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RECTIFICATION COLUMN: CRITERIA OF EFFICIENCY

KRYTERIA OCENY EFEKTYWNOŚCI DZIAŁANIA KOLUMN REKTYFIKACYJNYCH

Abstract

In the paper analysis of the factors influencing the efficiency of the rectification process is provided. The authors show that existing dependences, equivalent to HETP, cannot be used for calculating rectification plate columns and columns with vortex nozzles, in which the coefficients of mass transfer are substantially dependent on the irrigation density and the gas/steam velocity. The dependence of the efficiency of the packet vortex nozzle as a function of the hydrodynamic regimes of the vapour and liquid phases is proposed.

Keywords: HETP, efficiency, distillation column, rectification, packing

Streszczenie

W pracy przedstawiono analizę czynników efektywności procesu rektyfikacji. Autorzy wykazali, że istniejące zależności oparte o WRPT nie mogą być stosowane do kolumn półkowych i z dyszami wirowymi gdzie współczynniki transportu masy są powiązane z wielością zraszania i prędkością gazu/pary. Zaproponowano zależność efektywności dla dysz wirowych jako funkcji parametrów hydrodynamicznych pary i cieczy.

Słowa kluczowe: WRPT, efektywność, kolumny destylacyjne, rektyfikacja wypełnienia

DOI:

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1. Introduction

The efficiency of liquid mixture separation, which defines both the quality of the distillate and the size of the column (height), is the most important characteristic for any rectification column. If the diameter of the column is determined through the optimal vapour velocity in the upper and bottom parts of its column, which mainly depends only on the structural design of auxiliaries, then the height (as the most difficult parameter to define) depends on both the heat- and mass-transfer rate on each tray (or height of transfer unit in case of packed column) and the distribution of temperatures along the column length. The heat- and mass-transfer rate on a tray or on the packing is associated with optimal vapour velocity on different column heights, which is usually determined by means of experiments for each type of the mass-transfer devices.

Numerous simulation models exist for batch distillation, but many of these employ theoretical stage models. Some of these models consider tray efficiency, but only as a tuning parameter, obtained by the trial and error method, for matching simulation results with actual experimental data.

The quality of separation of both binary and multicomponent liquid mixtures depends on many factors: the difference between boiling temperatures of the components and the presence of the azeotropic mixtures; reflux flow rates; distribution of temperatures along the column length; structural design of the auxiliaries, which influence not only the quality of separation, but also the column height.

1.1. Factors affecting tray efficiency

The factors affecting the efficiency of a tray include: mechanical design factors (tray type and size, hole size, weir height), operation conditions (liquid and vapour rates) and the characteristics of the mixture on the tray. Some of these factors, which have been considered [1, 2, 3], include outlet weir height, hole size, liquid mixing, entrainment, flow regimes, reflux ratio, composition and surface tension of the components of the mixture.

1.1.1. Hole Size and Outlet Weir Height

The effect of the hole size in sieve trays and weir height (in other trays) on the tray efficiency is usually associated with its holdup characteristics. An investigation [3] of the hole size influence in sieve trays on the tray efficiency showed that smaller holes exhibited higher efficiencies at low vapour rates, but at higher vapour rates, the hole size has not shown any effect. It was suggested that smaller holes at low vapour rates prevented liquid downfall due to the capillary surface tension effects, thus increasing the tray liquid holdup and efficiencies. Smaller flows are issued from smaller holes thus increasing the mass-transfer process.

On the contrary, outlet weir height is used to maintain an appropriate liquid depth (holdup) on the tray and, as it was expected, tray efficiency increases with increasing outlet weir height. Deeper liquid levels on the tray mean that the residence time and mass transfer time of the vapour bubble through the liquid is increased.

1.1.2. Reflux ratio and tray holdup

While studying the reflux ratio effect, Pigford [4] showed that in the presence of appropriate holdup, the effect of the reflux ratio on the sharpness of the separation was less

pronounced than in a column with negligible holdup. Langdon and Keyes [1] however, concluded on the basis of the results obtained from experimental data, using isopropyl-water mixture that changes in reflux ratio had a negligible effect on the tray efficiency. Other researchers [5] have found plate efficiency to vary appreciably with reflux ratio. Ellis and Hardwick's results were based on the results obtained from the distillation of a methylcyclohexane/toluene mixture, but their conclusions did not take into account the effect of concentration on the tray efficiency. The effect of holdup on actual tray efficiency is not reported, but its effect on the sharpness of separation gives an indication to its effect on the distillation operation.

1.1.3. Liquid viscosity

Barker and Choudhury [6] studied the effect of liquid viscosity on the mass transfer and plate efficiency. It was found that with increasing liquid viscosity a reduction in the interfacial area occurs, thus leading to a reduction in the gas-film efficiency. Viscosity can also increase the size of a bubble at bubble formation at the slot or orifice by retarding the rate of closure of the neck of the bubble. This change in bubble size could be detected by liquid holdup measurements for the tray. Liquid holdups decrease with increasing liquid viscosity.

A beneficial effect of liquid viscosity is the reduction of the bubble rise rate through the liquid on the plate, leading to an increased mass transfer. This effect, however, does not appear to be sufficient to balance the decrease in surface area obtained at higher liquid viscosities.

1.1.4. Concentration effects

The effect of concentration on the tray efficiency was demonstrated by Fane and Sawistowski in [2], where they showed a dependence of the benzene-cyclohexane system on the efficiency passing through a maximum. This dependence was found to be strong at medium weir height and at low vapour velocity.

Shilling [7] obtained tray efficiency data for the distillation of ethanol/water mixture (concentration of ethanol was between 0 and 70 mole %). He observed an efficiency maximum in the composition range of 35 to 60 mole percent ethanol with efficiency falling more sharply at the lower ethanol composition range. At very low ethanol concentrations, Murphree efficiency values were observed to exceed 100%, the ones the authors suggested to be erroneous.

2 Packed rectification column: the efficiency of mass-transfer devices

Most of the up-to-date industrial packed beds have very extended surface area. For example, Sulzer AG is currently engaged in the production of packed beds with the specific surface of more than $800 \text{ m}^2/\text{m}^3$, and Ingehim Company suggests randomly distributed packing with specific surface in the range of 1000 to $1200 \text{ m}^2/\text{m}^3$ [8]. All packing columns, except the vortex ones, certainly work in the film mode, where the nozzle surface area is a mass-transfer surface in case of full flooding. Such columns work in the small operating range for vapour velocities (at vapour velocities less than about 1.5 m/s) and low density irrigation (less than $10\div 15 \text{ m}^3/\text{m}^2\text{h}$). The mass-transfer surfaces in them are to be practically

constant on the whole range of operating vapour/liquid velocities. The only exception is a short range close to the liquid holdup mode, when emulsification arises [9], whereby the value of mass-transfer surface and mass transfer coefficient spontaneously rise.

Inasmuch as this regime is close to the flooding point, its retention is impossible even with the use of modern automation systems. As a result, all the nozzles, except for the ones we studied [10, 11, 12], work as the apparatus with thin-film, which ideally equals the nozzle surface area.

As most packed beds mainly work in the film mode, the main desirable requirements for the packing of distillation columns are: to promote a uniform distribution of gas and liquid, have a large surface area (for greater contact between the liquid and vapour phase) and have an open structure, providing a low resistance to the gas flow. Many types and shapes of packing can satisfactorily meet these requirements [13].

While determining the actual number of trays of mass-transfer apparatus, the concept of theoretical plate is used, where the equilibrium between the vapour and liquid concentration is achieved on a condition that the complete mixing of vapour and liquid phases occurs. On the n -tray, the changing of the light-volatile component from liquid to vapour phase takes place, and semi-volatile component changes from vapour to liquid state. Such alteration of concentration is named the theoretical step or theoretical plate. Drawing the steps between the tie and equilibrium lines in the given range of operating concentrations, the general number of steps of N_T (or number of theoretical plates) is found.

If the number of theoretical trays is known, then the number of actual N_D steps of separation is:

$$N_D = \frac{N_T}{\eta_T}, \quad (1)$$

where η_T is the averaged tray efficiency.

As we have noted before [14], many researchers defined the overall tray column efficiency by means of the tray efficiency. The most popular concept for evaluating the height of a packed column is the *HETP* (Height Equivalent to Theoretical Plate), defined by the following equation:

$$Z = HETP \cdot N, \quad (2)$$

where:

Z is a height of the packed bed required for achieving the separation equivalent to the N – number of theoretical plates [15].

Unfortunately, there are only a few generalised methods available in the open source literature for estimating the *HETP*. These methods are empirical and supported by vendor advice. The performance data published by universities are often obtained while using small columns and with not industrially important packing. When commercial-scale data are published, they usually are not supported by analysis or generalisation. Several correlations and empirical rules have been developed for *HETP* estimation in the last 50

years. Among the empirical methods, there is a rule of thumb for traditional random packing, that says:

$$HETP = \text{Column Diameter}$$

In Caldas's investigation, it has been shown that this rule holds only for small diameter columns.

Lockett [16] has proposed a correlation to estimate *HETP* in columns containing structured packing elements. It was inspired by Bravo's correlation [17] in order to develop an empirical relation between *HETP* and the packing surface area, operating at 80% flooding condition:

$$HETP = \frac{(4.82 \cdot (\rho_L - \rho_G)^{0.5} \mu_r^{-0.06})}{\alpha}, \quad (3)$$

which:

$$\alpha = a_p \cdot \left[\left(1 + 0.78 \cdot e^{0.00058 a_p} \right) \cdot \left(\frac{\rho_G}{\rho_L} \right)^{0.25} \right]^2, \quad (4)$$

where

- ρ_G – vapour density, kg/m³;
- a_p – specific surface of the packing, m²/ m³;
- μ_r – relation between liquid viscosity at the packing bed temperature and viscosity of water at the temperature of 20°C.

According to the Wang studies [18], *HETP* can be evaluated more accurately by the following expression:

$$HETP = \frac{\ln \lambda}{\lambda - 1} \left[\frac{u_{Gs}}{k_G \cdot a_e} + \lambda \cdot \frac{u_{Ls}}{k_L \cdot a_e} \right], \quad (5)$$

where:

- λ – inclination ratio between the equilibrium and operation straight;
- k_G – gas-phase mass-transfer coefficient;
- k_L – liquid-phase mass-transfer coefficient;
- a_e – effective interfacial area, m²/ m³;
- u_{Ls}, u_{Gs} – liquid/gas phase superficial velocity.

Therefore, the precision of *HETP* evaluation by the equation (5) depends on the accuracy of correlations used to predict the effective interfacial area as well as the vapour and liquid mass-transfer coefficients, which are easily defined by the experiments at the known values of the mass-transfer area.

2.1. Murphree vapour phase efficiency

Besides the calculation of height equivalent to a theoretical plate, a number of researchers evaluate that by using the Murphree efficiency. In particular, in Laptev's work [19], the comparison between the efficiencies of diverse packings was provided. Also, it

was shown that “Ingehim” packing has the highest efficiency at the same vapour velocities and reflux ratio.

The equation derived by Murphree is:

$$y_j = y_j^* - M \cdot (y_j^* - y_{j-1}), \quad (6)$$

or in its more familiar form:

$$E_i^{MV} = \frac{y_{i,j} - y_{i,j+1}}{y_{i,j}^* - y_{i,j+1}} \quad (7)$$

where:

- E_i^{MV} – is the Murphree vapour phase efficiency,
- $y_{i,j}^*$ – equilibrium vapour phase concentration of the volatile component;
- j – tray number;
- i – component number.

It should be noted that this equation (7) is easily applied to the case where the vapour resistance is negligible in comparison with the liquid film resistance. An equivalent equation for the liquid phase can be also easily derived. However, the given method of calculation of the efficiency just weakly reflects (only through y^*) the real hydrodynamics of the mass-transfer device and it does not give any understanding on the influence of its structural design.

2.2. Description of packed mass-transfer devices

As an objective, Bravo and Fair [17] had the development of a general project model to be applied in packed distillation columns, using a correlation that doesn't need validation for the different types and sizes of packing. Moreover, the authors did not intend to obtain the dependence on the flooding point, as the model of Bolles and Fair does. For this purpose, the authors used the Onda's model, with the database of Bolles and Fair, to give a better correlation based on the effective interfacial area for calculating the mass-transfer rate. The better correlation, for all the systems and packing tested, is given by:

$$\frac{a_e}{a_p} = 0.498 \cdot \left(\frac{\sigma^{0.5}}{Z^{0.4}} \right) \cdot (Ca_L \cdot Re_V)^{0.392}, \quad (8)$$

where:

- a_e – effective interfacial area, m^2/m^3 ;
- a_p – specific surface of the packing, m^2/m^3 ;
- Ca_L – capillary number;
- σ – liquid surface tension, N/m;
- Re_V – Reynolds number for vapour phase.

Recently, with the emergence of more modern packing, other correlations predicting the rate of mass-transfer in the packed columns have been studied. Wagner [20], for example,

developed a semi-empirical model, taking into account the effects of pressure drop and holdup in the column for the Nutter rings and IMTP, CMR and Flaximax packing. These packings have a higher efficiency, and therefore they have become more popular for new projects of the packed columns today. However, for the traditional packing, according to the author, only correlations of Cornell [21], Onda [22] and Bravo and Fair [17] can give reliable information for the industrial use in both distillation and absorption processes. The derived theoretical relation is described as:

$$HETP = H_o \cdot \frac{\ln \lambda}{\lambda - 1}, \quad (9)$$

where:

- H_o – height of a global mass transfer, m;
- λ – inclination ratio between the equilibrium and operation line.

Although the above equation was validated only for the cases of dilute solutions, constant molar flow rates, binary systems and equimolar countercurrent diffusion, it has been applied to systems with very different conditions even for multicomponent systems [5].

Henriques de Brito and his co-workers [23] measured the effective interfacial area of sheet metal structured packings, such as Mellapak 125Y, 250Y and 500Y. Their results have demonstrated that the effective area can be much higher than the packing surface area due to instabilities in liquid flow, such as ripples, waves, etc.

The resulting correlation for all measurements as a function of the Reynolds number for liquid phase is as follows:

$$\frac{a_e}{a_p} = 0.465 \cdot Re_L^{0.3}, \quad (10)$$

where:

- Re_L – Reynolds number for liquid phase.

It should be noted that the authors have not checked the correlation with fluids with different densities and viscosities of a liquid. Moreover, none of the aforementioned correlations examined the influence of the vapour flow; consequently, these dependences can be used only at the low velocities of the vapour phase.

3. The difficulties in determination of actual mass-transfer surface

Based on experimental data, many researchers define the mass-transfer coefficients fast enough, inasmuch as, if nozzle surface equals mass-transfer surface, then they present a single unknown in the mass-transfer equations.

$$M = K_v \cdot F_H \cdot \Delta_s, \quad (11)$$

where:

- K_V – mass-output coefficient, (kg mol)/(m² s);
- F_H – packing surface area, m²;
- Δ_s – driving force (an average concentration gradient).

In tray columns and columns with vortex nozzles, while changing both the vapour velocity and the irrigation density, the alteration of two parameters (effective surface area and mass-transfer coefficients) in the mass-transfer equations occurs. Consequently, it is impossible to carry out the tray efficiency calculations by equations 8 and 10, in which we need to know both of the parameters. Thus, the efficiency for these mass-transfer devices is to be calculated either by determining the volumetric mass-transfer coefficients or in the terms of hydrodynamic characteristics of the vapour and liquid phases [19].

$$1 - \eta_T = e^{\frac{-K_{oy} \cdot a \cdot f \cdot H}{V}} = e^{-N_{oy}}, \quad (12)$$

where:

- f – cross-sectional area, m²;
- N_{oy} – number of transfer units;
- H – height of the contact area, m;
- $(K_{oy} \cdot a)$ – volumetric mass-transfer coefficient.

The equation (12) shows that the height of transfer unit can be found, on condition that the value of volumetric mass-transfer coefficient is well-known.

The most interesting approach for determining the efficiency of mass-transfer device has been proposed in the work of Kafarov V. V. [9], where an empirical dependence of overall column efficiency was obtained by means of processing a large amount of data taken from the industrial bubble plate rectification columns.

$$\lg \eta = 1.67 + 0.3 \cdot l \cdot g \cdot \left(\frac{L}{V} \right) - 0.25 \cdot \lg(\mu_L \cdot \alpha) + 0.3 \cdot h_L, \quad (13)$$

where:

- L, V – vapour and liquid load, (kg mol)/h;
- μ_L – liquid viscosity, (Pa s);
- α – relative volatility;
- h_L – length from upper end of cut to upper end of weir height plus half length of cut, m.

4. The directions of intensification and energy- and resource conservation of rectification processes

Besides the criteria of efficiency of the packed columns, great interest is presented by the directions of intensification and energy- and resource conservation of rectification processes.

Three directions, which are developed currently by domestic and abroad scientists, should be mentioned:

1. The works concerned with the minimisation of the reflux ratio. These investigations are intensively conducted in Russia under the direction of Serafimov L. A. [24, 25, 26].
2. The researches under Kulov N. N., directed to the development of combined reactive-catalytic processes and distillation processes combined with the processes of crystallisation. The implementation of these processes allows to decrease the external dimensions of equipment and power inputs. The combined processes of catalytic rectification enable to overpass the thermodynamic limitations in equilibrium reactions and to use the heat from exothermic reactions for the separation of the resulting mixtures [27, 28, 29].
3. The works concerned with the development and investigation of the new mass-transfer devices, which allow to essentially increase the rates and accordingly – the mass- and heat-transfer coefficients [8, 10, 11].

This work belongs to the latter direction, as we propose to apply the established by us dependence of transfer unit efficiency of the new packet vortex nozzle on the Reynolds number for the vapour and liquid phases.

The packet bed of this nozzle represents a set of cells combined with each other in the horizontal plane. In contrast with the existing packings, the created nozzle has more developed nonlinear surfaces, which provide the formation of a large quantity of drops on the film surface at vapour velocities of more than 2 m/s. Unlike the packings, which work in the thin-film mode, this nozzle works in the near-emulsive regime, where the mass-transfer surface is much higher than the geometric nozzle surface at the range vapour velocity from 2 to 5.5 m/s and values of the irrigation density of over 120 m³/m²s. The vortex nozzle possesses a quality of self-distribution of liquid phase on the column cross section even by feeding by a single jet on the nozzle surface.

The high velocities in the vapour and liquid phases as well as extremely high mass- and heat-transfer coefficients in the volume unit of a nozzle allow essential decreasing of external sizes of rectification columns at a high quality and efficiency of continuous processes.

The processing of experimental data based on the alteration of vapour velocities and flow rates at the column length permitted to derive an explicit form for the efficiency of conditional plate (or packed bed) of the packet vortex nozzle, which depends on the vapour and liquid according to Reynolds criteria; the irrigation density was changed in the range from $0.6 \cdot 10^{-3}$ to $1.4 \cdot 10^{-3}$ m³/m²s and the vapour velocity – from 1.2 to 3.5 m/s.

$$\eta_i = 4.5 \cdot 10^{-3} \cdot Re_{iL}^{0.5} \cdot Re_{iV}^{0.72} \quad (14)$$

$$Re_{iL} = \frac{V_{iL} \cdot b \cdot \rho_{iL}}{\mu_{iL}} \quad (15)$$

$$Re_{iV} = \frac{V_{iV} \cdot b \cdot \rho_{iV}}{\mu_{iV}} \quad (16)$$

where:

- V – irrigation density, $\text{m}^3/\text{m}^2\text{s}$;
- b – width of a cell in a packet vortex nozzle, m;
- ρ_L, ρ_V – vapour and liquid density, kg/m^3 ;
- μ_L, μ_V – vapour and liquid dynamic viscosity, (Pa s);
- U_V – vapour velocity, m/s.

5. Conclusion

Literary data analysis shows that the commonly used Murphree efficiency factor can be applied for evaluating the efficiency of the rectification columns with binary mixtures. Besides, it is necessary to know or experimentally determine the concentration of a volatile component on each tray for calculating the Murphree factor.

In turn, for calculating the efficiency of the packed columns, which work in the film mode, knowledge of mass-transfer coefficients and specific contact surface is required. The latter is discussed in a large number of works, in which, according to some foreign studies, the value of the interface area is substantially smaller than the value of the geometric nozzle surface. This is why the calculation of the actual mass-transfer surface is rather difficult even for the nozzles, which work in the film mode.

Due to the fact that for the plate rectification columns and columns with the mass-transfer devices one can't clearly determine the actual mass-transfer surface, the efficiency of the mass transfer devices is to be calculated, either by determining the volumetric mass transfer coefficients or the hydrodynamic characteristics of the vapour and liquid phases, which depend on the structural design of plates or vortex nozzles.

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