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# Limitations and Dynamics Imposed on Multicomponent Batch Distillation by Tray Hydraulics

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Batch distillation models incorporating a complete set of hydraulic relations, energy balances, and tray efficiencies and simpler models based upon constant molar holdup and zero holdup (the Rayleigh equation) were used to obtain distillation curves and optimal reflux policies. The approach to hydraulic limitations was monitored in the complete hydraulic model. The results indicate that during a multicomponent batch distillation the combination of the changes in the liquid composition and liquid flow rates causes the molar holdup on the trays to vary with time and, consequently, the characteristic time constant of the trays changes. The predicted composition profiles for descriptions which incorporate tray hydraulics differ significantly from those obtained from simpler models. Optimal reflux policies generated from the complete model which includes tray hydraulics differ significantly from those obtained from simple models and may lead to lower product yield. The optimum reflux ratios obtained from the model which includes tray hydraulics and tray efficiencies may be greater than the reflux ratios obtained from a model without tray hydraulics but which includes tray efficiencies by as much as a factor of 1.3. Tray hydraulics must be included in the process model to obtain a reliable and effective optimum operating strategy for batch distillation.

## Introduction

Batch distillation is an important unit operation in the chemical, pharmaceutical, and allied industries. It is typically employed in multiproduct manufacturing facilities, in low-volume services where round-the-clock manning (for continuous distillation) is uneconomical, where multiple product cuts are required from a single feed, or where batch integrity must be maintained. Batch distillation may become an important separation method in the recovery and reprocessing of waste solvent mixtures as environmental regulations become more strict and disposal costs become more expensive.

Simple models have traditionally been used to describe batch distillation. Analogs of the Rayleigh equation for batch distillation with rectification, which incorporate stagewise composition analysis but ignore holdup and energy effects, have proven to be immensely popular and are still in use today for shortcut design procedures and for the computation of optimal reflux policies. Later, Huckaba and Danley [1960] developed an improved model based upon a stagewise set of differential mass and energy balances with a constant molar holdup. The computational requirements imposed by the solution of optimal reflux problems have led to models being developed for computational speed, such as the shortcut model developed by Diwekar [1996] which combines the Fenske and Underwood equations and the Gilliland correlation with differential mass balances for the overall inventory and species in the reboiler to obtain a reduced-order model. Although physically detailed models incorporating a reduced set or a complete set of tray hydraulic and pressure drop relations have been applied to continuous distillation problems to study transient phenomena [Gallun, 1979; Gani et al., 1986], few have been applied to batch distillation and none to optimal reflux problems [Tomazi, 1993].

Tray hydraulics has an important role in continuous distillation. A column must be designed to ensure that the desired separation is feasible with the tray layout [Kister, 1992] and estimates of the pressure drop and tray efficiency may be refined by incorporating hydraulic relations for holdup, entrainment, and degree of mixing. Tray hydraulics can have a greater influence upon batch distillation. Tray hydraulics determine the quantity of material residing upon the trays in addition to the feasibility, pressure drop, and tray efficiency issues. Stewart et al. [1973] found that the fraction of liquid charge held up within the column could have a beneficial, negligible, or detrimental effect on the separation depending upon the reflux ratio. As a result, the dynamics of a batch distillation may be significantly influenced by tray hydraulics.

The problem of batch distillation is further complicated by the inherent dynamic nature of the operation. The hydraulic relations are functions of composition (which change with time) and the flow of liquid and vapor through the column (which may change with time). The flow of vapor may decrease significantly, unless a reboiler policy is implemented to compensate for the changes in bubble point temperature and the heat of vaporization. The liquid flow (and therefore holdup) may change because the vapor flow changes or because the reflux ratio changes with time (which is the case for time-optimal and constant overhead composition policies). The purpose of this paper is to address these hydraulic effects and apply them to an optimal reflux problem.

## Models for Batch Distillation

The following assumptions and approximations were used in the complete model of batch distillation. The vapor holdup on each stage is taken to be very small relative to the liquid holdup. The reflux returning to the column is at its bubble point. Heat enters or leaves the column only through the reboiler and condenser. The liquid on the trays is perfectly mixed. Stages are

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numbered from the top of the column, beginning with the condenser as stage number 1 and ending with the reboiler. For example, a column with ten stages has eight trays, a condenser, and a reboiler.

A complete set of material, energy, thermodynamic, pressure drop, hydraulic, entrainment, and tray efficiency equations makes up the most rigorous model of batch distillation. For each stage in the distillation of a mixture containing  $c$  components, there is one overall material balance,  $c - 1$  species balances, one energy balance,  $c - 1$  VLE relationships,  $c - 1$  independent tray efficiency relationships, one set of pressure drop correlations (including pressure drop through dry trays, the weir equation, and downcomer backup), and one liquid entrainment equation.

The Murphree efficiency of each tray is computed at each step of the integration (when used). In addition, each tray is checked to see if any hydraulic limitation (entrainment flooding, weeping, or downcomer flooding) is exceeded at each step of the integration. During the startup phase, a similar procedure is followed. However, weeping is not checked in order to avoid false errors caused by zero vapor velocity at the start of the integration.

### Process Model Development

The following balances, which include the material, energy, efficiency, equilibrium, and hydraulic relations, are used to describe a batch distillation operation:

**1. Material Balances.** The overall mole balance on tray  $j$  is represented by the following equation:

$$\frac{dN_j}{dt} = V_{j+1} - V_j + L_{j-1} - L_j + E_{j+1} - E_j \quad (1)$$

For  $c$  species in a mixture, only  $c - 1$  component balances may be used because only  $c - 1$  independent mole fractions can exist. The component balance of species  $i$  on tray  $j$  is represented by the following equation:

$$\frac{dx_{ij}}{dt} = \frac{1}{N_j} [V_{j+1}(y_{ij+1} - x_{ij}) - V_j(y_{ij} - x_{ij}) + L_{j-1}(x_{i,j-1} - x_{ij}) + E_{j+1}(x_{i,j+1} - x_{ij})] \quad (2)$$

**2. Energy Balances.** The energy balance on stage  $j$  is represented by the following equation. The addition or removal (or loss) of energy from the stage is represented by the term  $q$ . For adiabatic columns,  $q = 0$  except for the reboiler and condenser stages:

$$\frac{dh_j}{dt} = \frac{1}{N_j} [V_{j+1}(H_{j+1} - h_j) - V_j(H_j - h_j) + L_{j-1}(h_{j-1} - h_j) + E_{j+1}(h_{j+1} - h_j) + q] \quad (3)$$

The molar enthalpy of the liquid must satisfy the following relation:

$$0 = -h_j + h_{\text{mix}} + \sum_{i=1}^c x_{ij} h_{ij} \quad (4)$$

**3. Vapor-Liquid Equilibria.** The equilibrium vapor composition is obtained from the following expression:

$$y_{ij}^* = \gamma_{ij} K_{ij} x_{ij} \quad (5)$$

The distribution coefficient,  $K$ , and the liquid phase activity coefficient,  $\gamma$ , are dependent upon the species in the mixture. For the hydrocarbon system presented in this work, the activity coefficient is unity.

If the liquid and vapor on the trays is not at equilibrium, then the actual vapor composition ( $y$ ) is not identical to the equilibrium vapor composition ( $y^*$ ). The deviation from equilibrium may be represented by using the stage efficiency. The Murphree efficiency [Murphree, 1925] is defined as the amount of purification achieved on a given stage:

$$E_{ij}^M = \frac{y_{ij} - y_{ij+1}}{y_{ij}^* - y_{ij+1}} \quad (6)$$

The Murphree temperature convention requires the stage temperature to be identical to the bubble point temperature of the liquid in equilibrium with its vapor:

$$0 = \sum_{i=1}^c \gamma_{ij} K_{ij} x_{ij} - 1 \quad (7)$$

Vaporization efficiency is defined by modifying the vapor-liquid equilibrium (VLE) relation:

$$0 = \sum_{i=1}^c E_{ij}^V \gamma_{ij} K_{ij} x_{ij} - 1 \quad (8)$$

The Murphree and vaporization efficiencies may be determined by the procedures recommended by the AIChE [1958]. The required multicomponent diffusion coefficients may be determined by the method recommended by Graham et al. [1973]. The AIChE correlations for sieve tray efficiency are applicable to the froth regime. Correlations for efficiency in the spray regime may be found elsewhere [cf. Zuiderweg, 1982].

**4. Accumulator Level Control.** The liquid density relation is approximated by the following expression:

$$\frac{1}{\rho_j^L} = \sum_{i=1}^c \frac{x_{ij} V_i}{M w_i} \quad (9)$$

In general, the volume occupied by a liquid mixture is equal to the sum of the products of the mole fraction of each component and its partial molar volume. The partial molar volume of each component in an ideal solution, however, is equal to the pure-component volume. This ideal solution approximation of the relationship between the density and composition of a mixture makes it possible to derive an expression for a constant accumulator volume in terms of the molar flows and the liquid composition.

$$\frac{d(\text{volume})}{dt} = \frac{d(N_1/\rho_M^L)}{dt} = 0 \quad (10)$$

$$0 = N_1 \frac{d}{dt} \left( \frac{1}{\rho_M^L} \right) + \frac{1}{\rho_M^L} \frac{dN_1}{dt} \quad (11)$$

$$0 = \frac{1}{\rho_M^L} N_1 \sum_{i=1}^{c-1} x_{i1}^L v_i + V_2 - L_1 - D \quad (12)$$

For a constant reflux ratio, the final result is expressed by the following equation:

$$0 = -L_1 + \left( V_2 + \frac{1}{\rho_M} N_1 \sum_{i=1}^{c-1} X_{i1} V_i \right) \left( \frac{1}{1 + D/L} \right) \quad (13)$$

**5. Pressure Drop.** The pressure drop across both bubble cap and sieve trays may be obtained from the following equation [Smith, 1963]:

$$0 = -P_{j+1} + P_j + h_{t,j} \rho_j^L \quad (14)$$

In the above expression,  $h_{t,j}$  is the sum of the dry tray pressure drop corrected for liquid entrainment,  $h_d$ , and the pressure drop of the vapor rising through the liquid,  $h_{aL}$ , on the tray.

$$h_t = h_d + h_{aL} \quad (15)$$

**a. Bubble Cap Trays.** For bubble cap trays, the dry tray pressure drop may be obtained from the following expression [Bolles, 1956]:

$$h_d = \left( 273.4 K_c \frac{\rho^V}{\rho_L} \left( \frac{Q_{\text{vapor}}}{A_{\text{riser}}} \right)^2 + h_{s0} \right) \left( 1 + 15 E \sqrt{\frac{\rho^V}{\rho_L}} \right) \quad (16)$$

The term  $h_{s0}$  is a function of the bubble cap slot geometry. For rectangular slots, it may be obtained from the following equation, presented by Bolles [Smith, 1963]:

$$h_{s0} = 7.59 \left( \frac{\rho^V}{\rho^L - \rho^V} \right)^{1/3} \left( \frac{Q h_{sh}}{A_s} \right)^{2/3} \quad (17)$$

Expressions for  $h_{s0}$  for various slot geometries are presented in Smith [1963]. Since the bubble caps (and slots) are not flush with the bottom of the tray deck, the pressure drop through the aerated liquid is less than the depth of liquid on the trays. If the caps contain slots, the pressure drop through the liquid is caused by the height of liquid between the top of the slots and the top of the weir (the "static seal") added to the average liquid height over the weir. The pressure drop through the aerated liquid may be obtained from the following equation [Smith, 1963]:

$$h_{aL} = \beta (h_{ss} + h_{ow} + \Delta/2) \quad (18)$$

In the above equation, the term  $\Delta$  represents the difference in clear liquid height between the inlet and outlet sides of a tray. A reasonable curve fit for the aeration factor,  $\beta$  [Bennett et al., 1983], is given by the following expression:

$$\beta = 0.58 + 0.42 \exp(-1.62 u_A \sqrt{\rho^V}) \quad (19)$$

The flow of liquid and the height of the liquid crest over the outlet weir on bubble cap trays are determined by a modification to the Francis weir formula corrected for segmental downcomers [Bolles, 1946].

$$h_{ow} = 664.8 F_w (Q/L_w)^{2/3} \quad (20)$$

It is implied in this equation that the rate of flow over the weir is a function of the height of clear liquid on the bubble cap tray. The correction to the height of liquid flowing over a weir into a segmental downcomer,

$F_w$ , is obtained from the following equation [Bolles, 1946]:

$$\frac{Q}{L_w^{2.5}} = 0.597 \left[ \frac{\sqrt{1 - R_w^2/F_w^3} - \sqrt{1 - R_w^2}}{R_w F_w} \right]^{3/2} \quad (21)$$

This correction is typically needed for towers with diameters less than 1 m.

The hydraulic gradient across a bubble cap tray,  $\Delta$  [Bolles, 1956], may be obtained from the relation:

$$\sqrt{\Delta'} \left( \Delta' \left( \frac{3N_r}{2} - 1.4 \right) + 3N_r [h_L + h_{sc}(R_{rc} - 1)] \right) = 23.97 \times 10^6 \frac{N_r^{3/2} Q}{C_d I_c} \quad (22)$$

A fit for the coefficient,  $C_d$ , is given by the following expression:

$$C_d = 14.76 \left( \frac{2Q}{D_t + L_w} \right)^{0.53213} \quad (23)$$

The true hydraulic gradient,  $\Delta$ , may be obtained from an empirical correction to the uncorrected hydraulic gradient,  $\Delta'$ , obtained from eq 22. For small towers (about 1 m in diameter) under typical operating conditions, this correction may be conservatively estimated to be unity without creating an error greater than about 10%.

The volume of liquid residing on a tray is the product of the active (bubbling) portion of a tray and the average clear liquid height, added to the liquid volume in the downcomer, and reduced by the volume occupied by the bubble caps. The bubble cap volume is approximately equal to the product of the cap circumference and the cap height. (If the bubble caps are mounted flush with the bottom of the tray deck, the bubble cap volume is exactly equal to the product of the cap circumference and the cap height.)

**b. Sieve Trays.** For sieve trays, the method proposed by Bennett et al. [1983] may be used to determine the liquid holdup, pressure drop, and flow of liquid over the weir. The pressure drop is composed of two components, the pressure drop through the dry trays and the pressure drop through the liquid:

$$h_t = h_h + h_L \quad (24)$$

The pressure drop through the dry trays is given by the orifice equation [Smith, 1963]:

$$h_h = 50.86 \frac{\rho^V}{\rho^L} \left( \frac{u_h}{C_v} \right)^2 \left( 1 + 15 E \sqrt{\frac{\rho^V}{\rho^L}} \right) \quad (25)$$

The discharge coefficient,  $C_v$ , is obtained from a curve fit of the correlation proposed by Leibson et al. [1957].

The rate of liquid flow over the weir of sieve trays is proportional to the froth height over the weir rather than the clear liquid height [Bennett et al., 1983]:

$$h_L = \phi_e \left[ h_w + C \left( \frac{Q}{L_w \phi_e} \right)^{2/3} \right] \quad (26)$$

$$= \phi_e h_{Fe}$$

The froth density and weir constant are obtained from the following expressions [Bennett et al., 1983]:

$$\phi_e = \exp \left[ -12.55 \left( u_A \left( \frac{\rho^V}{\rho^L - \rho^V} \right)^{1/2} \right)^{0.91} \right] \quad (27)$$

$$C = 501 + 439 \exp[-0.1378 h_w]$$

Bennett also includes an additional term for trays with exceptionally small perforations or for liquids with exceptionally high surface tensions.

The pressure drop through the reflux condenser was given by the correlations proposed by Lord et al. [1970a,b]. Here, the condenser vapor pressure drop (in mmHg) is obtained from the following expression:

$$\Delta P = 58.282(m_v^2/\rho^V) \quad (28)$$

**6. Downcomer Backup.** Liquid height in the downcomers may be obtained from the following expression [Smith, 1963]:

$$0 = -h_{dc_j} + h_{t_{j-1}} + h_w + h_{ow_j} + \Delta_j + h_{da_j} \quad (29)$$

The head loss for liquid flowing under the downflow apron is obtained from the following equation [Smith, 1963]:

$$h_{da} = 165.4(Q/A_{da})^2 \quad (30)$$

The hydraulic gradient is determined by eq 22 for bubble cap trays. For sieve trays, the hydraulic gradient is typically very small (about 1 mm) [Zuiderweg, 1982] and has therefore been excluded from the simulation.

The set of equations to be solved contains both ordinary differential equations and algebraic equations. The differential equations are the overall material balances (1) for each tray, the  $c - 1$  species balances (2) for each tray, and the enthalpy balances (3) for each tray. Algebraic equations for each tray are developed for the molar energy determination (4), the VLE relationships either by eqs 5–7 or by eq 8, the pressure drop (14), the weir formula (20) or (26), and the downcomer liquid level equation (29). For stage  $j$  (numbered top to bottom) of a column, the full set of equations, which have been renumbered for clarity, becomes

$$\frac{dN_j}{dt} = V_{j+1} - V_j + L_{j-1} - L_j + E_{j+1} - E_j \quad (31)$$

$$\frac{dx_{ij}}{dt} = \frac{1}{N_j} [V_{j+1}(y_{i,j+1} - x_{ij}) - V_j(y_{ij} - x_{ij}) + L_{j-1}(x_{i,j-1} - x_{ij}) + E_{j+1}(x_{i,j+1} - x_{ij})] \quad (32)$$

$$\frac{dh_j}{dt} = \frac{1}{N_j} [V_{j+1}(H_{j+1} - h_j) - V_j(H_j - h_j) + L_{j-1}(h_{j-1} - h_j) + E_{j+1}(h_{j+1} - h_j) \pm q] \quad (33)$$

The correlation for the liquid entrainment rate,  $E$ , was obtained from Fair and Matthews [1958] for bubble cap trays or from Kister and Haas [1988] for sieve trays.

$$0 = -h_j + h_{mix} + \sum_{i=1}^c x_{ij} h_{ij} \quad (34)$$

$$0 = \sum_{i=1}^c \gamma_{ij} K_{ij} x_{ij} - 1 \quad (35)$$

$$0 = -P_{j+1} + P_j + h_{ij} \rho_j^L \quad (36)$$

The following expression is used for the liquid flow from sieve trays [Bennett et al., 1983]:

$$0 = -L_j + \rho_M^L L_w \phi_e \left[ \frac{h_L}{\phi_e C} - \frac{h_w}{C} \right]^{1.5} \quad (37)$$

The following expression is used for the liquid flow from the bubble cap trays [Bolles, 1946]:

$$0 = -L_j + \rho_M^L L_w \left( \frac{h_{ow}}{664.8 F_w} \right)^{3/2} \quad (38)$$

The following expression is used for the height of liquid in the downcomers:

$$0 = -h_{dc_j} + h_{t_{j-1}} + h_w + h_{ow_j} + \Delta_j + h_{da_j} \quad (39)$$

### Comparison with Less Rigorous Models

An example distillation problem of a ternary system with relative volatilities of 4/2/1 and a reflux ratio ( $L/D$ ) of 4.0 is presented which has been simulated by four methods to illustrate the significance of tray hydraulics. Details of tray layouts and vapor–liquid equilibrium are presented in the appendix. The examples presented here are based upon columns with seven stages (five trays). Responses (Figure 1) from the complete model with hydraulics, energy balances, and equilibrium stages are compared to results from the Rayleigh analog (no holdup on the trays) with a reboiler energy balance, the constant molar holdup and overflow model with a reboiler energy balance, and the TUTSIM model with a simple energy balance and a reduced set of hydraulic equations (the tray holdup was estimated from eq 20 with a weir factor of unity). The model with the complete set of hydraulic relations does not achieve the same degree of distillate purity predicted by the simpler models and is initially more sluggish than the constant holdup and Rayleigh models but later begins to respond faster than the constant holdup model due to the decreasing inventory on the trays. The most sluggish response was predicted by the model with the reduced set of hydraulic equations, which is due in part because it overestimates the tray inventory by failing to account for frothy liquid on the trays. However, the integration algorithm used in TUTSIM is based upon fixed step size Euler's method. Although in practice the step size was reduced until successive trials yielded essentially identical distillation curves, the numerical method cannot be ruled out as a contributing cause of the results.

### Response of a Column to Step Changes in Reflux Ratio

An optimum reflux policy may require multiple reflux ratios during a single batch separation. Therefore, the response of a distillation column to a step change in reflux ratio is presented in Figures 2–5. When the reflux ratio changes, the change in liquid flow causes corresponding changes in the liquid holdup on the trays

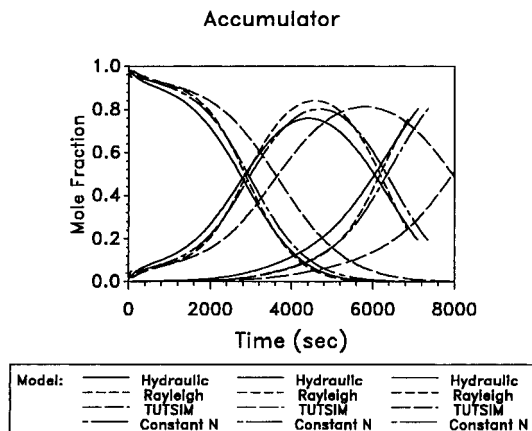


Figure 1. Composition profiles for various models, accumulator stage.

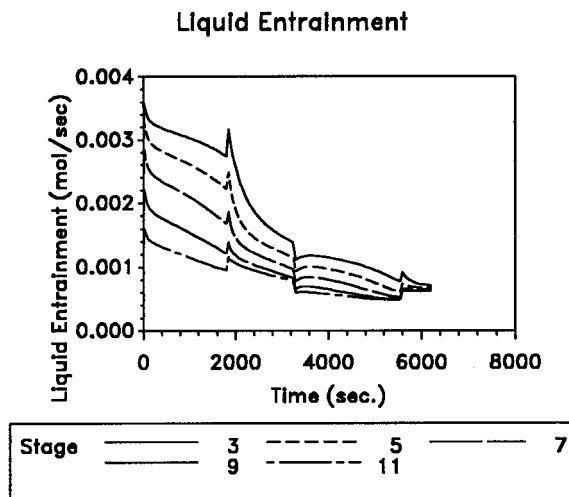


Figure 4. Liquid entrainment, step changes in reflux ratio.

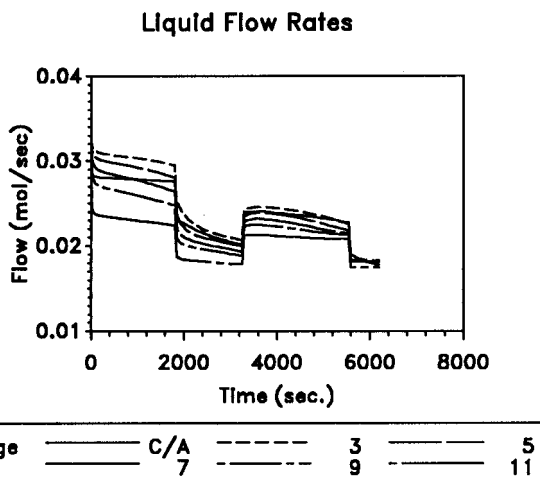


Figure 2. Liquid flow rates, step changes in reflux ratio.

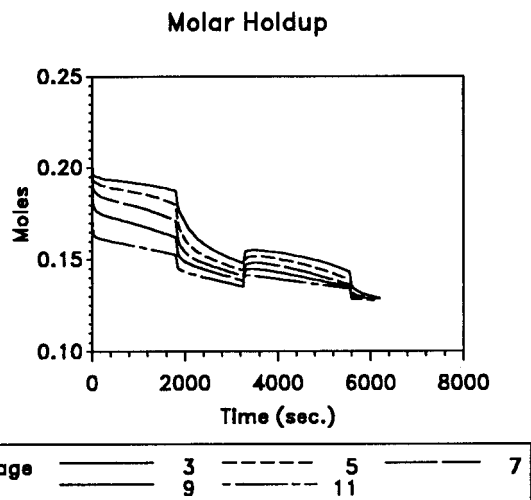


Figure 3. Molar holdup, step changes in reflux ratio.

and in the entrainment rate. Low liquid flows cause high entrainment rates and conversely. The characteristic time constant on the trays,  $N/V$ , changes as well, although the vapor flow may also change (but by a smaller amount). The responses of changes in the reflux ratio ( $r = L/D$ ) from 6.22 to 2.62 to 6.22 and back to 2.62 are shown for the molar liquid flow in Figure 2, the molar holdup in Figure 3, and the liquid entrainment in Figure 4. The changes in reflux ratios occur at approximately 1800, 3200, and 5400 s and correspond to an optimal reflux policy for a ternary system of ali-

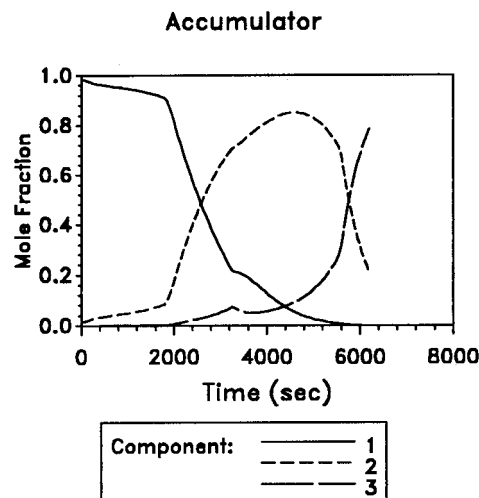


Figure 5. Composition, step changes in reflux ratio.

phatic hydrocarbons, as will be demonstrated below. The change in  $N/V$  is about -5% each time the reflux ratio is reduced. Lower reflux ratios tend to produce higher rates of entrainment, which reduces the overall ability of the column to make the separation. Neither the effect of the change in the entrainment rate nor the change in tray inventory is accounted for in the simpler models. These results are important in the development of optimal reflux strategies. Methods which overestimate the purity of the distillate may cause a designer to select a reflux ratio which is unrealistically low, and a model which fails to account for the true dynamics of the distillation column may lead to switching between product and slop cuts at the wrong times, leading to diminished profitability of the separation or even the inability of one or more product cuts to achieve the required purity specification. In addition, the designer must be aware that hydraulic limitations impose limits upon the operation of a column. A column may either approach or move away from a hydraulic limitation during a batch distillation because the boilup rate may change during the batch. In fact, the hydraulic limits may themselves change, since these limits are in part functions of the physical properties of the liquid and vapor.

### Optimal Operating Strategies

The difference in performance as indicated by simple distillation models and a model which incorporates tray

**Table 1. Aliphatic Hydrocarbons: Best Reflux Policies<sup>a</sup>**

| trays <sup>b</sup> | reboiler <sup>c</sup> | reflux policy <sup>d</sup> | product cut | slop cut <sup>e</sup> | X <sub>2</sub> switch | C <sub>f</sub> (% yield/h) | yield (%) | time <sup>f</sup> (s) | startup time <sup>g</sup> (s) |
|--------------------|-----------------------|----------------------------|-------------|-----------------------|-----------------------|----------------------------|-----------|-----------------------|-------------------------------|
| E, N               | V                     | r                          | 3.233       | SAPC                  | 0.6335                | 67.115                     | 76.96     | 4128                  | 0                             |
| E, N               | V                     | r                          | 3.611       | SAPC                  | 0.5867                | 49.764                     | 82.14     | 4442                  | 1500                          |
| E, N               | V                     | r                          | 4.115       | 0.833                 | 0.6499                | 71.854                     | 83.03     | 4160                  | 0                             |
| E, N               | V                     | r                          | 4.119       | 0.852                 | 0.6370                | 52.888                     | 83.20     | 4163                  | 1500                          |
| R, N               | V                     | r                          | 4.76        | 1.92                  | 0.6960                | 56.270                     | 71.94     | 4603                  | 0                             |
| E, H               | Q                     | r                          | 3.968       | SAPC                  | 0.5982                | 51.942                     | 80.07     | 5550                  | 0                             |
| R, H               | Q                     | r                          | 4.852       | SAPC                  | 0.6620                | 35.954                     | 65.89     | 6597                  | 0                             |
| E, H               | Q                     | r                          | 4.76        | 1.09                  | 0.6489                | 55.076                     | 82.41     | 5387                  | 0                             |
| R, H               | Q                     | r                          | 6.219       | 2.617                 | 0.7058                | 37.937                     | 65.18     | 6185                  | 0                             |

<sup>a</sup> Equivalent relative volatilities 4:2:1. Feed composition (mole fraction) 0.33, 0.33, 0.34. Sieve tray column. Product specifications: cut no. 1,  $x_1 \geq 0.95$ ; cut no. 2,  $x_2 \geq 0.80$ ; bottoms,  $x_3 \geq 0.95$ . <sup>b</sup> Trays are equilibrium (E) or "real" ( $E^M \neq 100\%$ ) (R) stages. Trays include hydraulics (H) or exclude hydraulics (N). <sup>c</sup> Reboiler policy is constant molar boilup (V) or constant duty (Q). <sup>d</sup> Reflux policy: reflux ratio ( $r = L/D$ ). <sup>e</sup> SAPC denotes the value of the reflux variable for the slop cut is identical to the value for the reflux variable chosen for the product cut. <sup>f</sup> "Time" refers to the amount of time elapsed during the product phase of the separation. <sup>g</sup> "Startup Time" refers to the amount of time required to achieve a close approximation to steady state during the startup phase.

hydraulics suggests that the optimal reflux policies obtained by these models may likewise differ. An optimal reflux strategy obtained from the model with the complete set of hydraulic and tray efficiency correlations, obtained by nonlinear programming, is presented in this section.

The measure used for the performance of a distillation column, that is, the objective function, is the capacity factor ( $C_f$ ). Luyben's [1971, 1988] capacity factor problem, the maximum number of moles of product from all distillate and bottoms cuts per time required for the distillation, can be written in the following form:

$$\text{Maximize } C_f = \frac{B + \sum_{i=1}^{c-1} \int_{t_{si}}^{t_{fi}} D(t) dt}{t_{su} + t_{th}} \quad (40)$$

subject to

$$\frac{\int_{t_{si}}^{t_{fi}} x_{D,i}(t) D(t) dt}{\int_{t_{si}}^{t_{fi}} D(t) dt} = x_{M,i} \geq x_{M,i}^* \text{ and } x_{M,B} \geq x_{M,B}^* \quad (41)$$

The "startup" time in this problem is defined as the time required to reach a reasonable approximation to steady state (no process variable changed by more than 2% between successive 100-s intervals) which may be acceptable tolerance in an industrial environment. Integration continued, however, until no process variable changed by more than  $1.0 \times 10^{-8}$ . To illustrate the effect of startup time upon the optimal reflux policy, the problem has been worked with and without the startup time included in the capacity factor.

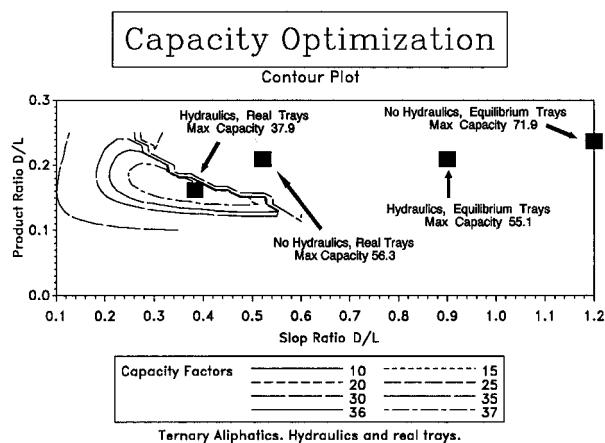
To better illustrate the sensitivity of the optimal policy to the process model and to minimize computational effort, a set of constant values was sought to optimize the objective function. The best set of reflux ratios (up to one for each product cut and a separate one for the slop cuts) and the composition of the distillate at which the intermediate product cut should begin is found by using various distillation models to illustrate the significance of tray hydraulics (and tray efficiency). The first model is based upon the commonly used approximations of constant molar holdup and overflow, constant molar boilup, and equilibrium trays. The second is similar to the first but includes nonequilibrium trays (based upon a 70% Murphree efficiency) and is presented to illustrate that nonequilibrium trays do not account for all of the differences between the optimal reflux policies obtained by simpler and by more

complete models. The third description is based upon a complete set of hydraulic balances and stage-to-stage energy balances but also includes the approximation of equilibrium stages. The fourth description is based upon a complete set of hydraulic balances and stage-to-stage energy balances and also includes tray efficiencies (computed at each step from the AIChE correlations) in order to represent a very close approximation to the performance of a real distillation column. Product specifications are (1) distillate cut no. 1 contains 0.95 mole fraction of the most volatile component, (2) distillate cut no. 2 contains 0.8 mole fraction of the intermediate component, and (3) the bottoms fraction contains 0.95 mole fraction of the least volatile component.

The generic operating policy is to withdraw the first product cut until the distillate product reaches its purity constraint, withdraw a slop cut until the distillate composition target is achieved, withdraw the second cut until its purity specification is met (or it is clear that it will never pass its purity specification), and then withdraw a second slop cut until the column inventory passes the bottoms cut specification. If the intermediate cut fails to achieve its purity specification, it becomes a slop cut. The danger of failing to obtain an intermediate product cut is very real. The concentration of the intermediate component in the distillate passes through a maximum. As a result, an operator may begin collecting the second product cut before the distillate composition achieves its purity specification. As the second cut is collected, the increasing distillate purity may eventually cause the aggregate purity of the second product cut to pass. However, if the second product cut commences at insufficient purity, the aggregate concentration may never achieve its purity specification.

Results for one separation are presented in Table 1. Other cases are presented in [Tomazi, 1993]. The constant holdup model with equilibrium stages and no hydraulics was used in order to compare the best reflux policy found by using a rigorous distillation model with the policy obtained by using the simple constant holdup and overflow model used by other authors.

The results presented above suggest that the optimal operating strategies obtained by simpler models tend to favor higher distillate rates than those obtained by the model incorporating the full set of hydraulic and efficiency equations. If the optimum reflux ratio and switching composition obtained from the model with constant holdup and boilup (see entry no. 1 in Table 1) are applied to the model with hydraulics and equilibrium trays and the constant reboiler duty policy is used, the capacity factor of the column is reduced to 34.36 from 67.115 and the yield is reduced to 46.68% from



**Figure 6.** Comparing the location of the optimum reciprocal reflux ratios (1 product, 1 slop) for three models.

76.96%. One reason for the reduction in the capacity factor is due to the failure of the intermediate distillate cut to achieve its purity specification. Since the hydraulic model was described by equilibrium trays in this instance, the failure to achieve an intermediate product that meets its purity specification is due to hydraulic effects, or possibly hydraulic and energy effects, rather than by tray efficiencies. If the reflux ratios are identical for both product cuts and are identical for both slop cuts, the best reflux ratios for the model that includes hydraulics and equilibrium trays used during the slop cuts are 1.3 times greater, while the best product cut reflux ratio is 1.15 times greater, than those found for the model without hydraulics. See entries 3 and 8 in Table 1. The maximum capacity factor when a column is described with hydraulics and "real" trays (Murphree efficiency included) is only 53% of the capacity factor obtained from a model which ignores hydraulics and real trays. Figure 6 shows the location of the best product cut and slop cut reflux ratios for the four models: hydraulics with real trays, hydraulics with equilibrium trays, no hydraulics with equilibrium trays, and no hydraulics but with a tray efficiency of 70%. The maximum capacity for the no hydraulic, real trays model is 56.27 and may be obtained with reflux ratios of 4.76 for the product cuts and 1.92 for the slop cuts, a switching composition of 0.696 mole fraction. The best set of reflux ratios is very different for the three models. The location of the reciprocal reflux ratios ( $r^{-1} = D/L$ ) for the two simpler models, including the model without hydraulics but with real trays, lies to the right of a cliff in the capacity factor for the model with hydraulics and real trays. The very steep drop in the profitability of the separation is due to the inability of the second product cut to meet its purity specification. These results suggest that if the reflux ratios and switching composition obtained from either of the two simpler models are applied to the rigorous model (and presumably a real distillation column), the capacity factor obtained from the separation will be lower than the best possible capacity factor. Similar results were obtained from a ternary mixture consisting of higher relative volatilities. In this case, a constant reboiler duty policy was required in order to avoid excessive weeping before the separation was completed. In addition, optimal reflux policies were obtained both with and without the inclusion of equilibration time in the batch cycle. Optimal reflux policies which account for the equilibration time have higher reflux ratios, which results in a

higher yield of product, than policies which ignore the equilibration time [Tomazi, 1993].

The simple reboiler policies presented in this work include constant steam temperature and constant duty. Constant reboiler duty may be used to avoid encountering hydraulic limitations during a batch separation. Although Luyben suggested using a high reboiler duty for optimal capacity, the reboiler duty may be a decision variable because high rates of entrainment encountered close to the flood point may decrease the profitability of the separation [Tomazi, 1993].

## Conclusions

Tray hydraulics have a significant effect upon a batch separation. Entrainment and the change in the characteristic time constants upon the trays ( $N/V$ ) cause the distillate product to be less pure and the column to respond in a more sluggish manner than predicted by the Rayleigh model and the constant holdup model. As a result, the single optimum reflux ratio, and switching composition for an example case found by using the constant holdup model, when applied to the model including the complete hydraulic equations results in a decrease of capacity of 48% and a decrease in yield of 40%. The decreases in yield and capacity occur primarily because the intermediate product cut fails to meet its purity specification. During the design of a reboiler and tray layout, it would be wise to examine the time behavior of the separation in order to avoid violating tray hydraulic boundaries. Before applying an optimal reflux policy found by using simple models, it is necessary to be sure this policy is not effected by the dynamic effects of entrainment and molar tray holdup.

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## Notation

- $A_a$  = active (bubbling) area of tray,  $A_t - 2A_d$ ,  $m^2$
- $A_d$  = downcomer area,  $m^2$
- $A_{da}$  = area under downcomer apron,  $m^2$
- $A_n$  = net area of tray,  $A_t - A_d$ ,  $m^2$
- $A_{riser}$  = area of bubble cap risers,  $m^2$
- $A_s$  = slot area,  $m^2$
- $A_t$  = tower cross-sectional area,  $m^2$
- $B$  = heel, or bottoms cut, mol
- $C$  = sieve tray weir factor
- $c$  = number of components in the mixture
- $C_t$  = capacity factor, mol/time
- $C_d$  = hydraulic gradient coefficient
- $C_v$  = sieve tray orifice coefficient
- $D$  = distillate rate, mol/s
- $D_t$  = tower diameter, m
- $E$  = liquid entrainment, liquid mass per unit mass vapor
- $E_j$  = liquid entrainment from tray  $j$ , mol/s
- $E^M$  = Murphree tray efficiency
- $E^V$  = vaporization efficiency
- $F_w$  = weir constriction factor
- $g$  = gravitational acceleration constant,  $m/s^2$
- $h_j$  = liquid molar enthalpy, stage  $j$ , J/mol
- $H_j$  = vapor molar enthalpy, stage  $j$ , J/mol
- $h_{ij}$  = pure liquid component enthalpy, species  $i$  on stage  $j$ , J/mol
- $h_{mix}$  = enthalpy change of mixing, J/mol



$h_{aL}$  = pressure drop through aerated liquid, mm of liquid  
 $h_d$  = dry tray pressure drop, mm of liquid  
 $h_{da}$  = head loss of liquid flowing under downcomer, mm of liquid  
 $h_{dc}$  = liquid height in downcomer, mm  
 $h_{Fe}$  = froth height, mm  
 $h_h$  = pressure drop through sieve tray holes, mm of liquid  
 $h_L$  = clear liquid height on tray, mm  
 $h_{ow}$  = height of liquid crest over weir, mm  
 $h_{sc}$  = cap skirt clearance, mm  
 $h_{sh}$  = slot height, mm  
 $h_{so}$  = pressure drop through bubble cap slots, mm of liquid  
 $h_{ss}$  = bubble cap static seal, mm  
 $h_t$  = total pressure drop across tray, mm of liquid  
 $h_w$  = weir height, mm  
 $K_c$  = bubble cap coefficient, dimensionless  
 $K_{ij}$  = equilibrium coefficient, species  $i$  on stage  $j$   
 $l_c$  = total free width between caps normal to flow, mm  
 $L_j$  = liquid flow leaving stage  $j$ , mol/s  
 $L_w$  = weir length, m  
 $m_V$  = mass flow rate of vapor, kg/s  
 $Mw_i$  = molecular weight, species  $i$   
 $N_j$  = number of moles on stage  $j$   
 $n$  = number of trays in the column  
 $N_r$  = number of rows of caps normal to flow  
 $P_j$  = pressure on stage  $j$  in consistent units  
 $q$  = heat added to each stage, including reboiler and condenser duty, J/s  
 $Q$  = liquid flow, m<sup>3</sup>/s  
 $Q_{vapor}$  = vapor flow, m<sup>3</sup>/s  
 $R$  = distillate ratio,  $D/L$   
 $r$  = reflux ratio,  $L/D$   
 $R_{rc}$  = ratio of distance between risers to distance between caps  
 $R_w$  = ratio of weir length to tower diameter  
 $t$  = time, s  
 $t_{th}$  = time required for heel purification, s  
 $t_{ti}$  = time at the end of product cut  $i$ , s  
 $t_{si}$  = time at the start of product cut  $i$ , s  
 $t_{su}$  = startup time, s  
 $u_A$  = superficial vapor velocity based upon active area, m/s  
 $u_h$  = vapor velocity through holes, m/s  
 $V_j$  = vapor leaving stage  $j$ , mol/s  
 $v_i$  = specific volume of component  $i$ , m<sup>3</sup>/mol  
 $x_{ij}$  = liquid mole fraction, species  $i$  on stage  $j$   
 $x_{M,B}$  = heel composition  
 $x_{M,B}^*$  = heel specification  
 $x_{M,i}$  = average distillate composition, cut  $i$   
 $x_{M,i}^*$  = distillate specification, cut  $i$   
 $y_{ij}$  = actual vapor mole fraction, species  $i$  on stage  $j$   
 $y_{ij}^*$  = equilibrium vapor mole fraction, species  $i$  on stage  $j$

#### Greek Letters

$\beta$  = aeration factor of liquid  
 $\gamma_{ij}$  = liquid phase activity coefficient, species  $i$  on stage  $j$   
 $\Delta$  = liquid gradient across tray, mm  
 $\Delta'$  = liquid gradient across tray, uncorrected, mm  
 $\rho^L$  = liquid density, kg/m<sup>3</sup>  
 $\rho_M^L$  = liquid molar density, kg mol/m<sup>3</sup>  
 $\rho^V$  = vapor density, kg/m<sup>3</sup>  
 $\phi_e$  = relative froth density

### Appendix: Column Specifications

The specifications of the basic tower design are presented in Table 2. The reboiler energy balance is expressed in terms of the product of the overall heat-transfer coefficient, the reboiler area, and the temperature difference between the hot fluid and the contents of the reboiler. For the sake of simplicity, it was assumed that the product of the heat-transfer coefficient and reboiler heat exchange area was a constant. The

**Table 2. Tower Specifications**

|   |                              |
|---|------------------------------|
| tower diameter, $D_t$                                 | 0.9144 m                     |
| accumulator diameter                                  | 0.9144 m                     |
| accumulator level                                     | 250 mm                       |
| weir length, $L_w$                                    | 0.6350 m                     |
| tray spacing, $S$                                     | 457.2 mm                     |
| downcomer per tray                                    | 1                            |
| foam factor, $F_s$                                    | 1.0                          |
| hole factor, $F_h$                                    | 1.0                          |
| downcomer clearance                                   | 25.4 mm                      |
| weir height, $h_w$                                    | 38.1 mm                      |
| hole diameter, $h_{dia}$                              | 6.35 mm                      |
| ratio of hole to tower area, $A_h/A_t$                | 0.1                          |
| tray thickness  | 1.982 mm                     |
| reboiler heat transfer, UA                            | 4919 W/K                     |
| reboiler duty at total reflux                         | 620.2 kW                     |
| steam temperature at total reflux                     | 144.4 °C                     |
| stages (including reboiler and condenser/accumulator) | 12 (unless otherwise stated) |

**Table 3. Data for Constant Holdup Model**

|                     |             |
|---------------------|-------------|
| relative volatility | 4/2/1       |
| vapor rate          | 0.034 mol/s |
| Murphree efficiency | 0.7         |
| tray holdup         | 0.227 mol   |
| accumulator holdup  | 2.20 mol    |

**Table 4. Antoine Coefficients Used in the Complete Model**

| component | A       | B       | C      |
|-----------|---------|---------|--------|
| 1         | 7.35156 | 1048.58 | 232.04 |
| 2         | 7.05053 | 1048.58 | 232.04 |
| 3         | 6.74950 | 1048.58 | 232.04 |

constant reboiler duty was taken to be the heat duty at total reflux and steady state using the steam temperature used in the constant steam temperature policies. Thus, at steady state, the reboiler duty and steam temperature are the same for both policies. The Murphree efficiency of each tray was computed at each integration step by the methods recommended by AIChE [1958]. In the case of constant molar holdup and real trays, a constant Murphree efficiency of 70% was assumed, which is approximately equal to the efficiency of the complete model at total reflux and steady state. Data for the constant holdup model are presented in Table 3. Physical properties for the system are identical to those of propane, butane, and pentane, except that Antoine coefficients were selected to obtain constant relative volatility behavior, and the surface tension was constant at 20 dyn/cm. See Table 4. A complete listing of all physical properties used in the various models is presented in [Tomazi, 1993]. Stage temperatures used in the energy balances in the constant relative volatility models, where used, were obtained from the Antoine equation.

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