2. The simulation, synthesis and design of reactive and azeotropic distillation. Such topics still constitute a gap in the knowledge of distillation technology.
3. Investigation of complex configurations for batch distillation processes.
4. Use of optimization methods for obtaining optimal configuration and design of batch and continuous distillation processes.
5. Online optimization and control of columns.

See also: **II/Distillation:** Batch Distillation; Theory of Distillation; Vapour-Liquid Equilibrium: Correlation and Prediction; Vapour-Liquid Equilibrium: Theory.

Further Reading

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Packed Columns: Design and Performance

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Use of Packing in Distillation

Use of packing in mass transfer has its origins in the early 1800s for simple applications such as alcohol distillation, and in sulfuric acid plant absorbers. Glass balls, coke or even stones were used as packing materials. Nevertheless packings for distillation were not established until the 1930s with the use of regular shape materials such as ceramic Raschig rings and Berl saddles, as well as the availability of distillation calculations such as the McCabe–Thiele and Ponchon–Savarit methods. Early in the second half of the century, the use of packing for distillation went through a transformation, producing the second-generation packings (see **Table 1**). Regular and improved shape of packings, such as pall rings, became available with larger open areas that permitted a substantial increase both in capacity and column efficiency. In the 1960s Sulzer introduced the wire-mesh packings with very high efficiency (low height equivalent to a theoretical plate, HETP), resulting in a new transformation in the use of packings. In the 1970s and 1980s all major mass-transfer equipment manufacturers developed structured packings. Compared to the traditional tray columns spectacular improvements in plant capacity were achieved, but also some projects were pitfalls, when the expected benefits did not materialize. Manufacturers started realizing that liquid distributors had to be improved, but there was no coherent understanding, nor correlations, that could lead to a safe distributor-column system design. Many manufacturers returned to trays, producing new improved designs, using the area under the downcomer for vapour flow: these trays are offered with new names that indicate their increased vapour flow capacity (Maxyflow, Superfrack, etc.). The need for good distribution and its effect on the column efficiency are now well understood, allowing safe design and efficient applications for random and structured packings in large industrial columns.

General Concepts

Distillation separation is based in relative volatility that makes it possible to concentrate the more volatile components in the vapour phase while the less volatile ones remain in the liquid phase. Distillation columns are countercurrent vapour–liquid mass-transfer devices, where the required separation and purification of components is achieved.

Table 1 Evolution of packing

	First generation, before 1950	*Second generation, 1950–1970*	*Third generation, after 1970*
Random packings	Rashing rings Lessing rings Saddles	Intalox® (Norton) Pall Rings[a]	IMTP® (Norton) CMR® (Koch Glitsch) Chempak®[b] Fleximax® (Koch Glitsch) Nutter Ring® (Nutter)
Grids		C-Grid (Koch Glitsch)[c] EF-25 (Koch Glitsch)[c]	
Structured packing		Wire-mesh type[d]	Sulzer BX and CY Mellapack® (Sulzer) Flexipack® (Koch Glitsch) Gempack® (Koch Glitsch) Intalox® (Norton) Montz packing (Montz)

[a]Developed by BASF, still marketed (or variations of it) by most packing manufacturers.
[b]Developed by Leva, marketed by Nutter.
[c]Variations of these grids are now offered by most packing manufacturers.
[d]Developed by Sulzer, they are now offered by other manufacturers.

The main variable influencing the column design requirements is the relative volatility, α. **Figure 1** illustrates the effect of α on the column performance:

- As α increases, the number of theoretical stages (NTS) required to achieve a fixed product quality decreases, since NTS is proportional to $1/\ln(\alpha)$. As α decreases and approaches 1, the number of stages required increases approaching infinity. At any given α, the minimum number of stages required to achieve a given separation corresponds to a total reflux operation. At total reflux all overhead vapours are condensed and returned to the column as reflux, so that there is no net product. The minimum reflux sets the limiting slope of the operating line, required to achieve a given separation.
- At constant α, the NTS increases as the product purity increases. The increase is proportional to the logarithm of the key components purity ratio.

It can be also demonstrated that:

- At constant product purity, the minimum reflux decreases as α increases.
- At constant product purity, the minimum number of stages decrease as α increases.
- At constant α, the minimum reflux decreases as the product purity decreases.

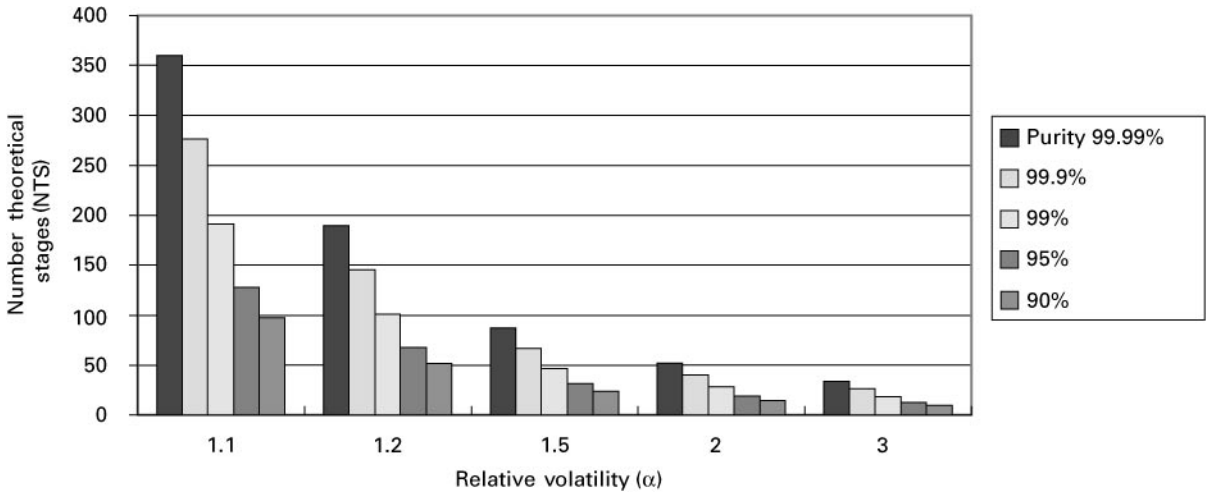

Figure 1 Number of stages required vs. relative volatility at several product purities.

- At constant α, the minimum number of stages increases as the product purity increases.

All these statement say that α defines the separation difficulty. For values around 1.1 and lower, separation by distillation becomes very difficult, requiring very large and expensive columns. For $\alpha = 1$ the mixture is azeotropic and would require the addition of selective entrainers if azeotropic or extractive distillation is to be applied.

Packed Column Description

Figure 2 illustrates a tower with structured packing. In addition to the packing itself, packed columns require other internals to assure the performance of the packing. These internals are:

- Liquid feed pipes to deliver the fluid to the liquid distributors, as seen at the top of the tower and at the intermediate distributor.
- Liquid collection and mixing as shown below the top bed.
- Liquid draw-off sump and pipe as shown below the top bed.
- Liquid redistributors, as presented between the two beds.
- Vapour feed pipes as shown at the vapour inlet nozzle, at the bottom of the tower.
- Packing support plates resting on beams and levelled rings welded to the vessel.
- Hold-down plates.

Incorrect design or incorrect installation of any of these elements can lead to tower failure. One of the most critical element, and often the culprit of tower failures, is the liquid distributor.

Packing Selection

Figures 3 and **4** illustrates random and structured packings. There are many parameters to be considered in the selection of packings; in some cases, there are one or two considerations that dictate the selection, such as capacity for a revamp, which could favour structured packing. There are also some considerations or applications, such as high-pressure distillation, that could make structured packing a questionable choice. **Table 2** gives some general guidlines on packing selection.

Pressure Drop in Packed Beds

The dry-bed pressure gradient is given by the following equation:

$$\Delta P_d = C_1 \rho_g u_g^2 \qquad [1]$$

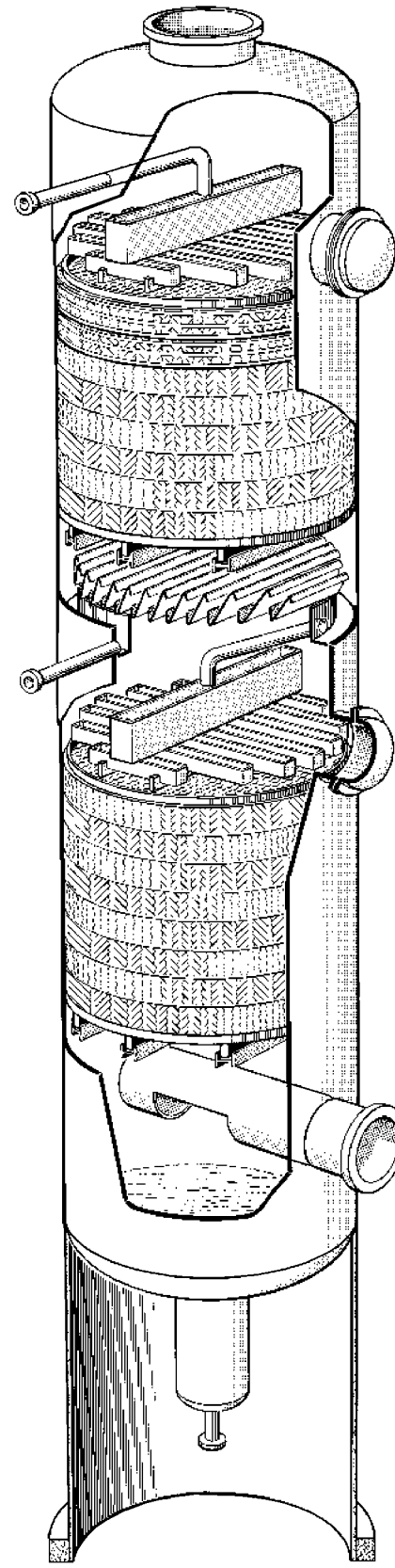

Figure 2 Packed tower illustration. (Photo courtesy of Sulzer Chemtech.)

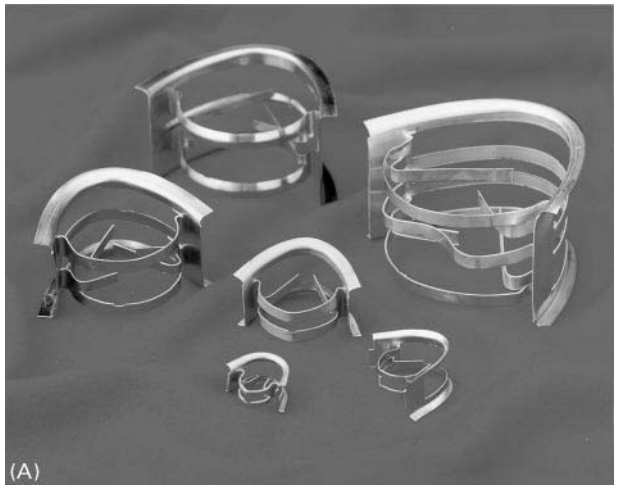

Figure 3 Random packings: (A) IMTP®. (Photo courtesy of Norton Chemical Process Products Corporation.) (B) Nutter Ring®. (Photo courtesy of Sulzer Chemtech.) (C) Cascade Mini-Rings® (CMR™) and Fleximax®. (Courtesy of Koch–Glitsch Inc.) (D) Pall Rings metal and plastic. (Courtesy of Koch–Glitsch Inc.)

Leva extended the correlation to irrigated beds:

$$\Delta P_i = C_1 10^{\beta u_1} \rho_g u_g^2.$$

Robbins developed the following set of general pressure-drop correlations:

$$\Delta P = C_2 G_f^2 \, 10^{C_3 L_f} + 0.4(L_f/20\,000)^{0.1}(C_2 G_f^2 10^{C_3 L_f})^4 \qquad [2]$$

where:

$$G_f = G(0.075/\rho_g)^{0.5}(F_p/20)^{0.5} 10^{0.024\,\rho_g}$$

(for pressures over 1 atm)*

*Note: in this correlation the original term $10^{0.3\rho_g}$ was replaced by $10^{0.024\rho_g}$ since the original correlation predicts too high a pressure drop.

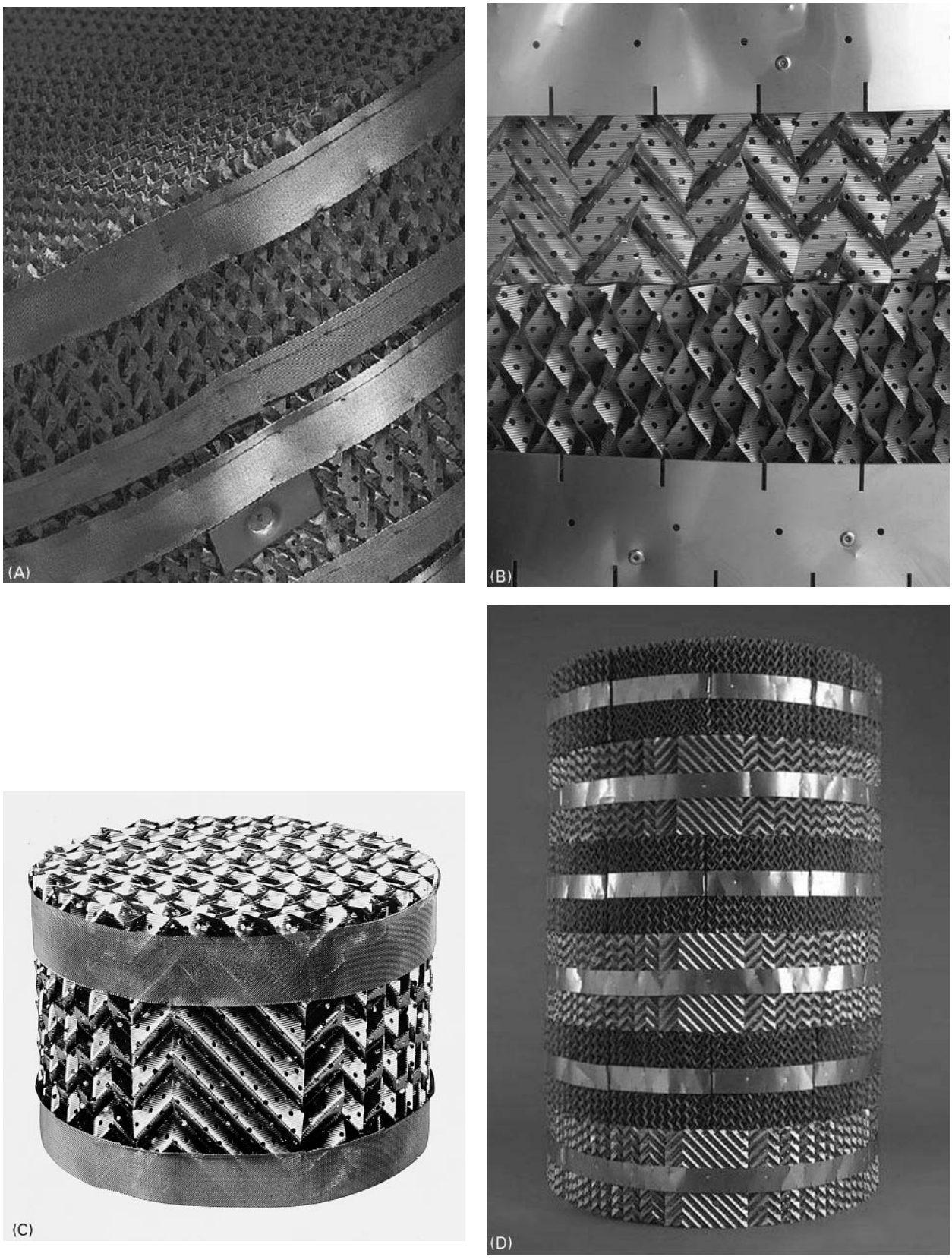

Figure 4 Structured packings: (A) Wire gauze structured packing. Close view, packing and wiper bands. (Photo courtesy of Koch–Glitsch Inc.) (B) Two structured packing layers rotated 90°. (Photo courtesy of Koch–Glitsch Inc.) (C) One structured packing element for small towers. (Photo courtesy of Sulzer Chemtech.) (D) Structured packed bed for a small tower. (Photo courtesy of Koch–Glitsch Inc.) (E) Packed bed for a large tower built in sections. (Photo courtesy of Norton Chemical Process Products Corp.)

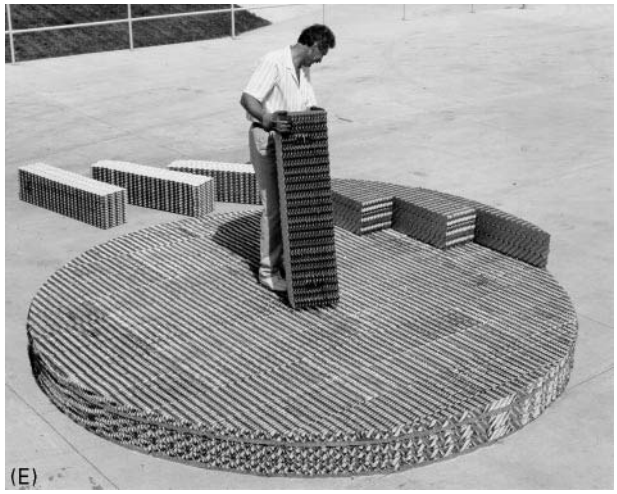

Figure 4 *Continued*

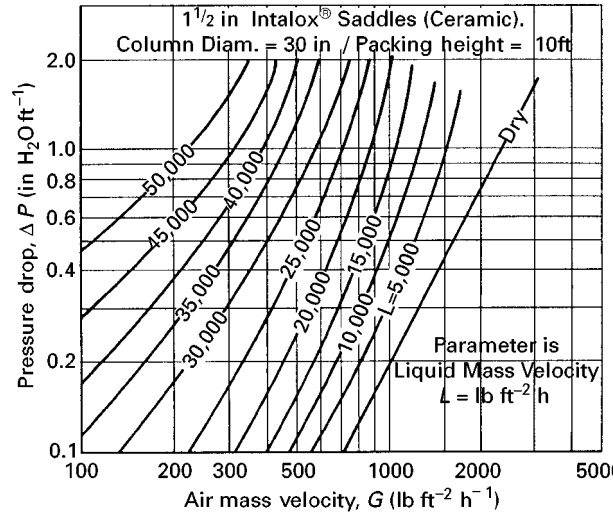

Figure 5 Bed ΔP vs. rates. (Permission from Gulf Publishing Company.)

$L_f = L(62.4/\rho_1)(F_p/20)^{0.5}\mu_l^{0.1}$ (for F_p over 15)

$C_2 = 7.4 \times 10^{-8}$ and $C_3 = 2.7 \times 10^{-5}$

For the case of dry packing $L_f = 0$, the pressure-drop equation reduces to:

$$\Delta P = C_2 G_f^2 = C_2(0.075/20)F_p G^2/\rho_g. \qquad [3]$$

Figure 5 presents a family of pressure drop-lines at constant liquid flow as a function of the vapour flow. The constant liquid rate lines start parallel to the dry-column line (which is a function of the drag only). Equation [3] allows calculation of the packing factor, F_p, by measuring the slope of the dry-packing pressure-drop data. As the vapour rate increases, the slope of the constant liquid rate lines increase; this increase is also proportional to the liquid rate. The initial departure from the dry-line slope indicates interaction between the vapour and liquid, and represents a loading point. Efficient mass-transfer operations can be achieved only above the loading

Table 2 Packing selection guidelines (trays included as a reference)

Application in distillation	*Random packing*	*Structured packing*	*Traditional trays*	*High-capacity trays*
Pressure drop/theoretical stage	2	1	3	3
Maximum capacity[a]	2	1	3	2
Efficiency at high pressure	2	4	2	1
Efficiency at low pressure	2	1	2	3
Efficiency at low liquid rate[c]	2	1	3	4
Efficiency at high liquid rate[d]	3	4	2	1
Low residence time	2	1	4	4
High residence time	3	4	1	1
Heat transfer	2	1	2	2
Foaming systems	2	2	3	3
Non-metallic services[b]	1	2	4	4
Fouling systems	4[f]	2[f]	1[e]	1[e]
Efficiency in high δ systems	2	4	1	1
Inspection and maintenance	3	4	1	1
Low cost	2	4	1	3

Application rating: 1, best; 2, good; 3, fair; 4, poor.
[a]Efficiency may be reduced at high capacities.
[b]As may be required based on corrosion protection considerations, such as ceramic.
[c]Systems below 5 gallons min^{-1} ft^{-2}.
[d]Systems over 15 gallons min^{-1} ft^{-2}.
[e]Applies to sieve trays, specially dual-flow, not to valve trays.
[f]It would require a fouling-resistant distributor, which may result in reduced efficiency.

point. For any given liquid rate, as the vapour rate further increases, the pressure-drop line slope increases rapidly until the line becomes near vertical. At this point the flow and ΔP are unstable, and the bed is flooded; the vapour flow does not allow the liquid to flow down the bed and there is massive entrainment of liquid in the vapour phase and mass transfer is no longer viable.

For most packings, bed flooding occurs between 1 and 2 inches of water-pressure drop per foot of packing. Pressure drop at flooding seems to be a function of the packing size. Kister cited Zenz and later Strigle and Rukovena observations indicating that flooding (ΔP_{fl}) is higher for smaller size packings, and proposed a correlation to determine the pressure drop at flooding as a function of the packing factor.

$$\Delta P_{fl} = 0.115(F_p)^{0.7} \quad [4A]$$

We also obtained by regression from data published by Strigle:

$$\Delta P_{fl} = 0.146F_p^{0.75} \text{ inch liquid ft}^{-1} \text{ or} \quad [4B]$$

$$\Delta P = 0.146S_gF_p^{0.75} \text{ inch } H_2O \text{ ft}^{-1} \quad [5]$$

Pressure drop at incipient loading may be estimated:

$$\Delta P_l = 0.072S_gF_p^{0.75} \quad [6]$$

and pressure drop at maximum efficiency loading may be estimated by:

$$\Delta P_e = 0.082S_gF_p^{0.75} \quad [7]$$

All the above correlations have been regressed for metallic random packings (Pall Rings and IMPT®).

For column design, it is well-accepted practice to assume flooding at 1 inch of water per foot of packing pressure drop and design the packing for an operation at 80% flood. However, when reliable packing-factor information is available, the use of the calculated ΔP_{fl}, using one of the eqns [4A], [4B] and [5], is a more accurate approach.

Caution: Presence of foam, even incipient foam, has a great impact on a packing column pressure drop and performance and should be avoided. Amines, insoluble fine solids (such as corrosion products), high-viscosity organic liquid (0.5–1 cP or higher) and immiscible liquids are known to foam. For these systems, or other systems known to be prone to foam, continuous or intermittent dosing of antifoam agents may be required to maintain an efficient packed-column operation. Nevertheless, uncontrolled antifoam injection is known to aggravate foaming problems. Filtration of liquids and adsorption of contaminants on activated carbon has proven valuable to control foaming in some systems such as amines.

Flooding Correlations

Several generalized flooding and pressure-drop correlations have been proposed for commercial packings. Sherwood, Shipley and Holloway presented the first correlation between a 'flow parameter' X defined as:

$$X = (L/G)\,(\rho_g/\rho_l)^{0.5} \quad [8]$$

and a 'flooding parameter' Y defined as:

$$Y_f = (u_g^2/g_c)(a/\varepsilon^3)(\rho_g/\rho_l)\mu^{0.2} = (G_f^2/g_c)(a/\varepsilon^3)\mu^{0.2}/(\rho_g\rho_l). \quad [9]$$

Sherwood and co-workers correlated dumped and stacked random packing data and found that Y_f is around five times higher for stacked than for dumped packing, which means that mass velocity at flood is over two times higher for stacked packing. This was the precursor idea for the later development of 'structured' packings.

Lobo and Friend presented a similar correlation of Y and X with indication of pressure-drop lines and flooding line.

Leva proposed a similar correlation with the same flow parameter given by eqn [8] and modified the flooding parameter $Y_f = (G_f^2/g_c)(a/\varepsilon^3)\mu^{0.2}\,(\rho_w/\rho_l)^2/\rho_l$. According to this correlation, minimum loading Y_m occurs at about one-third of Y_f which means that loading starts at 50% of the mass flow rates corresponding to the flooding point.

Eckert observed that the packing geometrical properties factor (a/ε^3) did not represent correctly the packing in the flooding correlations. He introduced a packing factor, F_p. The value of F_p is determined experimentally from pressure-drop data. The new flooding parameter became:

$$Y_f = (G_f^2/g_c)F_p\mu^{0.2}(\rho_w/\rho_l)^2/(\rho_g\rho_l) \quad [10]$$

and is correlated to the same flow parameter $X = (L/G)\,(\rho_g/\rho_l)^{0.5}$.

The most recent proposed correlation was presented by Strigle (see **Figure 6**):

$$Y = C_sF_p^{0.5}(\mu/S_g)^{0.05} = C_sF_p^{0.5}v^{0.05} \quad [11A]$$

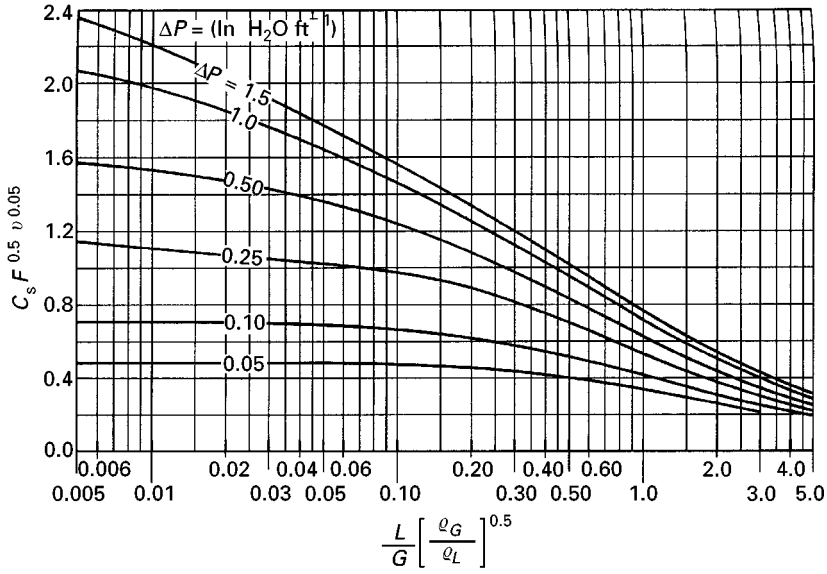

Figure 6 Striegle pressure drop chart. (Permission from Gulf Publishing Company.)

Y is the vapour flow parameter and is a function of vapour capacity factor $C_s = u_g(\rho_g/(\rho_l - \rho_g))^{0.5}$, the packing factor and the kinematic viscosity $v = \mu/S_g$. Note that at flooding $Y = Y_f$. Y is plotted in a linear ordinate as a function of the flow parameter X in a logarithmic abscissa and a family of constant ΔP lines. No flooding line is shown. The advantage of the linear ordinate is that it is easier to interpolate than the older log–log charts.

Figure 7 presents the flooding lines of packings as a function of the packing factor F_p and the flow parameter X. The ordinate is the modified flooding parameter Y_f^*, defined as follows:

$$Y_f^* = Y_f/F_p^{0.5} = C_s v^{0.05} \qquad [11B]$$

Y_f^* is plotted as a function of the flow parameter X, eqn [8], at constant packing factors.

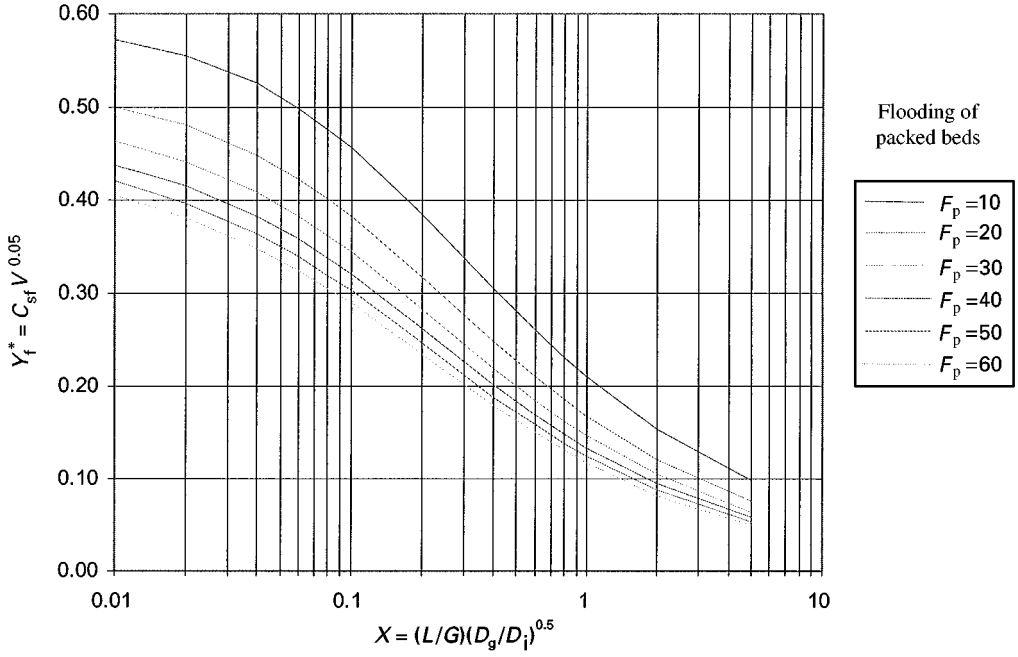

Figure 7 Modified flooding parameter as a function of the flow parameter.

Comparing Packed Column vs. Tray Tower Capacity

Table 5 presents packing capacities, calculated from the above relations, compared to tray flooding capacities at several tray spacings.

Packed Tower Diameter

Figures 6 or 7 can be used to determine the column diameter. Using Figure 7 the procedure is as follows:

1. Determine the value of the abscissa $X = L/G(\rho_g/\rho_l)^{0.5}$.
2. Obtain from the manufacturer the selected packing F_p value, or from Tables 3 or 4.
3. Determine the ordinate $Y_f^* = C_s v^{0.05}$ from Figure 7.
4. Calculate the capacity factor at flood C_s from the Y_f^* value, the gas velocity at flood $u_g = C_s(\rho_l - \rho_g)^{0.5}/\rho_g$ and the flooding gas mass velocity $G_{fl} = u_g\rho_g$.
5. Determine the column cross-sectional area $A_c = V/(0.8G_{fl})$, based on 80% of the G flooding rate. This is standard design practice for new column sizing, and allows for normal flow fluctuations that occur in actual operations and for process control requirements.
6. Determine the column diameter $D_c = 12(4A_c/\pi)^{0.5}$.

Turndown and Minimum Wetting Flow

In general, the turndown of a packed tower is limited to the turndown of the liquid distributor, which is its ability to reduce liquid load and still maintain a homogeneous distribution. Most standard liquid distributors can operate efficiently at 50% of its design liquid load; turndown as low as 25% can be achieved.

To operate efficiently as mass-transfer devices, packing should be homogeneously wetted to assure use of the total surface. Minimum recommended values of liquid irrigation depend on the packing material and surface wettability, as follows:

Random packing

Ceramic	0.2 gallons min^{-1} ft^{-2}
Surface-treated or rusted metals	0.5 gallons min^{-1} ft^{-2}
Glass, glassed ceramic and stainless steel	1.0 gallons min^{-1} ft^{-2}
Plastics	1.5–2.9 gallons min^{-1} ft^{-2}

Table 3 Random packing design parameters

Packing metal	*Nominal size*	*Packing factor (F_p)*	*Specific surface ft^2 ft^{-3} (a)*	*Void ft^3 ft^{-3} (ε)*	*Bulk density (lb ft^{-3})*
Pall Rings	0.625	81	103	0.918	39.9
	1	56	61	0.953	23.1
	1.5	40	39	0.971	14.3
	2	27	30	0.969	14.1
	3.5	18	18	0.972	13.9
CMR®	0	60	103	0.957	20.96
	1	38	76	0.968	15.51
	1.5	33	57	0.961	18.66
	2	26	44	0.970	14.29
	2.5	21	38	0.974	12.54
	3	14	32	0.979	10.22
	4	12	23	0.985	7.36
	5	8	15	0.989	5.46
IMTP®	No 15	51	88.7	0.961	17.9
	No 25	41	69.8	0.970	14.1
	No 40	24	46.9	0.969	14.6
	No 50	18	31.2	0.981	9.3
	No 60	16	25.3	0.982	8.7
	No 70	12	17.5	0.984	8.1
Nutter Rings®	0.7	N/A	69	0.978	11.0
	1.0	30	51	0.978	11.1
	1.5	24	38	0.978	11.3
	2.0	18	29	0.979	10.8
	2.5	16	25	0.982	9.0
	3.5	13	20	0.984	8.3

Table 4 Structured packing design parameters

Packing 45° Crimp angle	*Size*	*Packing factor*	*Specific surface $ft^2\ ft^{-3}$ (a)*	*Void fraction (ε)*	*Bulk density ($lb\ ft^{-3}$)*
Mellapack® (Sulzer)	125Y	10	35	0.989	5.09
	250Y	20	78	0.987	5.61
	350Y	23	107	0.983	7.8
	500Y	34	155	0.975	10.92
Sulzer BX (Gauze)	BX	21	150		
Gempack® (Koch Glitsch)	4A	55	138.1	0.942	17
	3A	23	91.4	0.962	9.9
	2A	15	67	0.972	6.3
	1A	9	35	0.977	4.7
Intalox® (Norton)	1T	28.0	95.2	0.980	10.14
	2T	20.0	65.3	0.984	8.23
	3T	15.0	51.9	0.987	6.55
	4T	13.5	40.6	0.986	6.75
	5T	12.0	27.0	0.991	4.5
Montz	B1-100		30		
	B1-200	20	61	0.94	
	B1-250		76		
	B1-300	33	91		

Structure packings

Surface-treated metals	0.2 gallons $min^{-1}\ ft^{-2}$
Plain surface metals	0.5 gallons $min^{-1}\ ft^{-2}$

Type of Liquid Distributors

Liquid distributors can be gravity or pressure fed depending on how the liquid is introduced to the distributor. Pressure distributors are limited to heat transfer and some simple mass-transfer operations, mainly in stripping or absorption. For distillation, especially for high-purity separations, only gravity distributions are used. **Table 6** illustrates the main type of distributors and the main factors to be considered for selection:

- *Pipe orifice headers* (*POH*) (**Figure 8**) consist of a pipe ladder arrangement with calibrated orifices drilled in the pipe laterals in a uniform layout. POH can be pressure or gravity fed.
- *Pan distributors* (*PAN*) (**Figure 9**) consist of a flat horizontal plate (tray) with uniformly spaced calibrated orifices that allow the passage of liquid to the packing below. Round or rectangular risers (chimneys), located within the orifice pattern, distribute the vapour to the packing above. The riser layout should be uniform and should not interfere with the uniformity of the orifice layout. PAN distributors are always gravity fed.
- *Narrow trough distributors* (*NTD*) (**Figure 10A** and **10B**). This distributor is composed of a series of narrow (3–4 inches) parallel troughs fed by one or more larger troughs (parting boxes) oriented at 90° from the narrow troughs. The narrow troughs distribute the liquid to the packing below, through calibrated orifices drilled at the bottom or at the wall. NTD are always fed by gravity.
- *Spray nozzle header* (*SNH*) (**Figure 11**). They are similar to POH but spray nozzles are used instead of orifices. The density of nozzles in the SNH is lower than the density of orifices in the POH. The SNH relies on the liquid cone leaving the nozzle for

Table 5 Relative capacity of packing and trays[a]

Tray spacing	*Ratio of packing to tray capacity according to packing factor (F_p)*					
	10	*20*	*30*	*40*	*50*	*60*
36 inches	1.15	0.96	0.87	0.81	0.76	0.73
24 inches	1.45	1.22	1.10	1.03	0.97	0.93
18 inches	1.90	1.60	1.44	1.35	1.27	1.22
12 inches	2.41	2.03	1.84	1.71	1.62	1.55

[a]Tray capacity based on the column full cross-sectional area, without discounting any area for downcomers (which implies high-capacity trays). For conventional trays the ratio of packing capacity/tray capacity will be higher. Tray capacity taken from the generalized correlation of tray flooding proposed by Fair JR and Matthews RL (*Petroleum Refiner* 37(4): 153). The packing capacity taken from the generalized correlations presented by RF Striegle Jr and Figure 6).

Table 6 Guidelines for distributor selection

	Gravity-fed distributors			*Pressure-fed distributors*	
	POH	*PAN*	*NTD*	*POH*	*SNH*
Uniformity	1	1	1	2	3
High-purity fractionation	1	1	1	3	3
Maximum drip points per area	2	1	1	2	2
For large diameter towers (over 10 ft)	1	3	1	1	1
Leakage potential	C	H[a]	C	C	C
For high liquid rates	2	1	2	2	1
For high vapour rates	1	3	1	1	1
Residence time	C	A	B	C	C
Solids handling	3	3	2[b]	2	1
Turndown	1	1	1	1	3
Easy installation and levelling	1	3	2	1	1
Cost	B	A	A	C	C

1, Good; 2, fair; 3, poor; A, high; B, medium; C, low.
[a] Unless it is seal-welded.
[b] Very good if a V-notch is provided at the top of the trough wall for liquid flow. Nevertheless, the quality and turndown of the distributor are affected.

further spreading. This results in either an overlap or a gap of the cone projection over the packed bed, and deteriorates the uniformity of the distribution. SNHs can handle very large liquid rates and are very efficient for heat transfer.

Liquid Mixing, Redistribution and Maximum Bed Height

Initial liquid distribution is essential to achieve good packed tower efficiency. Hoek suggested that at a given flow rate, each packing has its natural distribution determined by its radial spreading coefficient. Although this effect does spread the initial liquid distribution, this effect is not sufficient to correct poor initial distribution. Radial concentration gradients already established at the top of the bed cannot be compensated by additional packing. The result is permanent efficiency loss.

In general, if a good distribution is established at the top of the bed, the packing will develop its natural distribution and maintain it for bed depths of 10 NTS or more. Columns requiring more than 10 NTS per section should be subdivided into several packing beds to maintain coefficient HETP values. Liquid redistribution, and often mixing, are required between these bed sections.

Distributor Design Parameters

Distributor Liquid Level and Hole Diameter

The basic distributor design equation relates the total orifice open area, the liquid head and the volumetric flow:

$$Q = C_o n a_0 (h - h_d)^{0.5} \qquad [12]$$

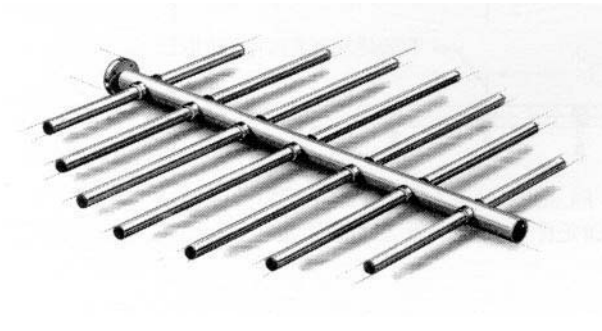

Figure 8 POH distributor. (Courtesy of Norton Chemical Process Products Corp.)

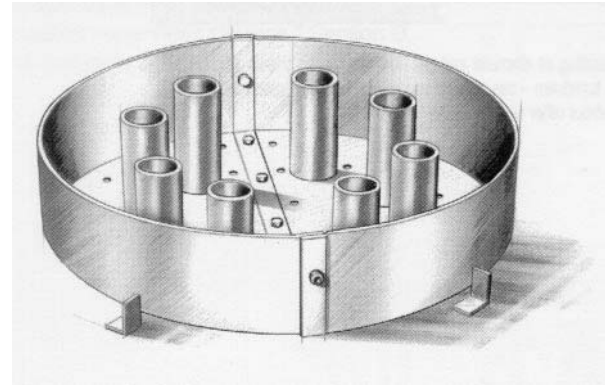

Figure 9 PAN distributor.

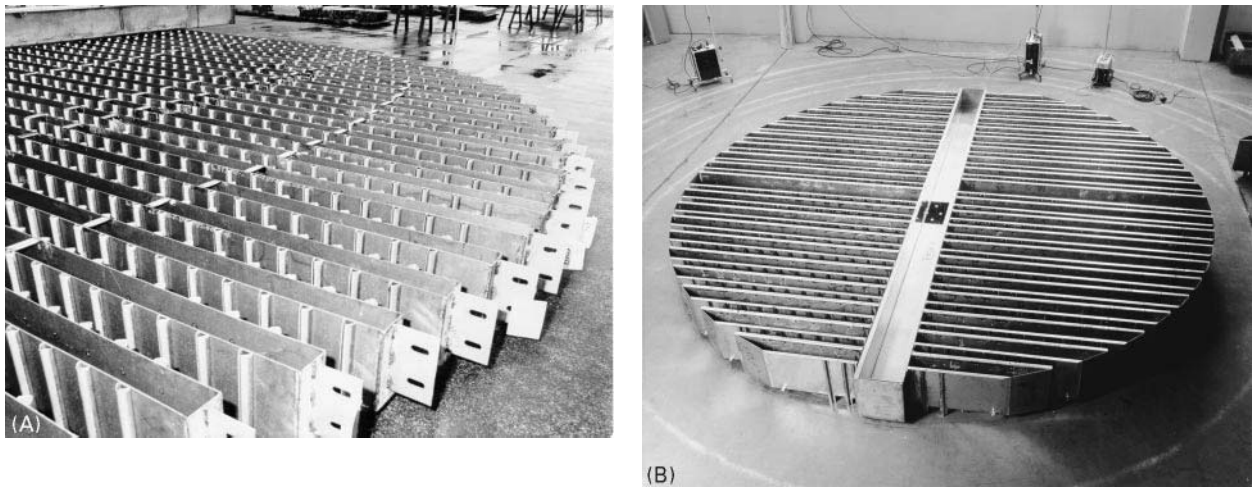

Figure 10 NTD distributor: (A) Photo courtesy of Norton Chemical Process Products Corp. (B) Photo courtesy of Koch–Glitsch Inc.

where Q is the volumetric flow rate, C_o the orifice flow coefficient, n the number of orifices, a_0 the open area of one orifice, h the liquid head over the orifice, and h_d the vapour-pressure drop across the distributor given in head of liquid. The value of C_o varies between 0.5 to 0.8 and is near 0.6 for most commercial distributors. Using this value, eqn [12] becomes:

$$Q = 4.0nd^2(h - h_d)^{0.5}$$

and:

$$n = 0.25Q/d^2(h - h_d)^{0.5} \qquad [13]$$

The minimum recommended orifice diameter, to prevent plugging, is 3/8 inch for carbon steel and 1/8 inch for stainless steel. The minimum recommended liquid level at minimum flow is 2 inches. If a 50% turndown is specified, the required liquid level at normal liquid load becomes 8 inches.

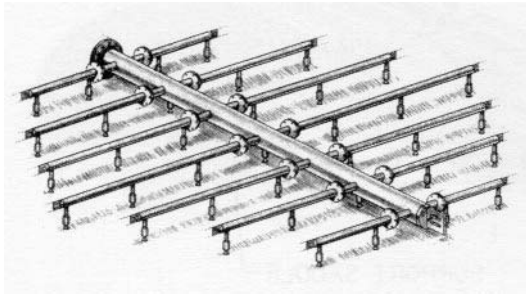

Figure 11 SNH distributor. (Courtesy of Norton Chemical Process Products Corp.)

Uniformity of the Drip Point Layout

Density of liquid drip points is not enough to assure a good distributor quality. The distribution must be homogeneous; the same amount of liquid should irrigate the packing at any fraction of the tower cross-sectional area. Areas near the tower wall should receive the same amount of liquid as areas near the centre.

Other Considerations

A number of factors need to be considered when selecting and designing packing and distributors.

Ratio Tower to Packing Size

The minimum recommended ratio of the tower diameter to the packing size is 8. In the case of structured packings, this ratio applies to the ratio of the tower diameter to the crimp size.

Fouling

Some solids are usually present even in 'clean systems' because of corrosion products, especially after maintenance shutdowns, when rust and debris can remain in the tower. The tower shell metallurgy should be adequate to prevent formation of scale or corrosion products that can plug distributors. Distributors with small orifices should be protected with filters in all liquid lines entering the tower. In other cases, solids are expected to be present because of the process itself. In these cases the distributor should be designed to handle the solids. A NTD distributor with V-notches for liquid overflow is adequate to handle

some slurries. SNHs can also handle slurries but their application is limited to heat transfer.

Vapour Distribution Requirements

Vapours entering the tower have a kinetic energy proportional to their velocity, which is converted into pressure as the vapour turns to start flowing upward in the tower. The resulting radial pressure profile is not uniform; areas of higher pressure would allow higher vapour up-flow. This is especially critical for low-pressure drop packings such as structured packings. Vapour radial velocity profiles are corrected by pressure drop and by diffusion devices. The following is the recommended practice for vapour distribution:

- Low vapour inlet velocity (velocity head below 0.5 inches of water): no inlet distributor required, provide as minimum $1\frac{1}{2}$ column diameters, or 36 inches between the top of the vapour inlet nozzle and the bottom of the bed.
- Intermediate vapour inlet velocity (velocity head between 0.5 and 1.5 inches of water): provide an inlet vapour diffuser directing vapour flow down the tower. This type of device can be a horizontal pipe with the bottom half cut as shown at the bottom of the column in **Figure 2**. Vertical baffles can be provided for better vapour distribution. The purpose of these baffles is to stop the horizontal velocity component of the vapour.
- High vapour velocity (velocity heads above 1.5 inches of water): provide an inlet vapour diffuser, as described above, plus a small riser chimney tray with a pressure drop of a minimum of 2 inches of water. The pressure drop can be created by orifices at the bottom of the risers. A vapour distributor, as shown in **Figure 12** is a good alternative to the vapour diffuser in critical systems.

Distributor Testing

Water test of assembled distributors at the manufacturer's workshop is always a good practice for all high-efficiency distributors. The test should determine liquid rate gradients under the distributor, liquid level in the distributor itself at design and turndown liquid rate, liquid level gradient in the trough and uniformity of the drip point layout. These parameters should be compared to the distributor design parameters and adjustments made to the distributor if necessary. **Figure 13** shows a distributor testing facility.

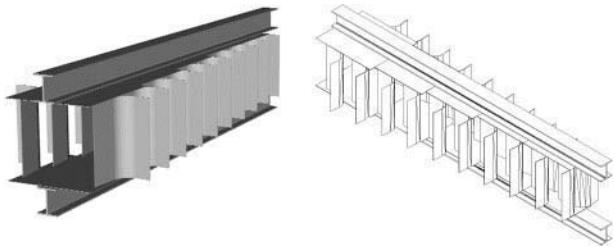

Figure 12 Vapour distributor. (Courtesy of Sulzer Chemtech.)

Packing Performance in Distillation

Factors to Consider in Determining the Column Design HETP

The height equivalent to a theoretical plate (HETP) is determined by the following main three factors:

Intrinsic geometric shape and size of the packing This factor determines the surface per unit of volume, and the packing capacity of establishing effective vapour liquid interfacial surface. It is a well-known fact that, for any packing, the smaller its particle size, the larger its surface : volume ratio, and the lower the HETP value. All the other factors being equal, numerous available data tend to indicate that the expected reference packing HETP_o may be correlated as follows:

$$\text{HETP}_o = K_p / F_p^f \qquad [14]$$

where F_p is the packing factor. The constants K_p and f for different types of commercial packing are correlated as in **Table 7** for a reference system.

Figure 13 Distributor testing facilities. (Photo courtesy of Koch–Glitsch Inc.)

Table 7 HETP correlation factors for a reference system, regressed by the authors for eqn [14]

Packing	*Constant K_p*	*Exponent f*
Structured packings		
Sulzer Mellapak®	126	0.73
Koch Flexipac®	100	0.69
Koch–Glitsch Gempak®	120	0.76
Average of above structured packings	106	0.70
Random metallic packings		
Koch–Glitsch CMR®	73	0.43
Norton IMPT®	198	0.69
Pall Rings	250	0.69
Average above random packings	110	0.50

System properties Numerous investigations have tried to correlate experimental HETP data with the distillation system fluid physical properties. The best and most consistent correlations tend to confirm that the HETP is proportional to reference $HETP_o$ and a factor proportional to the system physical properties:

$$HETP = HETP_o(\mu\alpha/S_g\delta)^n/(\mu\alpha/S_g\delta)_0^n \qquad [15]$$

when the n exponent best fit is between 0.15 and 0.21. Replacing $HETP_o$ from eqn [14] into eqn [15], and using the reference system we obtain:

$$HETP = (2.0K_p/F_p^f)\,(\mu\alpha/S_g\delta)^{0.2} \qquad [16]$$

In addition, theoretical considerations suggest that the HETP is related to $\lambda = m/(L/V)$, the ratio of the slopes of the equilibrium line and operating line, by the correlation:

$$HETP = \lambda \ln(\lambda)/(\lambda - 1)HTU$$

where HTU is the height of a transfer unit. Then:

$$HETP = (2.0K_p/F_p^f)\,(\mu\alpha/S_g\delta)^{0.2}\, f(\lambda) \qquad [17]$$

Packing loading **Figure 14** shows the pilot plant performance of Sulzer/Nutter ring No. 2.5 in

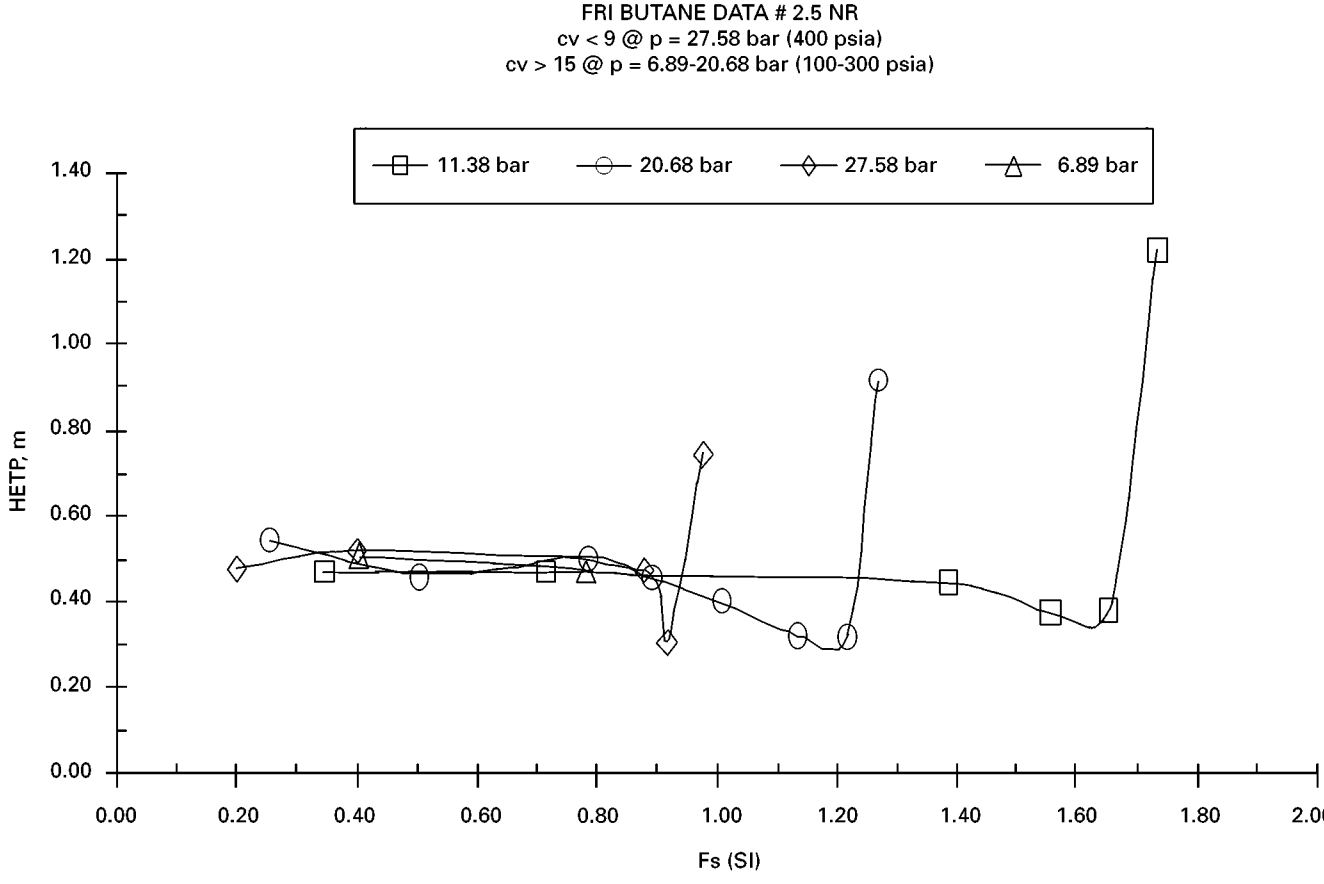

Figure 14 HETP vs. loads. (Courtesy of Sulzer Chemtech.)

isobutane–n-butane separation. Note that although only the vapour rate appears in the abscissa, actually both the vapour and the liquid rates increase in the same proportion since the chart was developed at total reflux. All packings present similar curves in small size experimental columns. The initial HETP is high (low efficiency) owing to the low loads that result in liquid maldistribution, poor packing wetting and little interaction between the vapour and the liquid (this left section of the curve is not shown in Figure 14). Nevertheless, the HETP continuously decreases as the loads increase. At a point, corresponding to the loading point of the packing, the HETP becomes constant over a range of loads. This range represents the operating range of the packing. As the loads continue to increase, the HETP shows a dip corresponding to high interaction between the fluids, followed by a rapid increase in the HETP caused by recirculation of liquid within the bed. This corresponds to the initial flooding of the bed.

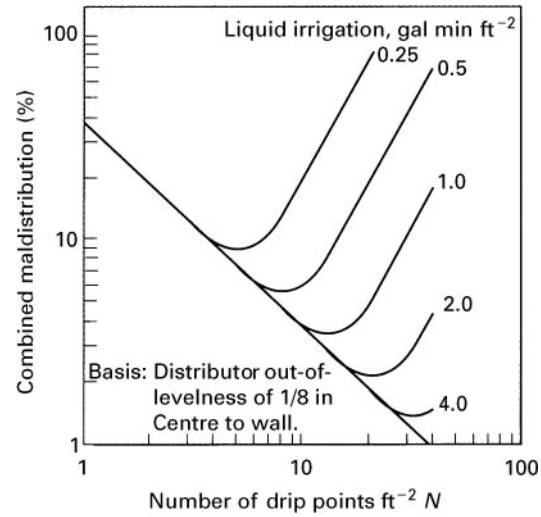

Figure 15 Effect of number of drip points and liquid irrigation rate on maldistribution. (Permission from Chemical Engineering Progress.)

Maldistribution

Liquid maldistribution has a very large effect on column distillation performance. Liquid maldistribution is originated by uneven liquid flow from the distributor to the top section of the packing. Some degree of maldistribution cannot be avoided and it is related to the following factors.

Drip points density (total drip points/column cross-section area) In principle, a smaller number of drip points equates to a higher initial maldistribution. This could be solved by constructing distributors with a high number of drip points. However, there are physical and mechanical limits that make it difficult to build distributors with more than 20 drip points ft^{-2}. It has also been demonstrated that if the distributor deck is not levelled, the resulting maldistribution effect may increase as the number of drip points is increased above an optimal number. The optimal number of drip points is related to the liquid irrigation flow as follows (**Figure 15**):

Liquid irrigation $g \cdot m^{-1} \cdot ft^{-2}$	0.25	0.5	1.0	2.0	4.0
Optimum number drip points per square foot	5	8	13	21	32

Furthermore, the drip points themselves may create additional maldistribution if they are not evenly distributed across the entire column cross-sectional area. Poor construction making holes of variable diameters or unlevelled installation of the distributor will also induce additional maldistribution. Operational problems such as plugging of the distributor deck areas will cause large sectors to be dry, thus producing a macroscopic or sectorial maldistribution.

Maldistribution and spreading factor Initial maldistribution produces a condition of uneven liquid/vapour flow ratio across the column cross-sectional area. Some areas or spots are underirrigated and some are overirrigated. The column packing does spread the liquid resulting in some correction or attenuation of the initial maldistribution. The overall weighted maldistribution is attenuated better in small diameter columns than in larger columns. This is determined by the nondimensional number (Z_b/CD_c^2), where Z_b is the bed height in feet, D_c the column diameter in inches, and C is the spreading factor in ft $\cdot$ in^{-2} units (see **Figure 16**). The spreading factor is related to the packing particle size and the liquid irrigation.

The lost column efficiency is proportional to the liquid maldistribution, and this effect is amplified by the number of theoretical stages required to achieve the separation. **Figure 17** presents a useful correlation for the calculation of the column efficiency in packed distillation columns.

Liquid distributor quality The liquid distributor intrinsic maldistribution, M_d (related to its design and manufacture) should be measured at the factory by a water test measuring the liquid flow under each subsection of the column cross-section. The smaller and more numerous the test area subdivisions, the more precise will be the maldistribution

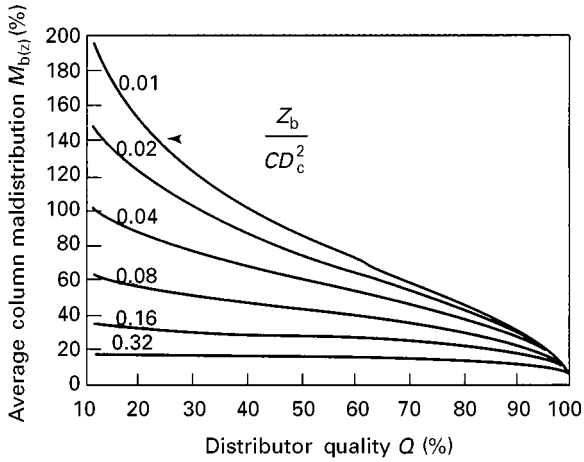

Figure 16 M vs. Z_b/CD_c^2. (Permission from Chemical Engineering Progress.)

measurement. The mathematical expression of the maldistribution is:

$$M_d = 100[\Sigma((L_i/L_{av} - 1)^2)/n]^{0.5} \quad [18]$$

for each point area subdivision from $i = 1$ to $i = n$. The following is the correlation between distributor quality and its maldistribution:

$$Q_d\,(\%) = 100/[1 + (M_d/100)^2] \quad [19]$$

If the distributor quality is 90%, the actual measured maldistribution should not exceed 33%. A 95% quality implied a maximum measured maldistribution of 23%.

Total maldistribution Additional maldistribution can originate from operational factors related to levelness and obstructions. The total initial maldistribution, M_o, can be calculated by:

$$M_o = (\Sigma M^2)^{0.5} \quad [20]$$

Assuming a maximum operational maldistribution $M_{op} = 15\%$, and using a 90% distributor quality ($M_d = 33\%$), the total effective operating maldistribution at the top of the packing is $M_o = (33^2 + 15^2)^{0.5} = 36.2$.

The effective bed attenuated maldistribution is calculated by the following equation:

$$M_{bz} = M_o/[1 + 0.16M_o(Z/CD_c^2)] \quad [21]$$

With this M_{bz} value, the bed efficiency E_z may be obtained from Figure 17. The calculated bed efficiency should be used to correct the packing HETP and obtain the bed operating $HETP_{op}$:

$$HETP_{op} = HETP\ E_z/100 \quad [22]$$

$$HETP_{op} = [(2.0\ K_p/F_p^f)(\mu\alpha/S_g\delta)^{0.2}\, f(\lambda)]E_z/100. \quad [23]$$

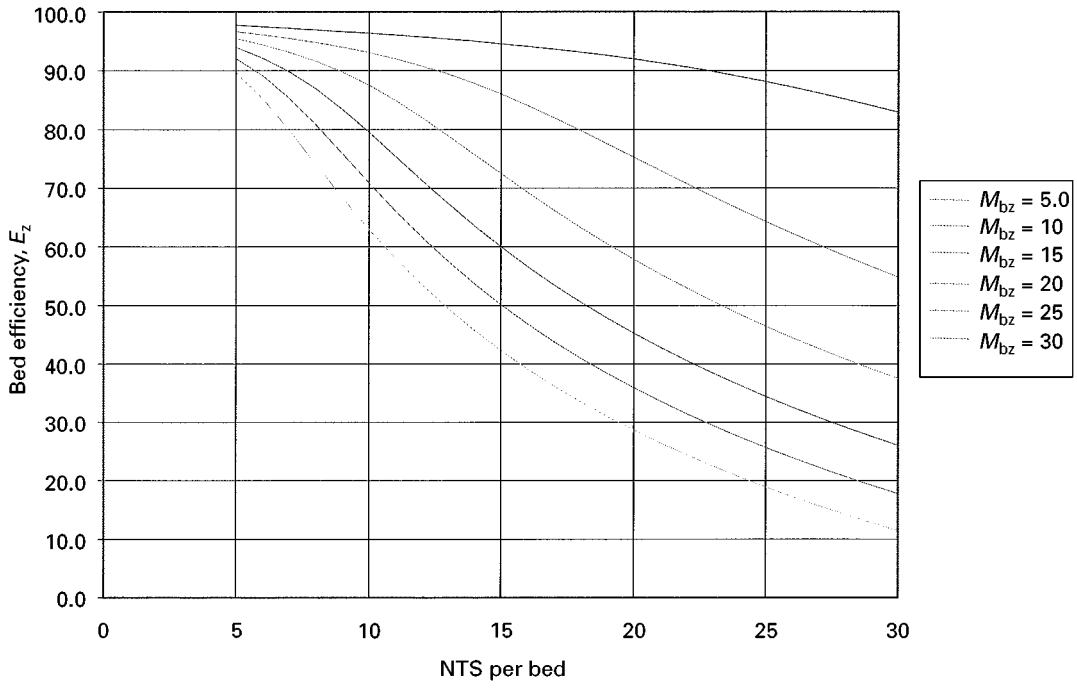

Figure 17 Efficiency vs. NTS.

The bed effective number of theoretical stages can be calculated by:

$$\mathrm{NTS} = Z/\mathrm{HETP}_{op}. \quad [24]$$

For a column requiring more than 10 NTS, it is in general advantageous to subdivide the packing in two or more beds and limit the NTS per bed to around 10. The lower the NTS per bed, the higher the resulting bed efficiency. The limiting factor of subdividing the column into a large number of redistributed beds, is the extra column height (or the effective packed height loss for column revamps) necessary to accommodate each redistributor, and the resulting increased cost. For new columns, the optimal number of beds is the one that results in the required performance at minimum cost, for revamps, it is often the one that results in the maximum available overall NTS. The best choice in each case is determined by an optimization.

Future Developments

With the ability to accurately design and predict the performance of packings in distillation, it is expected that the use of packings in distillation will become better accepted, not only for plant revamps but also for grass roots applications. The design and evaluation of liquid distributors needs to be better understood by users and equipment manufacturers; standard methods for distributor quality rating should be implemented based on the basic concepts presented in this contribution. Readers interested in further exploring the column design methods outlined in this article may download a free demo of BDSIM at url http://www.geocities.com/ ~ combusem/BDSIM.HTM

Nomenclature

A_c	Column cross-sectional area	ft^2
ΔP	Packing pressure drop	in ft^{-1}
C	Packing spreading factor	ft in^{-2}
C_o	Orifice flow coefficient	
C_1, C_2, C_3	Constants in pressure drop correlations	
d	Orifice diameter	in
D_c	Column diameter	in
E_z	Bed efficiency	%
C_s	Vapour capacity factor, defined by $C_s = u_g(\rho_g/(\rho_l - \rho_g))^{0.5}$	ft s^{-1}
ρ_g	Gas density	lb f^{-3}
ρ_l	Liquid density	lb f^{-3}
u_g	Vapour velocity	ft s^{-1}
h	Liquid head over distributor orifice	inches of liquid
h_d	Vapour pressure drop across liquid distributor	inches of liquid
G	Vapour flow mass velocity	lb ft^{-2} h^{-1}
G_f	Vapour flow mass velocity (in Robbins equation)	lb ft^{-2}
V	Vapour flow	lb h^{-1}
L	Liquid mass flow	lb ft^{-2} h^{-1}
L_f	Liquid mass flow	lb ft^{-2} h^{-1}
L_i	Liquid mass flow at point i	gallons min^{-1} ft^{-2}
L_{av}	Liquid average mass flow	gallons min^{-1} ft^{-2}
F_p	Packing factor	
K_p	HETP correlation factor	
n	Number of measured points (in distributor testing)	
HETP	Height equivalent of a theoretical plate	in
HTU	High of a transfer unit	in
μ_l	Liquid viscosity	cP
S_g	Liquid specific gravity	
X	Flow factor = $(L/G)\ (\rho_g/\rho_l)^{0.5}$	
Y	Vapour flow parameter. At flooding $Y = Y_f$	
Y_f, Y_f^*	Flooding parameters, defined by eqns [10] and [11]	
a	Packing surface area	ft^2 ft^{-3}
a_0	Open area of one drip point	
α	Relative volatility	
ε	Void fraction	
δ	Surface tension	dynes cm^{-1}
g_c	Gravitational constant	32.2 ft s^{-2}
NTS	Number of theoretical stages	
R	Reflux ratio	
R_m	Minimum reflux ratio	
M_d	Maldistribution originated by the distributor design	%
M_o	Total initial maldistribution	%
M_{bz}	Effective bed maldistribution	%
Q	Liquid flow	gallons min^{-1}
Q_d	Distributor quality	%
Z_b	Bed height	ft
v	Kinematic viscosity constant = μ_l/S_g	
λ	Ratio of the equilibrium curve slope to the operating line slope	

See also: **I/Distillation:** Historical Development; Modelling and Simulation; Theory of Distillation; Tray Columns: Performance; Tray Columns: Performance; Vapour-Liquid Equilibrium; Correlation and Prediction; Vapour-Liquid Equilibrium: Theory.

Further Reading

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Pilot Plant Batch Distillation

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Introduction

Laboratory distillation encompasses an operating range from millilitres in bench-top devices to pilot units with the capacity for producing several hundred kilograms of product per day. While the design of bench-top assemblies is generally geared towards the achievement of a specified purity grade of the desired product, quantitative predictions are not usually feasible for such equipment and their construction relies a great deal on ingenuity and craftsmanship. For dedicated applications, glassware companies offer off-the-shelf equipment. This article will therefore focus on the pilot-scale units, where the analytical principles of mass and heat transfer can be applied to the operation, design and optimization of the equipment.

The section on theory presents analytical descriptions of batch distillation for three different approaches in order of decreasing complexity. It starts with a comprehensive model for a nonadiabatic, nonzero hold-up, nonconstant molar overflow, nonideal multicomponent column. The second model presented neglects stage hold-ups and assumes adiabatic stages and constant molar overflows to arrive at a set of equations describing the transient behaviour of the equipment, which can be solved for a binary system using a simple spreadsheet. If constant relative volatility and operation at minimum reflux are further assumed, the derivation of a third model is possible, where the transient states within the equipment are given by direct analytical expressions.

The design of a batch column can be a challenging task because batch distillation presents unique considerations that are not addressed in most of the available literature, which is concerned with continuous operation. The section on design is a collection of advice and criteria for the design of batch columns. Specific information is given about equipment for batch distillation and accompanying instrumentation and safety circuitry. Details are drawn from a pilot-scale column that is installed in Penn State University's Department of Chemical Engineering. The section on column operation extends the scope of the two preceding sections by providing information on establishing operating strategies and operating protocols for batch runs. Much of this information is based on hands-on experience acquired with the column described in the subsection on equipment.

The last section is a synopsis of numerical techniques that have been developed in recent years to facilitate the optimization of the operation and design of batch columns. Inherent difficulties associated with the implementation of these numerical techniques into computer codes prevents their widespread use in equipment operation and design. However, it is likely that these techniques will be integrated into commercial simulators in the near future and be readily available to users with little knowledge of programming. The aim here is to introduce the reader to the topic, rather than to offer extensive coverage,