Chapter 5

Separation column: Distillation

The feasbbility of separation of mixtures by distillation, absorption, or stripping depends on the fact that the compositions of vapor and liquid phases are different from each other at equilibrium. The vapor or gas phase is said to be richer in the more volatile or lighter or less soluble components of the mixture. Distillation employs heat to generate vapors and cooling to effect partial or total condensation as needed.

5.1 Design of distillation column

The design of a distillation column can be divided into the following steps:

1 Specify the degree of separation required:set product specification

2 Select the operating conditions: batch or continuous; operating pressure

3 Determine the stage and reflux requirements: the number of requirement stages

4 Size the column: diameter, number of real stages

5 Design the column internals: plates, distributors, packing supports.

6 Mechanical design and internal fittings

7 Select the type of contacting device: plates or packing

The main step will be to determine the stage and reflux requirements.

5.2 Continuous distillation: process description

The separation of liquid mixtures by distillation depends on differences in volatility between the components. The greater the relative volatilities. Vapor flows up the column and liquid counter-currently down the column. Part of the condensate from the condenser is returned to the top of the column to provide liquid flow above the feed point (reflux), and part of the liquid from the base of the column is vaporised in the reboiler and returned to provide the vapor flow.

Above the feed, the more volatile components are stripped from the liquid and this is known as **stripping section**. The more volatile components above the feed is called the **rectifying section**.

If the process required to strip a volatile component from a relatively non-volatile solvent, the rectifying section will be omitted, and the column would then be called a **stripping column**.

5.2.1 Reflex consideration

The reflex ratio, R, is normally defined as:

$$R = \frac{\text{flow returned as reflux}}{\text{flow of top product taken off}} \tag{5.1}$$

The number of stages required for a given separation will be dependent on the reflux ratio used.

In an operating column, the effective reflux ratio will be increased by vapour condensed within the column due to heat leakage through the wall.

Note:

If the column is not well insulated, changes in the internal reflux due to the sudden changes in the external conditions, such as sudden rain storm, can have noticeable effect on the column operation and condition.

Total reflux

Total reflux is the condition when all the condensate is returned to the column as reflux: no product is taken off and there is no feed.

At total reflux, the total number of stages for a given separation will be the minimum.

Minimum reflux

As the reflux is reduced, the pinch point will occur at which the separation can only be achieved with infinite number of stages.

Optimum reflux ratio

Practical reflux ratios comes between the minimum and the total reflux. The optimum value include the performance and the cost. Increasing the reflux will reduce the number of stages and hence the capital cost, but it will increase the cost of maintenance and services.

Note:

For many systems, the optimum number of stages will be 1.2 to 1.5 times the minimum reflux ratio.

5.2.1.1 Feed point location

The precise location of the feed point will affect the number of stages required for a specified separation and the subsequent operation of the column. The feed should enter the column at the point that gives the best match feed composition and the vapour and liquid streams in the column.

5.2.1.2 Selection of column pressure

When distilling heat-sensitive materials, the main consideration when selecting column operating-pressure will be to ensure that the dew point of the distillate is above that which can be easily obtained with plant cooling water.

Vacum operation is used to reduce the column temperatures for the distillation of heat sensitive materials.

High operation temperature is used when it is required to distil relatively non-volatile materials.

5.2.2 Continuous distillation: basic principles

5.2.2.1 Stage equation

Figure 5.1 shows the material flows into and out of a typical stage n in a distillation column. Material and energy balance are given for any typical stage and component i:

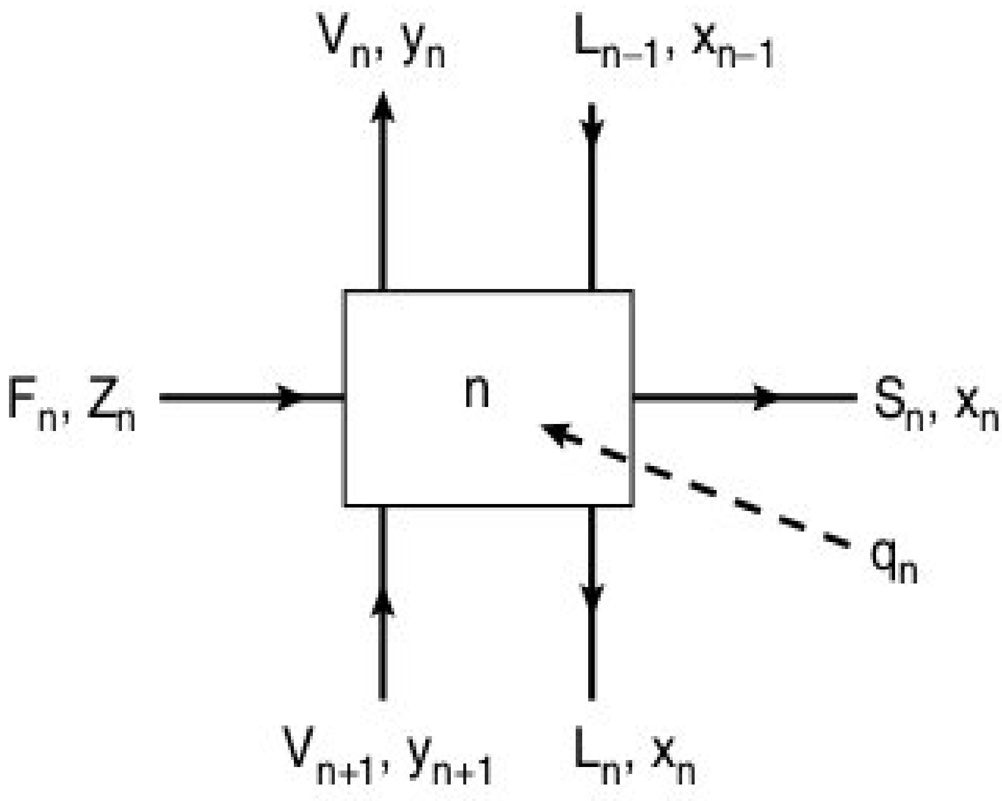

Figure 5.1: Stage flows

Equilibrium, **S**ummation and **H**eat (energy) balance, equations (MESH equations) for the stage:

1-The material balance :

$$V_{n+1}y_{n+1} + L_{n-1}x_{n-1} + F_n z_n = V_n y_n + L_n x_n + S_n x_n \tag{5.2}$$

2-The energy balance:

$$V_{n+1}H_{n+1} + L_{n-1}h_{n-1} + Fh_f + q_n = V_n H_n + L_n h_n + S_n h_n \tag{5.3}$$

$V_n=$ vapour flow from the stage

$V_{n+1}=$ vapor flow into the stage from the stage below

$L_n=$ Liquid flow from the stage

$L_{n-1}=$ lquid flow into stage from the stage above

$F_n=$ any feed flow into the stage

$S_n=$ any side stream from the stage

$q_n=$ heat flow into, or removal, from the stage

$n=$ any stage, numbered from the op of the column

$z=$ mol fraction of component i in the feed stream (note: feed may be two-phase)

$x=$ mol fraction of component i in the liquid streams

$y=$ mol fraction component i in the vapour phase

$H=$ specific enthalpy vapour phase

$h=$ specific enthalpy liquid phase

$h_f=$ specific enthalpy feed (vapour +liquid)

3- In terms of equilibrium constants:

$$y_i = Kx_i \tag{5.4}$$

$$\sum x_{i,n} = \sum y_{i,n} = 1 \tag{5.5}$$

MESH equations can be written for each stage, and for the reboiler and condenser. The solution of this set of equations forms the basis of the rigorous methods that have been developed for the analysis for staged separation processes.