Simple Method of Obtaining Pure Products by Batch Distillation

Massimiliano Barolo and Franco Botteon
Istituto di Impianti Chimici, Università di Padova, via Marzolo, 9 I-35131 Padova PD, Italy

Batch processes are becoming more important as a result of the renewed significance of the fine chemical industries, which are usually required to produce small amounts of products and high added value. Batch distillation often plays an important role in a batch process because, due to its intrinsic flexibility, a single batch column can separate a variety of feed mixtures, easily accommodating the frequent change of market demand. In many cases, the objective of the distillation is to recover both components of a binary mixture at a very high degree of purity. In this case, a convenient way to operate the batch column is that proposed independently by Bortolini and Guarise (1970) and by Treybal (1970). The feed charge is split between the reflux drum (H_D, kmol) and the reboiler (H_B), and the column is operated at total reflux for the whole duration of the batch (Figure 1a). If the reflux drum holdup has been chosen correctly and there are enough stages, the steady state approached by the column is characterized by the accumulation of the (essentially) pure light component in the reflux drum, and of the (essentially) pure heavy component in the reboiler. The advantages of this operating mode are clear: the column always operates at its maximum fractionating capacity (that is, less stages are required in comparison to operation at finite reflux ratio); column operation is very easy (only a level controller is necessary, so as to keep the reflux drum holdup at the prespecified desired value; no product switchovers are required); neither the yield nor the quality of the products are influenced by variations in the heating rate or interruption of the distillation.

However, one should take into account the fact that batch processes are frequently operated through repeated chains of operations. Thus, the actual composition of the feed charge during one batch distillation may not be known precisely, as it may result from the mixing of fresh feed and of recycling streams (slop cuts, for example). Therefore, the uncertainty on feed composition leads to an erroneous choice of the reflux drum holdup, and eventually results in the production of one off-specification product (either the lighter one or the heavier one) if no feedback adjustment of H_D is provided during the operation.

Correspondence concerning this article should be addressed to M. Barolo.

As early as 1970, Bortolini and Guarise indicated that the use of a middle-vessel batch column proves useful in order to "damp out" the uncertainties on the feed charge composition. These authors suggested charging part of the feed (H_M) to the middle vessel also, and to operate the column at total reflux and total reboil (Figure 1b) until the products are on specification; although industrial applications of this procedure were known by that time (Guarise, 1996), the authors did not report any experimental results in their article. Experimental evidence of this operating procedure has been provided only recently (Barolo et al., 1996). Clearly, the main disadvantage of this strategy is that part (H_M) of the whole feed charge is not separated, because it is "trapped" into the feed vessel. Also, presently it is not clear how to choose H_M for a given feed; in practice, given the feed nominal composition, one would like to know the *minimum* amount of such feed that should be charged to the middle vessel in order to guarantee that the final products are obtained pure through simple operation at total reflux and total reboil.

The objective of this article is to provide practical guidelines for the choice of H_M.

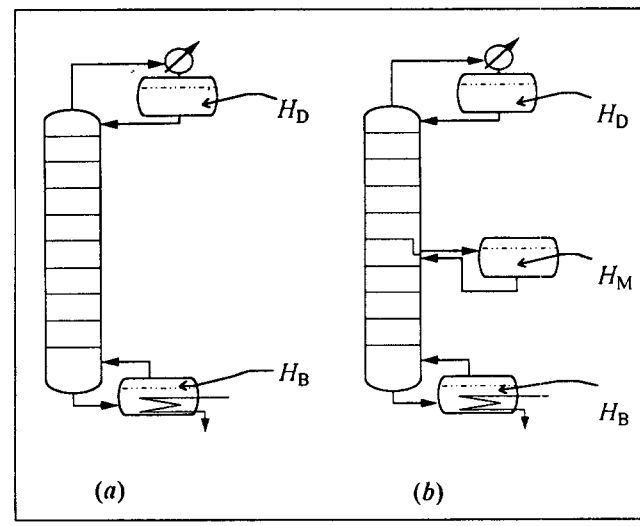

Figure 1. (a) Conventional and (b) middle vessel total-reflux batch distillation columns.

Development, Results and Discussion

In the following, we will assume that the tray holdup is negligible in comparison to the amount charged in the vessels, some considerations on the effect of tray holdup will be given later on. Also, the usual assumptions of constant molar overflows, perfect mixing on the vessels, total condensation with no subcooling, boiling feed, and perfect level control will be considered. As for the description of the thermodynamic equilibrium, a binary mixture with constant relative volatility α will be considered for the sake of simplicity; however, as will be shown, this assumption is not restrictive.

Consider a conventional total reflux operation (Figure 1a) in a column with a large number N of stages. Here, "large" has a relative meaning rather than absolute; in fact, depending on the system relative volatility, even a "small" number of theoretical trays will give the same result as the case with $N \gg 1$. For a feed of nominal composition z (mole fraction), the pure components can be recovered at the end of the batch distillation if the reflux drum and reboiler holdups are chosen according to the following criterion

$$\frac{H_D}{H_B} = \frac{z}{1-z} \tag{1}$$

This can be seen from the full lines of Figure 2, which refers to the system considered in Table 1; the dimensionless time τ in abscissa is defined as $\tau = tV/H_D$, where t is the time and V is the vapor boilup rate. However, if the actual feed composition z' differs from the expected nominal value z, the feed charge split calculated through Eq. 1 is not correct and only one product will be obtained pure. In the case of Figure 2 (broken lines), the feed is richer in the light component than expected ($z' > z$), so that some of the light component necessarily contaminates the bottom product; thus, an on-line adjustment of the reflux drum holdup should be performed (Wittgens et al., 1996), which may somewhat complicate the operation of the column.

As was noted by Bortolini and Guarise (1970), the insertion of a middle vessel in the column has the advantageous effect of "damping out" the uncertainties in the feed composition. This can be clearly seen by the curves represented in Figure 3; these curves refer to the separation of a feed of composition $z' = 0.65$ with H_D and H_B as in Figure 2. The practical implementation of a total-reflux middle vessel strategy is indeed simple, and can be easily implemented even in a column designed for continuous operations (Barolo et al., 1996). However, the main drawback of the middle vessel column is that not *all* the feed can be separated and one should determine the minimum amount $H_{M,\min}$ of feed that needs to be charged to the feed vessel in order to separate a given amount ($H_D + H_B$) of pure products from a feed of nominal composition z such that $z' = z + \Delta z$, where Δz is the uncertainty on the feed composition.

Table 1. System Parameters and Operating Conditions Used in the Simulations

α	z (Nominal)	V (kmol/h)	H_D (kmol)	H_B (kmol)
3	0.60	7	45	30

For the purpose of calculating $H_{M,\min}$, it is convenient to refer to the steady state to which the column moves. From a theoretical point of view, this steady state is reached after an infinite distillation time; however, it is clear (Figure 3) that a situation very close to that of the final steady state is reached after a limited time. Two cases can be outlined. In both of them, it is supposed that the amount charged to the reflux drum and to the reboiler meets the condition of Eq. 1.

- *Case 1.* $\Delta z > 0$ (feed lighter than expected).
  In this case, the reflux drum contains H_D kmol of pure light component at steady state ($x_D = 1$). However, since the amount of light component charged to the column is larger than H_D, the exceeding amount (E kmol) of this component will eventually accumulate in the middle vessel, and possibly in the reboiler. Depending on the amount charged to the middle vessel, one of the following three subcases can be encountered:

(a) $E > H_M$: the middle vessel composition is $x_M = 1$, and the bottom product is contaminated by the light component;

(b) $E = H_M$: the middle vessel composition is $x_M = 1$, and the bottom product is composed by pure heavy component ($x_B = 0$);

(c) $E < H_M$: the middle vessel composition is such that $x_M < 1$, and the bottom product is composed by pure heavy component.

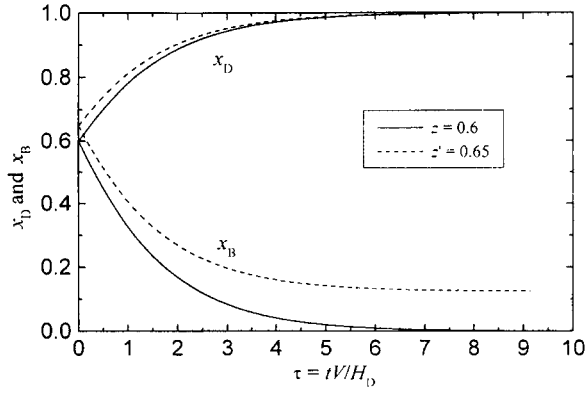

Figure 2. Total reflux operation in a conventional batch column for a correct (full lines) and incorrect (broken lines) split of the feed charge.

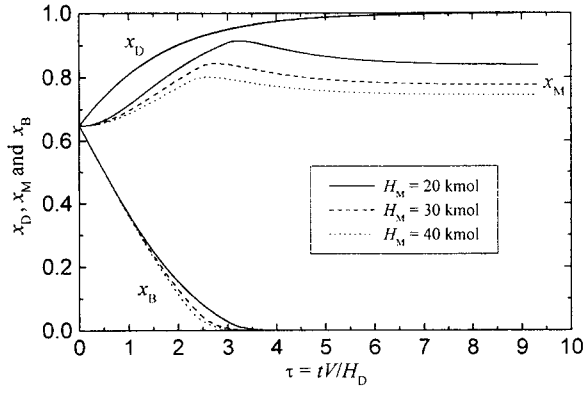

Figure 3. Composition profiles in the reflux drum, middle vessel and reboiler for different values in the middle vessel holdup.

When

$$H_0 = H_D + H_B \tag{2}$$

it follows that

$$E = (H_0 + H_M)(z + \Delta z) - H_D. \tag{3}$$

Subcase b refers to the minimum amount H_M^+ that needs to be charged to the middle vessel in order to ensure the sharp separation of the mixture components when an uncertainty $\Delta z > 0$ is expected in the feed composition. Thus, taking into account condition 1, Eq. 3 gives

$$H_M^+ = E = \frac{\Delta z}{1-(z+\Delta z)} H_0 \tag{4}$$

Note that H_M^+ is a function of the nominal feed composition, the expected feed composition uncertainty, and the total amount of products only.

- *Case 2.* $\Delta z < 0$ (feed heavier than expected).
  In this case, the reboiler contains H_B kmol of pure heavy component at steady state ($x_B = 0$). With a reasoning similar to that developed for the previous case, the amount E' of heavy component that will distribute between the middle vessel and the reflux drum is expressed by

$$E' = (H_M + H_0)(1 - z - \Delta z) - H_B \tag{5}$$

Depending on the middle vessel charge, one of the following three subcases can be encountered:

(a) $E' > H_M$: the middle vessel composition is $x_M = 0$, and the top product is contaminated by the heavy component;

(b) $E' = H_M$: the middle vessel composition is $x_M = 0$, and the top product is composed by pure light component ($x_D = 1$);

(c) $E' < H_M$: the middle vessel composition is such that $x_M > 0$, and the top product is composed by pure light component.

Application of the condition pertaining to subcase b leads to the following expression for the minimum amount H_M^- that needs to be charged to the middle vessel in order to ensure the sharp separation of the mixture components when an uncertainty $\Delta z < 0$ is expected in the feed composition

$$H_M^- = E' = \frac{-\Delta z}{z + \Delta z} H_0 \tag{6}$$

However, it should be noted that the sign of Δz is not known *a priori*. So, one should first estimate the *maximum* expected absolute uncertainty $|\Delta z_{max}|$ on the feed composition (that is, $z' = z \pm |\Delta z_{max}|$), and then use Eqs. 4 and 6 to calculate the values of H_M^+ and H_M^-. Finally, the amount to be charged to the middle vessel will "prudentially" be chosen as $H_M = \max\{H_M^+; H_M^-\}$. Thus, either Subcase c (Case 1) or Subcase c (Case 2) will generally be met.

Note that this result is independent of the system of relative volatility; more generally, it is independent of the form of the equilibrium relationship between the light and the heavy components. This is due to the fact that the number of stages of the column has been estimated to be large enough for a pinch point to be found in each section of the column at any time. However, the relative volatility will affect the time needed to approach the final steady state. As for the effect of column holdup, the tray holdup itself has a stabilizing effect, as was indicated by Bortolini and Guarise (1970). This was also verified through simulations with a more detailed column model. However, one should remember that more feed needs to be charged to the column in order to provide the necessary tray holdup.

Example

It is required to obtain $H_0 = 75$ kmol of very pure products from a feed mixture of mole fraction $z = 0.6 \pm 0.05$. According to Eq. 1, the feed should be split so that $H_D = 45$ kmol and $H_B = 30$ kmol are charged to the reflux drum and the reboiler, respectively. Since $|\Delta z_{max}| = 0.05$, by applying Eqs. 4 and 6 one gets: $H_M^+ = 10.7$ kmol and $H_M^- = 6.8$ kmol. Thus, the amount of feed that needs to be charged to the middle vessel in order to ensure the requested purity for both products is $H_M = \max\{H_M^+; H_M^-\} = 10.7$ kmol. Figure 4 illustrates three possible situations that can be met for this value of H_M. It is clear that if $z' \in [0.55; 0.65]$, then the desired purity is guaranteed in spite of the uncertainty on the actual feed composition; however, if $z' = 0.67$ is found, the bottom product will not be obtained pure ($E > H_M$; $x_M = 1$).

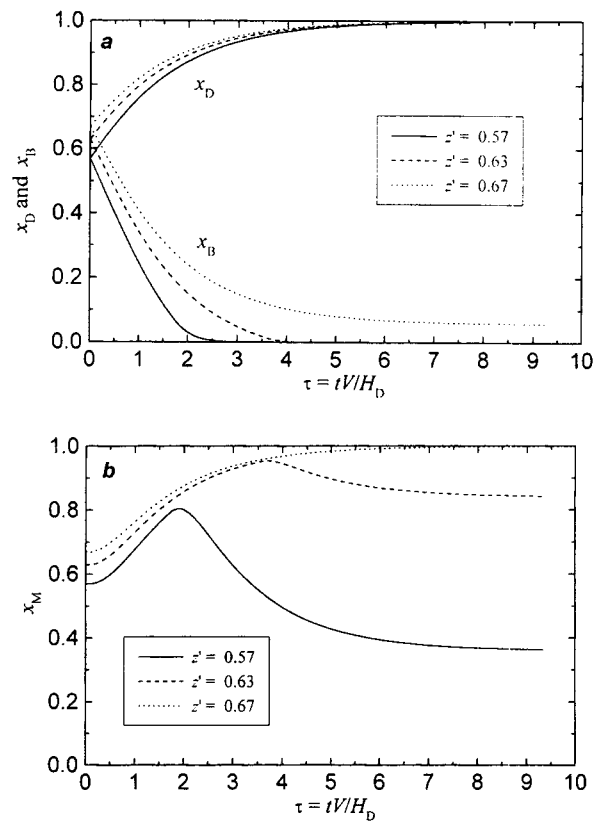

Figure 4. Effect of the feed charge composition on the attainable product purities for $H_M = 10.7$ kmol: composition profiles (a) in the reflux drum and reboiler and (b) in the middle vessel.

Conclusion

Two products at a very high degree of purity can be simultaneously obtained from batch distillation of a binary mixture, if part of the feed is charged to a middle vessel connected to the column. The column can be operated very easily at total reflux and total reboil. Practical guidelines for the choice of the amount of feed that need to be charged to the middle vessel have been provided. The engineering significance of the proposed method has been illustrated through a numerical example.

Acknowledgment

The financial support granted to this work by the Italian National Research Council (CNR, Progetto Strategico Tecnologie Chimiche Innovative) is gratefully acknowledged.

Literature Cited

Barolo, M., G. B. Guarise, S. A. Rienzi, A. Trotta, and S. Macchietto, "Running Batch Distillation in a Column with a Middle Vessel," *Ind. Eng. Chem. Res.*, **35**, 4612 (1996).

Bortolini, P., and G. B. Guarise, "A New Practice of Batch Distillation (in Italian)," *Quad. Ing. Chim. Ital.*, **6**, 150 (1970).

Guarise, G. B., personal communication (1996).

Treybal, R. E., "A Simple Method for Batch Distillation," *Chem. Eng.*, 95 (Oct. 5, 1970).

Wittgens, B., R. Litto, E. Sørensen, and S. Skogestad, "Total Reflux Operation of Multivessel Batch Distillation," *Computers Chem. Eng.*, **20**, S1041 (1996).

Manuscript received Jan. 9, 1997, and revision received May 16, 1997.