

DISTILLATION & HYDROCARBON PROCESSING PRACTICES

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Preface

This book is intended primarily for practicing process engineers and students of chemical engineering.

I have noticed that some process principles, their details, and design considerations are often not well-documented for use by the engineers engaged in process design and troubleshooting. My role in both process engineering as well as plant operation made me realize that such pieces of knowledge lie scattered over different textbooks, research papers, design codes, safety codes, and individual notes of experienced engineers and operators.

This work is my humble attempt to compile and elucidate the practices in the hydrocarbon industry and their fundamental principles in a concise form. The advanced practices in distillation are explained and illustrated with the help of examples, using process simulations along with the basic principles. The practical examples and design guidelines are based on all the knowledge and experience I have gathered in my professional career and from various other resources, including personal interaction with masters in the field.

I sincerely hope that this work shall add value to your academic and professional pursuits.

Heat balance with an inter-reboiler (Fig. 1.1.2) can be written as $Q_R + Q_F + Q$ inter-reboiler = $Q_D + Q_B + Q_C$. Thus, reboiler duty with an inter-reboiler will be less than reboiler duty without an inter-reboiler. Similarly, when an intercooler is added above the feed tray, the heat balance can be written as $Q_R + Q_F = Q_D + Q_B + Q_C + Q$ intercooler. Thus the condenser duty with an intercooler will be less than the condenser duty without an intercooler.

1.1.2 McCabe–Thiele method

The McCabe–Thiele method provides insight for the analysis of many distillation problems. The basic assumption of the McCabe–Thiele is equal molar heat of vaporization and condensation of components, and it reduces to equal molar liquid–vapor rates in all stages in the sections above the feed stage and equal molar liquid and vapor flow rates in all the stages below the feed stage. Fig. 1.1.3 shows the enrichment section and stripping section envelopes used to derive the enrichment section equation and stripping section equation.

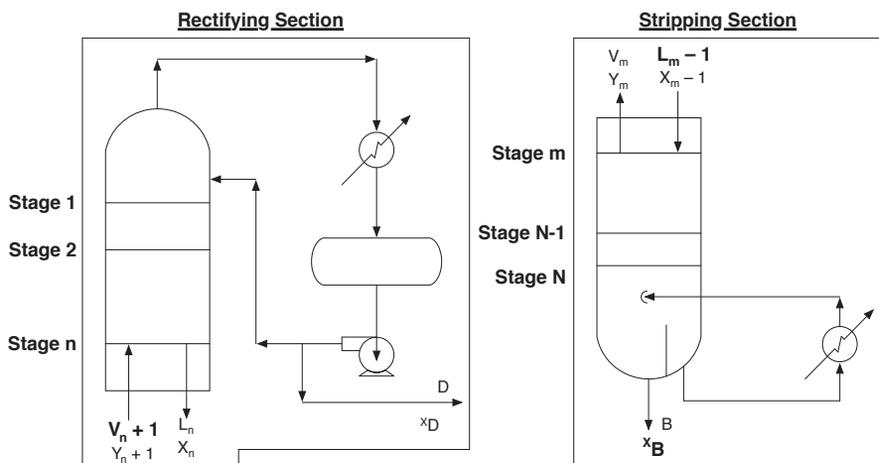


Fig. 1.1.3 Envelopes of the rectification and stripping section

Enrichment line equation

The material balance equation on the enrichment section is $V = L + D$.

$Y_{n+1} = (L/(L + D))X_n + (D/(L + D))X_D$ is the equation of the enrichment line for n^{th} stage.

The enrichment line equation can be rewritten as $Y_{n+1} = (R/(R + 1))X_n + (1/(R + 1))X_D$ with substitution of $R = L/D$ in the equation. Thus, $(R/(R + 1))$ is the slope and $(1/(R + 1))X_D$ is the intercept of the enrichment line and can be plotted on an X-Y chart.

The reason for the increase in reflux ratio is further examined using the McCabe–Thiele method.

Analysis of higher reflux requirement with increase of feed preheat with the McCabe–Thiele method

The phenomenon of the increase in reflux ratio with the increase in feed vaporization as seen above is further analyzed using the McCabe–Thiele method with the help of an example problem. The same problem as above (Problem 1.3.1) is worked out in a 15-stage column with feed stage 9.

Feed = 200 kmoles/hr (50:50 mole:mole benzene:toluene), benzene purity in top product is 95 mole %. Toluene purity in bottom product is 90 mole %.

Overall material (mole) balance (from earlier pages):

$$F = D + B \text{ or } 200 = D + B \quad (1)$$

Component (benzene) balance:

$$200 \times 0.5 = D \times 0.95 + B \times 0.1 \quad (2)$$

$$D = 94.1 \text{ kmoles/hr, } B = 200 - 94.1 = 105.9 \text{ kmoles/hr}$$

To further analyze the effect of feed condition on minimum reflux, the McCabe–Thiele method is used. The limiting reflux is calculated for three feed conditions: feed as a saturated liquid, feed 25% vaporized, and feed 40% vaporized.

Limiting reflux calculation using the McCabe–Thiele method for three conditions of feed

Feed as a saturated liquid

For saturated liquid where $q = 1$, the slope of the q line is ∞ . The vertical q line is drawn and the intersection with the equilibrium line is marked. The enrichment line and stripping line are drawn through the point where the q line and the equilibrium line intersect.

Intercept from the chart (similar to Fig. 1.1.5) for enrichment line = $0.42 = X_D \times D / (L + D)$.

Again, $V = L + D$; thus, $L = 118.7$ kmoles/hr and $V = 212.8$ kmoles/hr, and

$$V' = V - (1 - q)F \text{ or } V' = 212.8 - (0 \times 200) = 212.8 \text{ kmoles/hr.}$$

Feed 25% vaporized

Limiting reflux in the McCabe–Thiele method is calculated from the slope of the rectification line passing through the intersection of the q line and the equilibrium line. From the chart (Fig. 1.1.5), the limiting value of the intercept is 0.4 when the feed is 25% vaporized.

Fig. 1.5.4B shows that once the feed is split, the separation improves, and the reboiler duties, condenser duties, and reflux ratio(s) come down. The above concept (two-enthalpy feed) is further applied to a three-component distillation system: a benzene–toluene–*para*-xylene system.

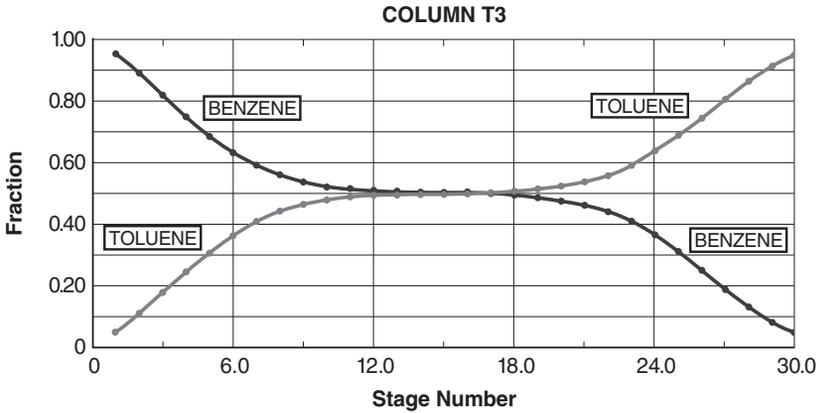


Fig. 1.5.4A Benzene–toluene liquid composition through the stages with a single feed

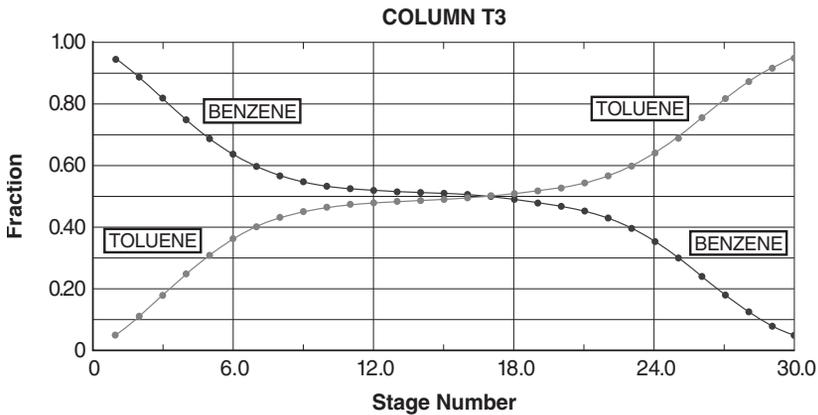


Fig. 1.5.4B Benzene–toluene liquid composition through the stages with a split feed

Some industrial applications of overhead heat coupling are shown below.

An overhead integration of a xylene column in a *para*-xylene complex is shown in Fig. 1.8.5.

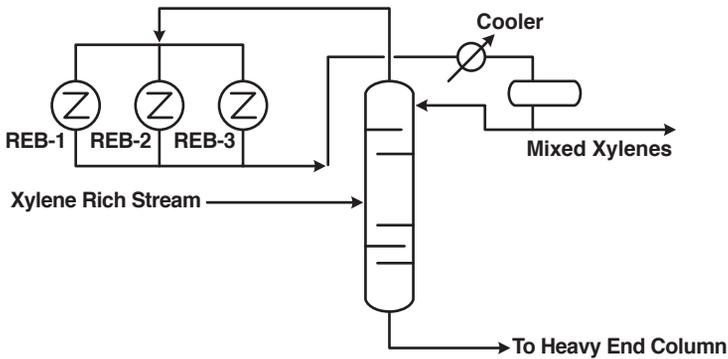


Fig. 1.8.5 Xylene column with overhead integration

In the scheme in Fig. 1.8.5, the overhead of the xylene column is supplying reboiler heat to various columns in the plant. The xylene fractionator is typically operated at about $7 \text{ kg/cm}^2(\text{g})$ to raise the overhead vapor dew point and bubble point temperatures so that overhead heat can be delivered to the reboilers of different columns.

A butene-1 plant with heat coupling with the reboiler of the first column is shown in Fig. 1.8.6.

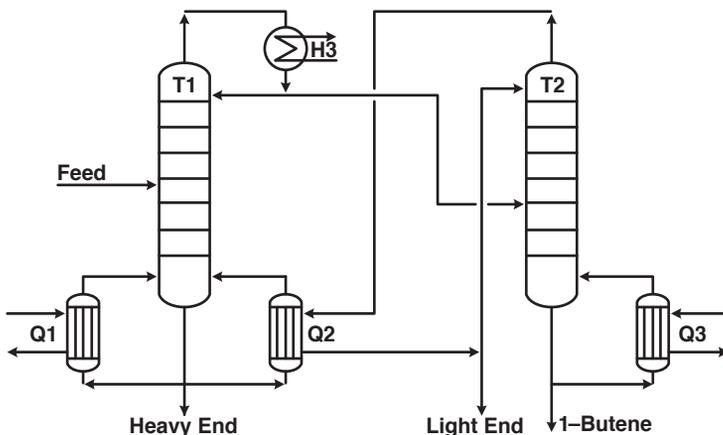


Fig. 1.8.6 Butene-1 plant with heat coupling with the reboiler of the first column

The difficulty with this scheme is to ensure the flow of vapor from one tower to the other when the both are operating at a similar pressure. Liquid can be introduced from one tower to the other by pumps, but vapor flow would need ejectors or compressors.

d) Modified Petlyuk schemes

The classic Petlyuk scheme has necessitated modifications for easier application. This particular modification uses two sequential towers. The first one can operate at higher pressure, thus ensuring the flow of vapor from first column to the second while pumps provide the flow of liquid from the second tower to the first. The reboiler is provided in the first column and the condenser in the second column. This modified Petlyuk scheme is shown in Fig. 1.9.8.

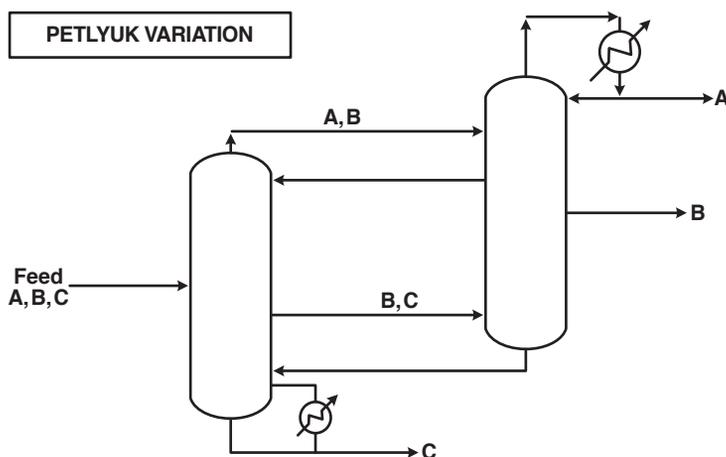


Fig. 1.9.8 Modified Petlyuk scheme

1.9.3 Benefits of using a TCDS

The TCDS is explained using the previous example of the benzene–toluene–*para*-xylene system and are compared with a sharp-cut distillation scheme.

Conventional scheme (sharp-cut)

The B–T–PX system is conventionally operated with two columns (sharp-cut). Benzene of the required purity is recovered as the top product of first column. The bottom of the first column is then fed to the second column. Toluene is recovered from the second column top with the required purity, and *para*-xylene is recovered as the bottom product of the second column.

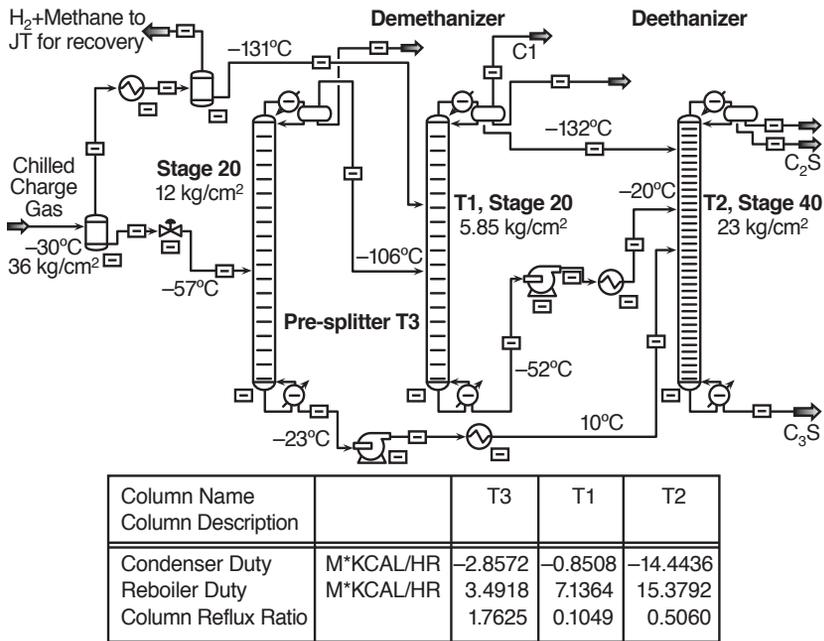


Fig. 1.9.19 Simulation of alternative scheme in DM, DE with a pre-splitter of 20 stages

Table 1.9.3 shows that the demethanizer is partly relieved by the installation of a pre-splitter 10 stages upstream. The pre-splitter operates at 12 kg/cm² pressure, an overhead temperature of -86°C, and a bottom temperature of -24°C. Thus, the demethanizer in the scheme (Fig. 1.9.18) requires a refrigerant of lower quality than in the conventional scheme. The demethanizer condenser and reboiler duties and the reflux ratio reduce marginally, though the total reboiler duty of the pre-splitter and the demethanizer increases.

Table 1.9.3 shows that the deethanizer load reduces significantly with the scheme shown in Fig. 1.9.19. In the scheme, the pre-splitter bottom product is fed to the deethanizer with the same minimum specification of methane as the demethanizer in the conventional scheme. The pre-splitter bottom is heated from -23°C to +10°C before it is fed to the deethanizer. Similarly, the demethanizer bottom is also heated from -52°C to -20°C before it is fed to the deethanizer at the upper location.

The colder feed is introduced at the upper location and warmer one to the lower location. The cold feed can be utilized for cooling any stream. In the scheme, the pre-splitter overhead drum temperature is -106°C and the pre-splitter bottom temperature is -23°C. The demethanizer load below the feed tray reduces significantly as a result of pre-splitting. Thus, the scheme can

1.12 Extractive Distillation

1.12.1 Introduction

Extractive distillation (ED) is often performed when the relative volatility of the keys are close to unity (1.0). A third component can be added to modify the relative volatility of the light or heavy key so that the key is separated as almost pure component. The other key remains, along with the third component added. This mixture is then sent to another column that separates the key and the third component.

In the alcohol–water system above (Fig. 1.11.7), the third component used is ethylene glycol, which alters the relative volatility of EtOH (one key) and separates pure EtOH from the top and a mixture of water and EG from the bottom (see Fig. 1.11.7). The water and EG phase is fed to the next column where it is separated, with water as the top product and EG as the bottom product. In the above depiction (Fig. 1.11.7) of an EtOH–water separation, EG is used as the extractive distillation solvent. Often in extractive distillation, salts are used as the entrainer to make the separation more energy efficient; for example, potassium acetate is used as the entrainer for ethanol–water systems.

The third component actually modifies the activity coefficients of the keys to make one more volatile and the other less volatile. The third component alters the relative volatility of a two-component system when the components have different polarity. If the third component (the entrainer) is highly polar, the activity coefficient of the polar component in the mixture will be close to unity (one). The other nonpolar (less polar) component will have a higher activity coefficient. Conversely, if a nonpolar entrainer is used for the same mixture of components, it will increase the activity coefficient of the polar component.

A very high proportion of entrainer (third component) is normally used to raise the relative volatility of one key in respect to the other key. The high proportion of entrainer used in the process allows the activity coefficient of the key components to approach the activity coefficient at infinite dilution.

Correlation of activity coefficient (γ) and relative volatility (α)

$$\text{Partial pressure: } (p_i) = \gamma_i \times P_i^s X_i = Y_i P_t \text{ or } Y_i P_t = \gamma_i P_i^s X_i \\ \text{or } Y_i = \gamma_i (P_i^s / P_t) \times X_i = \gamma_i K_i X_i$$

$$\text{Relative volatility: } \alpha_{ij} = (Y_i / X_i) / (Y_j / X_j) = (\gamma_i / \gamma_j) (P_i^s / P_j^s)$$

P_i, P_j = vapor pressures of i th and j th components

P_t = total pressure