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Shortcut Procedure for Inverted Batch Distillation Column (I) Multicomponent Ideal System

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Abstract Inverted batch distillation column(stripper) is opposed to a conventional batch distillation column(rectifier). It has a storage vessel at the top and products leave the column at the bottom. The batch stripper is favourable to separate mixtures with a small amount of light components by removing the heavy components as bottom products. In this paper, we are presenting a shortcut procedure based on our earlier work for design and simulation of the inverted batch distillation column, which is equivalent to the Fenske-Underwood-Gilliland procedure for continuous distillation. Given a separation task, we propose to compute the minimum number of stages($Nb_{\min}$) and the minimum reboil ratio($Rb_{\min}$) required in a batch stripper,which are the stages and reboil ratio required in a hypothetical inverted batch distillation column operating in total reboil ratio or having an infinite number of stages, respectively. Then, it is shown that the performance of inverted batch columns with a finite number of stages and reboil ratios could be correlated in Gilliland coordinates with the minimum stages $Nb_{\min}$ and the minimum reboil ratio $Rb_{\min}$.

Keywords inverted batch distillation column, stripper, shortcut procedure

1 INTRODUCTION

Batch distillation is widely used in specialty and fine chemicals, because of its inherent high flexibility. Some new configurations of batch distillation have been presented and paid more attention recent years[1−4], among which inverted batch distillation first presented by Robinson and Gilliland[5] (stripper, Fig. 1) is thought favourable for separation of mixtures with a small amount of light components by removing the heavy components as bottom products. Inverted batch distillation column is useful in synthesis batch distillation separation systems and essential to break azeotropes[6−8], especially when the minimum boiling azeotropes are present. In contrast to a conventional batch distillation, a stripper has its storage vessel at the top and the products leave the column at the bottom.

Although some commercial programs with rigorous simulation are available, shortcut procedure is still necessary to derive global properties, such as feasible regions of operation, which is important for optimization, optimal control, and synthesis problems. For multicomponents batch distillation, a shortcut model could avoid solving systems of stiff differential equations, which poses algorithmic problems, such as unconvergence. In this paper, we present shortcut procedures based on our eariler work[1,2] for inverted columns. For multicomponent ideal systems, our shortcut procedure is limited to constant relative volatilities and the liquid holdup on the plate is neglected. We first define the recovery of the separation task, the minimum number of stages and the minimum reboil ratio required by a batch stripper. Then, we use extensive simulation data to construct a correlation in Gilliland coordinates according to the minimum stages and reboil ratios. Finally, we summarize the design procedure and compare it with rigorous Hysys model for prediction of instantanous still and bottom compositions.

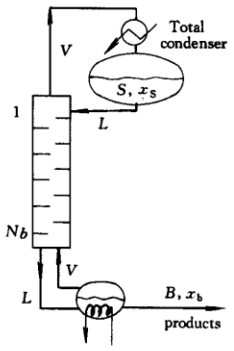

Figure 1 Inverted batch distillation column(stripper)

2 THEORETICAL FOUNDATION

2.1 Definition of recovery

The fractional recovery η_i is defined as the amount of component i in the bottom product after the separation, divided by the amount of component i in the still before the separation.

$$\eta_i = \frac{n_i^0 - n_i}{n_i^0} \tag{1}$$

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so

$$\mathrm{d}\,n_i = -n_i^0 \mathrm{d}\,\eta_i \tag{2}$$

where n_i^0 is the initial amount of i in the still, and n_i is the amount of i left in the still.

We set the stripping operation advance as

$$\varepsilon = \frac{S^0 - S}{S^0} \tag{3}$$

so

$$\mathrm{d}\,S = -S^0 \mathrm{d}\,\varepsilon \tag{4}$$

where S^0 is the initial total amount in the still, and S is the amount left in the still.

The material balance for component i is

$$\mathrm{d}\,n_i = x_{i,\mathrm{b}} \mathrm{d}\,S \tag{5}$$

Substituting Eqs. (2) and (4) into Eq.(5)

$$\frac{\mathrm{d}\,\eta_i}{\mathrm{d}\,\varepsilon} = x_{i,\mathrm{b}} \frac{S^0}{n_i^0} \tag{6}$$

since

$$x_{i,\mathrm{S}}^0 = \frac{n_i^0}{S^0} \tag{7}$$

Eq. (6) could be written as

$$\frac{\mathrm{d}\,\eta_i}{\mathrm{d}\,\varepsilon} = \frac{x_{i,\mathrm{b}}}{x_{i,\mathrm{S}}^0} \tag{8}$$

in which $x_{i,\mathrm{b}}$ is the mole fraction of component i in the bottom product, which varies with operating time. Therefore Eq. (8) should be integrated with the termination criterion , the recovery of the specified heavy key component.

2.2 The minimum stages

The differential material balances for each components are

$$\mathrm{d}\,n_i = x_{i,\mathrm{b}} \mathrm{d}\,S \tag{9}$$

where n_i is the moles of component i in the still, $\mathrm{d}\,S$ is the differential bottom product flowrate, and $x_{i,\mathrm{b}}$ is the mole fraction of component i at the bottom of the column, *i.e* the instantaneous composition of the bottom product. Choosing any component r as reference and dividing Eq. (9) by the equation corresponding to the reference gives

$$\frac{\mathrm{d}\,n_i}{\mathrm{d}\,n_r} = \frac{x_{i,\mathrm{b}}}{x_{r,\mathrm{b}}} \tag{10}$$

For a column operating at total reboil ratio (no product is taken out, so $V = L$), the distribution of components at any instant of the stripping is given by Fenske equation

$$\frac{x_{i,\mathrm{b}}}{x_{r,\mathrm{b}}} = \alpha_{i,r}^{-Nb} \frac{x_{i,\mathrm{S}}}{x_{r,\mathrm{S}}} \tag{11}$$

Eq. (11) relates the composition of any component i with that of component r through their relative volatilities $\alpha_{i,r}$ and the actual number of stages of this ideal total reboil column. Substituting Eq. (11) into Eq. (10),

$$\frac{\mathrm{d}\,n_i}{\mathrm{d}\,n_r} = \alpha_{i,r}^{-Nb} \frac{x_{i,\mathrm{S}}}{x_{r,\mathrm{S}}} \tag{12}$$

where subscript S refers to the top of the column(composition at the still). Multiplying and dividing the right hand side of Eq. (12) by the instantaneous holdup of the still and rearranging it, we have

$$\frac{\mathrm{d}\,n_i}{n_i} = \alpha_{i,r}^{-Nb} \frac{\mathrm{d}\,n_r}{n_r} \tag{13}$$

Integrating Eq. (13) between $n_i = S_i^0$ (feed to the stripper) and $n_i = S_i$ (amount of i left in the still after the separation) yields

$$\frac{S_i}{S_i^0} = \left(\frac{S_r}{S_r^0}\right)^{\alpha_{i,r}^{-Nb}} \tag{14}$$

Using the same definition of fractional recovery Eq. (1) and neglecting the holdup of the plate,we have

$$\frac{S_i}{S_i^0} = 1 - \eta_i \tag{15}$$

Substituting Eq. (15) into Eq. (14) yields

$$1 - \eta_i = (1 - \eta_r)^{\alpha_{i,r}^{-Nb}} \tag{16}$$

which is the partition functions for batch stripping. Note that Nb in Eq. (16) is the minimum number of stages required , because any batch stripper operating in a finite reboil ratio will require more stages to achieve the same separation. If the recoveries of two key components are set and substituted into Eq. (16), the value of the constant $Nb_{\min}$ can be computed.

$$Nb_{\min} = \frac{\ln\left[\dfrac{\ln(1-\eta_{\mathrm{h}})}{\ln(1-\eta_{\mathrm{L}})}\right]}{\ln \alpha_{\mathrm{L,h}}} \tag{17}$$

Then Eq. (16) can be applied setting $Nb = Nb_{\min}$ to predict the distribution of the remaining non-key components.

2.3 The minimum reboil ratio

The minimum reboil ratio for stripper is defined as the one required to achieve the desired separation with an infinite number of stages. For an inverted batch column(stripper), to get the minimum reboil ratio $Rb_{\min}$, Underwood equations for continuous distillation column are applied[9], *i.e.*

$$\sum_{i=1}^{n} \frac{\alpha_i x_{i,\mathrm{f}}}{\alpha_i - \phi} = 1 - q \tag{18}$$

$$-Rb_{\min} = \sum_{i=1}^{n} \frac{\alpha_i x_{i,\mathrm{b}}}{\alpha_i - \phi} \tag{19}$$

For a stripper, the feed composition in the Underwood equations can be replaced by still composition and feed at its boiling point, which means that q is unity. Therefore, the Underwood equations for the batch stripper could be written as

$$\sum_{i=1}^{n} \frac{\alpha_i x_{i,\mathrm{S}}}{\alpha_i - \phi} = 0 \tag{20}$$

$$-Rb_{\min} = \sum_{i=1}^{n} \frac{\alpha_i x_{i,\mathrm{b}}}{\alpha_i - \phi} \tag{21}$$

The integration of Eq. (8) for desired fractional recoveries should be performed by solving Eqs. (20) and (21) for the instantaneous still and bottom compositions. In the integration, the manipulated variable for converging to the desired fractional recoveries is Rb. The minimum reboil ratio $Rb_{\min}$ is obtained when Eq. (8) converges to the specified separation task. Here, we use the same procedure as the one in literature [1] to get the minimum reboil ratio for stripper.

Procedure for Computation of $Rb_{\min}$

Step 1 define the separation task $x^0_{i,\mathrm{S}}$, $\alpha_{i,\mathrm{h}}$, η^*_{L}, η^*_{h}

Step 2 assume a value of Rb

Step 3 integrate $\dfrac{\mathrm{d}\eta_i}{\mathrm{d}\varepsilon} = \dfrac{x_{i,\mathrm{b}}}{x^0_{i,\mathrm{S}}}$,

$\varepsilon = \dfrac{S^0 - S}{S^0}$, $0 \leqslant \varepsilon \leqslant 1$

down to $\eta_{\mathrm{h}} = \eta^*_{\mathrm{h}}$

Tracking $\eta_{\mathrm{h}} = \dfrac{n^0_{\mathrm{h}} - n_{\mathrm{h}}}{n^0_{\mathrm{h}}}$ with $x^0_{i,\mathrm{S}} = \dfrac{n^0_i}{\sum_{i=1}^{C} n^0_i}$, and $x_{i,\mathrm{b}}$ is calculated in subprocedure A.

Step 4 compute the light key fractional recovery

$$\eta_L = \frac{n^0_{\mathrm{L}} - n_{\mathrm{L}}}{n^0_{\mathrm{L}}}$$

Step 5 if $\eta_L <> \eta^*_{\mathrm{L}}$, assume a new value of Rb and go to step 3.

Step 6 $Rb_{\min} = Rb$

Subprocedure A

Step 1 given $x_{i,\mathrm{S}}$, $\alpha_{i,\mathrm{h}}$, Rb

Step 2 find the C-1 roots θ of

$$\sum_{i=1}^{C} \frac{\alpha_{i,\mathrm{h}} x_{i,\mathrm{S}}}{\alpha_{i,\mathrm{h}} - \theta} = 0$$

Step 3 obtain bottom compositions $x_{i,\mathrm{b}}$ solving the linear system

$$Ax = b$$

in which, $\alpha_{ij} = \dfrac{\alpha_j}{\alpha_j - \theta_i}$ i=1, $\cdots$, C-1; $a_{C,j} = 1$

$b_i = -Rb$ i=1, $\cdots$, C-1; $b_C = 1$

Step 4 if $x_{C,\mathrm{b}} < 0$,

then let $x_{C,\mathrm{b}} = 0$, C=C-1, and go to step 3

Step 5 return $x_{i,\mathrm{b}}$

3 SIMULATION MODEL

For a real inverted batch distillation column with the number of stages Nb, and assumed Nb, we can use the following method to simulate the column. We assume constant volatilities and constant molal overflow and neglect the column holdup.

The equilibrium relations and mass balances are given by as follows

the first stage,

$$px_{i,1} + k_{i,1}x_{i,1} - k_{i,2}x_{i,2} - px_{i,\mathrm{S}} = 0 \tag{22}$$

the jth stage,

$$px_{i,j-1} + k_{i,j+1}x_{i,j+1} - (p + k_{i,j})x_{i,j} = 0 \tag{23}$$

the reboiler,

$$(1 + Rb)x_{i,Nb} - (1 + Rbk_{i,\mathrm{b}})x_{i,\mathrm{b}} = 0 \tag{24}$$

in which $p = \dfrac{1 + Rb}{Rb}$, and subscript j corresponds to successive separation stages, starting from the top. At each integration step for Eq. (8), the instantaneous values of $x_{i,\mathrm{b}}$ are computed using Newton-Raphson method. The simulations are conducted by adjusting the reboil ratio Rb until the specified recoveries of both heavy and light key components are reached.

4 A CORRELATION

A correlation similar to Gilliland's correlation[10] is obtained for the stripper. We applied a lot of cases (all of them are mixtures with 5 components) in the simulation, which include relative volatilities ($1.0 \leqslant \alpha \leqslant 8.0$), reboil ratios ($2.7 \leqslant Rb \leqslant 20.0$), number of plates ($3 \leqslant Nb \leqslant 20$), and the key components recoveries($0.01 \leqslant \eta_{\mathrm{L}} \leqslant 0.15$, $0.85 \leqslant \eta_{\mathrm{h}} \leqslant 0.99$). The Xb and Yb factors are defined based on the original Gilliland correlation and plotted in Fig. 2.

$$Xb = \frac{Rb - Rb_{\min}}{Rb + 1},$$

$$Yb = \frac{Nb - Nb_{\min}}{Nb + 1}$$

An exponential curve fits well with the simulation results

$$Yb = 0.78(1.0 - Xb^{0.52}) \tag{25}$$

The above correlation completes the shortcut model for the batch stripper. From Fig. 2, we see that the

Gilliland coordinates fit a batch stripper very well. However, if the coordinates suggested by literature[4] is used a worse correlation is obtained(Fig. 3).

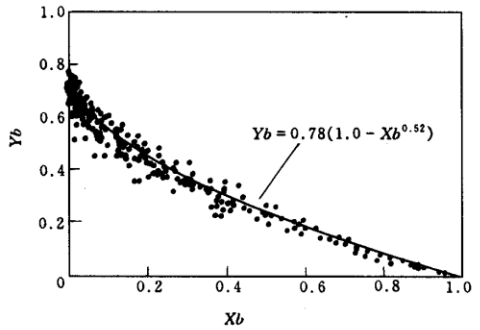


Figure 2 Gilliland's plot for a stripper

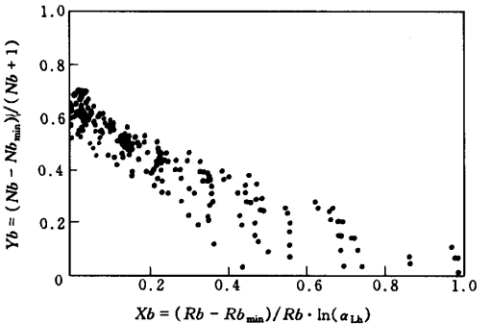


Figure 3 Modified Gilliland plot for a stripper

5 SUMMARY OF THE DESIGN PROCEDURE

For any given tasks, the design procedure can be summarized as follows:

(1) Define the separation task by specifying the feed compositions, the relative volatilities between components, and fractional recoveries for any two key components.

(2) Find $Nb_{\min}$ using Eq. (17), and find $Rb_{\min}$ by integration of Eq. (8) using the instantaneous still and bottom compositions solved from Eqs. (20) and (21). The procedure is outlined in preceding 2.4 and Table 1.

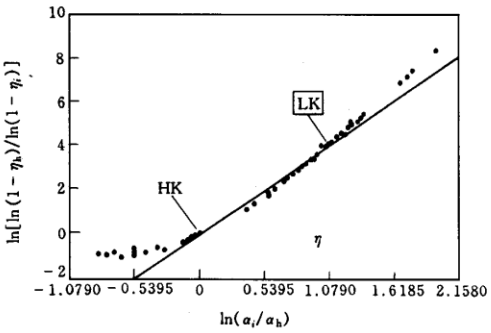
(3) Use the appropriate Yb *vs* Xb correlation (Eq. 25) for the inverted batch column design to get actual number of stages Nb and reboil ratio Rb with $Nb_{\min}$ and $Rb_{\min}$.

6 MODEL VALIDATION

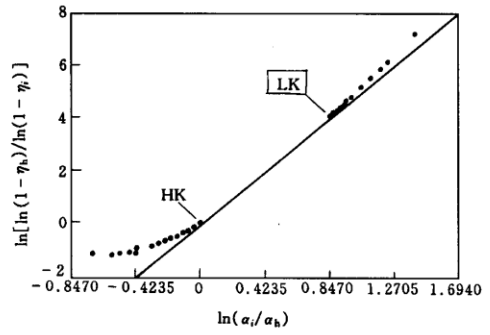
6.1 The distribution of non-key components

The mininum stages $Nb_{\min}$ for inverted batch distillation is given by Eq. (17), and the distribution of non-key components could be obtained from Eq. (16), in which the reference component r is usually the heavy key component, and Nb is $Nb_{\min}$.

We compared the non-key components distribution predicted by Eq. (16) with the present simulation results. In all the simulation cases, five components are used, in which the relative volatilities ($\alpha_L = 5.0$; $\alpha_h = 1.7$) and recoveries of the light key ($\eta_L = 0.05$) and heavy key ($\eta_h = 0.95$) are fixed. From Fig. 4(a) we can see that the straight line overestimates the recoveries of the components heavier than the heavy key and lighter than the light key, but it underestimates the recoveries of the components in between. If the two key components ($\alpha_L = 7.0$, $\alpha_h = 3.0$; $\eta_L = 0.05$, $\eta_h = 0.95$) are adjecent, Eq. (16) only overestimates the recoveries of the components lighter than the light key and heavier than the heavy key. Therefore the choice of the two key components for the batch stripper using Eq. (16) will result in discrepancy for distribution of the non-key components, but it should be noted that the discrepancy between simulated and predicted recoveries in Fig. 4 is not singnificant, which could also be concluded from the following comparison with the rigorous simulation model.



(a)



(b)

Figure 4 Distribution of non-key components in stripper

HK—heavy key component; LK—light key component

6.2 Comparison with rigorous model

To verify the accuracy of the shortcut procedure, several systematic comparisions with the rigorous model for the stripper are conducted. The rigorous simulations were performed with Hysys from Hyprotech Ltd(1997). The 12 representative cases are presented in Table 1, which also presents the average error in the prediction of the instantaneous bottom and still compositions. In all of the tests, excellent results were achieved with the shortcut model.

7 CONCLUSIONS

We have extended our shortcut procedure for rectifier to batch stripper. An analytical prediction of the minimum stages $Nb_{\min}$ for batch stripping distillation column is derived as a function of the specified fractional recoveries of the two key components. The prediction of $Nb_{\min}$ can be rearranged as a partition function for distribution of non-key components. The minimum reboil ratio $Rb_{\min}$ for a stripper column can be obtained according to the procedure for computation of $Rb_{\min}$. With a lot of real simulations of batch stripping columns with a finite number of stages and reboil ratios,we have derived an analytical function according to the Gilliland coordinates. Our shortcut procedure provides a fast and reasonably accurate design tool for inverted batch distillation columns.

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NOMENCLATURE

A, b	parameter of the equation
$a_{C,j}$	the parameters of C row(from 1 to j column)
B	bottom product flow rate, kmol·s^{-1}
C	the number of the components
$k_{i,j}$	vapor-liquid equilibrium constant of component i on the jth plate
L	liquid flow rate, kmol·s^{-1}
Nb	number of separation stages
$Nb_{\min}$	minimum number of stages to perform a separation task by a stripper
n_i	amount of component i in the still, kmol
q	thermal state of the feed
Rb	reboil ratio
$Rb_{\min}$	minimum reboil ratio to perform a separation task
r	reference component
S	amount of residue in the still, kmol
V	vapor flow rate, kmol·s^{-1}
Xb	Gilliland coordinate for reboil ratio
x	mole fraction of liquid phase
Yb	Gilliland coordinate for number of stages
y	mole fraction of vapor phase
α_i	relative volatility of component i
ε	stripper operation advance
η	mole fraction recovery
θ	root of Underwood equation
ϕ	root of Underwood equation

Superscripts

0	initial
*	specification

Subscripts

b	bottom
f	feed
h	heavy key component
i	component or intermediate composition in binary separation
j	number of stages starting from top
L	light key component
S	still

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Table 1 Test cases for validation of the shortcut method

Cases	$x^0_{i,S}$	Nb	Rb	Average error for x_S, %	Average error for x_b, %
1	0.2, 0.2, 0.6	10	8.5	0.431	0.443
2	0.2, 0.2, 0.6	14	2.25	0.363	0.743
3	0.2, 0.2, 0.6	6	4.5	0.276	0.024
4	0.3, 0.3, 0.4	12	1.25	0.206	2.491
5	0.3, 0.3, 0.4	8	3.4	1.918	5.228
6	0.3, 0.3, 0.4	5	8.0	1.889	0.0
7	0.1, 0.1, 0.1, 0.1, 0.6	8	2.1	1.143	6.225
8	0.1, 0.1, 0.1, 0.1, 0.6	6	5.0	1.705	4.663
9	0.1, 0.1, 0.1, 0.1, 0.6	4	5.0	1.44	1.191
10	0.2, 0.2, 0.2, 0.2, 0.2	4	2.2	1.714	2.771
11	0.2, 0.2, 0.2, 0.2, 0.2	4	7.5	3.388	2.197
12	0.2, 0.2, 0.2, 0.2, 0.2	4	5.0	3.149	3.642

cases 1—3, methanol, ethanol, 2-propanol, $p = 1.013 \times 10^5$ Pa; cases 4—6, methanol, ethanol, 1-propanol, $p = 1.013 \times 10^5$ Pa; cases 7—12, methanol, ethanol, 2-propanol, 1-propanol, 1-butanol, $p = 1.013 \times 10^5$ Pa;)

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