8].

7.5.4   Membrane Processes [1]

Membrane separation processes are one of the newer (relatively speaking) technologies being applied in practice. The subject matter is and has

been introduced into the chemical engineering curriculum. There are four major membrane processes of interest to the chemical engineer.

1. Reverse osmosis (hyperfiltration);
2. Ultrafiltration;
3. Microfiltration; and
4. Gas permeation.

The four processes have their differences. The main difference between reverse osmosis (RO) and ultrafiltration (UF) is that the size/diameter of the particles or molecules in solution to be separated is smaller in RO. In microfiltration (MF), the particles to be separated/concentrated are generally solids or colloids rather than molecules in solution. Gas permeation (GP) is another membrane process that employs a non-porous semipermeable membrane to "fractionate" a gaseous stream [12–14].

A detailed and expanded treatment of mass transfer operations is available in the following three references.

1. L. Theodore and F. Ricci, *Mass Transfer Operations for the Practicing Engineer*, John Wiley & Sons, Hoboken, NJ, 2010 [1].
2. R. Treybal, *Mass Transfer Operations,* McGraw-Hill, New York City, NY, 1955 [2].
3. Adapted from, L. Theodore, *Chemical Engineering: The Essential Reference*, McGraw-Hill, New York City, NY, 2014.

7.6   Illustrative Open-Ended Problems

This and the last Section provide open-ended problems. However, solutions *are* provided for the three problems in this Section in order for the reader to hopefully obtain a better understanding of these problems which differ from the traditional problems/illustrative examples. The first problem is relatively straightforward while the third (and last problem) is somewhat more difficult and/or complex. Note that solutions are not provided for the 43 open-ended problems in the next Section.

Problem 1: Provide a technical explanation of the essential difference between stagewise and continuous operation.
Solution: The essential difference between stagewise and continuous-contact operation may be summarized in the following manner. In the case of the stagewise operation, the flow of matter between the phases is allowed to

reduce the concentration difference. If allowed to contact for long enough, equilibrium can be established after which no further transfer occurs. The rate of transfer and the time (of contact) then determine the stage efficiency realized in any particular application. On the other hand, in the case of the continuous-contact operation, the displacement from equilibrium is deliberately maintained and the transfer between the phases may continue without interruption. Economics plays a significant role in determining the most suitable method. [1–3].

Problem 2: One of the more commonly used procedure for pressure drop and column diameter calculations is the U.S. Stonewaring [3] generalized pressure drop correlation as presented in Figure 7.3. Convert the generalized U.S. Stoneware pressure drop-flooding correlation into equation form. Comment: Refer to the literature [1–3].

Solution: There are several correlations available. Chen [16] developed the following equation from which the tower diameter can easily be obtained:

$$D = 16.28 \left(\frac{W}{\phi L} \right)^{0.5} \left(\frac{\rho_L}{\rho_G} \right)^{0.25} \tag{7.28}$$

with

$$\log_{10}\phi = 32.5496 - 4.1288\log_{10}\left(\frac{L^2 A_v \mu_L^{0.2}}{\rho L^2 \varepsilon^3} \right)^{0.5} \tag{7.30}$$

where (employing Chen's notation) A_v is the specific surface area of dry packing (ft²/ft³ packed column), L is the liquid flux (gal/min·ft² of superficial tower cross section), W is the mass flow rate of gas (lb/h), ε is the void fraction, μ_L is the liquid viscosity (cP), and the density terms are in lb/ft³.

Problem 3: Consider the absorber system shown in Figure 7.5. Ricci Engineers designed the unit to operate with maximum discharge concentration of 50 ppm. Once the unit was installed and running, the unit operated with a higher discharge concentration. Rather than purchase a new unit, what options are available to get the unit operating at the specified design concentration?
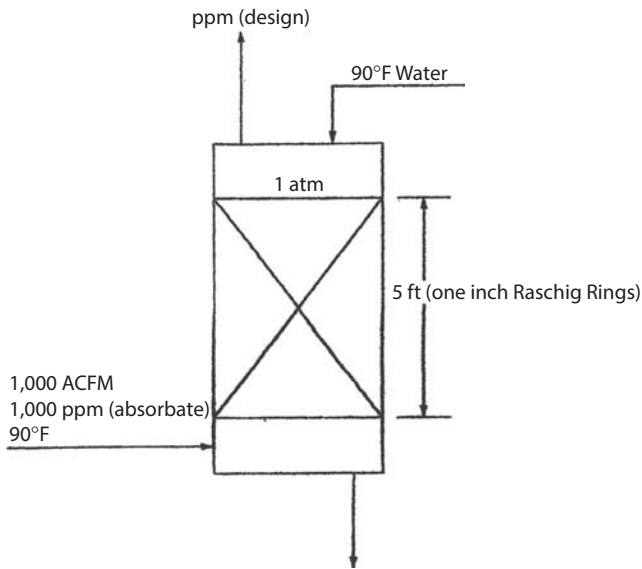
Figure 7.5 Absorber failure to meet design performance.

Solution: This is obviously an open-ended question. One may employ any one or a combination of the following suggestions [1]:

1. Increase the temperature.
2. Decrease the pressure.
3. Increase the height of the packing.
4. Place sprays at the inlet before the packing.
5. Increase the liquid flow rate, but check the pressure drop increase and any potential effect on the fan.
6. Change the liquid, but check the pressure drop increase and any potential effect on the fan.
7. Change packing size and/or type, but check the pressure drop increase and any potential effect on the fan.
8. Process modification: if possible reduce inlet absorbate concentration.
9. Process modification: reduce gas flow rate.
10. Design a new system.
11. Add more packing on top of existing packing (if possible), but check the pressure drop increase and any potential effect on the fan.
12. Fire the design engineer.
13. Shut down the plant.

7.7   Open-Ended Problems

This last section of the chapter contains open-ended problems as they relate to mass transfer operations. No detailed and/or specific solution is provided; that task is left to the reader, noting that each problem has either a unique solution or a number of solutions or (in some cases) no solution at all. These are characteristics of open-ended problems described earlier.

There are comments associated with some, but not all, of the problems. The comments are included to assist the reader while attempting to solve the problems. However, it is recommended that the solution to each problem should initially be attempted *without* the assistance of the comments.

There are 43 open-ended problems in this section. As stated above, if difficulty is encountered in solving any particular problem, the reader should next refer to the comment, if any is provided with the problem. The reader should also note that the more difficult problems are generally located at or near the end of the section.

1. Describe the early history associated with traditional mass transfer operations.
2. Describe the early history associated with traditional membrane technology.
3. Discuss the recent advances in traditional mass transfer technology.
4. Discuss the recent advances in traditional membrane technology.
5. Select a refereed, published article on mass transfer from the literature and provide a review.
6. Develop an original problem that would be suitable as an illustrative example in a book on mass transfer operations.
7. Prepare a list of the various books which have been written on mass transfer operations. Select the three best (hopefully including a book written by one of the authors) and justify your answer. Also select the three weakest books and, once again, justify your answer.
8. Discuss the advantages and disadvantages of batch versus continuous operation in mass transfer operations.
9. Describe the options that are available to represent a multicomponent diffusion operation via an average diffusion coefficient.
    Comment: Refer to Treybal's "Mass Transfer Operations" text [2].

10. Describe the advantages and limitations of employing the concept of an ideal stage in all the applicable mass transfer operations.

11. Discuss the merits of employing Henry's law in gas absorption calculations.

12. Discuss the merits of employing Raoult's law in distillations calculations.

13. Construct a vapor-liquid equilibrium diagram of x vs y for various relative volatility (γ) values. Include $\propto$ values of 0.0, 1.0, and ∞.

14. Discuss the merits of including the activity coefficient in liquid-vapor calculations.

15. Describe the various calculational problems that arise in many of the mass transfer processes that are not isothermal. Also extend the description to adiabatic operations.

16. Design a new packing that could be employed for absorption, adsorption, and distillation operations.

17. Suggest/develop a new support plate for an absorption column, adsorption column, and packed tower distillation unit.

18. List the various type of overhead separators employed in several unit operations of your choice.

19. Attempt to develop a new overhead separation device.

20. Develop a new liquid componential separation device.

21. Design a new membrane that could be employed in any of the four membrane separation processes discussed in the Overview section.

22. Develop a new method of drying solids.

23. Develop a new gas (or liquid) particulate separation device.

24. Describe the advantages and disadvantages of the various particulate separation devices that are employed in adsorption operations. Include those for both liquid and gaseous adsorption units.

25. Describe the various methods available to reduce the size of solids. Can a better method be developed?

26. Describe the various current methods employed for sedimentation operations.

27. Develop a generalized equation to describe the performance of a distillation column when one or more plates within the unit fail to perform any separation.

28. Outline a procedure to determine the optimum feel plate location(s) in a distillation tower. Compare your procedure to those employed in the past.

29. Discuss the problems associated with the various plate efficiencies employed in distillation calculations.

30. Convert the O'Connell correlation for overall distillation tray efficiency into equation form.
    Comment: Refer to L. Theodore and F. Ricci's *Mass Transfer Operations* text [1].

31. List some advantages and disadvantages of employing distillation versus another mass transfer operation to separate a two-component liquid stream.

32. Discuss the calculational problems that arise in absorption with chemical reaction.

33. Describe the difference between absorption and adsorption in both technical and layman terms.

34. Discuss the differences between drying, leaching, and extraction.

35. A dated plate distillation column is no longer delivering the degree of separation required for a process. Rather than replace the unit, you have been asked to recommend what other possible steps can be taken to the existing unit to get it back "on line".
    Comment: Refer to Problem 3 in the previous section.

36. An adsorber is designed to operate with a specified maximum discharge concentration. Once the unit is installed and running, the unit operated with a higher discharge concentration. Rather than purchase a new unit, what options are available to bring the unit into compliance with the specified design concentration?
    Comment: Refer to Problem 3 in the previous section.

37. You are an engineer at a water purification plant. The water is purified using a reverse osmosis (RO). One of the units at your plant is no longer operating at the required "separation" efficiency. What steps should be taken to avoid purchasing a new system?
    Comment: Refer to Problem 3 in the previous section.

38. Woist Engineers designed a cooling tower to operate at or below a maximum water discharge temperature. Once the tower was installed and running, the unit operated with a slightly higher temperature. Rather than purchase a new

tower, what options are available to bring the unit into compliance with the specified design temperature?
Comment: Refer to Problem 3 in the previous section.

39. A liquid-liquid extraction unit normally operates at a particular discharge concentration. Over time, the operating efficiency of the extractor has decreased. Rather than purchase a new unit, what options are available to bring the unit into compliance with the specified design concentration?
Comment: Refer to Problem 3 in the previous Section.

40. Fitzmaurice Engineers designed a rotary dryer to operate at or below a solids water discharge concentration. Once the dryer was installed and running, the unit operated with a higher concentration. Rather than purchase a new dryer, what options are available to bring the unit into compliance with the specified design solids concentration?
Comment: Refer to Problem 3 in the previous section.

41. You have been requested, as a part of a drying operation project, to convert the standard humidity chart into equation form.
Comment: Good luck.

42. Develop a design procedure for each of the various classes of dryers.

43. Develop a crystallization design procedure.

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