

# Multicomponent Batch Distillation with Distillate Receiver

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**Abstract**—Based on the assumption of adiabatic equilibrium stages, a rigorous calculation procedure applicable to a multicomponent batch distillation with a distillate receiver under total reflux condition was developed. Provided that the operating conditions including the desired product purity of the most volatile component in the receiver are specified, the proposed model estimates the volume of the distillate receiver, changes in compositions in the receiver and still, concentration profiles of the column, and the batch time required to complete the separation. In order to test the validity of the proposed model, experimental data for the separation of acetone-methanol-2-propanol mixture using a 10 cm I.D. column having six theoretical stages were compared with the simulation results for the two cases when the distillate receiver is initially empty and initially full.

**Key words:** Multicomponent Batch Distillation, Distillate Receiver, Total Reflux, Concentration Profile, Batch Time

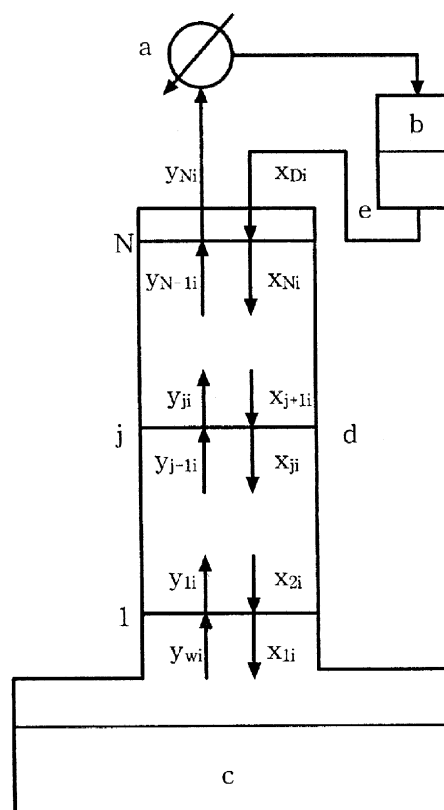
## INTRODUCTION

Batch distillation is the most widely used method of separating liquid mixtures in the batch processing industry. The flexibility in operation and the lower costs for the separation of relatively pure components are the advantages of batch distillation over continuous distillation. Moreover, the recent increase in the production of high added value, low volume specialty chemicals and biochemicals has created an additional interest in batch distillation.

There are two basic modes of operating a batch distillation column: (1) constant reflux and variable product composition, and (2) variable reflux and constant product composition. The behavior of these conventional columns can be analyzed either by rigorous stage-by-stage calculation methods [Meadows, 1963; Distefano, 1968; Boston et al., 1981; Choe and Luyben, 1987; Jang, 1993] or by shortcut methods [Farhat et al., 1990; Salomone et al., 1997; Lotter and Diwekar, 1997; Diwekar and Madhavan, 1991; Sundaram and Evans, 1993] assuming negligible holdups and constant molar overflows. Meadows [1963] developed the first rigorous multicomponent batch distillation model, based on the assumptions of adiabatic equilibrium stages and constant molar liquid holdups for stages and condenser. Distefano [1968] extended the model and developed a computer-based method for solving a set of differential mass and energy balance equations. Distefano's work forms the basis for almost all of the later work on rigorous modeling of batch distillation columns. Diwekar and Madhavan [1991] presented shortcut methods for the multicomponent batch distillation operation for the two cases of constant reflux and constant distillate composition. Sundaram and Evans [1993] also performed a simulation of the multicomponent batch distillation operations at constant reflux. Treating batch distillation as a sequence of continuous distillation, they calculated the vapor and liquid compositions at successive time steps using the Fenske-Underwood-Gilliland shortcut procedure for continuous distillation. Seader [1988] presented an excellent review of mod-

eling of batch distillation columns.

Another mode of operating a batch distillation column under total reflux condition was proposed by Treybal [1970]. A conventional apparatus with distillate receiver as depicted in Fig. 1 is used in this mode of operation. The equipment set-up has provision for total reflux. The batch charge is introduced into the still and the level of



**Fig. 1. Batch distillation column with a distillate receiver under total reflux conditions.**

(a) total condenser, (b) distillate receiver, (c) still, (d) column, (e) reflux line

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the overflow reflux line adjusted for the distillate receiver to hold a correct amount of distillate. Heat is then supplied and the distillation proceeds until the liquid in the distillate receiver becomes progressively richer in the light component reaching the desired composition. The operation can be performed with the distillate receiver either initially empty or filled-up with the original batch charge. The advantages of this operating mode are: (i) simplified equipment set-up because of no reflux control, and (ii) no influence by the variation in the heating rate on either the yield or the quality of the products. Moreover, since the column operates under total reflux condition, it operates at its maximum distillation capacity and fewer stages are required in comparison to the other mode of operation under finite reflux ratio. Applying this technique for separating a binary mixture, Treybal reported a shortcut calculation method for the dynamics of a binary column.

For the purpose of extending Treybal's mode of operation for a binary mixture to a multicomponent system, Kim and Ju [1999] proposed recently a shortcut calculation procedure assuming constant molar overflow, constant relative volatilities and negligible holdups. Comparing the simulation results with the experimental data obtained in the separation of a ternary mixture of benzene, toluene, and o-xylene, the authors proved the usefulness of the shortcut method for separating an ideal or nearly ideal multicomponent mixture using Treybal's mode of operation.

The objective of this study is to develop a rigorous calculation procedure for a batch distillation operation with a distillate receiver, which is applicable to separating a nonideal multicomponent mixture. Under the specification of the desired product concentration of the lightest component in the distillate receiver, the calculation procedure for determining the characteristics of the operation such as the volume of the distillate receiver, the variation of compositions in the distillate receiver and the still, and the stagewise concentration profiles are to be presented for two cases of startup policies of the distillate receiver. The validity of the proposed method is confirmed by comparing the theoretical concentration profiles with experimental results obtained for acetone-methanol-2-propanol system.

## ANALYSIS OF THE SYSTEM

### 1. Assumptions

$W^0$  mol of a multicomponent mixture, composed of  $n$  components 1, 2, ...,  $n$  in the order of decreasing volatility, are to be distilled in a batch column of  $N+1$  theoretical stages including the still as sketched in Fig. 1. Initial feed compositions are  $x_{wi}^0$ . The dynamic behavior of the column with a distillate receiver which operates under total reflux mode can be analyzed by using the following assumptions: (i) There are adiabatic equilibrium stages; (ii) there is same volume of liquid holdup on each plate; (iii) vapor holdup is negligible, and (iv) contents of the still and distillate receiver are well mixed.

### 2. Maximum Distillate Concentration of the Lightest Component ( $x_{D1}^{max}$ )

In order to perform a batch distillation operation with distillate receiver under total reflux condition, it is essential to find out the amount of the overhead product in advance of the startup of the operation since the level of the overflow reflux line must be adjusted so that the distillate receiver will contain the correct amount of the

distillate during the operation. The amount of the distillate product depends primarily upon its purity. Prior to specifying the product purity, it is necessary to examine the maximum product concentration obtainable by using the equipment at hand. Since the maximum distillate concentration of the lightest component ( $x_{D1}^{max}$ ) is the steady-state concentration attainable under total reflux condition without distillate receiver ( $D=0$ ), it is calculable by using the following equations.

Component balances for still ( $n$  equations):

$$L_1(x_{1i} - x_{wi}) - V_w(y_{wi} - x_{wi}) = 0 \quad i=1, 2, \dots, n \quad (1)$$

Component balances for column stages ( $nN$  equations):

$$V_{j-1}y_{j-1i} + L_{j+1}x_{j+1i} - V_jy_{ji} - L_jx_{ji} = 0 \quad i=1, 2, \dots, n \quad j=1, 2, \dots, N \quad (2)$$

Overall component balances ( $n$  equations):

$$W^0x_{wi}^0 - \sum_{j=1}^N U_j x_{ji} - \left( W^0 - \sum_{j=1}^N U_j - D \right) x_{wi} - Dx_{Di} = 0 \quad i=1, 2, \dots, n \quad (3)$$

Enthalpy balance for still (1 equation):

$$V_w H_w - L_1 h_1 - Q_R = 0 \quad (4)$$

Enthalpy balances for column stages ( $N$  equations):

$$V_{j-1}H_{j-1} + L_{j+1}h_{j+1} - V_jH_j - L_jh_j = 0 \quad j=1, 2, \dots, N \quad (5)$$

Flow rate identities for total reflux condition ( $N+1$  equations):

$$L_j = V_{j-1} \quad j=1, 2, \dots, N+1 \quad (6)$$

Concentration identities for total reflux condition [ $n(N+1)$  equations]:

$$y_{j-1i} = x_{ji} \quad i=1, 2, \dots, n \quad j=1, 2, \dots, N+1 \quad (7)$$

Condition for maximum concentration (1 equation):

$$D=0 \quad (8)$$

Eqs. (1)-(8) constitute a system of algebraic equations. The total number of equations is  $(2nN+3n+2N+3)$ . In these equations, values of  $W^0$ ,  $x_{wi}^0$ ,  $N$  and  $Q_R$  will be known since they are operating conditions. Since  $H_j$ ,  $h_j$  and  $U_j$  can be estimated from the literature correlations by using the composition, temperature and pressure for each stage, then the number of unknown variables, distributed as follows, is equal to the number of equations.

$x_{Di}$	$n$
$x_{ji}$ and $y_{ji}$	$2nN$
$x_{wi}$ and $y_{wi}$	$2n$
$V_j$	$N+1$
$L_j$	$N+1$
$D$	$1$
	<hr/>
	$2nN+3n+2N+3$

Hence Eqs. (1)-(8) can be solved by using an iterative solution procedure such as the Newton method. Value of  $x_{D1}$  obtained in this manner is the maximum distillate concentration ( $x_{D1}^{max}$ ). But if one desires this value as a distillate concentration, the volume of the distillate accumulated in the receiver will be null. Therefore, the desired product concentration ( $x_{D1}^F$ ) must be specified somewhat lower value than  $x_{D1}^{max}$  in order to obtain definite volume of distillate product.

### 3. Volume of the Distillate Receiver Corresponding to $x_{D1}^F$

The volume of the distillate product, or the volume of the distillate receiver (D mol) corresponding to a specified  $x_{D1}^F$  can be determined by solving once again the set of equations in the previous section. But in this case, Eq. (8) must be replaced by

$$x_{D1} = x_{D1}^F \quad (9)$$

### 4. Operation with a Distillate Receiver Initially Empty

#### 4-1. Analysis of the Regime for Rayleigh Distillation

Suppose initially the distillate receiver is empty. Then some time will be required to condense D mol of vapor to fill the receiver. During this time, there will be no reflux and the system undergoes a Rayleigh distillation. The changes in compositions and the variations of the amounts of the liquid in the distillate receiver and the still during this period can be estimated by the following equations.

Component material balances for the still (n equations):

$$\frac{dw_i}{dt} = -V_w y_{wi} \quad i=1, 2, \dots, n \quad (10)$$

Component material balances for the distillate receiver (n equations):

$$\frac{dd_i}{dt} = V_w y_{wi} \quad i=1, 2, \dots, n \quad (11)$$

There are 2n equations in 2n variables ( $w_i, d_i, i=1, 2, \dots, n$ ). An IMSL library routine such as IVPAG can be used to solve Eqs. (10) and (11) simultaneously using the following initial values:

$$w_i^0 = W^0 x_{wi}^0 \quad (12)$$

$$d_i^0 = 0 \quad (13)$$

The IVPAG routine solves an initial-value problem for first-order ordinary differential equations using the implicit Adams-Moulton method or Gear's BDF method. Then the volume and the composition of the distillate receiver and the still at any instant are determined by the relations

$$D_R = \sum_{i=1}^n d_i \quad (14)$$

$$x_{D1} = \frac{d_1}{D_R} \quad (15)$$

$$\text{and } W_R = \sum_{i=1}^n w_i \quad (16)$$

$$x_{wi} = \frac{w_i}{W_R} \quad (17)$$

During the calculation, the values of  $y_{wi}$  and  $V_w$  in Eqs. (10) and (11) are renewed at every time step ( $\Delta t=0.01$  min in this study) since the composition of the mixture in the still,  $x_{wi}$  varies continuously resulting in the variation of the latent heat of vaporization of the mixture.  $V_w$  is estimated from the division of the heating rate to the still by the latent heat of the mixture in the still, and  $y_{wi}$  is determined through the bubble point calculation of the mixture in the still. Since the contents of the distillate receiver increase continuously during Rayleigh distillation, the calculation is repeated until the volume of the receiver,  $D_R$  obtained by Eq. (14) grows to D. Multiplying the time increment  $\Delta t$  by this number of repetitions,

one can estimate the time required for Rayleigh distillation.

#### 4-2. Analysis of the Operation under Total Reflux

As the amount of the distillate in the receiver just becomes D mol by Rayleigh distillation, reflux runs down the column at the same molar rate as vapor boils up. From now on, the operation is conducted under total reflux mode. The dynamic behavior of the system can be described by using the following equations.

Component material balances for the distillate receiver, column stages, and still:

$$\frac{dx_{Di}}{dt} = \frac{V_N}{D} (y_{Ni} - x_{Di}) \quad i=1 \text{ to } n \quad (18)$$

$$\frac{dx_{ji}}{dt} = \frac{1}{U_j} [V_{j-1} y_{j-1i} + L_{j+1} x_{j+1i} - V_j y_{ji} - L_j x_{ji}] \quad i=1 \text{ to } n, j=1 \text{ to } N \quad (19)$$

$$\frac{dx_{wi}}{dt} = \frac{1}{W} [L_1 (x_{1i} - x_{wi}) - V_w (y_{wi} - x_{wi})] \quad i=1 \text{ to } n \quad (20)$$

Enthalpy balances for distillate receiver, adiabatic column stages, and still:

$$\frac{d(Dh_D)}{dt} = V_N H_N - L_D h_D - Q_C \quad (21)$$

$$\frac{d(U_j h_j)}{dt} = V_{j-1} H_{j-1} + L_{j+1} h_{j+1} - V_j H_j - L_j h_j \quad j=1 \text{ to } N \quad (22)$$

$$\frac{d(W h_w)}{dt} = Q_R + L_1 h_1 - V_w H_w \quad (23)$$

Phase equilibrium relations on column stages and in the still:

$$y_{ji} = K_{ji} x_{ji} \quad i=1 \text{ to } n, j=0 \text{ to } N \quad (24)$$

Mole fraction summations at column stages and in the still:

$$\sum_{i=1}^n y_{ji} = 1 \quad j=0 \text{ to } N \quad (25)$$

Molar holdups on the column stages:

$$U_j = G_j \rho_j \quad j=1 \text{ to } N \quad (26)$$

where  $G_j$  and  $\rho_j$  are the volume of the liquid holdup and the molar density of the liquid for j-th plate, respectively. Eqs. (18)-(26) constitute an initial value problem for a system of ordinary differential and algebraic equations. In this work, IVPAG routine was used to solve the set of equations at each time step. The initial stage compositions were taken by assuming linear variation between the compositions of the distillate receiver and the still at the end of Rayleigh distillation. K-values, vapor and liquid enthalpies, and liquid densities were estimated from Soave-Redlich-Kwong equation of state using the calculated values of stage compositions and temperatures. As the time step increases, the composition of the lightest component in the distillate receiver ( $x_{D1}$ ) varies continuously toward its ultimate value of  $x_{D1}^F$ . Counting the number of time steps, one can estimate the distillation time required for total reflux operation.

### 5. Operation Using the Distillate Receiver Initially Full

An alternate procedure is to start the operation with the distillate receiver full of the same liquid as the initial charge. In this procedure, there exists no region for Rayleigh distillation and the operation is carried out entirely under the condition of total reflux. Analysis of this mode of operation is analogous to that of operation with

**Table 1. Description of the batch column and operating conditions**

Distillation column
column: I.D. 10 cm, Pyrex glass, thermally insulated, 7 actual plates equivalent to 5 theoretical stages ( $N=5$ )
plate: cross-flow, sieve-plate type, number of holes/plate=251, hole diameter=0.2 cm, weir length=8.4 cm, weir height=0.5 cm, spacing=15 cm
still: 20 L, Pyrex glass, heated by electric mantle
Operating conditions
system: acetone(1)-methanol(2)-2-propanol(3)
initial charge: $W^0=220$ mol
feed composition: $x_{w1}^0=0.1449$ , $x_{w2}^0=0.3165$ , $x_{w3}^0=0.5386$
rate of heat transfer to still: $Q_R=1.47$ kW
product purity: $x_{D1}^F=0.66$
volume of the distillate receiver: $D=16.8$ mol

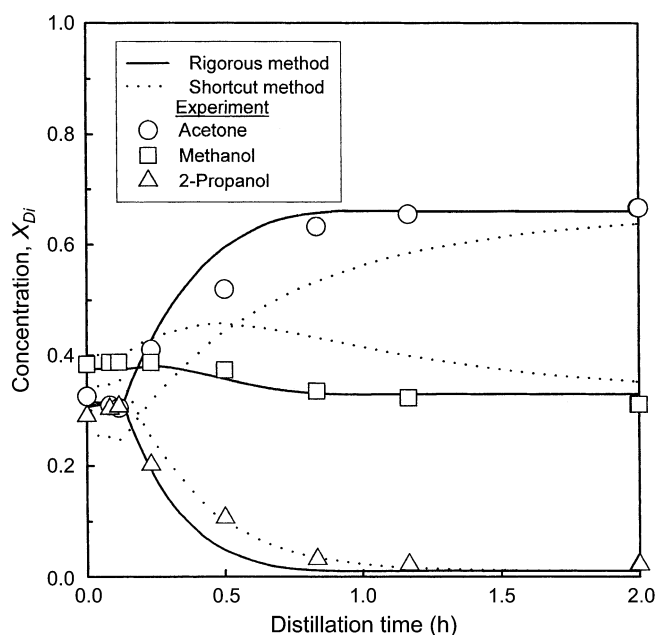
the receiver initially empty. Eqs. (18)-(26) are applied once again using  $x_{Di}^0$  and  $x_{wi}^0$  for the initial compositions of the receiver and the still.

### EXPERIMENTAL

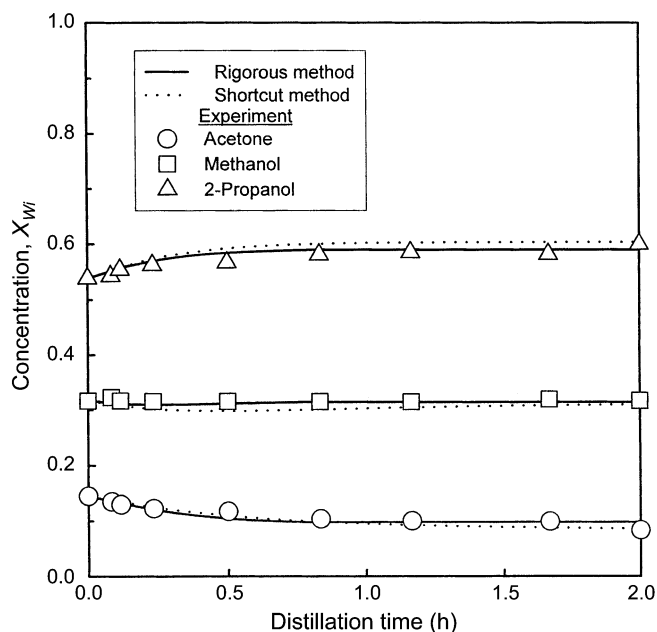
In order to test the validity of the calculation method proposed in this study, distillation experiments of a ternary system, a mixture of acetone, methanol, and 2-propanol, were undertaken by using a batch column depicted in Fig. 1. Two different modes of operation, that is, use of a distillate receiver initially empty and initially full with the original batch charge, were tested in these experiments. The details of the experimental apparatus and operating conditions are summarized in Table 1. In order to follow up the variation of the concentrations in the column, samples were taken from each stage, still and distillate receiver during experiments and analyzed by gas chromatography (Hewlett Packard 5890 series II plus).

### RESULTS AND DISCUSSION

By means of the calculation method proposed herein, one can estimate the maximum distillate concentration, the amount of the distillate product corresponding to the desired product purity, the variation of the compositions in the distillate receiver and the still, the stagewise concentration profiles at any instant during the distillation, and the batch distillation time required to complete the separation. Prior to the initiation of the operation, one must examine the ability of the equipment to deliver the specified product purity ( $x_{D1}^F$ ). If the product purity is attainable by the apparatus at hand, then the proposed method estimates the volume of the distillate receiver ( $D$  mol) corresponding to the specified purity. In case of the separation specified in Table 1, the maximum concentration of the component 1 (acetone) in the distillate receiver is 0.68, which is greater than the desired purity of  $x_{D1}^F=0.66$ . For this purity, the amount of the distillate product was calculated to be 16.8 mol. Fig. 2 shows the experimental variations of the compositions in the distillate receiver when the receiver is initially empty. Two different theoretical profiles, estimated by the rigorous calculation method proposed in this study and the shortcut method proposed by Kim and Ju [1999],

**Fig. 2. Changes in compositions in the distillate receiver. The receiver is initially empty.**

are also indicated in this figure. The agreement between the rigorous method and the experimental results is satisfactory while the differences between the shortcut method and the experimental data are considerable. It reveals that the shortcut method, developed originally for an ideal or nearly ideal multicomponent mixture, is not promising for the nonideal ternary mixture under consideration. Fig. 3 shows the variation of the compositions in the still when the receiver is initially empty. In this case, the conformity of the theoretical profiles with experimental results is excellent. Figs. 4 and 5 show

**Fig. 3. Changes in compositions in the still. The receiver is initially empty.**

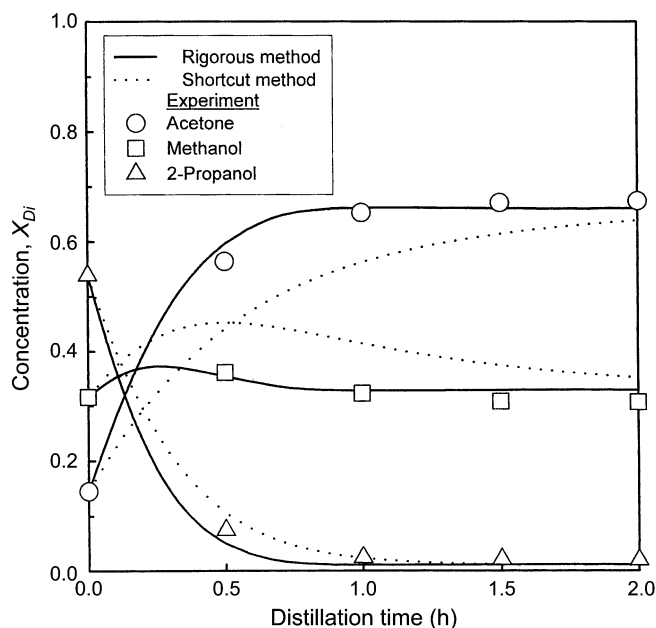


Fig. 4. Changes in compositions in the distillate receiver. The receiver is initially full with original batch charge.

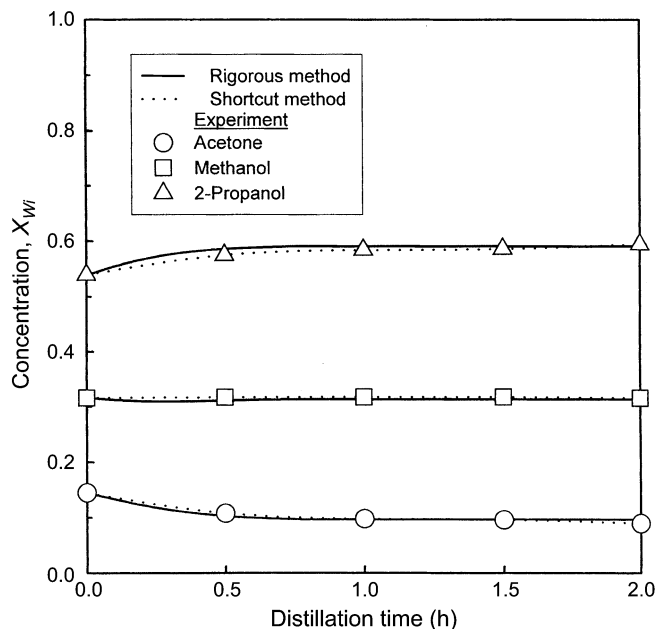


Fig. 5. Changes in compositions in the still. The receiver is initially full with original batch charge.

the trends of the composition change in the receiver and the still when the receiver is initially filled with the batch charge. Since the period of Rayleigh distillation does not exist in this case, the composition of acetone increases directly from the feed composition ( $x_{wi}^0$ ) to the desired product concentration ( $x_{Di}^F$ ). The predictions of the rigorous calculation method are excellent while the shortcut method gives unsatisfactory estimates in case of the distillate receiver. Fig. 6 represents the stagewise composition profiles of the liquid phase at the end of the distillation. Naturally, these final profiles are the same regardless of the initial condition of the distillate receiver.

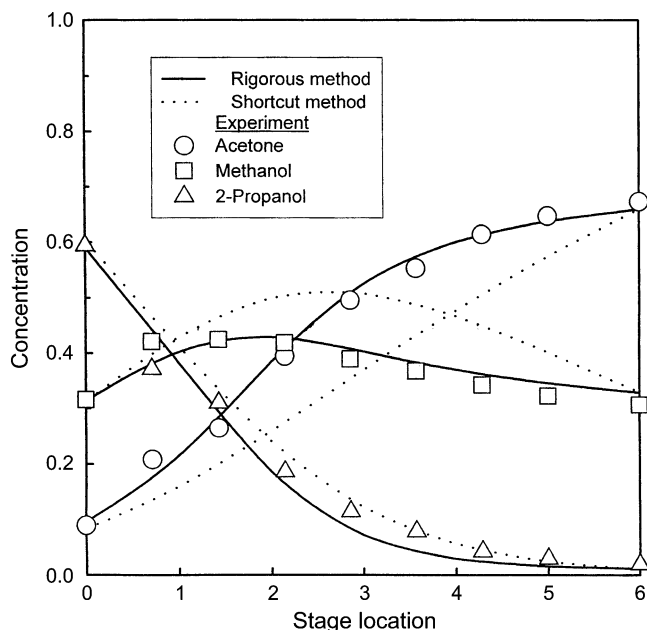


Fig. 6. Concentration profiles of the liquid phase at the end of distillation. Stage locations: 0 refers to the still, and 6, the distillate receiver.

In contrast with the shortcut method, the rigorous calculation method gives again an excellent estimation.

## CONCLUSIONS

Recently, we proposed a shortcut calculation method for batch distillation. This was to separate an ideal or nearly ideal multicomponent mixture by using a batch column equipped with a distillate receiver which operates under total reflux condition. Since the shortcut method was based on the assumptions of constant relative volatilities, constant molar overflows and negligible vapor and liquid holdups, it is not suitable for simulating a batch column of a nonideal multicomponent mixture. In this study, a rigorous calculation method was developed assuming negligible heat loss from the column wall. The behavior of the column was analyzed by solving a set of mass and energy balance equations.

To start the simulation, values of some parameters such as the number of equilibrium stages in the column, amount of the initial batch charge, initial feed compositions and the heating rate to the still have to be provided. The model first estimates the highest composition of the lightest component in the distillate receiver theoretically attainable under given operating conditions. In consideration of this maximum composition, desired distillate purity is specified as an input. Then the proposed model estimates the volume of the receiver, the variation of compositions in the receiver and the still, composition profiles of the column, and the distillation time required to complete the separation for the two cases of the receiver initially empty and initially filled-up with the original batch charge. The validity of the proposed method was demonstrated by comparing the simulation results with the experimental data, taking the separation of a nonideal acetone-methanol-2-propanol mixture. Judging from the fact that the agreement between the simulation and the experiment is excellent, it can be concluded that the calculation method

proposed in this study is sound and useful for separating a nonideal multicomponent mixture using a batch column with a distillate receiver.

### ACKNOWLEDGMENT

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### NOMENCLATURE

$D$	: volume of distillate receiver [mol]
$D_R$	: volume of distillate receiver during Rayleigh distillation [mol]
$d_i$	: amount of component $i$ in distillate receiver during Rayleigh distillation [mol]
$d_i^0$	: initial amount of component $i$ in distillate receiver [mol]
$G_j$	: volume of the liquid holdup in plate $j$ [ $\text{m}^3$ ]
$H_j$	: enthalpy of the vapor stream leaving plate $j$ [J/mol]
$H_w$	: enthalpy of the vapor stream leaving still [J/mol]
$h_D$	: enthalpy of the liquid in distillate receiver [J/mol]
$h_j$	: enthalpy of the liquid stream leaving plate $j$ [J/mol]
$i$	: component
$K_{ji}$	: equilibrium ratio of component $i$ in plate $j$
$L_D$	: flow rate of liquid stream leaving distillate receiver [mol/s]
$L_j$	: flow rate of liquid stream leaving plate $j$ [mol/s]
$N$	: number of equilibrium stages in the column
$n$	: number of components in feed mixture
$Q_C$	: rate of heat transfer from condenser [J/s]
$Q_R$	: rate of heat transfer to still [J/s]
$t$	: time [s]
$U_j$	: liquid holdup in plate $j$ [mol]
$V_j$	: flow rate of vapor stream from plate $j$ [mol/s]
$V_w$	: vapor boilup rate from still [mol/s]
$W$	: amount of liquid remaining in still [mol]
$W_R$	: amount of liquid remaining in still during Rayleigh distillation [mol]
$W^0$	: amount of the original batch charge [mol]
$w_i$	: amount of component $i$ in still during Rayleigh distillation [mol]
$w_i^0$	: initial amount of component $i$ charged in still [mol]
$x_{D1}^F$	: specified product purity of the lightest component in the distillate receiver
$x_{D1}^{\max}$	: maximum concentration of the lightest component in the distillate receiver
$x_{Di}$	: mole fraction of component $i$ in the distillate receiver
$x_{ji}$	: mole fraction of component $i$ in liquid stream leaving stage $j$

$x_{wi}$	: mole fraction of component $i$ in still
$x_{wi}^0$	: initial concentration of component $i$ in the batch charge
$y_{ji}$	: mole fraction of component $i$ in vapor stream leaving stage $j$
$y_{wi}$	: mole fraction of component $i$ in vapor stream leaving still

### Greek Letters

$\rho_i$	: density of component $i$ [ $\text{kg}/\text{m}^3$ ]
$\Delta t$	: time increment with a default value of 0.01 [min]

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