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SEPARATIONS

Shortcut Models and Feasibility Considerations for Emerging Batch Distillation Columns

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The transient nature and flexibility of batch distillation allow for configuring the column in a number of different ways. Some of the new configurations are an inverted column, a middle vessel column, and a multivessel column. These new column configurations have also provided new ways of operation. The preliminary analysis of these emerging columns has shown promising behavior, because of the added flexibility. This added flexibility is especially interesting for a chemical industry where the quantity and lifetime of the products are uncertain, but it has also made the analysis of the system more difficult. Shortcut procedures provide an easy way of understanding the global behavior of complex systems. In this paper we are presenting shortcut procedures for the newly described batch distillation column configurations. The transient profiles obtained by the proposed shortcut procedures and rigorous models are compared using extensive test cases. Global qualitative properties and feasibility criteria are derived for these new designs, and a detailed analysis of these configurations is also presented.

1. Introduction

Batch distillation is widely used and is an important unit operation in the batch processing industry. The flexibility of batch distillation combined with the inherent unsteady nature of the process poses challenging design and operation problems. The transient nature of the process allows for configuring of batch columns in a number of different ways, some of which are shown in Figures 1 and 2. The column at the right in Figure 1 is a conventional batch distillation column with a reboiler at the bottom and a condenser at the top, which essentially performs the rectifying operation. In contrast to a conventional batch distillation column, the inverted column (Figure 1, left) has its storage vessel at the top and the products leave the column at the bottom. Thus, mixtures with a small amount of the light component can be separated by removing the heavy component as the bottom product. These two columns are comparable to the rectifying and stripping parts of a continuous distillation column but with additional flexibility.

Devidyan et al. (1994) presented a batch distillation column that combines both the rectifying and stripping sections, the middle vessel column (Figure 2, left). Although this column has not been investigated completely, preliminary analysis has demonstrated that it provides high flexibility and that it is able to remove both light and heavy impurities by having three product vessels. For example, the composition of the most volatile component in a rectifier decreases with time, and the composition of the least volatile component in a stripper also decreases with time. In the middle vessel column, however, these effects can be reversed by setting the vapor ratios for the top and bottom parts of the column appropriately (Diwekar, 1995).

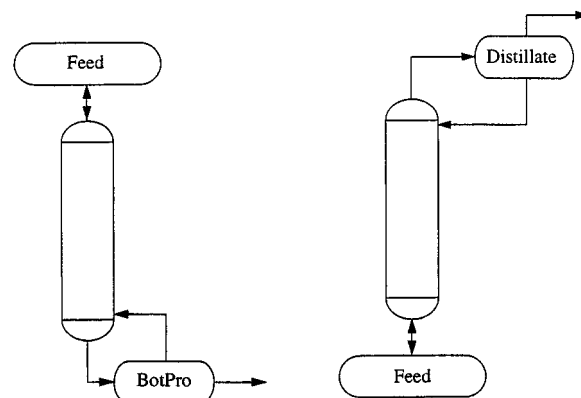


Figure 1. Inverted batch distillation column (stripper) and conventional batch distillation column (rectifier).

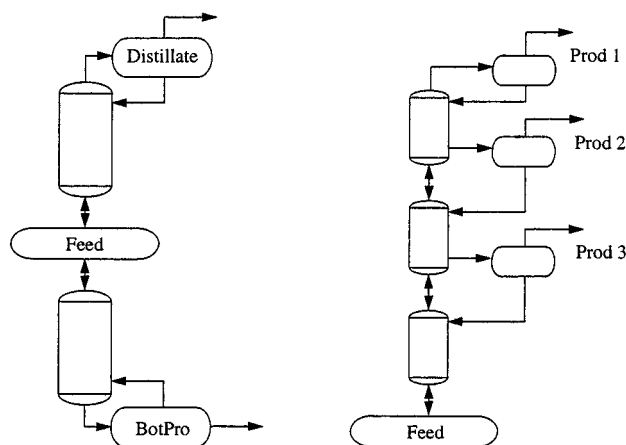


Figure 2. Middle vessel batch distillation column and multivessel batch distillation column.

Recently, Skogstestad et al. (1995) described a new column called a multivessel column (Figure 2, right) and showed that the column can obtain purer products at the end of a total reflux operation. With this column it

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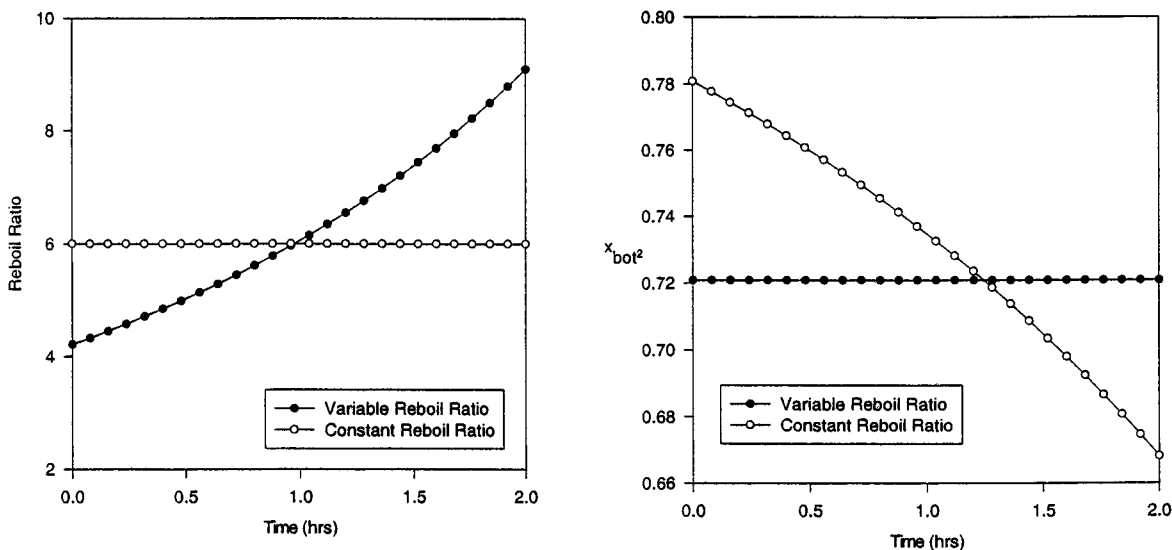


Figure 3. Two operating modes for a batch stripper.

is possible to separate more than three components at a time by installing enough intermediate vessels. Its design, however, is less flexible than the design of a conventional batch distillation column.

The two basic modes of operating batch rectifying columns are (1) constant reflux and variable distillate composition and (2) variable reflux and constant distillate composition of the key component. There is a third mode of operation for operating a batch rectifying column that is neither variable reflux nor constant reflux but instead is in between the two. This type of operation is known as optimal reflux or optimal control policy. The optimal reflux policy is essentially a trade-off between the two operating modes of constant reflux and variable reflux and is based on the ability to yield the most profitable operation. The calculation of this policy is a difficult problem and relies on optimal control theory (Diwekar, 1995). Although not reported in the literature, the stripper operating modes, like the batch rectifier, can be defined as (1) the constant reboil ratio and variable bottom composition, (2) the variable reboil and constant bottom composition of the key component, and (3) the optimal reboil policy. Figure 3 shows the first two operating modes for the stripper. For the middle vessel, the combination of the three rectifying and three reboil policies results in nine possible operating policies. Additionally, one can manipulate (at each time step) the parameter q' , or the ratio of vapor boilup rate in the rectifying to the stripping section, which is an important parameter for the middle vessel. For different kinds of columns combined with different modes of operation, the number of possible column configurations tends to be very high, which poses a bewildering problem of selecting proper configuration and feasible design in the face of complexities. Although computers have made it possible to solve large-scale models in a reasonable amount of time, shortcut models are necessary to derive global properties, such as feasible regions of operation, which are critical for optimization, optimal control, and synthesis problems. Even if this information was available in the solution of a rigorous model, the computational cost of iterative design, optimization, optimal control, or synthesis using rigorous models would be too high. In addition, with a shortcut model it is not necessary to solve systems of stiff differential equation, which poses an algorithmic problem. The shortcut method for the batch rectifier (Diwekar, 1988; Diwekar and Madhavan, 1991) is based

on the FUG equations (Fenske, Underwood, Gilliland) and has been shown to be successful in identifying the feasible parameter window for different operation modes, presenting criteria such as minimum reflux and minimum number of plates. The method is extremely efficient and involves only noninteger variables. This shortcut model delivers bounds (feasibility window) on design variables, especially the number of plates and the reflux ratio, and is thus useful in design and optimization.

In this paper, we are presenting shortcut procedures for the newly described batch distillation column configurations, namely, the inverted column and the middle vessel column. The models are limited to constant reflux and reboil operation but can be easily extended to other column configurations and operating modes. For both columns, the shortcut models are based on the assumption that a batch distillation column can be regarded as a continuous distillation column with changing feed at each time step, modified to suit the different configurations. This study is restricted to ideal systems in order to separate the thermodynamic complexity of nonideal and azeotropic mixtures and the complexities associated with the flexible, unsteady-state nature of the batch distillation column. The shortcut models presented here can be extended to azeotropic systems based on the variable transformation suggested in the earlier work (Diwekar, 1991; Kalagnanam and Diwekar, 1993). The transient profiles obtained by the proposed shortcut procedures and rigorous models are compared using extensive test cases. Global qualitative properties and feasibility criteria are derived for these new designs, and a detailed analysis of these configurations is also presented to help understand the novel batch distillation column configurations and their operating modes.

The shortcut procedures neglect the liquid holdups on the plate which can be a reasonable assumption for certain applications. However, it should be noted that, similar to the rectifier, holdup affects the batch stripper and the middle vessel in two ways, namely, the dynamic flywheel effect and the steady-state capacitance effect. It is possible to use the compartmental modeling approach to capture the capacitance effect with the shortcut procedures as suggested in Diwekar (1995). However, for the dynamic flywheel effect, the product composition profiles predicted by the shortcut method show rapid change initially as compared to the profiles

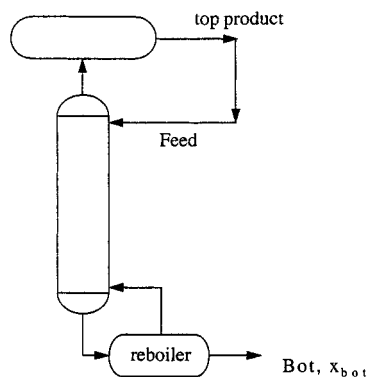


Figure 4. Shortcut method for the stripper.

of the column with large holdup effect, but as both show the same trends, the shortcut procedure can be used to predict the trends even for columns with large flywheel effect.

2. Inverted Batch Column (Stripper)

This section presents a shortcut model for the inverted batch distillation column (stripper), based on the shortcut model for the batch rectifier (Diwekar and Madhavan, 1991). A comparison between the results of rigorous simulation and the proposed shortcut method is made to validate the accuracy of the shortcut method. Using this method, design criteria and the window of feasible operation are provided.

2.1. Shortcut Model for the Stripper. Similar to the shortcut method for the batch distillation rectifier, the main assumption of the shortcut method for the stripper is that the batch distillation column is regarded as a continuous column with changing feed composition at each time step, as shown in Figure 4. Other assumptions include constant relative volatility (ideal systems), equal molal overflow, and negligible plate holdups. Thermodynamic equilibrium is assumed between the vapor and liquid leaving each plate (theoretical plate), and the composition of the liquid leaving the still is assumed to be the same as that of the still (well-mixed reservoir). Although the basis of the shortcut method for the stripper is similar to the shortcut method for the rectifier, i.e., based on FUG (Fenske–Underwood–Gilliland) equations, there are differences in the column behaviors. The first difference is the definition of the reflux and reboil ratio. Unlike the reflux ratio, the reboil ratio does not vary between zero and infinity but between 1 and infinity. This is due to the definition of the reboil ratio (eq 1). This difference also reflects in

$$\begin{aligned} \text{reflux ratio, } R &= \frac{L}{dD/dt} \\ \text{reboil ratio, } Rb &= \frac{Vb}{dBot/dt} \end{aligned} \quad (1)$$

various equations for the shortcut procedure.

For the system shown in Figure 4 we assume that the distillation is carried out at constant boilup rate Vb . The constant molal overflow assumption leads to the following overall material balance equation:

$$\frac{dS}{dt} = -\frac{Vb}{Rb} \quad S_0 = F \quad (2)$$

where F is the feed. A material balance for the key component k over the differential time can be written as

$$x_{\text{bot}}^{(k)} \frac{Vb}{Rb} = -\frac{d(x_s^{(k)} S)}{dt} \quad (3)$$

$$\frac{dx_s^{(k)}}{dt} = \frac{Vb}{(Rb)S} (x_s^{(k)} - x_{\text{bot}}^{(k)}), \quad x_{s0}^{(k)} = x_F^{(k)} \quad (4)$$

where F is the feed to the still, S is the amount remaining in the still, and Rb is the reboil ratio. The still composition and bottom composition are given by x_s and x_{bot} , respectively. Equation 4 relates the change in the still composition to the bottom product composition. The above two mass balance equations can be converted to a time-implicit equation similar to the Rayleigh equation for simple distillation and for batch rectifier. We will call this equation the modified Rayleigh equation. The following is the finite difference approximation to this modified Rayleigh equation for n components expressed in terms of the key component k .

$$x_{s_{\text{new}}}^{(j)} = x_{s_{\text{old}}}^{(j)} + \frac{\Delta x_s^{(k)} (x_{\text{bot}}^{(j)} - x_s^{(j)})_{\text{old}}}{(x_{\text{bot}}^{(k)} - x_s^{(k)})_{\text{old}}}, \quad i = 1, 2, \dots, n \quad (5)$$

As seen in the case of the rectifier, the functional relationship between the end compositions is crucial for the simulation of the complete operation, and it is here that we need to use the design equations described by the modified FUG method.

Functional Relationship between x_{bot} and x_s . At each instant, there is a change in the still composition of the key component, resulting in changes in the still composition of all the other components calculated by the differential material balance equations (eq 5). For rectifier, Hengestebeck–Geddes' relation provides the relation between distillate composition and bottom composition. For stripper, this equation translates into the following equation:

$$x_{\text{bot}}^{(j)} = \left(\frac{\alpha_j}{\alpha_k} \right)^{-C_B} \frac{x_s^{(j)}}{x_s^{(k)}} x_{\text{bot}}^{(k)}, \quad i = 1, 2, \dots, n \quad (i \neq k) \quad (6)$$

where C_B is the Hengestebeck–Geddes' (HG) constant. Fenske (1932) derived an equation to calculate the minimum number of plates for a continuous distillation column with constant relative volatilities in terms of the distillate composition and feed composition. It can be easily shown that the Fenske equation for a stripper is given by

$$Nb_{\text{min}} = \frac{\ln \left[\frac{x_s^{(j)} x_{\text{bot}}^{(k)}}{x_s^{(k)} x_{\text{bot}}^{(j)}} \right]}{\ln \left[\frac{\alpha_j}{\alpha_k} \right]} \quad (7)$$

The minimum number of plates is the number of equilibrium plates required for a separation at total reflux conditions and is thus a boundary of the operating conditions. If Hengestebeck–Geddes' equation is compared with the Fenske equation for the minimum number of plates Nb_{min} for two components, Nb_{min} has to be equal to C_B . Since the summation of all components must be equal to 1 ($\sum_{i=1}^n x_{\text{bot}}^{(i)} = 1$), an equation can be found which relates the new bottom product composition to the current still composition in terms of relative volatilities and the minimum number of plates (eq 8).

$$x_{\text{bot}}^{(k)} = \frac{1}{\sum_{i=1}^n \left(\frac{\alpha_i}{\alpha_k} \right)^{-C_B} \frac{x_s^{(i)}}{x_s^{(k)}}} \quad (8)$$

For a binary mixture, the separation is between only two components; in the case of a multicomponent mixture, however, the separation can be expressed in terms of a binary mixture of two key components, LK, the lightest component appearing in the bottom, and HK, the heavy key component defined as the heaviest component in the top. It should be noted that, for most of the cases, the heavy key component in the stripper is the least volatile component in the mixture, as the still contains all the feed. Unlike continuous distillation, the key components keep changing for batch distillation as time progresses. For example, the light key component will become lighter than light key, and the next component in the relative volatility hierarchy will become the light key component.

To get a second boundary of the operating condition, the minimum reboil ratio Rb_{min} , Underwood's (1948) equation for continuous distillation columns is applied to batch distillation columns. At minimum reboil conditions, an infinite number of equilibrium stages is required to achieve the desired separation.

$$\sum_{i=1}^n \frac{\alpha_i x_F^{(i)}}{\alpha_i - \phi} = 1 - q \quad (9)$$

$$-Rb_{\text{min}} = \sum_{i=1}^n \frac{\alpha_i x_{\text{bot}}^{(i)}}{\alpha_i - \phi} \quad (10)$$

The q in the above equations represents the feed condition and is defined as the ratio of heat required to vaporize 1 mol of the feed to the molar latent heat of the feed. The ϕ is the root of the Underwood equation which lies between the α_{LK} and α_{HK} . The shortcut method assumes that batch distillation can be considered as continuous distillation with changing feed. In other words, the top product of one time step forms the feed for the next time step. This is equivalent to having the top plate as the feed plate and the feed at its boiling point, which means q is unity. Also, the feed composition in the Underwood equations can be replaced by still composition. Therefore, the Underwood equations for the batch stripper shown in Figure 4 are given by

$$\sum_{i=1}^n \frac{\alpha_i x_s^{(i)}}{\alpha_i - \phi} = 0 \quad (11)$$

$$-Rb_{\text{min}} = \sum_{i=1}^n \frac{\alpha_i x_{\text{bot}}^{(i)}}{\alpha_i - \phi} \quad (12)$$

The Fenske and Underwood equations above provide the limiting boundary conditions for a batch stripper in terms of the still and bottom compositions. These limiting conditions should be related to the design variables of the column such as the reboil ratio Rb and the number of plates Nb , to complete the analysis. In batch rectifier, Gilliland's correlation (eq 13, Gilliland (1940)) furnishes this information for relating the design variables N and R with R_{min} and N_{min} .

$$X = \frac{R - R_{\text{min}}}{R + 1}; \quad Y = \frac{N - N_{\text{min}}}{N + 1}$$

$$Y = 1 - \exp \left[\frac{(1 + 54.4X)(X - 1)}{(11 + 117.2X)\sqrt{X}} \right] \quad (13)$$

For continuous distillation, from where the shortcut procedure for the rectifier is derived, FUG equations supply the complete design equations including the stripping section of the column. This is because in the continuous distillation the rectifying and stripping sections, and hence reflux and reboil ratios, are connected to each other by a steady-state material balance equation. However, for the batch stripper or for the middle vessel column, the steady-state balance equation does not exist. Therefore, a correlation similar to Gilliland's correlation needs to be obtained for the stripper. To get this correlation, we conducted a large number of systematic experiments using the new sampling technique (Diwekar and Kalagnanam, 1996). The samples are selected from various parameters which include relative volatility ($1.0 \geq \alpha \leq 5.0$), reboil ratio ($3.0 \geq Rb \leq 12.0$), number of plates ($4 \geq Nb \leq 15$), and still composition ($0.05 \geq x_s^{(1)} \leq 0.95$). At first the X and Y factors were defined based on the original Gilliland correlation and plotted in Figure 5.

$$X = \frac{Rb - Rb_{\text{min}}}{Rb}; \quad Y = \frac{Nb - Nb_{\text{min}}}{Nb + 1}$$

Unlike Gilliland's original plot for continuous distillation, where points line up nicely to form a curve, the results of the batch stripper in Figure 5 are widely dispersed. The preliminary analysis of the equations suggested a relative volatility factor to be included in the stripper correlation. The dependence of relative volatility is visible in Figure 6, plotted using the new definitions of X and Y given below.

$$X = \frac{Rb - Rb_{\text{min}}}{Rb} \ln(\alpha_{\text{LK}}/\alpha_{\text{HK}}); \quad Y = \frac{Nb - Nb_{\text{min}}}{Nb + 1}$$

A logarithmic curve fit with the following correlation resulted in a value of R^2 of 0.92.

$$Y = 0.2478 - 0.0965 \ln(3.784X) \quad (14)$$

The above correlation completes the shortcut model for the batch stripper. The following procedure illustrates the shortcut method for a single time step in detail for constant reboil mode of operation. Note that choosing the proper light key and heavy key components is crucial for the success of this method.

2.2. Solution Procedure. At any time step the still composition, $x_s^{(i)}$, $i = 1, 2, \dots, n$, can be found from the previous time steps using eq 5. Then the following procedure is used to obtain the bottom compositions.

1. Assume the initial value of C_B ($0 < C_B \leq Nb$).
2. Calculate the bottom product composition of the key component k using the HG equation and summation of all composition.

$$x_{\text{bot}}^{(k)} = \frac{1}{\sum_{i=1}^n \left(\frac{\alpha_i}{\alpha_k} \right)^{-C_B} \frac{x_s^{(i)}}{x_s^{(k)}}}$$

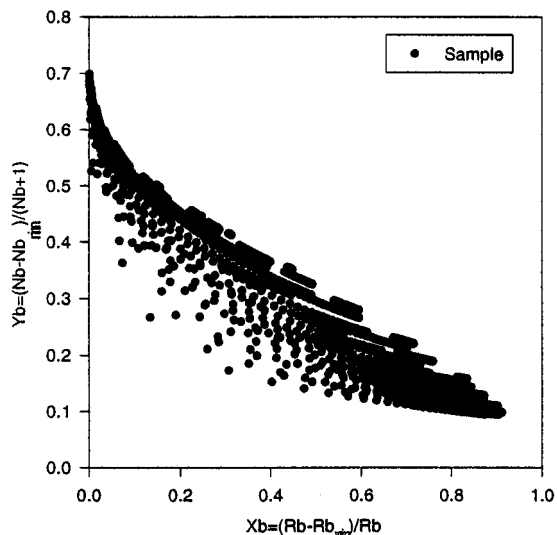


Figure 5. Gilliland's plot for a batch distillation stripper.

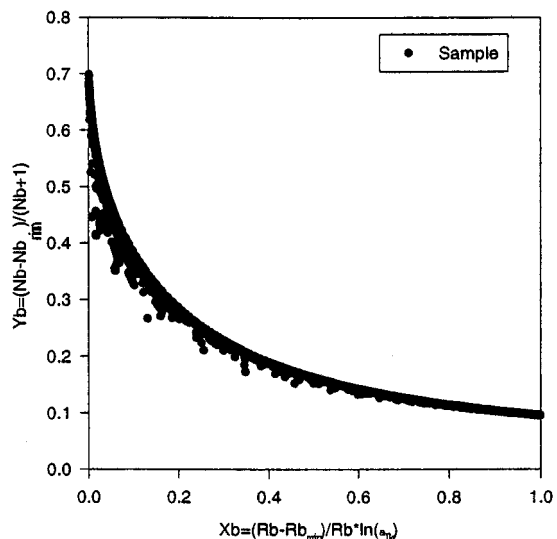


Figure 6. Modified Gilliland plot for a batch distillation stripper.

3. Substitute the value in Hengestebeck–Geddes' equation and find the bottom composition, $x_{\text{bot}}^{(i)}$, $i = 1, 2, \dots, n$.

$$x_{\text{bot}}^{(i)} = \left(\frac{\alpha_i}{\alpha_k} \right)^{-C_B x_{\text{bot}}^{(k)}} \frac{x_{\text{bot}}^{(k)}}{x_{\text{S}}^{(k)}} x_{\text{S}}^{(i)}, \quad i = 1, 2, \dots, n \quad (i \neq k)$$

4. Solve Underwood equations for ϕ and obtain the value of Underwood's minimum reboil ratio, Rb_{minu} .

$$\sum_{i=1}^n \frac{\alpha_i x_{\text{S}}^{(i)}}{\alpha_i - \phi} = 0$$

$$-Rb_{\text{minu}} = \sum_{i=1}^n \frac{\alpha_i x_{\text{bot}}^{(i)}}{\alpha_i - \phi}$$

5. Calculate Y using Nb_{min} equal to C_B and solve the modified Gilliland correlation for the stripper for X to obtain Gilliland's minimum reboil ratio, Rb_{ming} .

$$X = \frac{Rb - Rb_{\text{ming}}}{Rb} \ln(\alpha_{\text{LK}}/\alpha_{\text{HK}}); \quad Y = \frac{Nb - C_B}{Nb + 1}$$

$$Y = 0.2478 - 0.0965 \ln(3.784X)$$

6. For the correct value of C_B , Rb_{minu} should be equal to Rb_{ming} . Therefore, the value of the quantity Gb_c should be zero (within a tolerance).

$$Gb_c = \frac{Rb_{\text{minu}} - Rb_{\text{ming}}}{Rb}$$

7. Calculate Gb_c and find whether it is zero within a tolerance. If Gb_c is approximately zero, the solution is converged for this time step; otherwise, use the Newton–Raphson method to calculate the new C_B , and repeat all the steps from step 2.

2.3. Model Validation. To verify the accuracy of the shortcut model, several systematic experiments are conducted using the rigorous model (zero holdup) for the stripper implemented in *MultiBatchDS* (Diwekar, 1996) and the shortcut model presented here. At first binary mixtures of relative volatility varying from 4.0 to 1.15, reboil ratio from 3 to 12, and bottom plates from 4 to 15 are used. Table 1 presents the 10 representative cases from this study. Table 1 also presents the average error in the prediction of the bottom and still compositions. From the 10 cases in Table 1, details of the two cases (case 1 and case 9) are presented in Figures 7 and 8 which plot the composition profiles of the least volatile component. These two cases represent an easy (high α) and a difficult (low α) separation, respectively. Table 1 also reports four cases of the ternary mixture. It can be seen that the shortcut model predicted reasonably accurate results for binary as well as multicomponent systems. The accuracy of the shortcut method depends on the accuracy of the modified Gilliland correlation; therefore, for specific applicability one can improve this empirical correlation further.

Figures 7 and 8 also show that the bottom composition of the least volatile component decreases as the distillation progresses, a condition used for deriving one of the feasibility criteria for the stripper.

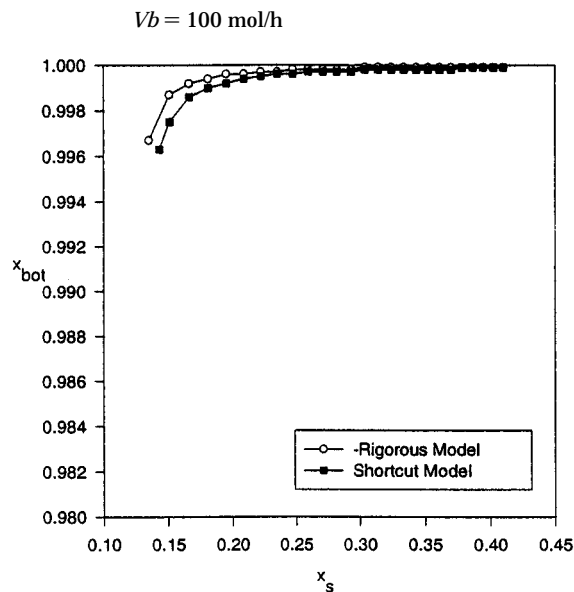
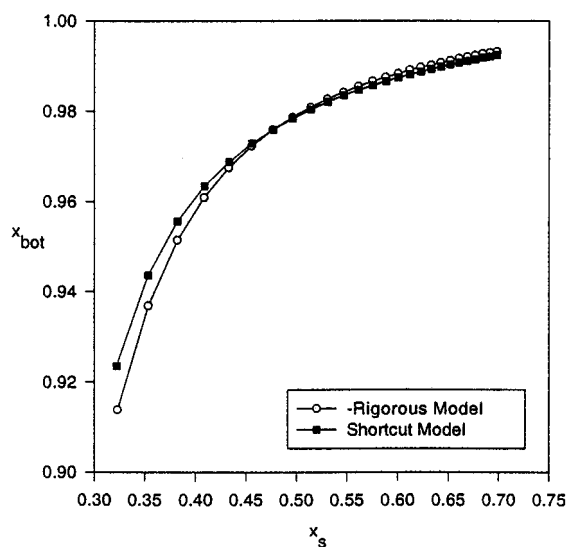
2.4. Feasibility of the Stripper. The determination of feasibility regions is crucial for optimization, synthesis, design, or control of a column. It is possible to easily identify the feasibility region with the help of the shortcut method and impose bounds. This section presents the feasibility criteria for the inverted column operating under constant reboil ratio mode and illustrates it with an example.

For specified average bottom product composition, the inverted batch distillation column has a lower bound Rb_{MIN} on the reboil ratio and the Fenske minimum number of plates provides the lower bound Nb_{min} on the number of plates. For the stripper, the initial composition of the least volatile component is the highest and decreases as the distillation progresses (as observed in Figures 7 and 8). If the initial value of Rb is such that the bottom composition of the least volatile component is less than the specified average, then the goal of attaining the specified average purity is impossible to meet for the given number of plates. This criterion provides the lower limit Rb_{MIN} on the value of Rb , where Rb_{MIN} is defined as the value of Rb required to obtain the bottom composition of the least volatile component equal to the specified average composition, for the given Nb . The reboil ratio at constant reboil policy has to be at least Rb_{MIN} to reach the desired product composition at initial conditions even if in this case the amount of product is almost zero. Rb_{MIN} is calculated by obtaining Nb_{min} and Rb_{min} from Fenske's and Underwood's equations by equating the bottom composition at the initial condition to the average specified composition and then using the modified Gilliland correlation to calculate the

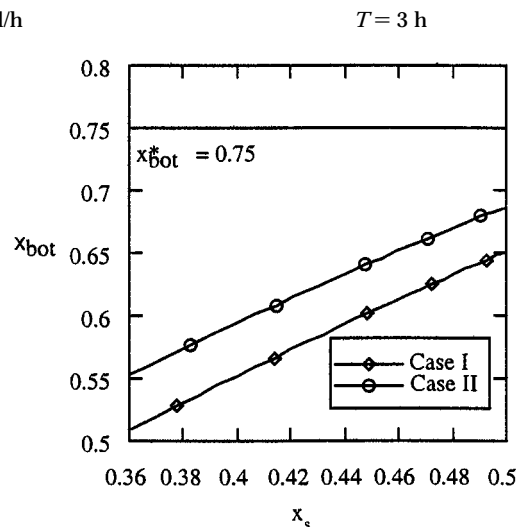
Table 1. Test Cases for the Validation of the Shortcut Method

test	α_1	α_2	Nb	Rb	$x_F^{(1)}$	average error for x_s (%)	average error for x_{bot} (%)
1	3.85	1.0	7	9.75	0.59	0.003	0.01
2	3.55	1.0	10	10.875	0.23	0.002	0.01
3	2.95	1.0	5	11.437	0.374	0.054	0.11
4	2.8	1.0	14	6.937	0.194	0.003	0.11
5	2.35	1.0	13	10.312	0.518	0.004	0.01
6	2.05	1.0	6	8.062	0.158	0.065	0.106
7	1.75	1.0	8	11.718	0.662	0.447	0.951
8	1.6	1.0	4	7.218	0.482	0.558	1.104
9	1.45	1.0	14	9.468	0.302	0.078	0.552
10	1.15	1.0	6	10.593	0.806	2.845	6.269

test	α_1	α_2	α_3	Nb	Rb	average error for x_s (%)	average error for x_{bot} (%)
1	1.7	1.2	1.0	3	8	0.257	0.077
2	2.5	1.3	1.0	7	6	6.00	1.600
3	4.5	2.3	1.0	5	6	1.277	0.571
4	5.6	3.3	1.0	5	4	0.183	0.741

**Figure 7.** Validation of the shortcut method for a batch stripper.**Figure 8.** Validation of the shortcut method for a batch stripper.

Rb from the given Nb and the values of Rb_{min} and Nb_{min} . It should be remembered that this Rb which is equal to Rb_{MIN} is different from Underwood's Rb_{min} , which stands for the minimum reboil ratio at an infinite number of plates and is also lower than Rb_{MIN} . The minimum reboil ratio Rb_{min} increases with time, thus showing that the separation becomes more difficult. Since the actual reboil ratio has to be higher than Rb_{MIN} ,

 $F = 100 \text{ mol/h}$ **Figure 9.** Concept of Nb_{min} .**Table 2. Feasible Region for a Batch Stripper (Constant Reboil Mode)**

final still composition	$0 \leq x_s^{(HK)} \leq x_{bot}^{(HK)}$
bottom composition	$x_{bot}^{(HK)} \leq x_{bot}^{(HK)} \leq 1$
reboil ratio	$1 \leq Rb / Rb_{MIN} \leq \infty$
number of plates	$Nb_{min} \leq Nb$

Table 3. Input for Illustration of the Nb_{min} and Rb_{MIN} Concept

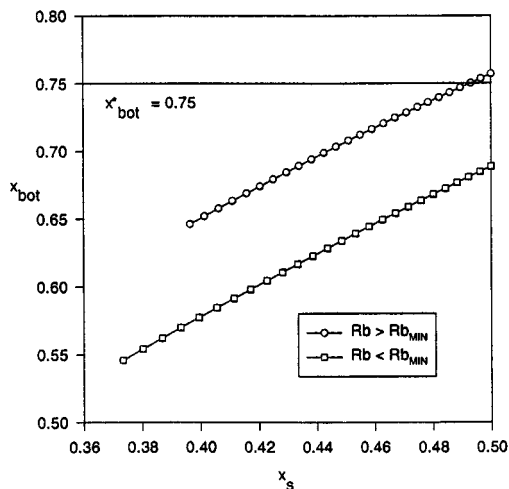
parameter	case I	case II	case III	case IV
Rb	5	50	3.0	2.0
Nb	1	1	6	6
Vb	100	100	100	100.0
α_1	1.5	1.5	1.5	1.5
α_2	1.0	1.0	1.0	1.0

the minimum reboil ratio serves as an indicator for the difficulty of a separation and gives information for the question if a column can be realized economically. Table 2 shows the feasibility region for the stripper.

The example presented in Table 3 serves as an illustration of the feasibility concept. An equimolar binary mixture is fed into an inverted batch distillation column and stripped off of the less volatile component. The purity of the bottom product was specified to be $x_{bot-ave}^{(2)} = 0.75$. At first consider case I, the composition profile of the bottom product for this case is shown in Figure 9. One can easily see that the composition profile cannot achieve the product purity specification. The figure also shows the results of case II, where the reflux ratio is 10 times higher, and it is still not possible to achieve the given specification. The reason can be found easily by calculating the lower bound of Nb , given

Table 4. Comparison of the Middle Vessel, Rectifier, and Stripper

column	α_1	α_2	R	Rb	Nt	Nb	Vt	Vb	$x_{\text{Dav}}^{(1)}$	$x_s(1)$	$x_{\text{bot-ave}}^{(2)}$
rectifier	1.7	1.0	4.0		8		50		0.9194	0.3202	
stripper	1.7	1.0		5.0		8		50		0.6690	0.8942
middle vessel	1.7	1.0	4.0	5.0	8	8	50	50	0.9522	0.4941	0.9427

**Figure 10.** Concept of Rb_{MIN} .

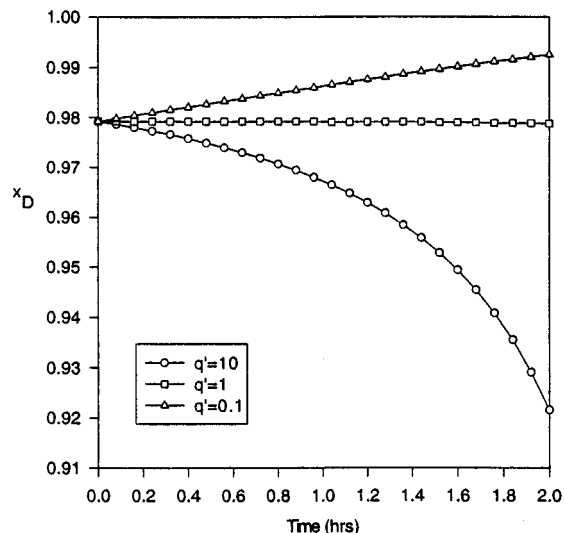
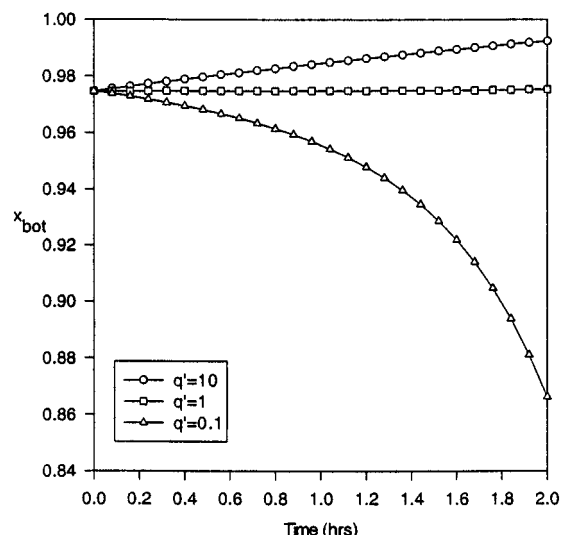
by Nb_{min} , and it was found to be 2.7 (including the reboiler). Since Nb should be greater than Nb_{min} (actually $Nb_{\text{min}} - 1$ when reboiler is excluded), it was impossible to attain a given separation. Now consider cases III and IV where Nb was increased from 1 to 6. The lower bound for the reboil ratio, Rb_{MIN} , was found to be 2.668 for the given value of Nb , and rigorous simulations with $R \geq Rb_{\text{MIN}}$ (case III) and $R \leq Rb_{\text{MIN}}$ (case IV) are computed. Figure 10 shows that, in case IV, the specified product conditions could not be reached because the reboil ratio was too small, whereas in case III, the desired composition could be reached at the beginning of the operation. To obtain a higher amount of product, the actual reboil ratio has to be increased appropriately.

3. Middle Vessel Column

The middle vessel column was proposed by Devidyan et al. (1994). This column consists of a rectifier and an inverted column connected by a still vessel (Figure 2). Hasebe et al. (1995) describe this connection as a heat integration for the rectifier and stripper. This configuration enables the simultaneous separation of light and heavy impurities and therefore offers more flexibility. In previous works, the directions a separation can take have been examined and the existence of equilibrium plateaus has been proved (Devidyan et al., 1994).

Rigorous simulations of this column with *MultiBatchDS* (Diwekar, 1996) show a promising behavior, as it can reach higher purities than conventional batch distillation columns and offers greater operation flexibility. If, for example, a binary mixture specified in Table 4 with an equimolar feed is distilled for 3 h to achieve 30 mol of distillate and bottom product, the results will differ from similar operations with a rectifier or a stripper. The average compositions of distillate and bottom product for the middle vessel are about 4% higher, even if none of the other parameters were changed and the same amount of distillate and bottom product was gathered, compared to the compositions of similar operations with a rectifier and a stripper.

The middle vessel column makes it possible to copy the behavior of a column operated at variable reflux

**Figure 11.** Distillate composition profiles for constant reflux and constant reboil operating mode.**Figure 12.** Bottom product composition profiles for constant reflux and constant reboil operating mode.

without having to change the reflux or reboil ratio. The goal of variable reflux operation is to maintain a specified product composition by changing the reflux ratio. This can be achieved in a middle vessel column by keeping the composition of the middle vessel constant. This means that the vapor boilup rates for the top and bottom part of the column and reflux and reboil ratios have to be designed in a way to make up the difference in mass balance equation (e.g., Figures 11 and 12 for $q' = Vt/Vb = 1$). Since this column consists of a rectifier and an inverted column both connected by the reservoir in the middle, its behavior becomes less predictable than a conventional column. For example, at constant reflux and reboil operation, the composition of the heavy key in the bottom product can increase with time (Figure 12, $q' = 10$), whereas in an inverted batch distillation column, it would decrease. A similar effect can be shown for the distillate (Figure 11, $q' = 0.1$). This behavior results from an increase of the composition of the light key component in the middle vessel due to

removal of the heavy components through the stripping part of the middle vessel column. The average product composition is higher than the composition resulting from a calculation where the product composition decreases with time.

3.1. Shortcut Model for the Middle Vessel Column. The shortcut model for the middle vessel column can be easily derived from the rectifier and the stripper shortcut model and is described below.

Since the mass balance around the middle vessel constitutes the main difference between a middle vessel column, an inverted column, and a rectifier, it is responsible for the change in column behavior and has therefore to be examined thoroughly.

$$\frac{dS}{dt} = \frac{-Vb}{Rb} - \frac{Vt}{R+1}, \quad S_0 = F \quad (15)$$

$$\frac{dBot}{dt} = \frac{Vb}{Rb}, \quad Bot_0 = 0.0 \quad (16)$$

$$x_{bot}^{(k)} \frac{Vb}{Rb} + x_D^{(k)} \frac{Vt}{R+1} = - \frac{d(x_s^{(k)} S)}{dt}, \quad x_s^{(k)} = x_F^{(k)} \quad (17)$$

$$\frac{dx_s^{(k)}}{dt} = \frac{Vb}{(Rb)S} (x_s^{(k)} - x_{bot}^{(k)}) + \frac{Vt}{(R+1)S} (x_s^{(k)} - x_D^{(k)}), \quad x_{s0}^{(k)} = x_F^{(k)} \quad (18)$$

where Vt is the vapor boilup rate for the top part of the column and $x_D^{(k)}$ is the distillate composition of the key component. The combination of the overall and component mass balance leads to an equation that updates the still composition in terms of the distillate and the bottom product composition.

$$x_{s_{new}}^{(j)} = x_{s_{old}}^{(j)} + \frac{\Delta x_s^{(k)} (x_{bot}^{(j)} - x_s^{(j)})_{old}}{(x_{bot}^{(k)} - x_s^{(k)})_{old}} + \frac{\Delta x_s^{(k)} (x_D^{(j)} - x_s^{(j)})_{old}}{(x_D^{(k)} - x_s^{(k)})_{old}}, \quad i = 1, 2, \dots, i \neq k, n \quad (19)$$

Once the composition of the distillate and the bottom product is known, the new still composition can be calculated. These two compositions result from Hengestebek-Geddes' equation, with C_B and C_T standing for the minimum number of plates for the top, Nt_{min} , and the bottom part, Nb_{min} , of the column, respectively.

$$x_D^{(j)} = \left(\frac{\alpha_j}{\alpha_k} \right)^{C_T} \frac{x_s^{(j)}}{x_s^{(k)}} x_D^{(k)}, \quad x_{bot}^{(j)} = \left(\frac{\alpha_j}{\alpha_k} \right)^{-C_B} \frac{x_s^{(j)}}{x_s^{(k)}} x_{bot}^{(k)}, \quad i = 1, 2, \dots, n \quad (20)$$

$$Nt_{min} = \frac{\ln \left[\frac{x_D^{(j)} x_s^{(k)}}{x_D^{(k)} x_s^{(j)}} \right]}{\ln \left[\frac{\alpha_j}{\alpha_k} \right]}, \quad Nb_{min} = \frac{\ln \left[\frac{x_s^{(j)} x_{bot}^{(k)}}{x_s^{(k)} x_{bot}^{(j)}} \right]}{\ln \left[\frac{\alpha_j}{\alpha_k} \right]} \quad (21)$$

The bottom composition of the key component and the distillate composition of the key component can be expressed in terms of the still composition.

$$x_D^{(k)} = \frac{1}{\sum_{i=1}^n \left(\frac{\alpha_i}{\alpha_k} \right)^{C_T} \frac{x_s^{(i)}}{x_s^{(k)}}}, \quad x_{bot}^{(k)} = \frac{1}{\sum_{i=1}^n \left(\frac{\alpha_i}{\alpha_k} \right)^{-C_B} \frac{x_s^{(i)}}{x_s^{(k)}}} \quad (22)$$

Similarly, Underwood's equations can predict Rb_{min} and R_{min} .

$$\sum_{i=1}^n \frac{\alpha_i x_s^{(i)}}{\alpha_i - \phi} = 0 \quad (23)$$

$$R_{min} + 1 = \sum_{i=1}^n \frac{\alpha_i x_D^{(i)}}{\alpha_i - \phi}, \quad -Rb_{min} = \sum_{i=1}^n \frac{\alpha_i x_{bot}^{(i)}}{\alpha_i - \phi} \quad (24)$$

The relationships between R , R_{min} , Nt , and Nt_{min} for the top and between Rb , Rb_{min} , Nb , and Nb_{min} for the bottom are given by empirical relations such as the Gilliland correlation (shown in Figure 13). Since, in the case of middle vessel, we have to solve the equations iteratively for both the stripping section and the rectifying section of the column, we simplified this empirical correlation further by assuming linear correlations given below. These correlations are obtained by fitting the data for several columns as shown in Figures 14 and 15. From the figures it can be seen that the linear correlation is only valid between $Yt \leq 0.6$ for the rectifier and $Yb \leq 0.55$. For wider applicability one can always use eq 13 for the rectifying section and eq 14 for the stripping section.

$$Yt = 0.5515 - 0.5948Xt \quad (25)$$

$$Yb = 0.6187 - 0.5655Xb \quad (26)$$

$$Xt = \frac{R - R_{min}}{R + 1}, \quad Yt = \frac{Nt - Nt_{min}}{Nt + 1}$$

$$Xb = \frac{Rb - Rb_{min}}{Rb}, \quad Yb = \frac{Nb - Nb_{min}}{Nb + 1}$$

Thus, eqs 15–26 form the shortcut model for the middle vessel column. At each time step the model needs to solve three differential equations (eqs 15, 16, and 18) and the algebraic equations consisting of eq 19, HG equations, and FUG equations iteratively. The following subsection describes this procedure for constant reflux/constant reboil mode of operation.

3.2. Solution Procedure. At any time step the still composition, $x_s^{(j)}$, $i = 1, 2, \dots, n$, can be found from the previous time steps using eq 19. Then the following procedure is used to obtain the bottom and top compositions.

1. Assume the initial values of C_T and C_B . Remember that C_T and C_B should be greater than zero but less than the number of plates in the respective sections.

2. Calculate the distillate and bottom product compositions of the key component k by using the corresponding Hengestebek-Geddes equation and summation of all compositions.

$$x_D^{(k)} = \frac{1}{\sum_{i=1}^n \left(\frac{\alpha_i}{\alpha_k} \right)^{C_T} \frac{x_s^{(i)}}{x_s^{(k)}}}, \quad x_{bot}^{(k)} = \frac{1}{\sum_{i=1}^n \left(\frac{\alpha_i}{\alpha_k} \right)^{-C_B} \frac{x_s^{(i)}}{x_s^{(k)}}}$$

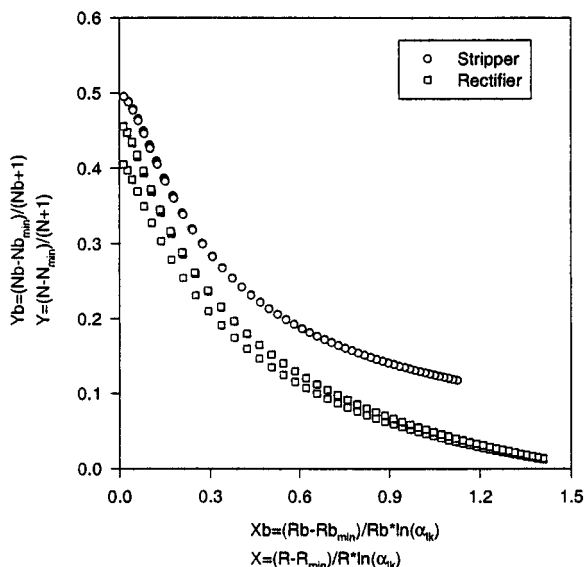


Figure 13. Gilliland's plot for the middle vessel column.

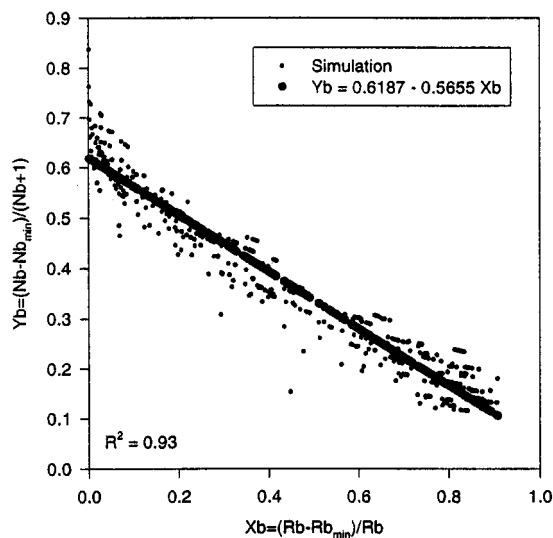


Figure 14. Linear curve fit for the stripping section in the middle vessel column.

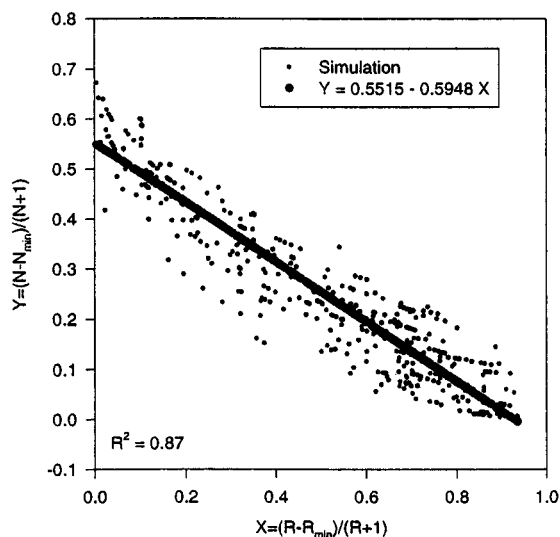


Figure 15. Linear curve fit for the rectifying section in the middle vessel column.

3. Find all other distillate and bottom compositions by using HG equations.

$$X_D^{(j)} = \left(\frac{\alpha_j}{\alpha_k} \right)^{C_T} \frac{X_D^{(k)}}{X_S^{(k)}} X_S^{(j)}, \quad X_{\text{bot}}^{(j)} = \left(\frac{\alpha_j}{\alpha_k} \right)^{-C_B} \frac{X_{\text{bot}}^{(k)}}{X_S^{(k)}} X_S^{(j)}, \quad i = 1, 2, \dots, n \quad (i \neq k)$$

4. Solve the Underwood equations for ϕ and obtain the value of Underwood's minimum reflux ratio, R_{minu} , and minimum reboil ratio, Rb_{minu} .

$$\sum_{i=1}^n \frac{\alpha_i X_S^{(i)}}{\alpha_i - \phi} = 0$$

$$R_{\text{minu}} + 1 = \sum_{i=1}^n \frac{\alpha_i X_D^{(i)}}{\alpha_i - \phi}, \quad -Rb_{\text{minu}} = \sum_{i=1}^n \frac{\alpha_i X_{\text{bot}}^{(i)}}{\alpha_i - \phi}$$

5. Calculate Yt and Yb using Nt_{min} (equivalent to C_T) and Nb_{min} (equivalent to C_B) and solve the modified Gilliland correlations for the Xt and Xb , respectively.

$$Xt = \frac{R - R_{\text{ming}}}{R + 1}, \quad Yt = \frac{Nt - C_T}{Nt + 1}$$

$$Yt = 0.5515 - 0.5948 Xt$$

$$Xb = \frac{Rb - Rb_{\text{ming}}}{Rb}, \quad Yb = \frac{Nb - C_B}{Nb + 1}$$

$$Yb = 0.6187 - 0.5655 Xb$$

6. For the correct values of C_T , R_{minu} should be equal to R_{ming} ; hence, Gt_c should be equal to zero. Similarly, Rb_{minu} should be equal to Rb_{ming} . Therefore, the value of the quantity Gb_c should be zero.

$$Gt_c = \frac{R_{\text{minu}} - R_{\text{ming}}}{R + 1}$$

$$Gb_c = \frac{Rb_{\text{minu}} - Rb_{\text{ming}}}{Rb}$$

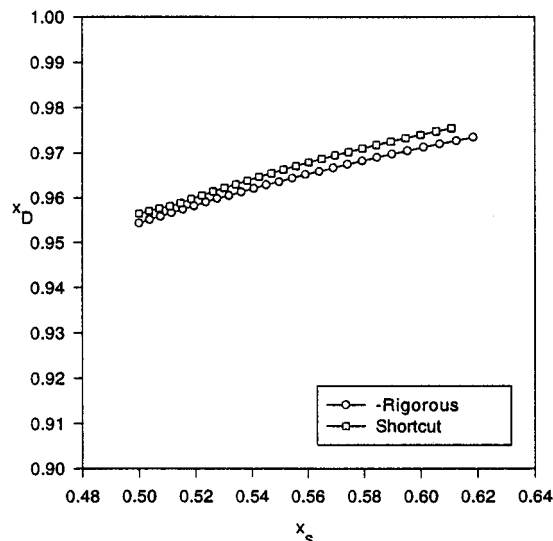
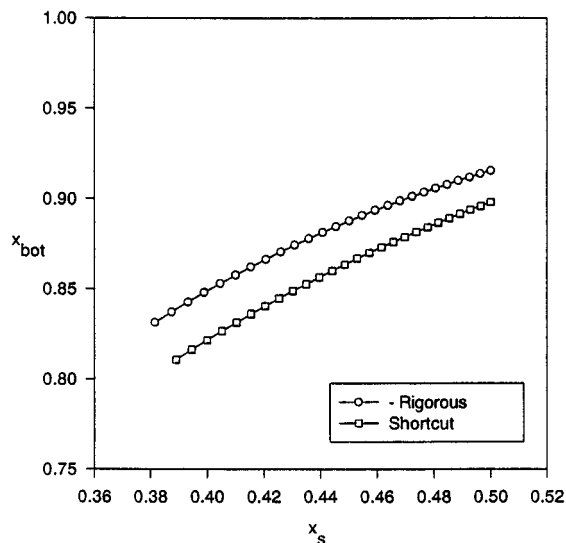
7. For the top column iterate on C_T till Gt_c is zero within a tolerance, and for the bottom portion of the column iterate on C_B for Gb_c to be negligible.

3.3. Model Validation. The middle vessel column offers greater flexibility to batch distillation, and there are a large number of various design variables which can be changed to achieve the desired separations. In the validation of the shortcut model for the middle vessel column, a large number of parameters are thus varied. First, a binary mixture is tested and then testing is extended to multicomponent mixtures. The relative volatility of the light component (α_1), the reflux ratio (R), the reboil ratio (Rb), the number of equilibrium plates for the top and bottom section (Nt , Nb), and the ratio between the vapor rates ($q' = Vt/Vb$) are changed (Table 5). To cover as many variations as possible, a systematic sampling across all the variables is carried out using the HSS sampling technique (Diwekar and Kalagnanam, 1996). For the examples in Table 5, the feed mixture is equimolar and the second relative volatility is set at a constant.

The differences between the results of the rigorous simulation and the shortcut model are negligible for the binary feed mixture. The average product compositions vary 2% at most, whereas the compositions at each time step differ by less than 1% on average. Thus the shortcut procedure for the middle vessel simulates the distillation with satisfying accuracy for a binary mixture

Table 5. Test Cases for the Validation of the Shortcut Method for the Middle Vessel Column

test	α_1	α_2	Nt	Nb	R	Rb	q'	average % error in composition	max % error in composition
1	3.0175	1.0	8	6	6.0	6.57	1.64	0.085	0.18
2	2.835	1.0	6	9	5.0	5.86	1.46	0.047	0.12
3	2.6525	1.0	9	7	4.0	5.14	1.28	0.115	0.3
4	2.2875	1.0	8	8	6.8	3.71	0.92	1.139	4.45
5	2.105	1.0	5	7	5.8	7.90	0.74	0.437	0.67
6	1.9225	1.0	9	5	4.8	7.18	0.56	0.856	1.98
7	1.74	1.0	7	9	3.8	6.47	0.38	0.803	2.37
8	1.5575	1.0	8	8	7.6	5.76	0.20	1.141	2.53
9	1.375	1.0	5	6	6.6	5.04	1.98	0.904	1.74
10	1.1925	1.0	9	9	5.6	4.33	1.80	0.193	0.36

**Figure 16.** Validation of shortcut method for the middle vessel column.**Figure 17.** Validation of shortcut method for the middle vessel column.

at constant reboil and constant reflux policy (Figures 16 and 17). Note that, because of the limitation of the linear form of the Gilliland correlations for the top and bottom sections of the column, the test examples do not include extreme values of Xt or Xb .

For the shortcut procedure for multicomponent mixtures light and heavy key components have to be chosen properly and can also change during the simulation. To validate the shortcut model for the middle vessel column, several ideal ternary mixtures have been tried. For these test cases the average error is found to be less than 5%.

3.4. Feasibility Regions for the Middle Vessel Column.

Due to the nature of the middle vessel

column, finding feasibility regions is more difficult than for the batch distillation stripper. Three possible trends in composition profiles are observed when extensive test cases of a binary mixture are conducted. These three trends are also observed for ternary and multicomponent systems with particular combinations of parameters.

1. The distillate and bottom product compositions are constant throughout operation.

2. The distillate composition of the light key component increases, and the bottom product composition of the heavy key component decreases.

3. The bottom product composition of the heavy key component increases, and the distillate composition of the light key component decreases.

In the middle vessel column the dynamic behavior of the two sections is governed by the interactions of the two columns through the differential mass balance equation which is dependent on the design parameters Nt , Nb , R , and Rb as well as q' . Therefore, it is difficult to derive the feasibility regions for the middle vessel without detailed study of the parameter interactions. However, a preliminary judgment can be made for the feasibility windows for the three types of behavior (trends) given above.

The first case is similar to the continuous distillation where compositions are not changing significantly over the time period; the feasibility window can be specified in terms of the minimum reflux, minimum reboil ratio, and minimum number of plates given by $Nt \leq Nt_{min}$, $Nb \leq Nb_{min}$, $R \leq R_{min}$, and $Rb \leq Rb_{min}$. In the second case the bottom product composition for the heavy key component is decreasing, a behavior similar to that of the batch stripper. However, the top product composition of the light key component is increasing, a behavior different from that of a rectifier but desirable. Therefore, qualitatively one can argue that the limiting conditions in this case are due to the bottom column, and the stripper feasibility window for the bottom part of the column holds well. Similarly, for the third case the rectifying part of the column is limiting and a rectifier feasibility criterion is applicable.

4. Conclusions

In this paper shortcut models for the batch distillation stripper and middle vessel column are presented. These shortcut models provide a fast and reasonably accurate design tool for the emerging batch distillation columns. The models are based on the Fenske–Underwood–Gilliland method for continuous columns and hence are applicable to nearly ideal systems and columns with negligible holdups. Additionally, with these models it is possible to identify the feasibility window for operation of these new configurations. This paper also presented the feasibility criteria based on the shortcut model equations for these columns. This information

is crucial for iterative design, optimization, synthesis, and control problems.

Nomenclature

B_{ot} = amount in the reboiler (mol)
 C_B = constant in the Hengestebeck–Geddes equation for the stripper and stripping (bottom) part of the middle vessel
 C_T = constant in the Hengestebeck–Geddes equation for the rectifying (top) part of the middle vessel
 D = total distillate (mol)
 F = total feed (mol)
 HK = heavy key component
 LK = light key component
 n = number of components
 Nb = number of theoretical plates in the stripper or bottom part of the middle vessel
 Nt = number of theoretical plates in the top part of the middle vessel
 Nb_{min} = minimum number of plates in the stripper or bottom part of the middle vessel
 Nt_{min} = minimum number of plates in the top part of the middle vessel
 q = feed condition defined as the ratio of heat required to vaporize 1 mol of the feed to the molar latent heat of the feed
 q' = ratio of the vapor rate in the top section of the middle vessel to the vapor rate in the bottom section [Vt/Vb]
 R = reflux ratio
 R_{min} = minimum reflux ratio
 R_{MIN} = minimum feasible reflux ratio for the constant reflux operations
 R_{ming} = minimum reflux ratio given by the Gilliland correlation
 R_{minu} = minimum reflux ratio given by the Underwood equations
 Rb = reboil ratio
 Rb_{min} = minimum reboil ratio
 Rb_{MIN} = minimum feasible reboil ratio for the constant reboil operations
 Rb_{ming} = minimum reboil ratio given by the Gilliland correlation
 Rb_{minu} = minimum reboil ratio given by the Underwood equations
 S = amount remaining in the still (mol)
 S_0 = initial amount in the still (mol)
 t = integration time (h)
 $x_{bot-ave}^{(i)}$ = average bottom product composition of component i (mole fraction)
 $x_{bot}^{(i)}$ = liquid composition of component i in the bottom product (mole fraction)
 $x_D^{(i)}$ = distillate composition of component i (mole fraction)
 $x_{Dav}^{(i)}$ = average distillate composition of component i (mole fraction)

$x_F^{(i)}$ = feed composition of component i (mole fraction)

$x_S^{(i)}$ = liquid composition of component i in the still (mole fraction)

Vb = vapor rate in the bottom section of the column (mol/h)

Vt = vapor rate in the top section of the column (mol/h)

Greek Symbols

α_i = relative volatility of component i

ϕ = Underwood constant

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