

Chapter 4

4.4 Distillation with Reflux

4.4a Introduction: Distillation with reflux can be considered to be a process in which equilibrium stages are arranged in a series in such a manner that the vapor and liquid product from each stage flow counter-current to each other. Each stage receives the liquid flow from the stage above and the vapor flow from the stage below. The liquid and vapor are mixed in each stage and they are assumed to be in equilibrium when they leave a stage. Figure 4.4-1 shows the process flow diagram for a distillation tower containing sieve trays.

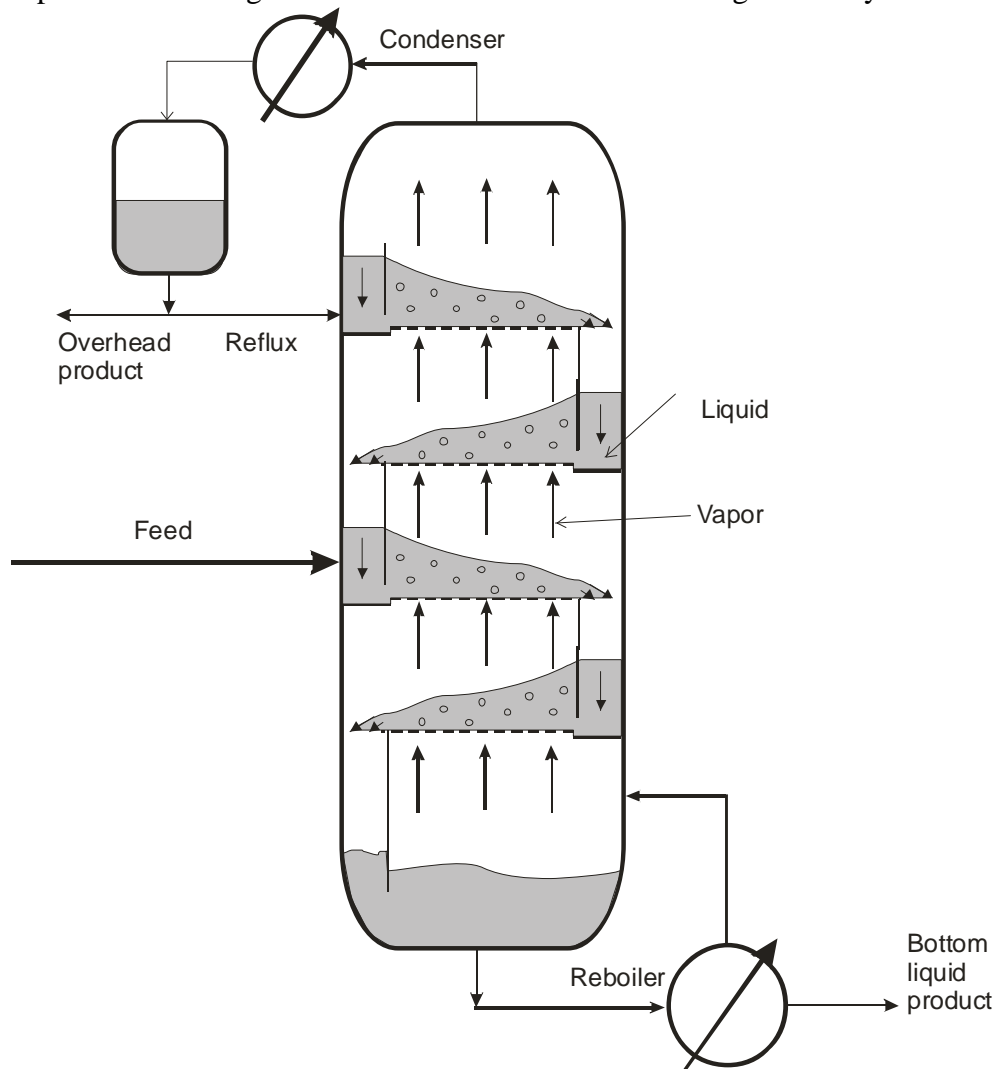


Figure 4.4-1 Process flow of a distillation tower containing sieve trays.

The feed is normally introduced into the distillation at a location between the top and the bottom trays. For a total condenser, the vapor from the top tray is condensed and part of the condensate is returned to the top of the column to provide liquid flow above the feed point. Part of the liquid from the bottom tray is vaporized in the reboiler and returned to the bottom of the column to provide the vapor flow.

4.4b McCabe-Thiele Method

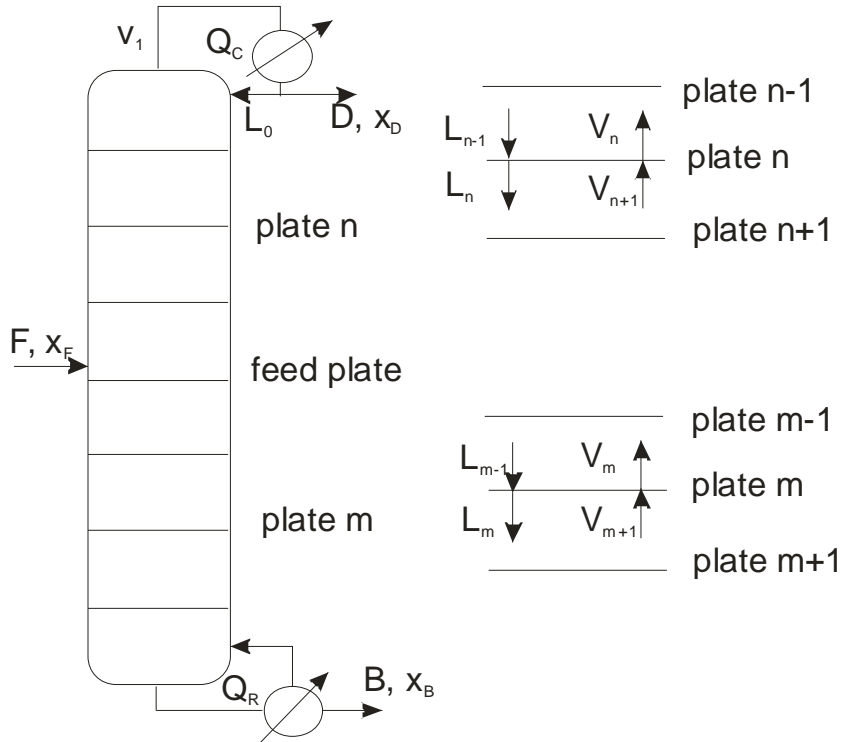


Figure 4.4-2 Distillation column notations.

The McCabe Thiele method is a graphical procedure to solve alternately the equilibrium relation and the material balance for binary distillation. Consider the distillation column shown in Figure 4.4-2 with the tray above and below the feed tray using the index n and m , respectively. The section of the column above the feed tray is the rectifying section and the section below the feed tray is the stripping section. The two streams (L_n and V_n or L_m and V_m) leaving a theoretical (or equilibrium) tray are in equilibrium. The two passing streams (for example L_{n-1} and V_n) are related by the material balance or operating line. A total material balance for tray n gives

$$V_{n+1} + L_{n-1} = V_n + L_n \quad (4.4-1)$$

A component balance on the more volatile component, A, gives

$$y_{n+1}V_{n+1} + x_{n-1}L_{n-1} = y_nV_n + x_nL_n \quad (4.4-2)$$

In this expression, y_{n+1} is mole fraction of component A in V_{n+1} and x_{n-1} is the mole fraction of component A in L_{n-1} and so on. The McCabe-Thiele method is valid when we have constant molal overflow in both the rectifying and stripping sections of the distillation column. Hence

$$V_1 = V_2 = V_3 = \dots = V_n = V_{n+1} = \text{constant}$$

$$L_0 = L_1 = L_2 = \dots = L_{n-1} = L_n = \text{constant}$$

$$V_{m-1} = V_m = V_{m+1} = \text{constant}$$

$$L_{m-1} = L_m = L_{m+1} = \text{constant}$$

Even for CMO, $V_n \neq V_m$ and $L_n = L_m$. The constant molal overflow is justified if the heat of mixing is negligible for the two components in the mixture (ideal solution) and the molar heats of vaporization for the two components are almost the same.

Equations for rectifying section

Figure 4.4-3 shows the rectifying or enriching section which is the distillation tower section above the feed. The vapor from the top tray with a composition y_1 is condensed in the total condenser so that the resulting liquid is at the boiling point. Part of the liquid is taken out as the overhead product D and the remaining liquid is returned to the first tray with flow rate L_0 .

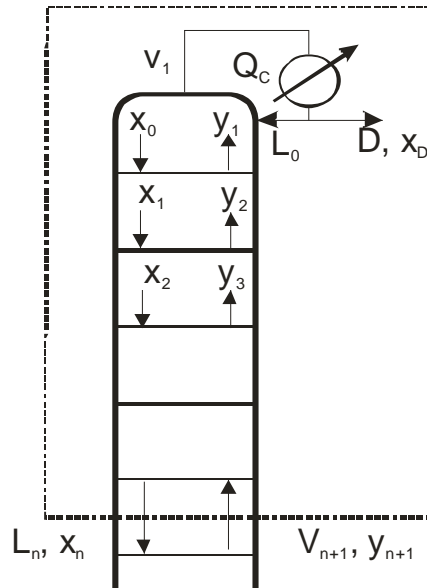


Figure 4.4-3 Schematic of the rectifying section with a total condenser.

Making a balance over the top part of the tower (the dashed-line section) on component A gives

$$y_{n+1}V_{n+1} = x_nL_n + x_D D \quad (4.4-3)$$

Since CMO is assumed, $V_{n+1} = V = \text{constant}$ and $L_n = L = \text{constant}$. Eq. (4.4-3) can be written as

$$y_{n+1} = x_n L/V + x_D D/V \quad (4.4-4)$$

From the total balance $V = L + D \Rightarrow D/V = 1 - L/V$. In terms of the reflux ratio $R = L/D$

$$\frac{L}{V} = \frac{L}{L+D} = \frac{L/D}{L/D+1} = \frac{R}{R+1}$$

$$\frac{D}{V} = \frac{D}{L+D} = \frac{1}{L/D+1} = \frac{1}{R+1}$$

Eq. (4.4-4) becomes

$$y_{n+1} = \frac{R}{R+1}x_n + \frac{1}{R+1}x_D \quad (4.4-5)$$

This equation which is the material balance for component A is called the operating line. Given x_D , x_F , R , and the equilibrium data, the equilibrium stages can be determined by successively solving the equilibrium relation and the material balance at each stage. Starting at x_D , y_1 is evaluated, for a total condenser $y_1 = x_D$. The liquid composition x_1 is then determined from the equilibrium curve at $y = y_1$. This is a dew point calculation with known y_1 . Next y_2 is determined from the mass balance or operating line

$$y_2 = \frac{R}{R+1}x_1 + \frac{1}{R+1}x_D$$

x_2 is again determined from the equilibrium curve and y_3 is determined from

$$y_3 = \frac{R}{R+1}x_2 + \frac{1}{R+1}x_D$$

This process continues until $x_n < x_F$. The number of equilibrium stages in the rectifying section is n including the feed tray, n is the number of equilibrium calculations during the process.

Example 4.4-1 -----

A distillation column receives a feed that is 40 mole % n-pentane and 60 mole % n-hexane. Feed flow rate is 2,500 lbmol/hr and feed temperature is 30°C. The column is at 1 atm. A distillate that is 97 mole % n-pentane is desired. A total condenser is used. Reflux is a saturated liquid. The external reflux ratio is $L_0/D = 3$. Bottoms from the partial reboiler is 98 mole % n-hexane. Determine the number of equilibrium trays and their compositions for the rectifying section.

Data: Vapor pressure, P^{sat} , data: $\ln P^{\text{sat}} = A - B/(T + C)$, where P^{sat} is in kPa and T is in K.

Compound	A	B	C
n-pentane (1)	13.9778	2554.6	- 36.2529
n-hexane (2)	14.0568	2825.42	- 42.7089

Heat of evaporation for n-pentane, $\lambda_{C5} = 11,369$ Btu/lbmol, $C_{pL,C5} = 39.7$ Btu/lbmol·°F
 Heat of evaporation for n-hexane, $\lambda_{C6} = 13,572$ Btu/lbmol, $C_{pL,C6} = 51.7$ Btu/lbmol·°F

Solution -----

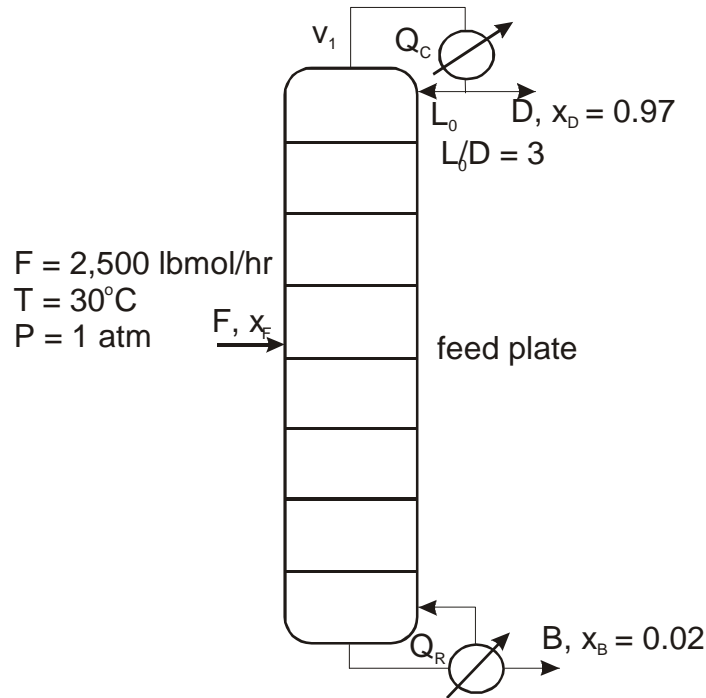
Overall material balance over the entire tower gives

$$D + B = 2,500$$

Material balance for n-pentane over the entire tower gives

$$0.97D + 0.02B = (0.4)(2,500) = 1,000 \Rightarrow 0.97(2,500 - B) + 0.02B = 1,000$$

Solving for B and D from the above equations we have $B = 1,500$ lbmoles/hr and $D = 1000$ lbmoles/hr.



Since the column has a total condenser, $y_1 = x_D = 0.97$. The liquid composition x_1 is then determined from the equilibrium curve at $y = y_1 = 0.97$. The equilibrium curve y versus x for n-pentane and n-hexane system is plotted in Figure E-1a where x and y are the mole fraction of n-pentane in the liquid and vapor phase, respectively. The equilibrium curve is expanded near the region $x = 1$ in Figure E-1b. At $y = y_1 = 0.97$, $x = x_1 = 0.9107$. We can also perform a dew point temperature calculation with $y = y_1 = 0.97$.

We start with the equation

$$x_1 + x_2 = 1 \tag{E-1}$$

Substituting $x_i = y_i P / P_i^{sat}$ into equation (E-1) yields

$$\frac{y_1 P}{P_1^{sat}} + \frac{y_2 P}{P_2^{sat}} = 1 \tag{E-2}$$

With the numerical values for mole fractions and pressure, equation (E-2) becomes

$$.97 \times 101.325 / \exp(13.9778 - 2554.6 / (T - 36.2529)) + 0.03 \times 101.325 / \exp(14.0568 - 2825.42 / (T - 42.7089)) = 1 \quad (E-3)$$

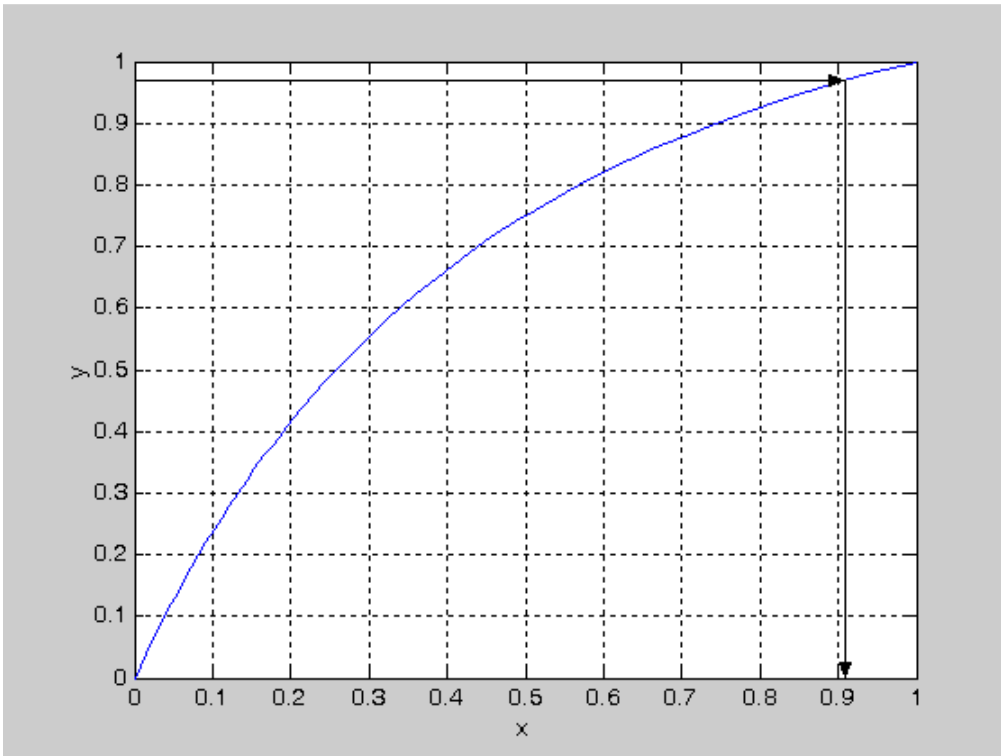


Figure E-1a: Equilibrium curve for n-pentane and n-hexane system.

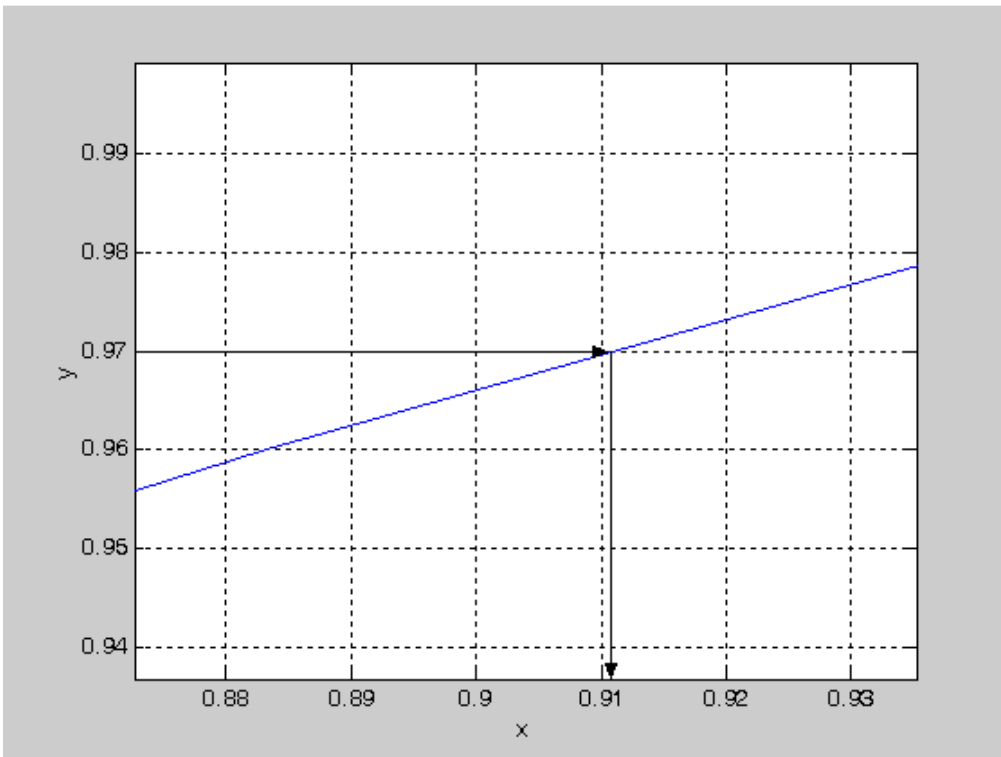


Figure E-1b: Equilibrium curve for n-pentane and n-hexane system near $x = 1$.

The dew point temperature should be between the boiling points of n-pentane and n-hexane given by

$$T_1^{\text{boil}} = \frac{2554.6}{13.9778 - \ln(101.325)} + 36.2529 = 309.20 \text{ K}$$

$$T_2^{\text{boil}} = \frac{2825.42}{14.0568 - \ln(101.325)} + 42.7089 = 342.06 \text{ K}$$

The solution of the nonlinear algebraic equation (E-3) can be determined using Matlab function `fsolve` with inline function as follows:

```
>> fun=inline('0.97*101.325/exp(13.9778-2554.6/(T-36.2529))+
0.03*101.325/exp(14.0568-2825.42/(T-42.7089))-1');
>> T=fsolve(fun,309.5,optimset('Display','off'))
```

T = 311.0479

The dew point temperature of the n-pentane and n-hexane mixture is **311.0479 K**. At this temperature, the vapor pressure of n-pentane is

$$P_1^{\text{sat}} = \exp(13.9778 - 2554.6/(311.0479 - 36.2529)) = 107.9228 \text{ kPa}$$

The mole fraction of n-pentane in the liquid stream leaving state 1 is then

$$x_1 = \frac{y_1 P}{P_1^{\text{sat}}} = \frac{(0.97)(101.325)}{107.9228} = \mathbf{0.9107}$$

Next y_2 is determined from the mass balance or operating line

$$y_2 = \frac{R}{R+1}x_1 + \frac{1}{R+1}x_D = \frac{3}{3+1}x_1 + \frac{1}{3+1}x_D = 0.75x_1 + 0.25x_D$$

$$y_2 = (0.75)(0.9107) + (0.25)(0.97) = 0.9255$$

x_2 is again determined from the equilibrium curve and y_3 is determined from

$$y_3 = \frac{R}{R+1}x_2 + \frac{1}{R+1}x_D$$

This process continues until $x_n < x_F$. The following Matlab program alternately solves the equilibrium relation and material balance for the mole fraction of n-pentane leaving each equilibrium stage:

----- Table 4.4-1: Matlab program e2d4d1b -----

```
% Example 4.4-1
%
A=[13.9778 14.0568];B =[2554.6 2825.42];C=[-36.2529 -42.7089];
xd=.97;xb=.02;xf=.4;R=3;
am=R/(R+1);bm=xd/(R+1);
P=101.325;pl=log(P);
Tb=B./(A-pl)-C;
dT=.01;
yi=xd;
for i=1:20;
    % Assume a temperature for the dew point calculation
    T=yi*Tb(1)+(1-yi)*Tb(2);
    % Solve for the dew point temperature using Newton's method
    for n=1:20;
        f=P*(yi/exp(A(1)-B(1)/(T+C(1)))+(1-yi)/exp(A(2)-B(2)/(T+C(2))))-1;
        T1=T+dT;f1=P*(yi/exp(A(1)-B(1)/(T1+C(1)))+(1-yi)/exp(A(2)-B(2)/(T1+C(2))))-1;
        fp=(f1-f)/dT;eT=f/fp;T=T-eT;
        if abs(eT)<0.001,break, end
    end
    % Solve for the mole fraction in the liquid phase using equilibrium relation for ideal
    system
    xi=yi*P/exp(A(1)-B(1)/(T+C(1)));
    fprintf('Stage #%g x = %8.5f , y = %8.5f\n',i,xi,yi)
    % Solve for the mole fraction in the vapor stream leaving a stage
    yi=am*xi+bm;
    if xi<xf, break, end
end
```

```
>> e2d4d1b
Stage #1 x = 0.91070 , y = 0.97000
Stage #2 x = 0.79889 , y = 0.92552
Stage #3 x = 0.63454 , y = 0.84167
Stage #4 x = 0.46085 , y = 0.71840
Stage #5 x = 0.32841 , y = 0.58814
```