products. Final purification of the ester product is carried out in a rectification column with only a few stages. The entrainer is recovered using a one-step liquid-liquid extraction (LLE) column with water as the extractant.

Wuest *et al*. found they could prepare ethyl chloroacetate by addition of ethanol to a melt of chloroacetic acid and *p*-toluene sulfonic acid and purify the ester product using azeotropic distillation. The ester and water form a minimum boiling binary azeotrope and therefore affect the ester/alcohol separation. Water and ethyl chloroacetate are removed overhead and the product is decanted. In this manner the ester is practically alcohol-free. This process avoids formation of the ternary ester/alcohol/water azeotropes which are difficult to separate.

Hills *et al*. found that azeotropic distillation is useful in the purification of esters produced using an enzymatic route. For example, isopropyl myristate can be prepared with lipase or immobilized lipase from myristic acid and propane-2-ol. The water produced in the reaction is removed by azeotropic distillation.

### **Conclusion**

For almost 100 years azeotropic distillation has been an essential tool in the separation of purification of many industrial chemicals. There are distinct advantages to using azeotropic distillation, including energy savings, increased recovery and ability to separate components contained in close boiling, pinch point and azeotropic mixtures. Azeotropic distillation will undoubtedly remains a viable alternative for simplifying difficult separations found in industry.

See also: **I/Distillation. II/Distillation:** Energy Management; High and Low Pressure Distillation; Historical Development; Instrumentation and Control Systems; Modelling and Simulation; Multicomponent Distillation; Theory of Distillation; Vapour-Liquid Equilibrium: Correlation and Prediction; Vapour-Liquid Equilibrium: Theory.

## **Further Reading**

- *Catalogue of Successful Hazardous Waste Reduction*/*Recycling Projects*. Energy Pathways Inc. and Pollution Probe Foundation (1987) Prepared for Industrial Programs Branch, Conservation & Protection Environment Canada, March, p. 84.
- Guinot H and Clark FW (1938) Azeotropic distillation in industry. *Transactions of the Institute of Chemical En*gineers 16: 189-199.
- Hills GA, Macrae AR and Poulina RR (1989) Eur. Pat. Appl. EP383,405, 16 Feb.
- Horsely LH (1973) *Azeotropic Data III*. Washington DC: American Chemical Society.
- Mato Vazques F, Bonilla Alonso D and Gonzalez Benito G (1990) Purification of commercial acetic acid esters by azeotropic distillation. Part I. Selection of azeotropic agents. *Quim*. *Ind*. (*Madrid*) 36(5): 444-449.
- Mato Vazques F, Bonilla Alonso D and Gonzalez Benito G (1990) Preparation of high-purity esters and recovery of azeotropic agents. Part II. Purification of commercial acetic acid esters by azeotropic distillation. *Quim*. *Ind*. (*Madrid*) 36(6): 543-547.
- Ohura H, Uchida Y, Yasaki A and Kishimoto S (1993) EP 0 529 413 A2. 13 Mar.
- Taylor AM and Wheelhouse RW (1992) EP 0 467 531 A1. 22 Jan.
- Te C and Shih TT (1989) Development of an azeotropic distillation scheme for purification of tetrahydrofuran. Fluid Phase Equilibria. *5th International Conference on Fluid Properties and Phase Equilibria for Chemical Process Design*, 30 Apr-5 May, Vol. 52, pp. 161-168. Banff, Alberta, Canada.
- Wuest W, Leischner H and Esser H (1989) Eur. Pat. Appl. EP 315,096, 10 May.
- Young S (1902) The preparation of absolute alcohol from strong spirit. *Journal of the Chemical Society* 81: 707-717.

## **Batch Distillation**

M. Barolo, Università di Padova, Padova, Italy

Copyright  $\odot$  2000 Academic Press

## **Introduction**

The interest in batch distillation has been steadily increasing in the last few years. This is a natural reflection of a change in industrial trends. In fact, the shift towards the production of high value-added and low volume speciality chemicals and biochemicals has been much more marked in the last decade than in the past. Within firms producing specialized chemicals and biochemicals, batch distillation is a ubiquitous method for separating products from process inventories, for recovering components that are used in one of the process steps (like solvents, for example), and for removing undesired components

from an effluent stream. Examples of industrial applications of batch distillation include: the recovery of chloroform, butanol, water, organic and inorganic acids from process broths in the pharmaceutical industry; the concentration of acetone from dilute aqueous mixtures and the removal of water from butyl alcohol in the fine chemistry industry and the manufacture of alcoholic beverages from fermented mixtures in the food and beverages industry.

The success of batch distillation as a method of separation is undoubtedly due to its flexibility. In fact, a single batch column can separate a multicomponent mixture into several product cuts within a single batch but if the separation were carried out continuously, either a train of columns or a multipass operation would be required. Also, whenever completely different mixtures must be processed from day to day, the versatility of a batch column is still unexceeded. These attributes are crucial for responding quickly to a market demand characterized by short product lifetimes and severe specification requirements.

## **Equipment Arrangements and Process Modelling**

#### **Simple Distillation**

The simplest batch distillation apparatus is shown in **Figure 1**. The operation is known as simple distillation or differential distillation. This is the oldest manifestation of distillation itself; it was used by many ancient cultures as a way of producing alcoholic beverages, essential oils and perfumes.

With regard to the separation of a binary mixture, the batch apparatus is run as follows. The pot is continuously heated, so that a vapour rich in the more volatile component is produced, condensed and collected in the external receiver. Indicating with  $x_B$  and *y* the instantaneous mole fractions of the more volatile component in the liquid and vapour phases in equilibrium, and with  $H_B$  the molar amount of boiling liquid remaining in the pot, the following material balances hold during any small time interval:

$$
dH_V = - dH_B \tag{1}
$$

$$
y dH_V = - d(H_B x_B)
$$
 [2]

where  $dH<sub>V</sub>$  is the molar amount of vapour produced during the same time interval. Combining [1] and [2], and integrating from the original feed conditions  $(H_F)$ moles of boiling liquid of mole fraction *z*) to the present conditions, leads to the so-called Rayleigh equation:

$$
\ln \frac{H_{\rm B}}{H_{\rm F}} = \int_{z}^{x_{\rm B}} \frac{\mathrm{d}x'_{\rm B}}{y - x'_{\rm B}} \tag{3}
$$

Eqn [3] can be integrated numerically or graphically. It relates the amount of liquid remaining in the pot to the composition of this liquid. The molar amount  $H<sub>D</sub>$  and average composition  $\bar{x}<sub>D</sub>$  of the liquid accumulated in the receiver can be determined from the following material balances:

$$
H_{\rm F} = H_{\rm B} + H_{\rm D} \tag{4}
$$



**Figure 1** Apparatus for simple distillation.

and:

$$
H_{\rm F}z = H_{\rm B}x_{\rm B} + H_{\rm D}\bar{x}_{\rm D} \tag{5}
$$

If the system displays a constant relative volatility  $(\alpha)$ , the integration of eqn [3] can be performed analytically, leading to:

$$
\ln \frac{H_{\rm B}}{H_{\rm F}} = \ln \frac{1-z}{1-x_{\rm B}} + \frac{1}{\alpha - 1} \ln \frac{x_{\rm B}(1-z)}{z(1-x_{\rm B})} \qquad [6]
$$

A graphical representation eqn [6] is presented in **Figure 2**. It is shown that, for a given fractional purification  $x_B/z$  of the feed, a larger amount of liquid can be recovered at the end of the batch when the feed is leaner in the more volatile component. It is also clear that it is not possible to obtain a finite amount of the pure heavy component, whatever the feed composition.

For a mixture of *Nc* components, *Nc* Rayleigh equations of the form [3] can be derived, and an iterative procedure is usually employed in order to determine the composition profile of each component as a function of the amount of liquid remaining in the still. In the case of constant relative volatility mixtures, direct calculation of the composition profiles can be accomplished by defining the amount  $b_i$  of the *i*th component remaining in the pot  $(b_i = H_B x_{B,i})$ . It can be shown that:

$$
\frac{b_i}{f_i} = \left(\frac{b_j}{f_j}\right)^{\alpha_{i,j}}\tag{7}
$$

where *j* is an arbitrarily chosen reference component,  $f_i = H_F z_i$ , and  $\alpha_{i,j}$  is the relative volatility of the *i*th component with respect to the reference one. From



**Figure 2** Simple distillation: fraction of the initial charge recovered after the purification of three different binary feeds from a light impurity.

the definition of  $b_i$ , it follows that:

$$
x_{B,i} = \frac{b_i}{\sum_{i=1}^{N_c} b_i}
$$
 [8]

Differential distillation is a very simple way of separating a mixture of several components into cuts of different purities, for example for laboratory applications. However, it permits only rough separations, even with large energy consumption (it is a one-theoretical-stage operation). This is the reason why multistaged columns with reflux are employed whenever a sharper separation is needed.

#### **Batch Rectification**

A typical batch distillation column (a batch rectifier) is shown in **Figure 3**. The feed mixture is charged to a large reboiler, to which a heating medium (e.g. steam) is also fed. The vapour reaching the top of the column is condensed, and partially returned as reflux. A distillate stream is withdrawn, which sequentially feeds a series of tank receivers. The column may be a staged or a packed one.

Since in practice it is much more frequently requested to operate an existing column rather than to design a new one, only the issues of modelling and operating an existing rectifier will be addressed in the following sections.

Regardless of the operating mode, the columnmodelling approaches can be grouped into three main classes: short-cut modelling, approximate modelling and rigorous modelling.

**Short-cut models** Short-cut models usually assume that the column dynamic behaviour can be approximated as a sequence of steady states (pseudo-steadystate approximation), which in turn is true when the column and reflux drum hold-ups can be neglected compared to that of the reboiler. This assumption allows the short-cut models that have been devised for the design of continuous columns to be employed at every time step in the simulation of batch columns. When binary mixtures are considered, a graphical representation of the process can be obtained by means of classical McCabe–Thiele diagrams. For multicomponent mixtures, a modification of the Fenske-Underwood-Gilliland design approach is often employed for simulating the batch column operation.

The main advantage of these models is their speed of computation; the main limits are their poor accuracy, and the fact that most of them are limited to constant relative volatility mixtures. They are mostly employed in preliminary process design and



**Figure 3** Sketch of a batch rectifier.

optimization studies, when a large number of cases need to be evaluated quickly, and a result of limited accuracy is acceptable. Rigorous results can be obtained by the subsequent use of more accurate models.

**Approximate models** Approximate models are used whenever a more realistic representation of the column dynamics is required. In this case, the tray and reflux drum liquid hold-ups are explicitly taken into account, even though they are usually assumed to be constant throughout the whole operation. Other usual assumptions are: boiling feed, total condensation without subcooling, perfect mixing in all parts, negligible vapour hold-ups, constant boil-up rate, constant molar overflows, ideal tray behaviour and negligible pressure drops and heat losses.

The set of equations usually employed to model a column with a separating capacity of *N* ideal trays is as follows:

**Reboiler**

$$
\frac{dH_{\rm B}}{dt} = L - V \tag{9}
$$

$$
\frac{d(H_Bx_{B,j})}{dt} = Lx_{1,j} - Vy_{B,j} \quad j = 1, 2, ..., Nc - 1 \quad [10]
$$

**Bottom tray** (**tray 1**)

$$
H_1 \frac{dx_{1,j}}{dt} = L(x_{2,j} - x_{1,j}) - V(y_{1,j} - y_{B,j})
$$
  

$$
j = 1, 2, ..., Nc - 1
$$
 [11]

**Tray i**

$$
H_{i} \frac{dx_{i,j}}{dt} = L(x_{i+1,j} - x_{i,j}) - V(y_{i,j} - y_{i-1,j})
$$
  

$$
j = 1, 2, ..., Nc - 1
$$
 [12]

**Top tray** (**tray N**)

$$
H_{N} \frac{dx_{N,j}}{dt} = L(x_{D,j} - x_{N,j}) - V(y_{N,j} - y_{N-1,j})
$$
  

$$
j = 1, 2, ..., Nc - 1
$$
 [13]

**Reflux** drum

$$
\frac{dH_{\rm D}}{dt} = 0 \quad (V = L + D) \tag{14}
$$

$$
H_{\rm D} \frac{dx_{\rm D,j}}{dt} = V(y_{\rm N,\,j} - x_{\rm D,\,j}) \quad j = 1, 2, \, \dots, Nc - 1
$$
\n[15]

**Thermodynamic equilibrium in stage** *i*

$$
y_{i, j} = K_{i, j} x_{i, j} \quad j = 1, 2, ..., Nc - 1
$$
 [16]

**Summation constraints**

$$
x_{i, Ne} = 1 - \sum_{k=1}^{Ne-1} x_{i, k} \qquad [17]
$$

$$
y_{i, Nc} = 1 - \sum_{k=1}^{Nc-1} y_{i, k}
$$
 [18]

In the above equations,  $H_\mathrm{i}$ ,  $H_\mathrm{D}$  and  $H_\mathrm{B}$  are the tray, reflux drum and reboiler hold-ups respectively;  $x_{i,j}$  is the mole fraction of component *j* in stage *i*; *L*, *V* and *D* are the liquid, vapour and distillate molar flow rates (respectively);  $K_{i,j}$  is the vapour/liquid equilibrium ratio of component *j* in stage *i*; and *t* is the time. Any suitable thermodynamic model can be used for the representation of the equilibrium ratio; deviations from the ideal tray behaviour can be taken into account using the Murphree tray efficiency concept. Due to possible wide ranges in the relative volatilities and/or large differences in tray and reboiler hold-ups, the system of differential equations is frequently stiff. As a starting point for the integration, it is often assumed that the reboiler, reflux drum and all trays are filled with the boiling liquid feed at the beginning of the operation.

Approximate models provide a fairly accurate description of the column dynamics, and are frequently used in process design and process optimization applications.

**Rigorous models** When a still more accurate description of the process dynamics is needed (for example, when it is required to model accurately the operation of an existing column), a rigorous model should be employed. In this case, almost all of the previous assumptions are relaxed, which considerably increases the computational load. The determination of the system and equipment parameters (like the Murphree tray efficiency, for example) is done by fitting some of the modelled profiles to actual plant data.

#### **Other Column Arrangements**

In the attempt to improve the flexibility of batch distillation operation as a whole, a closer look at alternative column configurations has been undertaken in the last few years. These different kinds of columns have been known since the 1950s, but a thorough understanding of their dynamic behaviour and features has only recently been achieved.

In the batch stripper represented in **Figure 4**A, the feed is charged to the reflux drum, while multiple cuts are withdrawn from the bottom of the column sequentially. In the middle-vessel batch column (Figure 4B), the feed is charged to an intermediate vessel, and the product/impurity removal is accomplished simultaneously from both the top and the bottom ends. The reboilers of both columns may be considerably smaller than the reboiler of a batch rectifier.

Potential improvements in column productivity, with respect to conventional batch rectifiers, may be very large when using these alternative configurations. However, to date it is not possible to provide general guidelines for determining *a priori* the best column configuration for a certain separation.



**Figure 4** Sketch of (A) a batch stripper and (B) a middle-vessel batch column.

A preliminary simulation with a simplified model (or even with a short-cut one) should be used to assess the most profitable configuration for separating a given feed (amount and composition), subject to the given purity constraints.

## **Column Operation**

The operation of a batch rectifier only will be considered in here. The most popular operating modes are: constant reflux ratio (with variable distillate composition), constant distillate composition (with variable reflux ratio) and total reflux (no product take-off). The former two operations are in fact semibatch operations; the latter one is truly a batch operation. Operation at optimal reflux ratio should be mentioned too, but  $-$  since it is often accomplished through a series of constant reflux ratio sequences  $-$  this operating mode will not be considered here. In the following, the components are numbered in order of decreasing ease of separation (i.e. component 1 is the most volatile one, component 2 is the second most volatile, and component *Nc* is the least volatile).

From a practical point of view, regardless of the operating policy, the operation of a multicomponent column is carried out in three steps: (i) a start-up phase; (ii) a main cut production phase; and (iii) a slop cut removal phase. The last two steps are repeated sequentially. The following actions take place during the start-up phase: the column and receivers are possibly cleaned from the materials processed in the previous batch; cooling of the condenser is started; steam is fed to the reboiler, so that the material to be processed is heated up to its bubble point and starts to vaporize; as soon as the overhead vapour has been condensed and has filled the reflux drum, liquid is returned to the top of the column, and the column is run at total reflux; consequently, the column trays are sequentially filled with liquid.

The start-up procedure is very general, and goes on until either the steady state is approached, or the lighter product purity specification is reached in the reflux drum. The other two phases vary according to the operating policy employed.

#### **Constant Reflux Ratio Operation**

In the case of the separation of a binary mixture, a short-cut modelling approach is useful in order to have a graphical representation of the process. In a McCabe–Thiele diagram, at a given value  $r = L/D$ of the reflux ratio, the slope of the operating line is fixed during the operation (**Figure 5**).



**Figure 5** Binary batch distillation at constant reflux ratio (threeideal-stage column).

While the distillation is proceeding, the operating line shifts down along the diagonal line. The mole fraction of the more volatile component in the distillate is continuously decreasing, and, at any instant of time, the reboiler composition is determined by stepping off *N* stages along the operating/equilibrium lines.

If both the top and the bottom purity specifications  $(x_D^{\text{spec}}$  and  $x_B^{\text{spec}}$ , respectively) are assigned, the reflux ratio should be chosen such that the final amount  $H<sub>B</sub><sup>final</sup>$  of bottom product, as calculated through eqns [4] and [5] with  $x_B = x_B^{\text{spec}}$  and  $\bar{x}_D = x_D^{\text{spec}}$ , is the same as the amount calculated by means of the Rayleigh equation:

$$
\ln \frac{H_{\rm B}^{\rm final}}{H_{\rm F}} = \int_{z}^{x_{\rm B}^{\rm spec}} \frac{dx_{\rm B}}{x_{\rm D} - x_{\rm B}} \tag{19}
$$

The integral of eqn [19] can be evaluated numerically or graphically. The total amount  $Q_r$  of energy to be supplied at the reboiler is given by:

$$
Q_{\rm r} = \lambda (r+1) H_{\rm D}^{\rm final} \tag{20}
$$

where  $\lambda$  is the molar latent heat of vaporization of the mixture, and  $H<sub>D</sub><sup>final</sup>$  is the final amount of distillate product collected.

For a multicomponent mixture, after starting up the column, the product removal is started at a constant and finite reflux ratio, and it continues until the average composition of the accumulated product  $(product 1)$  falls below the specification. At this point (called the cut-off point), the distillate composition is

neither sufficiently rich in component 1 to allow further accumulation of product 1, nor is it sufficiently rich in component 2 to allow beginning the withdrawal of product 2. Therefore, the distillate stream is diverted to another tank, and the collection of slop cut 1 is started. When the concentration of component 2 in the reflux drum meets the second product's specification, the distillate stream is diverted to a third receiver, and the accumulation of product 2 is initiated. The operation proceeds this way until all the products but the heaviest have been collected in the receivers. The heaviest product (product *Nc*) is recovered in the reboiler at the end of the operation. Therefore, at the end of the operation *Nc* main cuts (products) and at most  $Nc - 1$  slop cuts are obtained. The slop cuts need to be reprocessed in subsequent batches.

It should be noted that, unless this issue has been addressed at the column design stage, the whole tray hold-up drains to the reboiler when the steam supply is stopped. This must be taken into account when estimating online the final reboiler composition in order to detect the time when the distillation should be interrupted.

The plot of a typical evolution of the profiles of the average product composition, product amount and instantaneous distillate composition for the separation of a nonazeotropic ternary mixture at constant reflux ratio is shown in **Figure 6**. The profiles were determined with the approximate model described previously, and a constant relative volatility mixture was considered.

The dynamic behaviour of the column is characterized by a 'front' of each component sequentially moving from the bottom to the top of the column. If the column has enough stages, the top trays virtually separate a binary mixture made of the current most volatile component and its adjacent



**Figure 6** Typical evolution of the profiles of the average product composition, product amount and instantaneous distillate composition for an ideal ternary mixture under constant reflux ratio operation.

component in the scale of relative volatilities. This may prove advantageous for the control of the operation.

Operation at constant reflux ratio is the most popular in industry, because it is very easy to accomplish. However, some considerations deserve attention at this point. In order to implement this operating policy practically, a measure of the distillate and reboiler compositions should be available online, which is not usually the case. Therefore, the product composition needs to be inferred from secondary measurements, which should be available online quickly and at a low cost (like temperature and flow rates, for example). Also, operating the column at a constant reboiler duty is often easier than operating it at constant vapour boil-up, as was done in the previous numerical example. Finally, it should be noted that the column pressure may be decreased during the batch in order to improve the ease of separation of the less volatile components.

#### **Constant Distillate Composition Operation**

For the separation of a binary mixture under the quasi-steady-state assumption, a quick glance at the process can be obtained again by using a McCabe-Thiele representation, as shown in **Figure 7**.

In order to get the desire specification  $x_{\text{D}}^{\text{spec}}$  from the very beginning of the operation, the initial value of the reflux ratio must be such that the feed composition *z* is exactly obtained in the still when stepping off  $N$  stages from  $x_{\textrm{D}}^{\textrm{spec}}.$  Then, the reflux ratio (hence, the



**Figure 7** Binary batch distillation at constant distillate composition (three-ideal-stage column).

slope of the operating line) is continuously increased during the batch in order to keep the distillate composition on specification. The operation is interrupted when either the reboiler composition has reached the desired composition, or the reflux ratio is so high that continuing the operation would be uneconomical (or even impossible, in an actual column).

The determination of the total amount of energy that needs to be supplied to the still is obtained by the following equation:

$$
Q_{\rm r} = \lambda \int_0^{H_{\rm D}^{\rm final}} (r+1) \mathrm{d}H_{\rm D} \tag{21}
$$

whose integration requires knowing *r* as a function of  $H<sub>D</sub>$ . By combining eqns [4] and [5], with  $\bar{x}_{\text{D}} = x_{\text{D}}^{\text{spec}}$ , it is found that:

$$
H_{\rm D} = H_{\rm F} \frac{z - x_{\rm B}}{x_{\rm D}^{\rm spec} - x_{\rm B}} \tag{22}
$$

which enables relating the instantaneous reboiler composition  $(x_B)$  to the amount of product  $(H_D)$  collected up to the same instant. Therefore, since the reflux ratio can be related to  $x_B$  at any instant of time, eqn [21] can be easily integrated.

In a multicomponent separation, product 1 is collected until the reflux ratio reaches the prespecified maximum value. Then, the reflux rate is decreased in order to allow a quick removal of the remaining traces of the most volatile component (these traces are accumulated in slop cut 1). At the same time, this enables the front of the second lightest component to build up in the column so that, when the reflux drum composition meets the specification for product 2, the accumulation of this product is started. The production phase and the slop removal phase are then repeated sequentially; product *Nc* is eventually recovered from the reboiler.

Since operation at constant distillate composition is inherently a feedback operation, some kind of feedback from the plant is needed in order to be able to determine the correct value of the reflux ratio at any instant. When the distillate composition measurement is not available online, the most convenient feedback signal that can be used is a pilot tray temperature measurement. This strategy is similar to the one applied for one-point composition control in continuous columns. In principle, temperature control should be applied to binary mixtures only, but it can be extended to multicomponent mixtures, provided that the column has enough trays. Note that, similarly to continuous distillation, the measuring element must be placed somewhat down from the top of the column in order to ensure sensitivity of the temperature

response to changes in the distillate composition. Therefore, if good control at a constant set point is provided, the product purity usually increases while this product is being removed from the column. This is because the separating capacity of the column increases during the batch, due to the increase of the reflux rate.

From a practical point of view, this kind of operation is harder to implement than constant reflux ratio, even if a measure or an estimate of the distillate composition is available online. Because the dynamics of the process may change significantly as the batch proceeds, conventional proportional plus integral controllers often prove to be inadequate for tight composition control. Care should be taken when the reflux rate increases widely, because the liquid loading to the column might increase to a point that flooding conditions are reached.

#### **Total Reflux Operation**

This operation is the best suited for binary mixtures, whose components should be recovered at a relatively high degree of purity. The feed charge  $H_F$  is split between the reflux drum ( $H_D$  kmol) and the reboiler  $(H_B \text{ kmol})$ , and the column is operated at total reflux for the whole duration of the batch. Under the assumptions of quasi-steady-state operation and constant relative volatility, the operation can be analysed easily. For a given pair of product specifications, the minimum number  $N_{\text{min}}$  of theoretical stages necessary in order to achieve the desired separation can be determined from the Fenske



Figure 8 Binary batch distillation at total reflux (three-idealstage column).

equation:

$$
N_{\min} = \frac{1}{\ln \alpha} \ln \frac{x_{\rm D}^{\rm spec}(1 - x_{\rm B}^{\rm spec})}{x_{\rm B}^{\rm spec}(1 - x_{\rm D}^{\rm spec})} \tag{23}
$$

If the operation is feasible in the available column, the requested top and bottom hold-ups are calculated immediately by eqns [4] and [5], with  $x_B = x_B^{\text{spec}}$  and  $\bar{x}_D = x_D^{\text{spec}}$ .

At any time *t*, the operating line is parallel to the diagonal line in a McCabe–Thiele diagram (**Figure 8**), and moves towards the diagonal as time progresses according to the following dynamics:

$$
V(y - x) = HD \frac{dx_D}{dt}
$$
 [24]

or:

$$
V(y - x) = -HB \frac{dx_B}{dt}
$$
 [25]

Since, at time *t*, the following material balance holds true:

$$
\frac{x_{\rm D} - z}{z - x_{\rm B}} = \frac{H_{\rm B}}{H_{\rm D}}\tag{26}
$$

the reflux drum and reboiler compositions at the same time instant can be determined by using the auxiliary line FQ, where the abscissa  $a<sub>Q</sub>$  of the auxiliary point Q on the *x*-axis is such that:

$$
\frac{a_{\rm Q} - z}{z} = \frac{H_{\rm B}}{H_{\rm D}}\tag{27}
$$

The segment  $\overline{BD}$  is drawn, where B is on the diagonal line with  $a_B = x_B$ , and D is on the auxiliary line FQ with  $a_D = x_D$ . The product compositions at time *t* are correct if the segment  $\overline{BD}$ , is parallel to the *x*-axis.

The total amount of heat to be supplied to the reboiler is given by:

$$
Q_r = \lambda H_{\rm D} \int_z^{x_{\rm D}^{\rm spec}} \frac{\mathrm{d}x_{\rm D}}{y - x} = -\lambda H_{\rm B} \int_z^{x_{\rm B}^{\rm spec}} \frac{\mathrm{d}x_{\rm B}}{y - x} \ [28]
$$

From a practical standpoint, the advantages of this operating mode are:

- the column always operates at its maximum fractionating capacity;
- column operation is very easy: only a level controller is necessary, which keeps the reflux drum holdup at the specified value; no product switchovers are required;
- neither the yield nor the quality of the products is influenced by variations in the heating rate or interruption of the distillation.

However, it should also be observed that unavoidable uncertainties in the feed amount, feed composition and column hold-up would act in such a way as to prevent reaching the desired purity specifications for both products. In order to ensure that the more valuable product is recovered on specification, the hold-up of the vessel containing this product should be set somewhat smaller than requested theoretically; a conventional temperature controller may be cascaded to the reflux drum level controller in order to adjust the level setpoint online, according to the desired purity requirements. Another way to reduce (and even eliminate) the effect of the uncertainties is using a middle-vessel batch column.

For multicomponent separations, a cycling operation may be employed: the column is operated at total reflux until a steady state is approached; distillate is then taken as total draw-off for a short period of time; then, the operation is switched back to total reflux, and the operating cycle is repeated.

## **Conclusion**

As was noted in the Introduction, batch distillation as a way of separating a given mixture in multiple fractions is becoming more and more frequent, especially in the fine chemicals, pharmaceutical, biochemicals, food and beverages industries. While it is true that batch distillation usually consumes more energy than continuous distillation, it nevertheless provides more flexibility and requires less capital investment. This is an economic incentive, particularly when small volume or high added-value products are to be recovered. Still, there is a certain reluctance among operators to use this process. In fact, its unsteady nature, together with the need for monitoring, switching and discharging a series of cuts, may unavoidably confuse plant personnel.

From an engineering perspective, modelling and simulation of the operation is no longer as hard a task as it was in the past. The understanding of the process is fairly comprehensive (at least for conventional columns), so that all the operating steps characterizing a batch distillation process can be modelled in some detail. The availability of computer hardware at low cost makes it possible to solve all the model equations in a reasonably short time. It should be stressed that the availability of a reliable process model is a crucial issue for profitably utilizing an existing batch column. If a good model is available, then it can be used to find out the optimal operating policy for the batch, i.e. to determine which are the sequence of reflux ratios during the batch and the strategy for reprocessing slop cuts between batches that would maximize a prescribed profit function. A process model can also prove useful for the closedloop control of the operation. Advanced control strategies employ process models to get online estimations of the product compositions, thus eliminating the need to resort to expensive hardware analysers; more efficient control laws than the classical proportional plus integral algorithm can also be developed if a process model is available. Finally, process models can be employed within a training programme to instruct the plant personnel on how to interpret the column dynamics, and its response to process uploads: this may prove useful in improving the operator's confidence about this unsteady unit operation.

See also: **II/Distillation:** Historical Development; Instrumentation and Control Systems; Multicomponent Distillation; Pilot Plant Batch Distillation; Theory of Distillation.

### **Further Reading**

- Diwekar UM (1995) *Batch Distillation*: *Simulation*, *Optimal Design*, *and Control*. Bristol: Taylor-and-Francis.
- Hart DR (1992) Batch distillation. In: Kister HZ (ed.) *Distillation Design*. New York: McGraw-Hill.
- Holland CD (1966) *Unsteady State Processes with Applications in Multicomponent Distillation*. Englewood Cliffs: Prentice-Hall.
- Luyben WL (1990) *Process Modeling*, *Simulation*, *and Control for Chemical Engineers*. New York: McGraw-Hill.
- Macchietto S and Mujtaba IM (1996) Design of operation policies for batch distillation. In: Reklaitis GV, Sunol AK, Rippin DWT and Hortaçsu Ö (eds) *Batch Processing Systems Engineering* } *Fundamentals and Applications for Chemical Engineering*. Berlin: Springer.
- Muhrer CA and Luyben WL (1992) Batch distillation. In: Luyben WL (ed.) *Practical Distillation Control*. New York: Van Nostrand Reinhold.
- Robinson CS and Gilliland ER (1950) *Elements of Fractional Distillation*. New York: McGraw-Hill.
- Rose LM (1985) *Distillation Design in Practice*. Amsterdam: Elsevier.
- Seader JD and Henley EJ (1998) *Separation Process Principles*. New York: John Wiley.
- Stichlmair JG and Fair JR (1998) Distillation: Principles and Practice. New York: Wiley-VCH.

# **Control Systems**

See **II / DISTILLATION / Instrumentation and Control Systems**