

5 Batch Distillation

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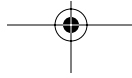
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5.1 INTRODUCTION

Distillation has been widely accepted for product separation, purification, and waste removal in chemical process industries. Depending on whether the industry is handling petrochemicals, bulk chemicals, specialty chemicals, or pharmaceuticals, the distillation process can be divided into two categories: (1) batch distillation, which is mainly used in specialty chemical, biochemical, and pharmaceutical industries; and (2) continuous distillation, which is primarily implemented in the petrochemical and bulk chemical industries. Figure 5.1a shows a conventional batch distillation column where the feed is initially charged into the reboiler at the beginning of operation. After a total reflux operation (i.e., all condensates are recycled to the column), the distillate is continuously withdrawn while the bottom residue with a high-boiling-temperature component is concentrated, making this a time-varying process. In continuous distillation (Figure 5.1b), the feed is constantly supplied to the column, and the top and bottom products are simultaneously obtained under a steady-state operation. The upper section of the feed point is referred to as the *rectifying section*, as a low-boiling-temperature component is enriched. The lower section is referred to as the *stripping section*, as a low-boiling-temperature component is stripped off.

Batch distillation is the oldest separation process and the most widely used unit operation in the batch industry. Batch distillation is highly preferable to continuous distillation when high-value-added, low-volume chemicals must be separated. It is also widely used in chemical processing industries where small quantities of materials are to be handled in irregularly or seasonally scheduled periods, and it is implemented when the feed composition varies widely from period to period or where completely different feed stocks have to be handled.

Theoretical studies on batch distillation began with a simple distillation still in a laboratory. In this type of distillation, a still is initially filled with a feed mixture, which evaporates and leaves the still in the vapor form. This vapor, which is richer in the more volatile component, is collected in the condenser at the top and accumulated in a receiver. In this operation, no liquid is refluxed back to the still, and no plates or packing materials are present inside the still. This simple distillation still is an example of a batch operation, often referred to as *Rayleigh distillation*¹ because of Rayleigh's pioneering theoretical work in simple distillation. The concept of reflux and the use of accessories such as plates and



Batch Distillation

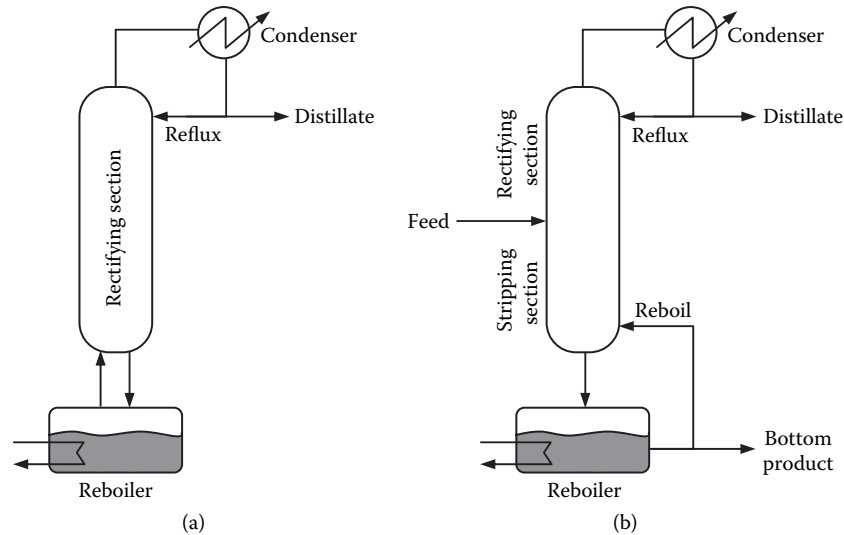


FIGURE 5.1 Types of distillation processes: (a) batch distillation, and (b) continuous distillation.

packing materials to increase the mass transfer converts this simple still into a batch distillation column, as shown in Figure 5.1a. Because this batch column essentially performs the rectifying operation, it is often referred to as a batch *rectifier*.

The most outstanding feature of batch distillation is its flexibility in operation. This flexibility allows one to deal with uncertainties in feed stocks or product specifications. In addition, one can handle several mixtures just by switching the operating conditions of the column. The basic difference between batch distillation and continuous distillation is that in continuous distillation the feed is continuously entering the column, while in batch distillation the feed is charged into the reboiler at the beginning of the operation. The reboiler in batch distillation gets depleted over time, so the process has an unsteady-state nature. A conventional batch column can be operated under the following operating conditions or policies:

- Constant reflux and variable product composition
- Variable reflux and constant product composition of the key component
- Optimal reflux and optimal product composition

Under conditions of constant reflux, the instantaneous composition of the distillate keeps changing because the bottom still composition of the more volatile component is continuously depleted. On the other hand, under variable reflux, the composition of the key component in the distillate can be kept constant by increasing the reflux ratio. The third type of operation, known as optimal reflux,

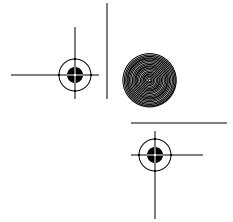


is neither constant nor variable; instead, this type of operation exploits the difference between the two operating modes. Thus, the optimal reflux policy is essentially a trade-off between the two operating modes, and is based on the ability of the process to yield the most profitable operation.

The flexible and transient nature of batch distillation allow us to configure the column in a number of different ways, some of which are shown in Figure 5.2.² The column in Figure 5.2a is a conventional batch distillation column, with the reboiler at the bottom and condenser at the top. A single column can be used to separate several products using the multifraction operation of batch distillation presented in Figure 5.2b. Some cuts may be desired and others may be intermediate products. These intermediate fractions can be recycled to maximize profits or minimize waste generation. Figure 5.2c shows a periodic operation in which each charge consists of a fresh feed stock mixed with recycled off-specification material from the previous charge. Figure 5.2d represents a stripping column for separating a heavy component as the bottom product where the liquid feed is initially charged into the top. In 1994, Davidyan et al.³ presented a batch distillation column that has both stripping and rectifying sections embedded in it (Figure 5.2e). Although this column has not been investigated completely, recent studies demonstrated that it provides added flexibility for the batch distillation operation. Recently, Skogestad et al.⁴ described a new column configuration referred to as a *multivessel column* (Figure 5.2f) and showed that the column can obtain purer products at the end of a total reflux operation. These emerging column designs play an important role in separations of complex systems such as azeotropic, extractive, and reactive batch distillation systems. The batch rectifier configuration for such separations may be very restrictive and expensive.

These emerging designs, combined with different possible operating modes similar to the ones described earlier for the rectifier, provide greater flexibility but result in a large number of column configurations. Because of the unsteady-state nature of the operation, embedded in the design problem is the optimal control problem of deciding time-dependent variables such as reflux ratios, reboil ratios, vapor flow rates, and vessel holdups. Given this flexibility, batch distillation poses a difficult synthesis problem involving the selection of optimal column configurations and optimal operating conditions. Complex systems, such as azeotropic, extractive, and reactive batch distillation systems, add another dimension to the synthesis problems as the cuts (fractions) in the multifraction operation can have significantly different characteristics depending on the feed mixture of these systems. The complexity in design, synthesis, and analysis of batch distillation due to the (1) unsteady-state nature, (2) operational flexibility, and (3) emerging column design can only be handled systematically using computer-aided design techniques and recently developed software tools.

This chapter presents a complete review of batch distillation starting from the first analysis in 1902 by Rayleigh to the current state-of-the-art, computer-aided design techniques. The chapter introduces an early theoretical analysis of simple distillation and various operating policies in Section 5.2. Section 5.3 examines the challenges involved in rigorous modeling of batch distillation



Batch Distillation

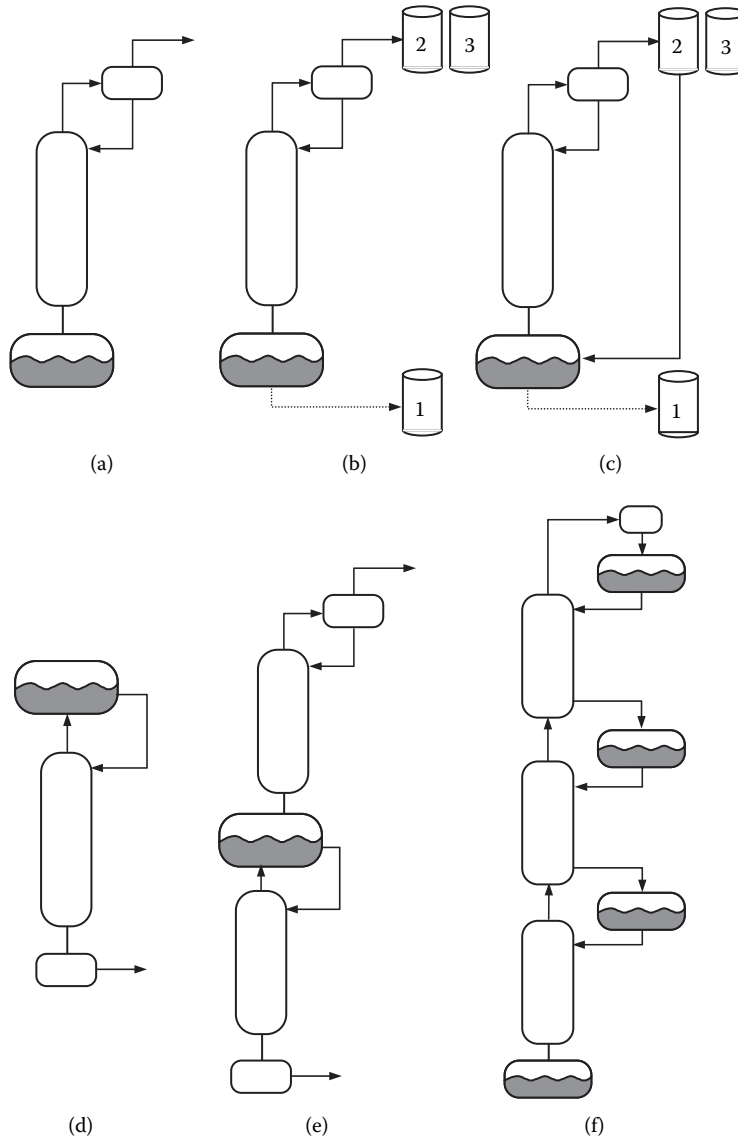
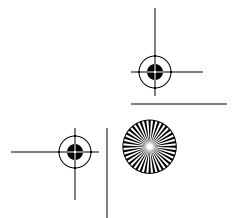
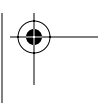


FIGURE 5.2 Examples of ways to configure the batch distillation column.

dynamics and provides a hierarchy of models of varying complexity and rigor. Recent advances in optimal design and control problems are discussed in Section 5.4. Emerging columns, complex systems, and batch synthesis are described in Section 5, followed by an overview of available software packages. The last section provides overall conclusions and addresses the direction of future research.





5.2 EARLY THEORETICAL ANALYSIS

This section presents early theoretical analysis of simple distillation, which was first analyzed by Rayleigh.¹ The limitations of simple distillation that led to the development of the batch rectifier are discussed, as is the operational flexibility of batch distillation with regard to the type of operation.

5.2.1 SIMPLE DISTILLATION

The analysis of simple distillation presented by Rayleigh in 1902 marks the earliest theoretical work on batch distillation. Simple distillation, also known as *Rayleigh distillation* or *differential distillation*, is the most elementary example of batch distillation. In this distillation system, the vapor is removed from the still during a particular time interval and is condensed in the condenser. The more volatile component is richer in the vapor than in the liquid remaining in the still. Over time, the liquid remaining in the still begins to experience a decline in the concentration of the more volatile component, while the distillate collected in the condenser becomes progressively more enriched in the more volatile component. No reflux is returned to the still, and no stages or packing materials are provided inside the column; therefore, the various operating approaches are not applicable to this distillation system.

The early analysis of this process for a binary system, proposed by Rayleigh is given below. Let F be the initial binary feed to the still (mol) and x_F be the mole fraction of the more volatile component (A) in the feed. Let B be the amount of compound remaining in the still, x_B be the mole fraction of component A in the still, and x_D be the mole fraction of component A in the vapor phase. The differential material balance for component A can then be written as:

$$x_D dB = d(Bx_B) = B dx_B + x_B dB, \quad (5.1)$$

giving:

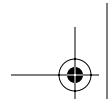
$$\int_F^B \frac{dB}{B} = \int_{x_F}^{x_B} \frac{dx_B}{x_D - x_B}, \quad (5.2)$$

or:

$$\ln\left(\frac{B}{F}\right) = \int_{x_F}^{x_B} \frac{dx_B}{x_D - x_B}. \quad (5.3)$$

In this simple distillation process, it is assumed that the vapor formed within a short period is in thermodynamic equilibrium with the liquid; hence, the vapor composition (x_D) is related to the liquid composition (x_B) by an equilibrium





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relation of the form $x_D = F(x_B)$. The exact relationship for a particular mixture may be obtained from a thermodynamic analysis depending on temperature and pressure. For a system following the ideal behavior given by Raoult's law, the equilibrium relationship between the vapor composition y (or x_D) and liquid composition X (or x_B) of the more volatile component in a binary mixture can be approximated using the concept of constant relative volatility (α), which is given by:

$$y = \frac{\alpha x}{(\alpha - 1)x + 1} \quad (5.4)$$

Substitution of the above equation in Equation 5.3 results in:

$$\ln\left(\frac{B}{F}\right) = \frac{1}{\alpha - 1} \ln\left[\frac{x_B(1 - x_F)}{x_F(1 - x_B)}\right] + \ln\left[\frac{1 - x_F}{1 - x_B}\right] \quad (5.5)$$

Although the analysis of simple distillation historically represents the theoretical start of batch distillation research, a complete separation using this process is impossible unless the relative volatility of the mixture is infinite. Therefore, the application of simple distillation is restricted to laboratory-scale distillation, where high purities are not required, or when the mixture is easily separable.

Example 5.1

A mixture of components A and B with 0.6 mole fraction of A and relative volatility of 2.0 is distilled in a *simple* batch distillation column. The feed is 133 mol, and 29.3% of the mixture is distilled. Find the distillate composition (derived from Converse and Gross⁵).

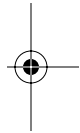
Solution

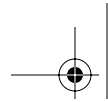
Because 29.3% of the feed is distilled, the residue amount is 94.031 mol. The bottom composition can be found using Equation 5.5:

$$\ln\left(\frac{94.031}{133}\right) = \frac{1}{2 - 1} \ln\left[\frac{x_B(1 - 0.6)}{0.6(1 - x_B)}\right] + \ln\left[\frac{1 - 0.6}{1 - x_B}\right] \Rightarrow x_B = 0.4793$$

Then the distillate composition ($x_D = y$) can be obtained from Equation 5.4, resulting in a distillate composition of 0.6480.

Because this distillate composition is quite low for separation purposes, simple batch distillation cannot be used in real practice. To obtain products with high purity, multistage batch distillation with reflux has been used. As seen in Figure 5.1a, the batch rectifier is comprised of multiple thermodynamic stages (manifested by internal trays or packings) inside the rectifying section. The feed is normally charged to the reboiler at the beginning of the operation. Although





the top products are removed continuously, no bottom product withdrawal occurs in batch distillation, and the reboiler becomes depleted over time. This makes batch distillation an unsteady-state but flexible operation.

5.2.2 OPERATING MODES

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The two basic modes of batch distillation are (1) constant reflux and (2) variable reflux, resulting in variable distillate composition and constant distillate composition, **respectively**. The third operating model of a batch distillation, optimal reflux or optimal control, is neither constant nor variable but is between the two. Similar operating modes are also observed in the emerging batch distillation columns. For example, a stripper can also have three operating modes: (1) constant reboil ratio, (2) variable reboil ratio, and (3) optimal reboil ratio. For a middle vessel column, the combination of the three reflux and three reboil modes results in at least nine possible operating policies. The operating modes of a multivessel column can be derived based on the middle vessel column, but this column configuration requires additional considerations with respect to operating variables such as the holdup in each vessel. The total reflux mode can be also considered especially in the middle vessel and multivessel columns. As these column designs are still under extensive research, early analyses of operating modes are mainly restricted to the batch rectifier and are discussed below.

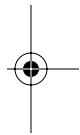
5.2.2.1 McCabe–Thiele Graphical Method

The difference between simple distillation and batch distillation operations is the relation between the distillate composition (x_D) and the bottom composition (x_B) due to the presence of reflux and column internals. The graphical analysis presented by McCabe and Thiele⁶ for continuous distillation provided the basis for analyzing batch distillation operating modes. They suggested a graphical method to calculate this relation using the following procedure. In the McCabe and Thiele method, the overall material balance with no holdup is considered from the condenser to the j th plate. This leads to the following operating equation:

$$y_j = \frac{R}{R+1} x_{j-1} + \frac{1}{R+1} x_D. \quad (5.6)$$

This operating equation represents a line through the point $y_j(x_{j+1} = x_D)$ with a slope of $R/(R+1)$. Starting from this point (x_D, x_D), which corresponds to the distillate composition, Equation 5.6 and the equilibrium curve between y_j and x_j can be recursively used from top plate 1 to the reboiler (the reboiler can be considered as the $(N+1)$ th plate). This procedure relates the distillate composition (x_D) to the still composition (x_B) through the number of stages.

In the case of batch distillation, however, the still composition (x_B) does not remain constant, as observed in continuous distillation, thus the instantaneous distillate composition (x_D) is also changing. This necessitates using the recursive





scheme several times. If this scheme is used while keeping the reflux ratio constant throughout the operation, just like normal continuous distillation, the composition of the distillate keeps changing. This is the constant reflux mode of operation. On the other hand, the composition of the key component in the distillate can be maintained constant by changing the reflux, resulting in the variable reflux mode of operation. The third mode of operation of batch distillation, optimal reflux or optimal control, is designed to optimize a particular perk mode such as maximum distillate, minimum time, or maximum profit functions.

5.2.2.2 Constant Reflux Mode

Smoker and Rose⁷ presented the first analysis of the constant reflux operation of a binary batch distillation with no holdup. They used the Rayleigh equation in conjunction with the McCabe–Thiele graphical method to capture the dynamics of the batch distillation column. In their procedure, the relationship between x_D and x_B is recursively determined by the McCabe–Thiele graphical method, then, the right-hand side of the Rayleigh equation (Equation 5.3) is integrated graphically by plotting $1/(x_D - x_B)$ vs. x_B . The area under the curve between the feed composition (x_F) and the still composition (x_B) now gives the value of the integral, which is $\ln(B/F)$. The average composition of the distillate can be obtained from the following equation:

$$x_{D,avg} = \frac{F x_F - B x_B}{F - B}. \quad (5.7)$$

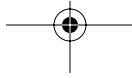
Although Smoker and Rose presented the calculation method independent of time, time can be introduced through the vapor boilup rate (V) of the reboiler. The resulting equation for determining batch time is given by:

$$T = \frac{R+1}{V} (F - B) = \frac{R+1}{V} D. \quad (5.8)$$

This operation policy is easy to implement and is commonly used.

Example 5.2

We have seen in Example 5.1 that the purity obtained by simple distillation is not satisfactory. Let us add four stages and make this a batch distillation column operating under constant reflux of 1.82. Using the McCabe–Thiele graphical method, find the distillate and still composition when 29.3% of the feed mixture is distilled. What is the average distillation composition? If the feed is 133 mol and the vapor boilup rate is 110 mol/hr, what is the total time required to complete the distillation operation and what is the average distillate composition?



Solution

From Equation 5.3, we have:

$$\ln\left(\frac{B}{F}\right) = \ln\left(\frac{0.707}{1}\right) = -0.3467 = \int_{x_B}^{x_F} \frac{dx_B}{x_D - x_B}$$

For various values of x_D , operating lines (Equation 5.6) are drawn to obtain the x_B values using the McCabe–Thiele graphical method (see Figure 5.3a). Then, values of x_B vs. $1/(x_D - x_B)$ are plotted in Figure 5.3b, where the area under the curve is equal to the right-hand side of the Rayleigh equation:

$$\int dx_B / (x_D - x_B)$$

The operation is stopped when the integral is -0.3467 , which is equivalent to $\ln(B/F)$. From Figure 5.3b, $x_{B,final}$ is 0.4755, which satisfies the above integral. The average distillate composition becomes:

$$x_{D,avg} = \frac{F x_F - B x_B}{F - B} = \frac{133 \times 0.6 - 94.031 \times 0.4755}{133 - 94.031} = 0.9001$$

Even though the bottom compositions are similar, the distillate composition of batch distillation with multiple stages and reflux is significantly increased from 0.6480 to 0.9001. The time required for the distillation, as given by Equation 5.8, is 0.999 hr.

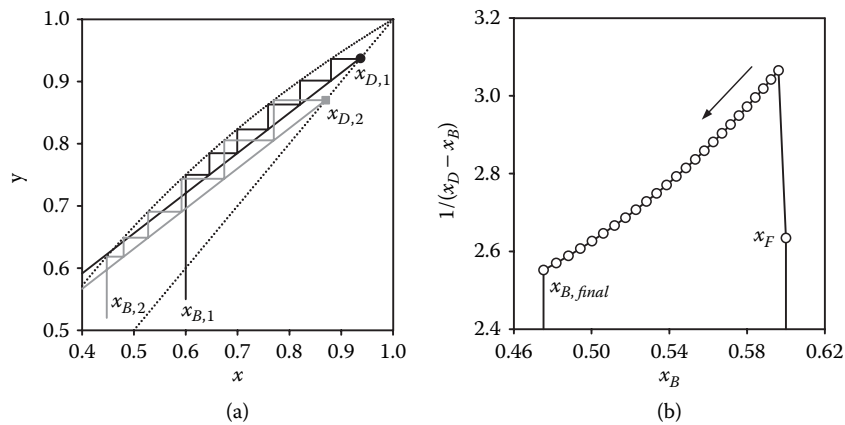


FIGURE 5.3 (a) McCabe–Thiele method for plate-to-plate calculations, and (b) graphical integration of the right-hand side of the Rayleigh equation.



5.2.2.3 Variable Reflux Mode

In 1937, Bogart⁸ presented the first analysis of the variable reflux policy for a binary system. The steps involved in calculating the variable reflux mode are similar to those in the case of the constant reflux mode; however, for variable reflux, the reflux ratio is varied instead of the distillate composition at each step. Moreover, the Rayleigh equation, though valid for the variable reflux condition, takes a simplified form. Because the distillate composition remains constant (remember that we are considering binary systems here) throughout the operation, the Rayleigh equation reduces to the following equation:

$$\frac{B}{F} = \frac{x_D - x_F}{x_D - x_B}. \quad (5.9)$$

The second step is to establish the relation between R and x_B using the McCabe–Thiele graphical method. Several values of R are selected, operating lines are drawn through the fixed point (x_D, x_D) with slope $R/(R + 1)$, and steps are drawn between the operating line and the equilibrium curve to obtain the bottom composition (x_B) . This recursive scheme is repeated until the desired stopping criterion is met, thus B and x_B can be found at each value of the reflux ratio. The time required for this operation at a given product purity is calculated by plotting the quantity $(R + 1)V \times \{F(x_D - x_F)\} / (x_D - x_B)^2$ vs. x_B in the following equation and then finding the area under the curve:

$$T = \int_{x_B}^{x_F} \frac{R + 1}{V} \frac{F(x_D - x_F)}{(x_D - x_B)^2} dx_B. \quad (5.10)$$

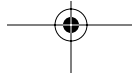
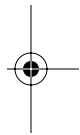
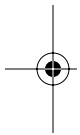
The variable reflux operation policy is commonly used with a feedback control strategy because the reflux ratio is constantly adjusted to keep the distillate composition constant. Section 5.4.2 presents a detailed description of the control strategy involved in this operating mode.

Example 5.3

Rework the problems in Example 5.2 for the variable reflux mode. For the various iterations of R , use the following 10 reflux ratios: 0, 1.3343, 1.4057, 1.5091, 1.6234, 1.7498, 1.9283, 2.0902, 2.2718, and 2.5926.

Solution

Because the type of operation is variable reflux mode (Figure 5.4), the distillate composition is held constant at $x_D = x_{D,avg} = 0.9001$. The bottom composition can be obtained from the McCabe–Thiele graphical method, while the distillate can be obtained from the Rayleigh equation for the variable reflux condition (Equation 5.9):



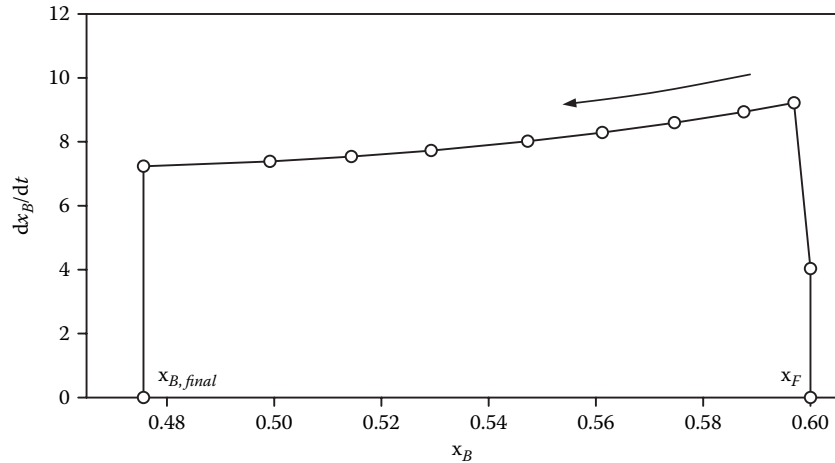


FIGURE 5.4 Graphical integration for batch time under the variable reflux mode.

$$D = F - B = F \left(1 - \frac{x_D - x_F}{x_D - x_B} \right).$$

Then the resulting x_B and D are:

| | | | | | | | | | | |
|-------|-----|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| R | 0 | 1.3343 | 1.4057 | 1.5091 | 1.6234 | 1.7498 | 1.9283 | 2.0902 | 2.2718 | 2.5926 |
| x_B | 0.6 | 0.59694 | 0.58757 | 0.57463 | 0.56119 | 0.54728 | 0.52926 | 0.51440 | 0.49920 | 0.47550 |
| D | 0 | 1.3421 | 5.2893 | 10.3663 | 15.2282 | 19.8725 | 25.3683 | 29.5152 | 33.4386 | 38.969 |

The operation is stopped when the amount of distillate is greater than or equal to 38.969 mol, when the bottom composition at this condition is 0.4755. The time required for the distillation is given by Equation 5.10. By plotting the quantity $dx_B/dt = (R + 1)V \times \left\{ F(x_D - x_F) \right\} / (x_D - x_B)^2$ vs. x_B , the area under the curve between x_B equal to 0.6 and 0.4755 is the batch time. The time required is 0.994 hours.

5.2.2.4 Optimal Reflux Mode

The optimal reflux mode is a third mode of operation in which neither the distillate composition nor reflux is kept constant, as shown in Figure 5.5. This operating mode is a reflux profile that optimizes the given indices of column performance chosen as the objectives. The indices used in practice generally include the minimum batch time, maximum distillate, or maximum profit functions. This reflux mode is essentially a trade-off between the two operating modes and is based on being able to yield the most profitable operation from optimal

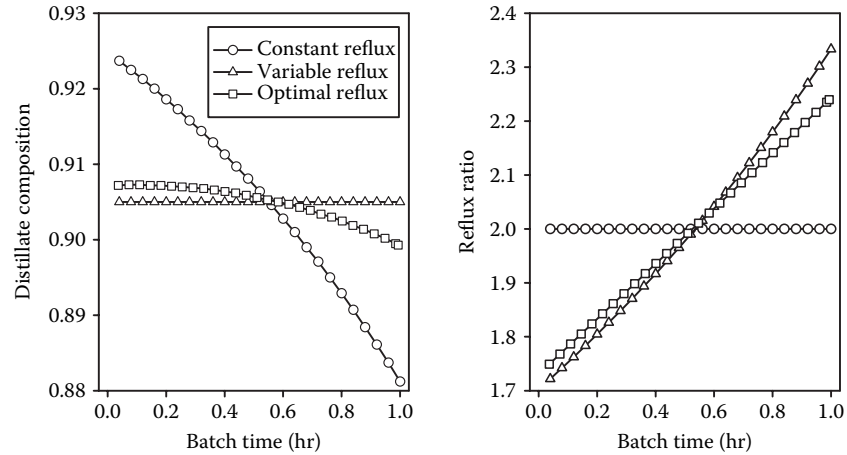


FIGURE 5.5 Three operating modes of batch rectification column.

performance. The calculation of this policy is a difficult problem and relies on optimal control theory. The batch distillation literature is rich in papers on this policy; therefore, a separate section (Section 5.4.1) is dedicated to discussing the solution procedures for this operating mode. Although the first optimal reflux policy was discussed as early as 1963, practical implementation of this procedure has only been possible recently because of the advent of computers.

Example 5.4

Rework the problems in Example 5.2 for the optimal reflux mode. Consider the following reflux profiles for optimal batch operation.

| | | | | | | |
|-------|---------|---------|---------|---------|---------|---------|
| x_D | 0.97959 | 0.91127 | 0.90750 | 0.90228 | 0.89402 | 0.88141 |
| R | 0 | 1.5479 | 1.6699 | 1.7919 | 1.9343 | 2.0919 |

Solution

Because the operation is neither constant composition nor variable reflux, the bottom compositions are calculated at each given reflux profile using the McCabe–Thiele graphical method, resulting in:

| | | | | | | |
|-------|-----|---------|---------|---------|---------|---------|
| x_B | 0.6 | 0.59632 | 0.57311 | 0.54781 | 0.51533 | 0.47558 |
|-------|-----|---------|---------|---------|---------|---------|

The distillate is calculated using the Rayleigh equation, in which the area under the curve of $1/(x_D - x_B)$ vs. x_B (Figure 5.6) is equivalent to



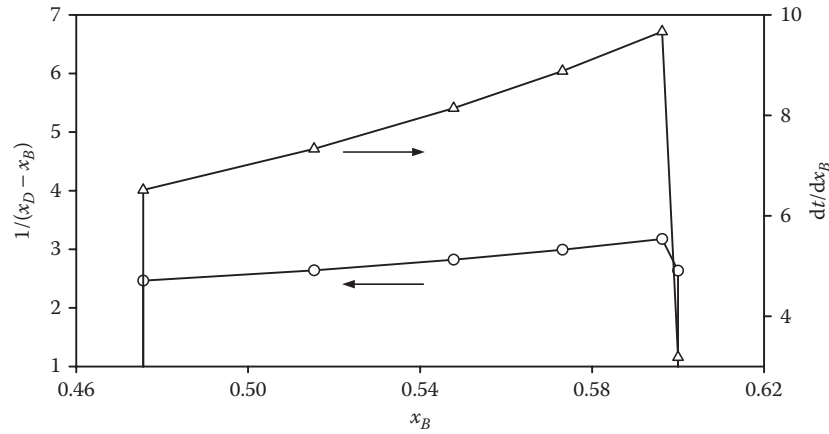


FIGURE 5.6 Graphical integration for Rayleigh equation (open circle) and batch time (open triangle) for the optimal reflux model.

$$\ln\left(\frac{B}{F}\right) = \int_{x_F}^{x_B} \frac{dx_B}{x_D - x_B} = -\text{Area under the curve of } \frac{dx_B}{x_D - x_B} \text{ versus } x_B = -0.3467,$$

$$D = F\left(1 - \frac{B}{F}\right) = 38.969 \text{ moles.}$$

Thus, we can see that the distillates are equal under the three types of operation. The average distillate composition calculated using Equation 5.7 results in $x_{D,avg} = 0.9001$, which is exactly the same result as for the constant and variable reflux modes. Batch time T required when neither the distillate composition nor reflux is constant is found to be:

$$\int_0^T dt = \int_B^F \frac{R+1}{V} dB \quad \text{and} \quad dB = B \frac{dx_B}{x_D - x_B}$$

$$\Rightarrow T = \int_{x_B}^{x_F} \frac{B}{V} \frac{R+1}{x_D - x_B} dx_B.$$

So, the time required is the area under the curve of $(B/V)((R + 1)/(x_D - x_B))$ vs. x_B , as shown in Figure 5.6. The value of T is found to be 0.992 hours, smaller than for the constant and variable modes of operation.

5.3 HIERARCHY OF MODELS

As seen in Section 5.2, the earlier models of the batch rectifier were built on assumptions of negligible liquid holdup and ideal binary systems. Computers have played an important role in relaxing these assumptions, especially the negligible

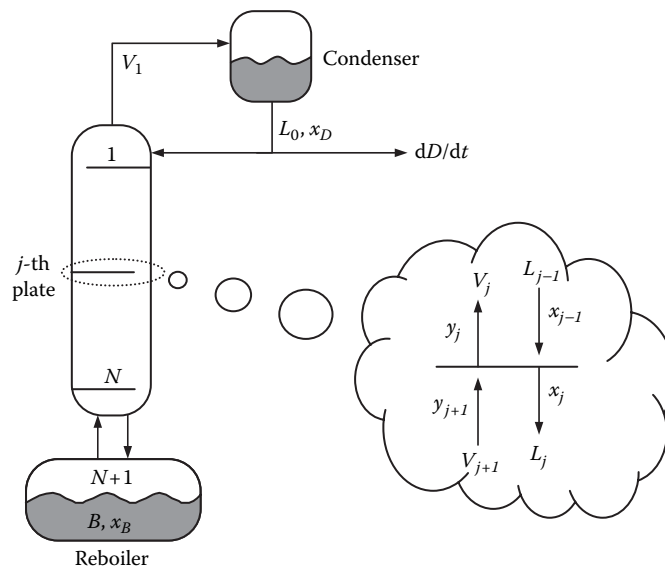


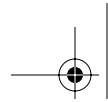
FIGURE 5.7 Schematic of a batch distillation column.

holdup assumption. Distefano⁹ analyzed the numerical differential equations for multicomponent batch distillation in 1968 for the first time. The rigorous models of batch distillation in current state-of-the-art computer packages are based on his pioneering work; however, it is recognized that, due to the severe transients in batch distillation, a hierarchy of models is necessary to capture the dynamics of this flexible operation. This section presents the hierarchy of models ranging from the rigorous model similar to the one presented by Distefano to the simplest shortcut model.

5.3.1 RIGOROUS MODEL

A rigorous model in batch distillation involves consideration of column dynamics along with the reboiler and condenser dynamics. A detailed analysis of the characteristics of differential mass and energy balances associated with the complete dynamics of a multicomponent batch distillation column was presented by Distefano.⁹ He pointed out that the system of equations presented for batch distillation is much more difficult to solve than that for continuous distillation due to several factors. For example, in the case of batch distillation, plate holdup is generally much smaller than reboiler holdup, while in continuous distillation the ratio of reboiler holdup to plate holdup is not nearly as great. In addition, in batch distillation severe transients can occur, unlike continuous distillation, where variations are relatively small. Distefano's work forms the basis for almost all of the later work on rigorous modeling of batch distillation columns.

Figure 5.7 represents a schematic of a batch distillation column, where the holdup on each plate is responsible for the dynamics of each plate. For an arbitrary



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TABLE 5.1
Complete Column Dynamics for a Rigorous Model⁴⁴

Assumptions

Negligible vapor holdup
Adiabatic operation
Theoretical plates
Constant molar holdup
Finite difference approximations for the enthalpy changes

Composition Calculations

Condenser and accumulator dynamics:

$$\frac{dx_D^{(i)}}{dt} = \frac{V_1}{H_D} (y_1^{(i)} - x_D^{(i)}), \quad i = 1, 2, \dots, n$$

Plate dynamics:

$$\frac{dx_j^{(i)}}{dt} = \frac{V_1}{H_D} [V_{j+1}y_{j+1}^{(i)} + L_{j-1}x_{j-1}^{(i)} - V_jy_j^{(i)} - L_jx_j^{(i)}], \quad n; \quad j = 1, 2, \dots, N$$

Reboiler dynamics:

$$\frac{dx_B^{(i)}}{dt} = \frac{1}{B} [L_N(x_N^{(i)} - x_B^{(i)}) - V_B(y_B^{(i)} - x_B^{(i)})], \quad i = 1, 2, \dots, N$$

Flow Rate Calculations

At the top of the column:

$$L_0 = R \frac{dD}{dt}; \quad V_1 = (R + 1) \frac{dD}{dt}$$

On the plates:

$$L_j = V_{j+1} + L_{j-1} - V_j; \quad j = 1, 2, \dots, N$$

$$V_{j+1} = \frac{1}{J_{j+1} - I_j} [V_j(J_j - I_j) + L_{j-1}(I_j - I_{j-1}) + H_j \delta I_j], \quad j = 1, 2, \dots, N$$

At the bottom of the column:

$$\frac{dD}{dt} = L_N - V_B$$

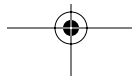
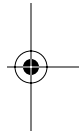




TABLE 5.1 (CONTINUED)
Complete Column Dynamics for a Rigorous Model⁴⁴

Heat-Duty Calculations

Condenser duty:

$$Q_D = V_1(J_1 - I_D) - H_D\delta I_D$$

Reboiler duty:

$$Q_B = V_B(J_B - I_B) - L_N(I_N - I_B) + B\delta I_B$$

Thermodynamics Models

Equilibrium relations:

$$y_j^{(i)} = f((x_j^{(k)}, k = 1, \dots, n), T_{E,j}, P_j)$$

Enthalpy calculations:

$$I_j = f((x_j^{(k)}, j = 1, \dots, n), T_{E,j}, P_j)$$

$$J_j = f((x_j^{(k)}, j = 1, \dots, n), T_{E,j}, P_j)$$

plate j , the total mass, component, and energy balances yield the governing equations, summarized in Table 5.1. This table lists all the equations involved in the dynamic analysis of the batch column and the assumptions behind these equations.

As the governing equations represent a generalized form of the batch rectifying column, treatment of an individual operating mode (i.e., constant reflux, variable reflux, or optimal reflux) exploits the same governing equations but with different specifications. Furthermore, the governing equations of the stripper, middle vessel column, and multivessel columns can be similarly derived.

From the system of differential equations in Table 5.1, we can easily see that the problem has no analytical solution, and we must resort to numerical solution techniques. The governing differential equations of batch distillation often fall into the category of stiff differential equations. The solution of stiff differential equations contains a component that contributes very little to the solution but can cause errors that accumulate over time, resulting in an incorrect solution. Most recent batch distillation codes^{10,11} use stiff numerical methods based on a backward difference formula (BDF), and one of the well-known BDF techniques is the LSODE¹² method.

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Because the computational intensity of the stiff algorithms is generally more severe than for non-stiff algorithms, it is better to switch to non-stiff algorithms. The quantifying measures, such as the stiffness ratio or computational stiffness, both based on eigenvalue calculations, can be used to decide whether or not to





switch;² however, eigenvalue calculations are computationally expensive and are not normally used for large systems of differential equations. Further, it should be noted that, for highly stiff systems, it is difficult to apply any numerical integration method unless the system is transformed in some way to reduce the stiffness of the system. This can happen in batch distillation of wide boiling systems or for columns where the holdup inside the column is significantly smaller than that of the still. The semirigorous model can be used to circumvent this problem.

5.3.2 LOW HOLDUP SEMIRIGOROUS MODEL

For columns where the plate dynamics are significantly faster than the reboiler dynamics (due to very small plate holdups or wide boiling components), the stiff integrator often fails to find a solution (see Example 5.4 in this section). The solution to this problem is to split the system into two levels: (1) the reboiler, where the dynamics are slower, can be represented by differential equations; and (2) the rest of the column can be assumed to be in the *quasi-steady state*. Thus, the composition changes in the condenser and accumulator ($dx_D^{(i)}/dt$), the composition changes on plates ($dx_j^{(i)}/dt$), and the enthalpy changes in the condenser and on plates (δ, I_D and δ, I_j) in Table 5.1 can be assumed to be zero. This results in a zero holdup model, so this approach can be used for simulating the semirigorous model of batch distillation. Bernot et al.^{13,14} developed and compared semirigorous models of the batch rectifier and stripper for the behavior of multicomponent azeotropic distillation. Diwekar and coworkers^{2,11} developed the software packages, BATCH-DIST and MultiBatchDS, in which a semirigorous model is available for cases when the rigorous model fails to obtain solutions, as can be seen in Example 5.5.

The holdup effects can be neglected in a number of cases where this model approximates the column behavior accurately. This model provides a close approximation of the Rayleigh equation, and for complex systems (e.g., azeotropic systems) the synthesis procedures can be easily derived based on the simple distillation residue curve maps (refer Section 5.2 for details). Note, however, that this model involves an iterative solution of nonlinear plate-to-plate algebraic equations, which can be computationally less efficient than the rigorous model.

Example 5.5

An equimolar mixture containing 100 mol of a four-component mixture having relative volatilities of 2.0, 1.5, 1.0, and 0.5 is to be distilled in a batch distillation column. The column has 10 theoretical plates with a holdup of 0.001 mol per plate and a condenser holdup of 1 mol. The vapor boilup rate (V) of the reboiler is 100 mol/hr. The column is operating under a constant reflux mode with a reflux ratio equal to 5.0. Simulate a 1-hr operation of the column using the rigorous model presented in Table 5.1. Repeat the simulation using the semirigorous model.



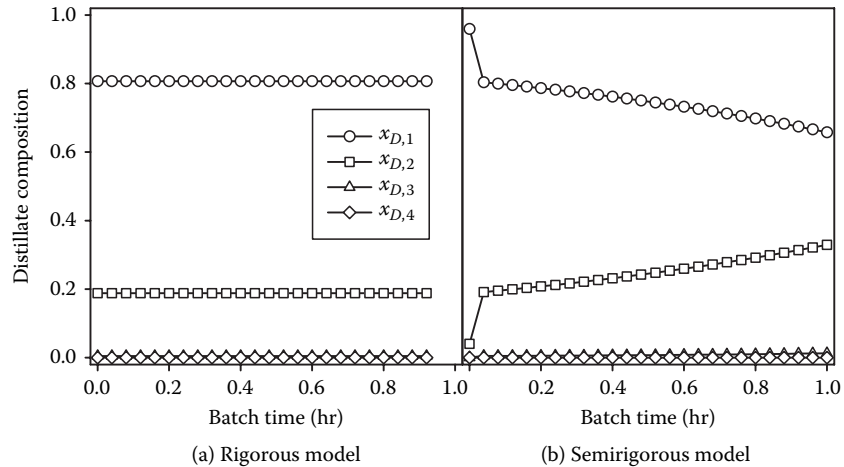


FIGURE 5.8 Transient composition profiles for (a) rigorous model, and (b) semirigorous model.

Solution

Figure 5.8a shows the transient profiles obtained using the rigorous model, and Figure 5.8b was obtained using the semirigorous model. The rigorous model could not integrate the column because the step size became so small that rounding errors dominated the performance, thus switching to the semirigorous model is required in this case.

5.3.3 SHORTCUT MODEL AND FEASIBILITY CONSIDERATIONS

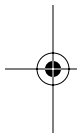
As seen in Section 5.1, the rigorous model of batch distillation operation involves a solution of several stiff differential equations. The computational intensity and memory requirement of the problem increase with an increase in the number of plates and components. The computational complexity associated with the rigorous model does not allow us to derive global properties such as feasible regions of operation, which are critical for optimization, optimal control, and synthesis problems. Even if such information is available, the computational costs of optimization, optimal control, or synthesis using the rigorous model are prohibitive. One way to deal with these problems associated with the rigorous model is to develop simplified models such as the shortcut model and the collocation-based model. These simplified models are abstractions of the rigorous model, and their accuracy depends on the simplifying assumptions embedded within them. The process of abstraction can be viewed as a trade-off between simplicity and accuracy. The usefulness of abstracted models depends on the ease with which they can be analyzed for global behaviors without compromising accuracy. Moreover, the abstracted models are expected to be computationally simpler to analyze.



The shortcut model of batch distillation proposed by Diwekar¹¹ is based on the assumption that the batch distillation column can be considered equivalent to a continuous distillation column with the feed changing at any instant. Because continuous distillation theory is well developed and tested, the shortcut method of continuous distillation is modified for batch distillation, and the compositions are updated using a finite-difference approximation for the material balance (based on the Rayleigh equation). The other assumptions of the shortcut method include constant molar overflow and negligible plate holdups. As described earlier, the functional relationship between the distillate composition (x_D) and the bottom composition (x_B) is crucial for the simulation, and the Fenske–Underwood–Gilliland (FUG) method is used for estimating this relation.

Shortcut methods have also been modified to incorporate holdup issues using a compartmental modeling approach and extended to complex mixtures containing binary and ternary azeotropes.¹⁵ Lotter and Diwekar¹⁶ applied a similar shortcut approach to emerging batch columns, such as stripper and middle vessel columns.

The shortcut model is very useful in feasibility analysis. In order to maintain the feasibility of design, we must place certain constraints on the variables, especially for the design variables such as the number of plates (N) and reflux ratio (R). The shortcut model helps to identify these bounds on the design parameters. The bounds on the parameters depend on the operating modes. The feasible region of operation has been identified using the short-cut model and is summarized in Table 5.2. In this table, R_{\min} is the Underwood minimum reflux ratio, which is different from R_{MIN} . R_{MIN} is defined as the value of R required to obtain the distillate composition of the key component equal to the specified average

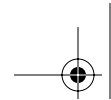


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TABLE 5.2
Feasible Region for Multicomponent Batch
Distillation Columns⁴⁵

| Variable Reflux | Constant Reflux | Optimal Reflux |
|--|-----------------|-------------------------------------|
| Final still composition: | | |
| $0 \leq x_{B,\text{initial}}^{(1)} \leq x_D^{(1)}$ | | |
| Distillate composition: | | |
| $x_B^{(1)} \leq x_D^{(1)} \leq 1$ | | |
| Reflux ratio: | | |
| $R_{\min} \leq R_{\text{initial}} \leq R_{\text{max}}$ | | $R_{\text{MIN}} \leq R \leq \infty$ |
| Number of plates: | | |
| $N_{f,\min} \leq N$ | | $N_{\min} \leq N$ |





distillate composition at the initial conditions for the given N . Recently, Kim and Diwekar¹⁷ defined new performance indices, such as the N -feasibility index and the R -feasibility index, for analyzing feasible regions of various column configurations. These new indices can identify distinctive feasibility regions for various configurations and provide useful guidelines for optimal column selection.

The shortcut model has been found to be extremely efficient and reasonably accurate for nearly ideal mixtures and for columns with negligible holdup effects. For further details, please refer to Diwekar.²

5.3.4 COLLOCATION-BASED MODELS

The next simplified model in the simulation hierarchy is the reduced-order model based on the orthogonal collocation approach. The collocation approach was first proposed in the context of continuous staged separation processes by Cho and Joseph.¹⁸ The collocation approach to model reduction is based on approximating the column stage variables by using polynomials rather than discrete functions of stages; thus, it is widely used for packed batch column design. The orthogonal collocation technique can change partial differential equations to ordinary differential equations (ODEs) or algebraic equations, and ODEs to a set of algebraic equations. In the case of batch distillation, we encounter ordinary differential equations, and the orthogonal collocation technique can be used to reduce this system of ODEs into nonlinear algebraic equations.

Srivastava and Joseph¹⁹ developed the orthogonal collocation method of a simplified packed batch column using the fourth-order polynomial. For a quasi-steady-state batch distillation with total reflux, Aly et al.²⁰ used the Galerkin method as the weighting function over the finite elements. Even though the Galerkin method is one of the best known approximation methods for weighted residuals, this method is difficult to implement.

Note that the orthogonal collocation model can also be used to reduce the order of optimization problems. It is not always advantageous to convert ordinary differential equations to nonlinear algebraic equations. The converted large systems of algebraic equations are computationally time consuming. Instead of using orthogonal collocation to reduce the ODEs to nonlinear algebraic equations, one can use it to reduce the order of ODEs.²¹ This model is especially useful when other simplified models cannot be used to describe the column (e.g., for highly nonideal systems or systems for which constant molar flow assumptions cannot be used).

5.3.5 MODEL SELECTION GUIDELINES

So far several batch distillation simulation models with varying complexity have been presented. Figure 5.9 shows general guidelines to choosing the best one among the hierarchy of batch distillation models. With these models as the basis, numerous batch distillation tasks such as optimization and optimal control



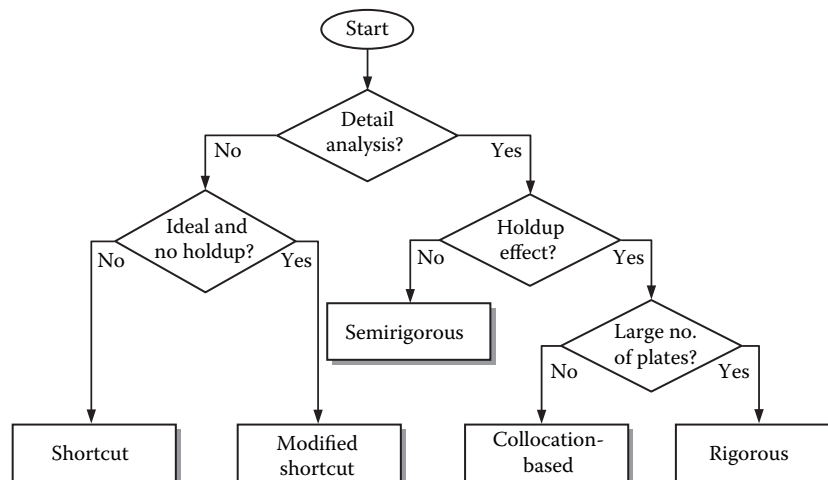


FIGURE 5.9 Model selection guidelines for batch distillation simulation.

(Section 5.4), emerging batch column configurations (Section 5.5), and complex batch distillation systems (Section 5.5) have been developed.

5.4 OPTIMIZATION AND OPTIMAL CONTROL PROBLEMS

The previous sections concentrated on the design and simulation of batch distillation columns using a hierarchy of models. Optimal design and operation in a batch distillation process are challenging decision-making problems that involve several time-dependent and -independent decisions in the face of operating and thermodynamic constraints. Mathematical optimization theory makes the decision-making process easier and more systematic. With the advent of computers, it is possible to exploit these theories to the maximum extent, provided that the problem is properly formulated in terms of the objective functions and constraints and the suitable solution method from the optimization theory is identified. Optimization methods are also used in solving and implementing control problems in batch distillation. This section presents design optimization, optimal control, and closed-loop control problems.

Literature on the optimization of the batch column is focused primarily on the solution of optimal control problems, including optimizing the indices of performance such as maximum distillate, minimum time, and maximum profit; however, literature on the optimal design of batch distillation for performing specified operations using the constant reflux or variable reflux modes is very limited. This section describes optimal control problems within the context of performance indices and optimization techniques. Some recent articles address the problem of design and optimal control policy together by combining optimal control theory and numerical optimization methods. This approach for



simultaneous optimal design and operation is described later. Closed-loop control of columns is presented at the end of this section.

5.4.1 OPTIMAL CONTROL PROBLEMS

This subsection is devoted to optimal control problems in batch distillation, which have received considerable attention in the literature. In general, control refers to a closed-loop system where the desired operating point is compared to an actual operating point and a knowledge of the error is fed back to the system to drive the actual operating point toward the desired one; however, the optimal control problems we consider here do not fall under this definition of control. Because the decision variables that will result in optimal performance are time dependent, the control problems described here are referred to as optimal control problems; thus, use of the control function here provides an open-loop control. The dynamic nature of these decision variables makes these problems much more difficult to solve as compared to normal optimization, where the decision variables are scalar.

These problems are categorized by: (1) performance indices and (2) solution methods. The following subsection discusses the performance indices for optimal control problems (maximum distillate, minimum time, and maximum profit) and is followed by a subsection on mathematical techniques used to solve optimal control problems: calculus of variations, Pontryagin's maximum principle, dynamic programming, and nonlinear programming (NLP) techniques. The first three techniques treat the decision variables as vectors, while the NLP approach requires the variables to be transformed into scalars. For details about these methods, please refer to Diwekar.^{22,23}

5.4.1.1 Performance Indices for Optimal Control Problems

Optimal control problems can be classified as:

- *Maximum distillate problem*, where the amount of distillate of a specified concentration for a specified time is maximized.^{5,24–27} This problem can be represented as follows:

$$\max_{R_t} J = \int_0^T \frac{dD}{dt} dt = \int_0^T \frac{V}{R_t + 1} dt, \quad (5.11)$$

subject to the material and energy balances. Converse and Gross⁵ first reported the maximum distillate problem for binary batch distillation, which was solved using Pontryagin's maximum principle, the dynamic programming method, and the calculus of variations. Diwekar et al.²⁴ extended this optimization model to multicomponent systems and used the shortcut batch distillation model along with the maximum principle to calculate the optimal reflux policy. Logsdon et al.²⁵ used





the orthogonal collocation approach on finite elements and NLP optimization techniques over the shortcut model, and they extended this method to the rigorous batch distillation model,²⁷ in which they considered the effect of column holdups on optimal control policy.

- *Minimum time problem*, where the batch time required to produce a prescribed amount of distillate of a specified concentration is minimized.^{22,28} Although there are several different formulations for the minimum time problem, Diwekar²² derived the following formulations to establish a unified theory for all the optimal control problems:

$$\min_{R_i} J = \int_0^T \frac{dt^*}{dt}. \quad (5.12)$$

where t^* is a dummy variable as a state variable.

- *Maximum profit problem*, where a profit function for a specified concentration of distillate is maximized.^{25,29-31}

Much of the recent research on optimal control problems can be classified into this problem. Kerkhof and Vissers²⁹ were the first to use the profit function for maximization in batch distillation, and they solved the optimal control problem. They obtained the following simple objective function:

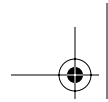
$$\max_{R_i, T} J = \frac{DP_r - FC_F}{T + t_s}, \quad (5.13)$$

subject to purity constraints and column modeling equations.

Diwekar et al.²⁴ used a different objective function to solve the profit maximization problem under the constant and variable reflux conditions. Logsdon et al.²⁵ formulated a new profit function and solved the differential algebraic optimization problem for optimal design and operation. Li et al.³⁰ developed a detailed dynamic multifraction batch distillation model, discretized the model using the orthogonal collocation method on finite elements, and finally solved the maximum profit model using an NLP optimizer. Mujtaba and Macchietto³¹ considered a rigorous reactive distillation system for the maximum conversion problem, which can also be classified as the maximum profit problem. The detailed dynamic system is then reduced by using polynomial curve-fitting techniques and solved by using an NLP optimizer.

A variant of this objective function is to minimize the mean rate of energy consumption when the market size for the product is fixed by the current demand. The objective function is given by Furlonge et al.:³²





$$\min J = \frac{\int_0^T Q_R(t) dt}{T + t_s}, \quad (5.14)$$

$$\text{s.t. } x_{D,avg} \geq x^*,$$

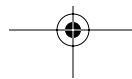
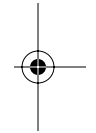
$$D \geq D^*,$$

where Q_R is the reboiler heat duty. They used this objective function for optimal control of multivessel columns for the first time. Hasebe et al.³³ also presented the optimal operation policy based on energy consumption for the multivessel column.

5.4.1.2 Solution Techniques

To solve the optimal control problems, the following four solution techniques have been used in the literature; of these, Pontryagin's maximum principle and nonlinear programming techniques are commonly used today:

- *Calculus of variations* — The theory of optimization began with the calculus of variations, which is based on the vanishing of the first variation of a functional ($dJ = 0$) according to the theorem of minimum potential energy, which involves the definition of stationary values for a function. This leads to the Euler equation and natural boundary conditions.⁵
- *Pontryagin's maximum principle* — The maximum principle was first proposed in 1956 by Pontryagin.³⁴ The objective function formulation is represented as a linear function in terms of the final values of a state vector and a vector of constants. Like the calculus of variations, this method is only applicable to optimal control problems for fixed scalar variables. The maximum principle necessitates repeated numerical solutions of two-point boundary value problems, thereby making it computationally expensive. Furthermore, it cannot handle bounds on the control variables.
- *Dynamic programming* — The method of dynamic programming is based on the principle of optimality, as stated by Bellman.³⁵ In short, the principle of optimality states that the minimum value of a function is a function of the initial state and the initial time. This method is best suited for multistage processes; however, the application of dynamic programming to a continuously operating system leads to a set of nonlinear partial differential equations.
- *NLP optimization techniques* — NLP optimization techniques are the numerical tools used by models involving nonlinear algebraic equations. Obviously, applying NLP techniques to optimal control problems





involves discretization of the control profile by applying either the orthogonal collocation on finite elements,^{25,30,33} the control vector parameterization approach,^{36,32} or the polynomial approximation.²⁶ These discretization approaches add nonlinearities to the system as the number of nonlinear equations increase; therefore, they require good initializations and may result in suboptimal solutions. On the other hand, the polynomial approximation methods depend on the crucial decision of choosing the right type and order of polynomials for approximating the control profile.

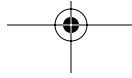
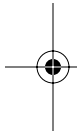
A new approach to optimal control problems in batch distillation, proposed in a paper by Diwekar,²² combines the maximum principle and NLP techniques. This algorithm reduces the dimensionality of the problem (caused by NLP techniques) and avoids the solution of the two-point boundary value problems (caused by the maximum principle). Furthermore, it was shown that for batch distillation problems, bounds could be imposed on the control vector by virtue of the nature of the formulation.

5.4.2 CLOSED-LOOP CONTROL

The two traditional batch operation policies, constant reflux and variable reflux policies, involve different control strategies. For the constant reflux policy, where the distillate composition is continuously changing, the average distillate composition can only be known at the end of operation unless proper feedback from the operation is obtained. The control of the average distillate composition is, then, of an open-loop control nature; however, the variable reflux policy is inherently a feedback operation because the reflux ratio is constantly adjusted to keep the distillate composition constant. The purpose of designing a closed-loop control scheme is to reduce the sensitivity of the plant to external disturbances. Because batch distillation begins with total reflux to obtain a steady state and the distillate is withdrawn after that point, the reflux ratio and distillate composition may oscillate if a controller gain is not properly selected. This is the reason why the constant composition control proves to be very challenging. This subsection describes recent research efforts on closed-loop control problems.

Quintero-Marmol et al.³⁷ proposed and compared several methods for estimating the online distillate composition by feedback control under constant reflux operating mode in a batch rectifier. An extended Luenberger observer for tracking the distillate composition profile proved to provide the best result.

Bosley and Edgar³⁸ considered modeling, control, and optimization aspects of batch rectification using nonlinear model predictive control (NMPC) and implemented an optimal batch distillation policy that was determined *a priori* by the offline optimization. NMPC can determine the set of control moves that will yield the optimal trajectory and allow explicit constraints on inputs, outputs, and plant states. It is known that NMPC is one of the best approaches for distillate composition control; however, the control scheme is computationally intensive





because optimization problems are solved inside this control loop. This work was further studied by Finefrock et al.,³⁹ who studied nonideal binary batch distillation under the variable reflux operating policy. Because the gain space can be changed significantly after a switch to the production phase, they suggested a gain-scheduled proportional and integral (PI) controller based on NMPC if the instantaneous distillate composition is known.

Besides NMPC, Barolo and Berto⁴⁰ provided a framework for obtaining composition control in batch distillation using a nonlinear internal model control (NIMC) approach. NIMC can exactly linearize the system input-output map and be easily tuned by using a single parameter for each component. The distillate composition is estimated by the selected temperature measurements. They also used an extended Luenberger observer for a composition estimator. Although this approach can be reliable and easily implemented, the authors pointed out the problem of selecting the best temperature measurement locations and the problems with using the extended Luenberger observer for a batch column with a large number of trays. For a tighter composition control, more research is necessary to develop a robust and fast closed-loop control scheme.

Closed-loop control schemes have also been applied to new column configurations and complex batch systems. For the control of the middle vessel column, Barolo et al.⁴¹ first proposed and examined several control schemes with or without product recycling. They showed the experimental results of the proposed control structures for dual composition control with or without impurity. Farschman and Diwekar⁴² proposed dual-composition control in which the two composition control loops can be decoupled if the instantaneous product compositions are known. The degree of interaction between the two composition control loops can be assessed using the relative gain array technique.

Hasebe et al.⁴³ proposed a single-loop cascade control system to control the composition of each vessel in the multivessel column. The vessel holdup under total reflux is the manipulated variable, and the reflux flow rate from each vessel is, then, controlled by a simple PI controller. Skogestad et al.⁴ developed a simple feedback control strategy in which the temperature at the intermediate vessel is controlled by the reflux rates from the vessels, thereby adjusting the vessel holdups indirectly. Further, Furlonge et al.³² compared different control schemes, including optimal control problems, in terms of energy consumption.

Future work in closed-loop control problems can involve identifying the proper temperature measurement locations, easy parameter tuning, and focusing on tracing the optimal profiles, as well as on-spec products.

5.5 EMERGING BATCH COLUMNS, COMPLEX SYSTEMS, AND SYNTHESIS

In the previous sections, we described various aspects of batch distillation, including the development of a hierarchy of models ranging from simplified to rigorous, optimization, and optimal control of the batch distillation (rectification) operation.





This section presents discussion on alternative emerging column configurations and thermodynamically or kinetically complicated batch distillation systems such as azeotropic, extractive, and reactive distillations. In addition, this section describes how these complex batch column configurations and complex systems result in difficult batch distillation synthesis problems.

5.5.1 EMERGING BATCH COLUMNS

Figure 5.2 shows a batch stripper (Figure 5.2d), a middle vessel column (Figure 5.2e), and a multivessel column (Figure 5.2f) as emerging batch columns. These column configurations and their advantages are described here.

5.5.1.1 Batch Stripper

Although the batch stripper, often called an *inverted batch column* and originally proposed by Robinson and Gilliland,⁴⁴ is not a true emerging batch column, it has gained much attention in recent literature. In this column configuration, the feed mixture is charged into the top reflux drum, and the products are withdrawn at the bottom reboiler.

Bernot et al.¹⁴ developed a semirigorous model of the batch stripper for multicomponent azeotropic distillation and showed that the batch stripper, compared to the rectifier, is essential to break a minimum boiling point azeotrope. Sørensen and Skogestad⁴⁵ compared the batch stripper with the batch rectifier in terms of batch time and proposed that the inverted column configuration is better than the regular column for separations where the light component in the feed is present in a small amount. They also reported that in some cases the stripper can separate feed mixtures while the rectifier design is infeasible for that separation. Kim and Diwekar,¹⁷ based on this shortcut model, derived more generalized heuristics for column selection using various performance indices — namely, product purity and yield, feasibility and flexibility, and thermodynamic efficiency.

Example 5.6

From Example 5.2, the bottom product composition of a heavy component (B) of the batch rectifier is 0.5247. Repeat the simulation using a batch stripper when the product throughput is the same (i.e., $B = 38.969$ mol), and compare the bottom product compositions of the heavy component (B).

Solution

The reflux ratio of the batch rectifier in Example 5.2 should be converted to the reboil ratio (R_B) of the batch stripper. Because we assume a constant boilup rate (V), the relationship between the reflux and reboil ratios is:

$$R = \frac{L}{dD/dt} = \frac{V - dD/dt}{dD/dt} = \frac{V}{dD/dt} - 1 = \frac{V}{dB/dt} - 1 = R_B - 1$$



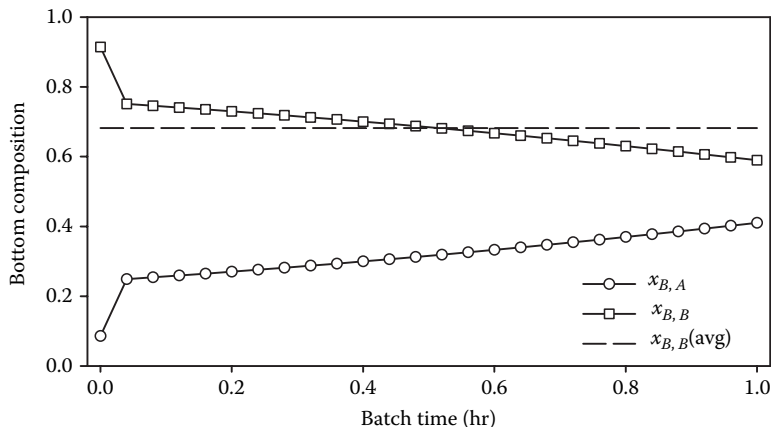


FIGURE 5.10 Bottom composition profiles of a batch stripper from Example 5.6.

So, the reboil ratio becomes 2.82, and the other design and operating conditions remain the same. The bottom composition profiles are shown in Figure 5.10, in which the composition of a heavy component ($x_{B,B}$) is higher than that from the batch rectifier. The average bottom product composition is 0.6819. Thus, if a bottom product is a main concern, it would better to use a batch stripper to obtain highly pure bottom product. For detailed comparisons of the batch rectifier and stripper, please refer to the literature by Sørensen and Skogestad⁴⁵ and Kim and Diwekar.¹⁷

5.5.1.2 Middle Vessel Column

This column configuration consists of a middle vessel between two sections of the batch column. The feed is initially charged into the middle vessel, and the products are simultaneously withdrawn from the top and the bottom of the column. The middle vessel column can be an ideal configuration for ternary batch systems. This column configuration has been known since the 1950s; however, only recently has an analysis of this column configuration been published. Davidyan et al.³ analyzed the dynamic behavior of the middle vessel column for ideal binary and ternary and azeotropic ternary systems. They found additional steady states that are stable or unstable singular points of a dynamic system describing the column. They also introduced a new parameter (q'), which is the ratio of the vapor boilup rate in the rectifying section to the vapor boilup rate in the stripping section. Depending on the value of variable q' , the column shows a qualitatively different behavior for a domain of the reflux and reboil ratio. Figure 5.11 shows the effect of q' on the top and bottom product purities. For $q' = 1$, the distillate composition of the more volatile component increases with time, and this is a favorable trend for the light key distillate; however, the bottom composition of the least volatile component is decreasing. These trends are opposite to those of batch rectification, for which the trends are similar to the

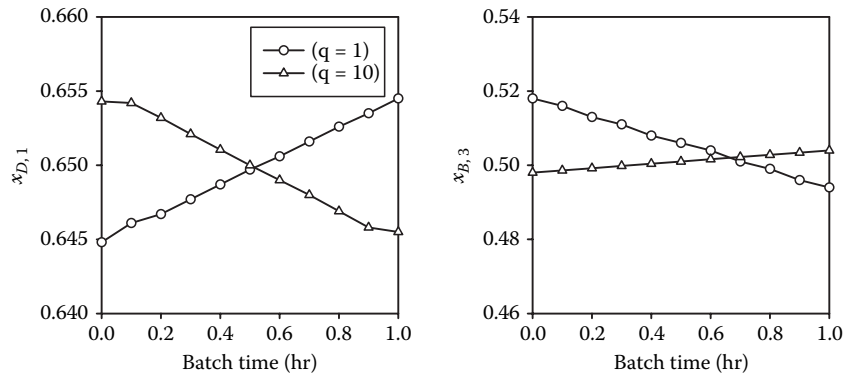
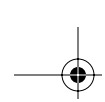


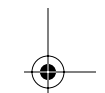
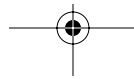
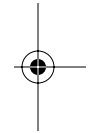
FIGURE 5.11 The effect of q_0 on the top and bottom product purities in the middle vessel column.

case of $q' = 10$; therefore, the new degree of freedom (q') is an important parameter to be used in optimizing the operation. Meski and Morari⁴⁶ extended their previous work under the infinite separation and the minimum reflux conditions and suggested that the middle vessel column always outperforms the rectifier and stripper in terms of batch time. For a binary separation system, they also found that the steady-state operation corresponding to $q' = 1$ is the optimal control policy.

This column configuration is very flexible and effective; hence, one can, in theory, simultaneously obtain very pure components in the top, bottom, and middle vessel columns. For example, Safrit et al.⁴⁷ investigated extractive distillation in the middle vessel column and found that this column can recover all of the pure distillate product from an azeotropic feed with a relatively small size of reboiler, while a rectifier alone would require a still pot of infinite size.

5.5.1.3 Multivessel Column

Similar to a middle vessel column is the multivessel column. Hasebe et al.⁴³ presented a heat-integrated, multieffect batch distillation system (MEBDS) as an alternative to continuous distillation (Figure 5.12). The feed was initially distributed among all the middle vessels and operated in total reflux mode. They proposed a composition control system in which the vessel holdups are manipulated by level controllers. They concluded that this new emerging column configuration can have better separation performance than continuous distillation for systems having a larger number of products. Hasebe et al.⁴⁸ published an optimal operation policy for this column using variable holdup modes. They optimized the liquid flow rates in order to minimize the batch time and concluded that the varying holdup mode resulted in up to 43% more distillate than that of the constant holdup mode. Recently, Hasebe et al.³³ optimized the holdup of each vessel as a function of time for the total reflux multivessel system. When they compared the optimal reflux mode with the constant reflux and variable reflux modes, they found that the performance index, defined as the amount of products per batch



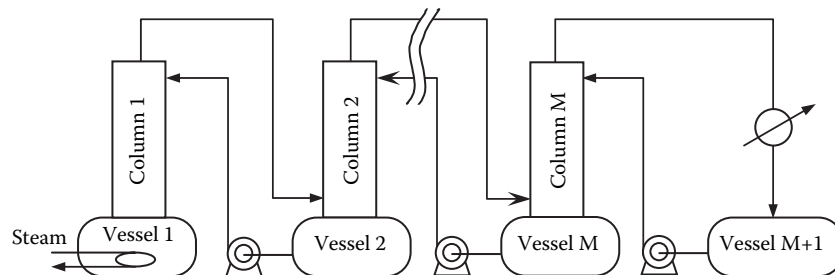


FIGURE 5.12 Multiple-effect batch distillation system. (From Hasebe, S. et al., Comparison of the Separation Performances of a Multieffect Batch Distillation System and a Continuous Distillation System, preprints of IFAC Symposium on Dynamics and Control of Chemical Reactors, Distillation Columns, and Batch Processes (DYCORD '95), Helsingor, Denmark, 1995, pp. 249–254. With permission.)

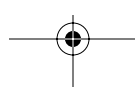
per total batch time, for a variable reflux ternary system was approximately 18 to 38% greater.

Skogestad et al.⁴ reported a new column configuration they referred to as a *multivessel column*. This column is operated under total reflux conditions. They showed that the steady-state compositions in the intermediate vessels could be maintained regardless of the initial feed composition by controlling the liquid rate from the middle vessel so the temperature of the tray just below the middle vessel remained constant. This operation policy can be the ideal operation policy of batch distillation, especially for the middle vessel and multivessel columns. The total reflux mode is commonly used for the multivessel column⁴ because multiple products can be accumulated in each vessel according to their relative volatilities. As a variant of this operating mode, the cyclic operation mode has also been studied. Some literature can be found on the cyclic operation policy, which is essentially a variant of the total reflux condition. Recently, Sørensen⁴⁹ presented a comprehensive study on optimal operation of the cyclic operating mode of the batch rectifier, stripper, and middle vessel columns. The computational results and experiments showed a significant savings in batch time for some separations.

Furlonge et al.³² extended their previous study to optimal control problems and developed more detailed rigorous equations with dynamic energy balance equations, liquid and vapor holdups, and dry and wet head losses on each tray. They compared various operating modes in terms of a mean energy consumption rate and found that the optimal initial feed distribution greatly improves the column performance, resulting in an energy consumption rate half that of the rectifier.

5.5.2 COMPLEX BATCH DISTILLATION SYSTEMS

Thermodynamically and kinetically complex systems such as azeotropic, extractive, and reactive systems pose additional bottlenecks in the design and operation





of batch columns. The operational flexibility offered by batch distillation, along with new emerging designs, can provide promising alternatives for circumventing the bottlenecks. The following sections describe the methods for analyzing these complex systems. These methods also provide heuristics for the synthesis of these columns, especially in terms of the different cuts obtained in a single column or performance comparison of the complex columns.

5.5.2.1 Azeotropic Batch Distillation

Azeotropic distillation is an important and widely used separation technique as a large number of azeotropic mixtures are of great industrial importance. Despite their importance, azeotropic distillation techniques remain poorly understood from a design standpoint because of the complex thermodynamic behavior of the system. Theoretical studies on azeotropic distillation have mainly centered around methods for predicting the vapor–liquid equilibrium data from liquid solution models and their application to distillation design; however, only during the past two decades has there been a concerted effort to understand the nature of the composition region boundaries. Doherty and coworkers^{13,14} in their pioneering works proposed several new concepts in azeotropic distillation. They established the use of ternary diagrams and residue curve maps in the design and synthesis of azeotropic continuous distillation columns. In batch distillation, they outlined a synthesis procedure based on the residue curve maps.

The residue curve map graphs the liquid composition paths that are solutions to the following set of ordinary differential equations:

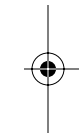
$$\frac{dx_i}{d\xi} = x_i - y_i \quad i = 1, 2, \dots, n - 1, \quad (5.15)$$

where n is the number of components in the system, and the independent variable, warped time (ξ), is a monotonically increasing quantity related to real time. One can see that Equation 5.15 is one form of the Rayleigh equation described earlier. The residue curve map occupies a significant place in the conceptual design stage of column sequencing in continuous distillation and fractions (cuts) sequencing in batch distillation.^{13,14,50}

Despite the advances in the thermodynamics for predicting azeotropic mixture, feasible distillation boundaries, and sequence of cuts, the azeotropic batch distillation system is still incipient in terms of design, optimization, and optimal control. The design problems of these complex systems are described in Section 5.5.3.

Example 5.7

A residue curve map of the propylamine–acetonitrile (ACN)–water system are given in Figure 5.13, in which the curves show liquid composition profiles from the lightest component to the heaviest component. Find the batch distillation regions and define the product cuts for each region.



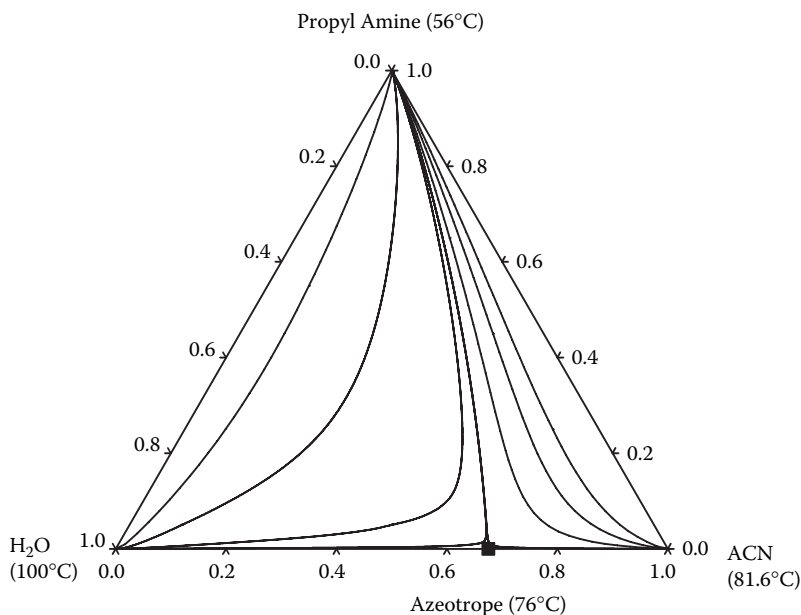


FIGURE 5.13 A residue curve map of the propylamine–acetonitrile–water system for Example 5.7.

Solution

Because the curve from the propylamine apex to the ACN–water azeotrope distinguishes two different product paths, this system has two distillation regions. For the left distillation region, the product sequence is propylamine, ACN–water azeotrope, and water. For the right region, the product sequence is propylamine, ACN–water azeotrope, and ACN. This example shows that conventional distillation cannot obtain pure water and pure ACN cuts at the same time due to the distillation barrier; therefore, this system requires a mass separating agent to cross over this barrier, resulting in a novel batch distillation synthesis problem.

5.5.2.2 Extractive Batch Distillation

Extractive batch distillation can provide advantages of both batch distillation and extractive distillation; thus, this process can be very useful for separation and recovery of waste solvent streams that generally form multicomponent azeotropes. However, most of the recent research efforts on this kind of distillation have been limited to feasibility analysis. Safrit et al.⁴⁷ and Safrit and Westerberg⁵¹ investigated batch extractive distillation in the middle vessel column. They showed that the extractive process is comprised of two steps (operations 2 and 3) and requires a much smaller still pot size. They also identified feasible and infeasible regions and showed that, by varying column conditions such as the

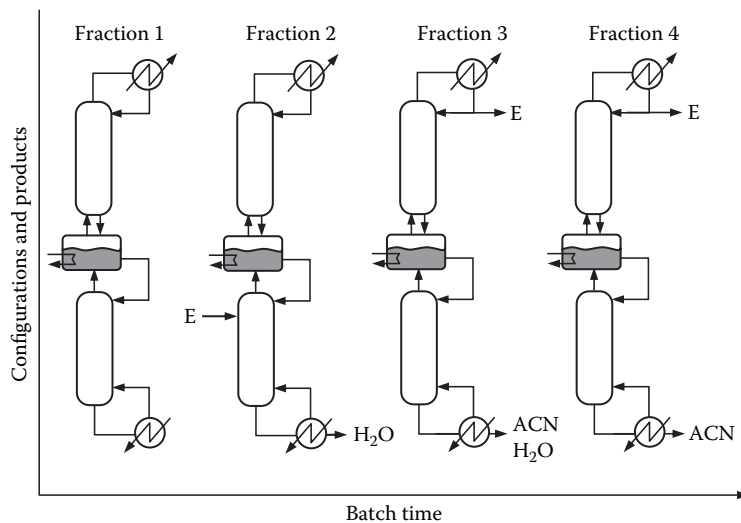


FIGURE 5.14 Operational fractions of batch extractive distillation in a middle vessel column.

product rate, reflux ratio, and reboil ratio, one can steer the middle vessel composition to avoid an infeasible region.

Figure 5.14 shows operational fractions of batch extractive distillation using a middle vessel column, recently developed by Kim,⁵² in order to separate acetonitrile from an aqueous mixture. Fraction 1 is a total reflux and total reboil condition for startup without entrainer (E) feeding. The entrainer is fed to the bottom section of the middle vessel column in fraction 2, where the entrainer can increase relative volatility and separate highly pure water as a bottom product. In this fraction, the middle vessel column operates as a stripper. Fraction 3, where entrainer feeding stops, recovers the entrainer as a top product and a waste cut as a bottom product. Fraction 4 separates the entrainer and ACN as top and bottom products, respectively. This example shows the usefulness of batch extractive distillation using a middle vessel column. This process can provide flexibility in selecting a mass separating agent (entrainer) over batch azeotropic distillation and in obtaining proper product sequences and can exhibit seamless batch distillation operation between fractions. Detailed advantages of this process are explained in Example 5.8.

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5.5.2.3 Reactive Batch Distillation

Although reactive distillation was acknowledged as a unit operation as early as in the 1920s, it has gained its research interest as an excellent alternative to both reaction and separation since the 1980s. For example, most of the new commercial processes of methyl-*tert*-butyl ether (MTBE, an anti-knocking agent) are based on continuous reactive distillation technologies. The analysis of a reactive batch



distillation model in a staged column was first published by Cuille and Reklaitis.⁵³ Using a stiff integrator for the differential and algebraic equations, they presented a numerical solution technique for the esterification of 1-propanol and acetic acid. Wajge et al.⁵⁴ developed a new solution technique based on the orthogonal collocation method and the finite-element method for the reactive batch distillation of a packed column. The differential contactor model of a packed column, originally designed by Hitch and Rousseau,⁵⁵ was then reduced to low-order polynomials with the desired accuracy. They compared the results with those from the finite difference method and global collocation method for nonreactive packed-bed batch distillation systems and showed that their approach was more efficient. Wajge and Reklaitis⁵⁶ extended their previous work to the optimal campaign structure for reactive batch distillation, which can offer reasonably sharp separations between successive cuts and reduce the amount of waste off-cuts. To obtain the optimal reflux policies or profiles for the maximum distillate or minimum time problem, multiperiod reflux optimization can be applied. Macchietto and Mujtaba⁵⁷ showed that, for the same production rate, the waste generation can be significantly reduced under the optimal campaign structure.

An efficient optimization approach for reactive batch distillation using polynomial curve fitting techniques was presented by Mujtaba and Macchietto.³¹ After finding the optimal solution of the maximum conversion problem, polynomial curve-fitting techniques were applied over these solutions, resulting in a nonlinear algebraic maximum profit problem that can be efficiently solved by a standard NLP technique. Four parameters in the profit function (maximum conversion, optimum distillate, optimum reflux ratio, and total reboiler heat load) were then represented by polynomials in terms of batch time. This algebraic representation of the optimal solution can be used for online optimization of batch distillation.

A dynamic rate-based model for packed-bed batch distillation was recently presented in which a solid catalyst was used first in the reactive batch distillation modeling.⁵⁸ The pilot-scale experiments were conducted with strong anion-exchange resins. The results were compared with the experimental data and with results from its counterpart, the equilibrium-based model. The rate-based model provides more accuracy, much higher physical significance, and more predictability of the experimental data even though the formulation of the rate-based model is complicated.

5.5.3 BATCH DISTILLATION SYNTHESIS

The complexity of batch distillation design and operation is also reflected in the batch distillation synthesis problem. In continuous distillation, optimal column sequencing is the main focus of synthesis research. Several past reviews are available on this subject.^{59,60} Unlike continuous distillation synthesis, the area of batch distillation synthesis is complicated by its transient nature. Decisions such as cut selection, operating mode, configuration type, and column sequencing enter into the synthesis problem. For complex systems such as azeotropic, extractive, and reactive distillation, identifying the distillation boundaries and steering



toward feasible and optimal regions add further complications to the problem; however, as seen in the previous section, theoretical and geometric analyses can point toward optimal synthesis solutions in this area.^{13,14,50}

Example 5.8

feed point?

For the residue curve map of the propylamine–acetonitrile–water system given in Example 5.7, find the necessary batch distillation cuts to separate all three pure components if the initial feed (F_0) is (0.58, 0.20, 0.22). Discuss which column configuration is best for this separation.⁵²

Solution

Because the initial feed is located in the left distillation region, draw a line from a stable node (H_2O apex) through the feed point to the distillation boundary. The top product (D_1) is on the distillation barrier. Draw a line from an unstable node (propylamine apex) through D_1 (i.e., now F_2) to the ACN–water boundary line, which will be B_2 . Now there are only two components in the system, thus the top and bottom product cuts are ACN–water azeotrope and ACN, respectively. The product cuts are summarized in Table 5.3.

If a batch rectifier is used, shutdown and setup time is required before cut 2 because the top product from cut 1 is the feed to the bottom reboiler in the next cut. Similarly, if a batch stripper is used, shutdown and setup time is required before cut 3. Because a middle vessel column performs both rectifying and stripping operations, a middle vessel column configuration is the optimal column configuration. The operational fractions or cuts of this batch synthesis problem are shown in Figure 5.?.

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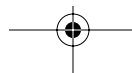
State-of-the-art techniques used in the solution of synthesis problems include: (a) a heuristic approach, which relies on intuition and engineering knowledge; (b) a physical-insight approach, which is based on exploiting basic physical principles; and (c) an optimization approach. In this section, two common approaches, heuristics and optimization, are discussed.

The recent literature in batch distillation has been devoted to comparing emerging column configurations with the conventional one, thereby obtaining heuristics for optimal column configuration, optimal design, and optimal operating conditions.^{17,43,45,46,61} In these studies, parameters such as product purity, batch

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TABLE 5.3
Product Cuts for Example 5.8⁴⁶

| Cut No. | Feed | Bottom Product Cut | Top Product Cut |
|---------|-------------|---------------------------|------------------------------------|
| 1 | $F_1 = F_0$ | $B_1 = \text{pure water}$ | $D_1 = \text{propylamine-ACN}$ |
| 2 | $F_2 = D_1$ | $B_2 = \text{ACN-water}$ | $D_2 = \text{pure propylamine}$ |
| 3 | $F_3 = B_2$ | $B_3 = \text{pure ACN}$ | $D_3 = \text{ACN-water azeotrope}$ |





time, or total cost were evaluated to compare the performance of column configurations. Chiotti and Iribarren⁶¹ compared the rectifier with the stripper in terms of annual cost and product purity. They noted that the rectifier is better for the more volatile component products while it is more economical to obtain less volatile component products using the stripper. Meski and Morari⁴⁶ compared three column configurations in terms of the batch time under fixed product purity and infinite number of plates. They observed that the middle vessel column always had the shortest batch time, and the rectifier had the next shortest time. Sørensen and Skogestad⁴⁵ studied two competing column configurations, rectifier and stripper, in the context of minimum optimal operating time and also described the dynamic behavior of these columns. They concluded that the stripper is the preferred column configuration when a small amount of the more volatile component is in the feed and that the rectifier is better when the feed has a high amount of the more volatile component. Although several studies support the same heuristics, some studies present contradictions among the suggested heuristics. For example, the batch time studies of Meski and Morari⁴⁶ and Sørensen and Skogestad⁴⁵ give conflicting results with respect to feed composition. This is due to the limited ranges of parameters and systems considered, as well as the complexity and difficulty of the problem of column selection.

In order to elicit comprehensive heuristics, the analysis must cover a wider range of column configurations, operation policies, and design variables, and various performance indices must be included. Kim and Diwekar¹⁷ extended the column configuration problem using four performance indices: product purity, yield, design feasibility and flexibility, and thermodynamic efficiency. It is generally observed that the rectifier is a promising column configuration for the more volatile component product and that the stripper is better in the opposite case. Feasibility studies based on the minimum number of plates and minimum reflux ratio addressed the flexibility of such a high-purity configuration for changing operating conditions. It was found that the rectifier and the stripper have distinctive feasibility regions in terms of the feed composition. Thermodynamic efficiency indicates how close a process or system is to its ultimate performance and also suggests whether or not the process or system can be improved. The rectifier can also be a promising column configuration in terms of thermodynamic efficiency, but in some conditions higher efficiencies of the stripper or the middle vessel column can be observed. Furthermore, for the middle vessel column, the thermodynamic efficiency is greatly affected by an added degree of freedom (q'). This systematic and parametric **study** concluded that the trade-offs between performance indices should be considered within a multiobjective framework.

which one
(by whom?)

5.5.4 COMPUTER-AIDED DESIGN SOFTWARE

It is difficult to analyze batch distillation without using computers due to the two reasons stated before: (1) the process is time varying, so one has to resort to complex numerical integration techniques and different simulation models for obtaining the transients; and (b) this ever-changing process also provides





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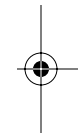
TABLE 5.4
Batch Distillation Software Requirements⁴⁷

| Features | Why |
|----------------|--|
| Windows | User-friendly state-of-the-art input/output interface |
| Databank | Ability to generate data from structural information |
| Operations | Yield improvement due to operational flexibility; systematic optimization/optimal control methods |
| | Constant reflux |
| | Variable reflux |
| | Fixed equation optimal |
| | Optimal reflux |
| Models | Hierarchy of models for numerical stability, design feasibility, and advanced system designs |
| | Shortcut |
| | Semirigorous |
| | Design feasibility |
| | Optimization |
| Options | Advanced feature |
| | Reactive distillation |
| | Three-phase distillation |
| | Uncertainty analysis |
| Configurations | Emerging designs provide promising directions for effective designs to obtain purer products |
| | Semibatch |
| | Recycle waste cut |
| | Rectifier |
| | Stripper |
| | Middle vessel column |

flexibility in operating and configuring the column in numerous ways. Based on the current state of the art in batch distillation techniques and computer simulation technology, Table 5.4 identifies the required functionality and the rationale behind it. Several commercial software packages are available for simulations, optimizations, and optimal controls of batch distillation (see Table 5.5). These include Bdist-SimOPT (Batch Process Technologies), BatchSim (Simulation Sciences), BatchFrac (Aspen Technology, based on Boston et al.¹⁰), and MultiBatchDS (Batch Process Research Company). Bdist-SimOPT and MultiBatchDS are derived from the academic package BATCHDIST.¹¹ Most of these packages, except MultiBatchDS, are usually limited to conventional systems as they were developed in early or late 1980s. The educational version of MultiBatchDS can be obtained from the AIChE CACHE website (<http://www.che.utexas.edu/cache/>).

5.6 SUMMARY

This chapter presented a complete review of the batch distillation literature, beginning from the first theoretical analysis to the current state of the art in computer-aided design and optimization methods. The new advances in batch distillation include novel column configurations, optimal designs, optimal operation policies, and new methods of analysis. These new advances can increase





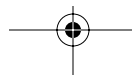
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TABLE 5.5
Batch Distillation Software Comparison⁴⁸

| Features | BATCHSIM | BatchFrac | MultiBatchDS |
|--------------------------|---------------|---------------|----------------|
| Windows | Yes | No | Yes |
| Databank | SIMSCI | Aspen Plus | Cranium/Others |
| Operations | | | |
| Constant reflux | Yes | Yes | Yes |
| Variable reflux | Yes (limited) | Yes (limited) | Yes |
| Fixed equation optimal | No | No | Yes |
| Optimal reflux | No | No | Yes |
| Shortcut | No | No | Yes |
| Models | | | |
| Semirigorous | No | No | Yes |
| Reduced order | No | No | Yes |
| Rigorous | Yes | Yes | Yes |
| Design feasibility | No | No | Yes |
| Optimization | No | No | Yes |
| Options | | | |
| Reactive distillation | No | Yes (limited) | Yes (limited) |
| Three-phase distillation | No | Yes | Yes |
| Uncertainty analysis | No | No | Yes |
| Semibatch | No | No | Yes |
| Recycle waste-cut | No | No | Yes |
| Configurations | | | |
| Rectifier | Yes | Yes | Yes |
| Stripper | No | No | Yes |
| Middle vessel | No | No | Yes |

the possibility of using batch distillation profitably for a wide variety of separations, but they also present a bewildering array of problems regarding the selection of proper configurations, the correct operating mode, and optimal design parameters. Thus, we will certainly see future researchers working on various aspects of design, analysis, and synthesis of batch distillation, some of which are outlined below:

- A more extensive analysis for each column configuration must be carried out. For example, the effect of q' on the performance of the middle vessel column has not been fully investigated. For the multi-vessel column, no general guidelines or heuristics exist for column holdups and operating modes because of the additional degrees of freedom.
- Azeotropic, extractive, and reactive distillations and off-cut recycling operations have been studied extensively in recent years, but they are in the developing stage. For instance, for continuous reactive distillation, solid catalysts are commonly used, but only a few applications of solid catalysts in batch reactive distillation exist in theory and practice.

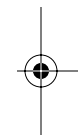




- Comprehensive heuristics for optimal design and synthesis should be derived. Several heuristics and trade-offs between heuristics can be found, but they are still limited to the systems considered.
- Batch processes often encounter feed composition variations and other operational uncertainties. Consideration of uncertainties at various stages of design and operation can provide useful and cost-effective solutions to the batch processing industries.
- An important research area in this field is batch distillation synthesis. Based on future advances in batch distillation, batch distillation synthesis from a superstructure can lead to the most promising and flexible column configuration with the appropriate operation mode and conditions.

NOTATION

| | |
|-------------|--|
| α | relative volatility |
| B | amount of bottom residue (mol) |
| dB/dt | bottom product flow rate or change of bottom product (mol/hr) |
| D | amount of distillate (mol) |
| dD/dt | distillate rate (mol/hr) |
| E | entrainer feed rate (mol/hr) |
| F | amount of feed (mol) |
| H_j | molar holdup on plate j (mol) |
| H_0, H_D | condenser holdup (mol) |
| I_D | enthalpy of the liquid in the condenser (J/mol) |
| I_j | enthalpy of the liquid stream leaving plate j (J/mol) |
| J_j | enthalpy of the vapor stream leaving plate j (J/mol) |
| L_j | liquid stream leaving plate j (mol/hr) |
| L_0 | liquid reflux at the top of the column (mol/hr) |
| n | number of components |
| N | number of plates |
| q' | ratio of the top vapor flow rate to the bottom vapor flow rate |
| Q_R | reboiler heat duty |
| R | reflux ratio (L/D) |
| R_t | reflux ratio as a function of time |
| T | batch time (hr) |
| V_j | vapor stream leaving plate j (mol/hr) |
| x | liquid-phase mole fraction |
| x_D | liquid-phase mole fraction of the distillate |
| $x_{D,avg}$ | average distillate mole fraction |
| x_F | liquid-phase mole fraction of the feed |
| y | vapor-phase mole fraction |





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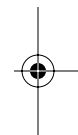
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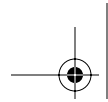
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