Packed distillation columns

Packed columns are used for distillation, gas absorption and liquid-liquid extraction.

The gas-liquid contact in a packed column is continuous, not stage-wise, as in a plate column. The liquid flows down in the column over a packing surface and the vapor (or the gas) moves counter-currently, up the column. The performance of a packed column is very dependent on the maintenance of good liquid and gas distribution through the packed bed, and this is an important consideration in packed column design.

There are some advantages and disadvantages for the plate and the packed columns.

1. Plate columns can be designed to handle a wider range of liquid and gas flow rates than packed columns.

2. Packed columns are not suitable for very low liquid flow rates.

3. The efficiency of a plate can be predicted more accurately than the equivalent terms of packings (*HETP* or *HTU*).

4. For corrosive liquids a packed column will usually be cheaper than the equivalent plate column.

5. The liquid hold-up is lower in a packed column than in a plate column. This can be important when the hold-up of toxic or flammable liquids must be kept as small as possible for safety reasons.

6. Packed columns are more suitable for handling foaming systems.

7. The pressure drop can be lower in a packed column than the equivalent plate column.

The design procedure of a packed column consists of the following steps:

1. Select the type and size of packing.

2. Determine the column height required for the specified separation.

3. Determine the column diameter (capacity), to handle the liquid and vapor flow rates.

4. Select and design the column internal features: packing support, liquid distributor and redistributors.

The packing types can be divided into two broad classes: structured and random packings. Earlier the random packings were more commonly used in the industry (Raschig-, Pall- and HyPack rings, and Berl- and Intallox saddles).

1. Determination of the column height

1.1. The theoretical stage method

If at a point of the distillation column the concentration of the liquid flow is x, and the concentration of the counter-current vapor flow at the same point is y, then there is a height above this point where the concentration of the vapor flow (y^*) is in equilibrium with x. This section is the Height Equivalent a Theoretical Plate (*HETP*).

The height of the packing is calculated by Eq. 1.

$$H = HETP \cdot n \qquad\qquad (1)$$

where
n	number of theoretical stages (determined by the McCabe-Thiele method)	[-]
$HETP$	Height Equivalent a Theoretical Plate	[m]
H	height of the packing	[m]

The value of the *HETP* can be determined by the modified Granville[1] equation (Eq. 2).

$$HETP = 28 \cdot d_p \cdot m_a \cdot \frac{V}{L} \cdot \left(\frac{H}{2.4}\right)^{1/3} \qquad\qquad (2)$$

where
d_p	diameter of the packing	[m]
m_a	average slope of the equilibrium curve	[-]
V	molar vapor flow rate	[kmol/h]
L	molar liquid flow rate	[kmol/h]

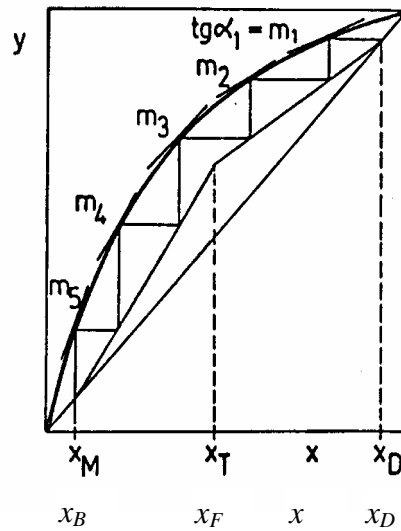The value of m_a can be determined by the following method:

Figure 1. The calculation of m_a

$$m_a = \frac{\sum\limits_{i=1}^{n} m_i}{n} \qquad\qquad (3)$$

where m_i local slope of the equilibrium curve at theoretical plate i [-]

1.2. The transfer unit method

The transfer unit is a part of column height where the change in vapor concentration equals to the average driving force. The driving force is the difference between the equilibrium (y^*) and the actual (y) vapor concentration.

The column height can be computed by Eq. 4.

$$H = HTU \cdot NTU \tag{4}$$

where HTU Height of a Transfer Unit   [m]

   NTU Number of Transfer Units   [-]

NTU can be determined by Eq. 5.

$$NTU = \int_{y_B}^{y_D} \frac{dy}{y^* - y} \tag{5}$$

where y_D vapor concentration at the top of the column   [-]

   y_B vapor concentration at the bottom of the column   [-]

The relation between the equilibrium concentrations (y^*) and the actual concentrations (y) is shown in Figure 2. NTU can be determined by graphical integration, as it can be seen in Figure 3.
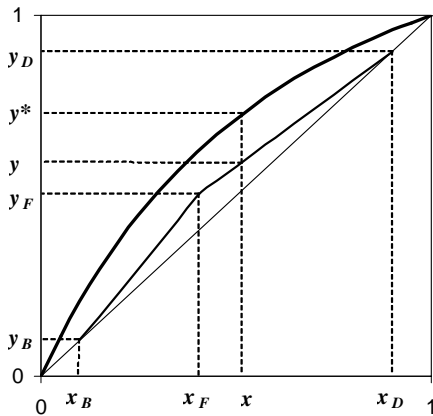
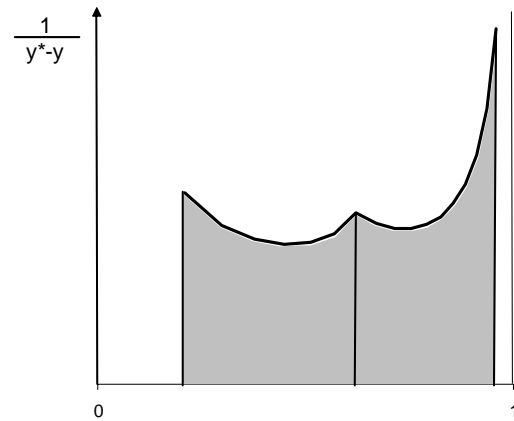Figure 2. Relation between y^* and y · · · · · Figure 3. Graphical integration

There is no entirely satisfactory method for predicting the height of a transfer unit. In practice the value for a particular packing will depend not only on the physical properties and flow rates of the vapor and liquid, but also on the uniformity of the liquid distribution throughout the column, which depends on the height and diameter of the column. Experimental values for several systems are given by Eckert[2] and Cornell et al.[3.]

Kasatkin et al.[4] published useful correlations for the mass-transfer coefficients K_V which can be used to calculate H. First, the equivalent diameter of the packing (d_e) must be calculated:

$$d_e = 4\frac{\varepsilon}{\omega}$$ (6)

where ε fractional voidage   [m^3 free volume / m^3 column volume]

    ω interfacial surface per unit volume of column   [m^2/m^3]

After then the Archimedes (Ar) and the Reynolds (Re) numbers can be determined:

$$Ar = \frac{d_e^3(\rho_L - \rho_V)\rho_V g}{\eta_V^2}$$ (7)

$$Re = 0.15 \cdot Ar^{0.57}\left(\frac{V}{L}\right)^{0.43}$$ (8)

where ρ_L liquid density   [kg/m^3]

    ρ_V vapor density   [kg/m^3]

    η_V dynamic viscosity of vapor   [Pa s]

The linear vapor velocity (v, [m/s]) can be calculated:

$$v = \frac{Re\,\eta_V}{d_e\,\rho_V}$$ (9)

After that the mass transfer coefficient (K_V, [1/s]):

$$K_V = \frac{0.35Re^{0.8}\left(\dfrac{\eta_V}{\rho_V D_V}\right)^{0.35} D_V}{d_e^2}$$ (10)

where D_V diffusivity of vapor   [m/s]

The Height of the Transfer Unit is given by:

$$HTU = \frac{v}{K_V}$$ (11)

2. Column diameter (capacity)

The capacity of a packed column is determined by its cross-sectional area:

$$\Phi = \sqrt{\frac{3 \cdot W_V}{\pi \cdot v}} \tag{12}$$

where Φ column diameter   [m]

W_V volumetric flow rate of vapor   [m^3/s]

v linear vapor velocity   [m/s]

The volumetric flow rate can be determined from the material balance:

$$F = D + B \tag{13}$$

$$Fx_F = Dx_D + Bx_B \tag{14}$$

where F molar flow rate of the feed   [kmol/s]

D molar flow rate of the distillate   [kmol/s]

B molar flow rate of the bottom product   [kmol/s]

If the distillate flow rate is known, the vapor flow rate can be calculated.

$$V = (R+1)D \tag{15}$$

where R reflux ratio   [-]

Assuming ideal vapor phase, from the value of V, the volumetric flow rate W_V can be calculated by the gas law. The actual vapor velocity can be taken to 60% of the flooding velocity, which can be computed from the following equation[5,6]:

$$\log \frac{v_0^2 \omega \rho_V \eta_L^{0.16}}{g \varepsilon^3 (\rho_L - \rho_V)} = -0.125 - 1.75 \left(\frac{L}{V}\right)^{0.25} \left(\frac{\rho_V}{\rho_L - \rho_V}\right)^{0.125} \tag{16}$$

where v_0 flooding velocity of the vapor   [m/s]

g gravitational constant   [m/s^2]

3. Instructions for the experiments

The target is to determine the separation ability and capacity of a column, packed with 5 mm glass Raschig-rings, and of a column packed with Sulzer EX structured packing. The parts of the equipment are: heating plate with manageable power, boiling flask with sampler, packed column, column head with thermometer and device for measuring volume, deflegmator. The equipment can be seen in Figure 4.
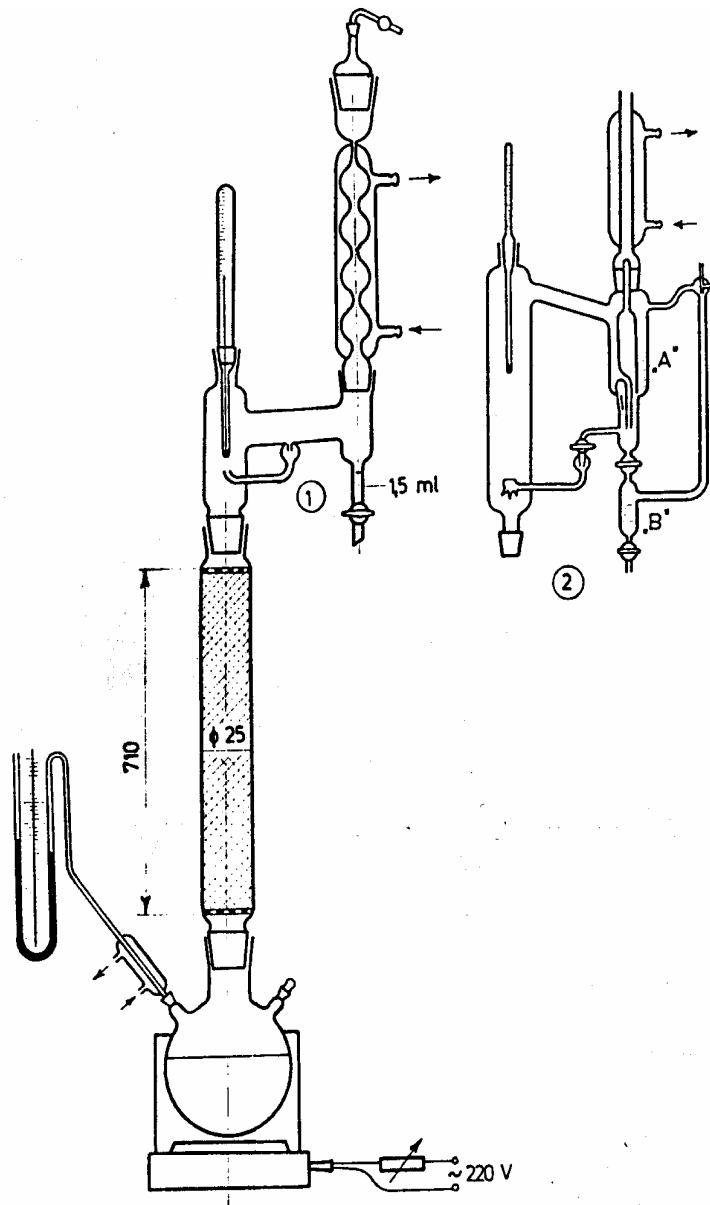
Figure 4. Packed distillation column

The system contains methylcyclohexane and *n*-heptane.

First, open the cooling water, and then turn on the heating of the boiling flask. If the vapor reaches the top of the column, wait about 30 minutes operating with total reflux until the quasi-steady state. Then, take a sample from the bottom and from the top. Determine the concentration of the samples by refractometer. Measure the time of the flow of 1 ml reflux. During the experiment, the following data have to be noted:

temperature at the bottom   °C

temperature at the top   °C

refraction coefficient of the bottom sample

refraction coefficient of the top sample

temperature of the oil bath   °C

flow time of 1 ml distillate   s

mole fraction of the bottom sample

mole fraction of the top sample

Data of the column containing Raschig-rings:

column height:	0.71 m
column diameter:	25 mm
diameter of the packing:	5 mm
interfacial surface per unit volume of column:	1000 m^2/m^3
fractional voidage:	0.62 m^3/m^3

Data of the column containing Sulzer EX packing:

column height:	0.5 m
column diameter:	25 mm

4. Calculation tasks

The following calculations should be performed.

4.1. Column containing Raschig-rings

1. Calculation of *HETP* based on the measured data using a computer program.

2. Calculation of *HETP* using the Granville equation (Eq. 2).

3. Calculation of the linear vapor velocity.

4. Calculation of the flooding velocity of the vapor and the optimal linear velocity of the vapor using a computer program.

5. Calculation of *NTU* and *HTU* using a computer program.

6. Calculation of *HTU*, based on the column height, and on the previously calculated *NTU* (Eq. 4).

4.2. Column containing Sulzer EX packing

1. Calculation of *HETP* using Eq. 1.

2. Calculation of the linear vapor velocity.