MASS-TRANSFER EFFICIENCY OF A SMALL-SIZE TEFLON BUBBLE-CAP TRAY

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The combined effect of flow conditions, geometry and physical properties on distillation efficiency of a newly constructed small-size Teflon bubble-cap tray for production of high-purity substances is experimentally investigated. The limits of vapour loading required for a steady hydrodynamic tray operation are found. The results evidence an influence of the liquid entrainment on plate efficiency at elevated superficial vapour velocities and higher downcomers. The comparison of the plate efficiency values determined experimentally with those from different correlations for large columns shows that the equation of MacFarland *et al.* and the point AIChE theoretical method are the best for predicting mass-transfer efficiency of the small-size bubble-cap Teflon tray used.
Key words: Plate efficiency; Small-size bubble-cap tray; Teflon column.

Mass-transfer efficiency is a basic parameter in the distillation column design and simulation. It depends on various factors, such as composition and properties of the fractionated mixture, flow conditions, type, construction details and size of the trays.

The contact devices intended for high-purity distillation are of the form and size that differ substantially from the conventional metal units met in industry. Their features are defined by the small diameter related to the low production capacity of high-purity substances. Their construction also strongly depends on the specific ways of machining the material used, *i.e.*, borosilicate glass, quartz of Teflon which determine the appropriate shape, thickness, mode of disposition and connection of column sections and internals.

In comparison with the case of the common large steel plates, the specifics of small-size tray geometry cause a change in the hydraulics of bubble contact and affect the tray efficiency. For small tray diameters and unimportant variation in the radial and axial concentration profile inherent to high-purity distillation, the point, plate and column efficiencies are approximately equal ($E_{mv} \approx E_{mV} \approx E_0$). Several constructions of laboratory-scale glass plate columns are described in literature[1]. Nevertheless, no corre-

lation for plate efficiency in designing of small-size distillation columns has been proposed.

In a number of cases, in the production of equipment for precise separation, Teflon is preferable to glass or quartz for its bending and impact strength. So far, however, Teflon has been used to produce merely packings for small-scale columns. No information about constructions of Teflon trays and correlations of respective plate efficiencies are known to the authors.

The present work is devoted to :

– experimental analysis of the combined effect of flow conditions, geometry and physical properties on the distillation efficiency of a newly developed construction of the small-size Teflon bubble-cap tray (D = 80 mm);

– finding the limits of vapour loading required for stable hydrodynamic conditions on the tray construction proposed;

– testing the published efficiency prediction methods against experimental plate efficiency data of the small-size Teflon tray construction elaborated.

EXPERIMENTAL

Tray efficiency was studied experimentally by using the model binary mixture hexane–tetrachloromethane, both components of reagent grade.

The experimental set-up is illustrated in Fig. 1. The Teflon tray 4 under investigation was of the bubble-cap type with internal downcomer. Details and geometrical parameters of the tray are shown at the right-hand side of Fig. 1. Another Teflon tray 3 (without a cap) at tray spacing of 40 mm was mounted over the tray 4 for vapour condensate sampling. The 5-litre reboiler 6 was made of glass

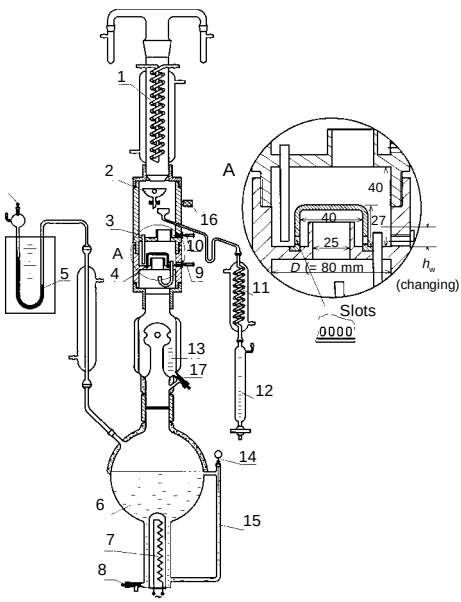

FIG. 1

Experimental Set-up: 1 Total reflux condenser; 2 automatic reflux divider; 3 Teflon tray without cap; 4 Teflon tray under investigation; 5 pressure drop contact manometer for control; 6 reboiler; 7 electric heater; 8 Teflon valve; 9, 10 sampling syringes; 11 distillate cooler; 12 measuring cup for vapour condensate flow rate; 13 measurement device for liquid flow rate; 14 platinum electric resistance component; 15 thermosiphon circulation unit; 16 solenoid coil; 17 Teflon valve. Number of slots: 40, slot width 2 mm, slot height 6 mm

(Simax) with the built-in electric heater 7 and the thermosiphon circulation unit 15. The total reflux condenser 1 is a water-cooled helical-type heat exchanger made of glass and open to the atmosphere.

The liquid temperature in the reboiler was measured by the platinum electric resistance component 14 giving an accuracy of 0.1 °C. Different heating intensities corresponding to different column vapour loadings were obtained by changing the power input to the electric heater. The control of vapour loading was carried out by means of the pressure drop contact manometer 5 connected with the electric heater by means of a relay. Below the total reflux condenser 1, the automatic reflux divider 2 with the solenoid coil 16 was mounted to allow the measurement of the volumetric condensate flow rate Q_L by collecting in the measuring cup 12. The volumetric reflux flow rate measured directly by the device 13, after closing up the Teflon valve 17, is equal to the volumetric condensate flow rate Q_L at total reflux ($L/V = 1$) and adiabatic conditions.

The bottom liquid sampling was carried out by Teflon valve 8. The tray liquid sampling was accomplished by the syringe 9. Sample of vapour condensate was taken by the syringe 10.

The experimental conditions obtained by changing the values of the variables considered are given in Table I. The study included variations of:

1. Concentration x_f of the more volatile component C_6H_{14} in the initial feed mixtures from 0.06 to 0.9 mole fraction. This change leads to different values of the physical properties of vapour and liquid on the plate investigated in the limits:

$$\mu_L = 2.06 \,.\, 10^{-3}\text{–}4.31 \,.\, 10^{-3} \text{ kg m}^{-1} \text{ s}^{-1};$$

$$\rho_L = 652.4\text{–}1342.8 \text{ kg m}^{-3};$$

$$\sigma_L = 1.37 \,.\, 10^{-2}\text{–}1.92 \,.\, 10^{-2} \text{ kg s}^{-2};$$

$$\mu_V = 7.66 \,.\, 10^{-6}\text{–}1.08 \,.\, 10^{-5} \text{ kg m}^{-1} \text{ s}^{-1};$$

$$\rho_V = 3.35\text{–}5.07 \text{ kg m}^{-3};$$

$$\alpha = 1.15\text{–}1.62.$$

The properties of the pure components were obtained from literature data or empirical equations as a function of temperature[2,3]. The properties of the binary mixtures were calculated by means of generally accepted additive correlations.

2. Superficial vapour velocity w_V in limits of 0.06–0.3 m s^{-1}. The superficial vapour velocity w_V was computed from the volumetric vapour flow rate Q_V ($Q_V = Q_L\rho_{L/}\rho_V$; $w_V = Q_V/A$).

3. Weir height h_w from 6 to 21 mm. The change of the weir height h_w was realized by vertical moving and fixing of the downcomer at different levels.

The experiments were carried out at total reflux, at atmospheric pressure and adiabatic conditions. Each experiment was of about 5 h duration to achieve steady-state conditions. After this period, liquid samples of about 5 ml were withdrawn from the sampling points 8, 9, 10 (see Fig. 1), and the concentrations of C_6H_{14} in the bottom liquid (x_b), in the tray liquid (x_p), and in the vapour condensate (x_d) were determined. The compositions of the liquid samples were analyzed using a Pulfrich refractometer with standard calibration, giving an accuracy of ±0.001 mole fraction.

These compositions were used to determine the Murphree vapour plate efficiencies for various experimental conditions, as:

$$E_{mV}^{exp} = \frac{y_{out} - y_{in}}{y_{out}^{*} - y_{in}} \,, \qquad (1)$$

where $y_{out} = x_d$ and $y_{in} = y_b^* = k_b x_b$ are the mole fractions of the more volatile component C_6H_{14} in the vapour leaving and entering the tray investigated; $y_{out}^* = k_p x_p$ is mole fraction of C_6H_{14} in the vapour in equilibrium with the liquid leaving the tray.

The equilibrium constant values k_b and k_p for the measured concentrations of more volatile components x_b and x_p were evaluated by using the vapour–liquid equilibrium data[4] tested for thermodynamic consistency (Herington test) and then correlated using the Wilson model for calculating the activity coefficients.

RESULTS AND DISCUSSION

The Murphree vapour plate efficiency values, obtained from the experiments, are presented in Table I. The results of the repeated runs 24 to 29 showed good reproducibility:

$$\mathrm{RMSD} = \left[\left(\sum_{j=24}^{29} (\bar{E}_{mV}^{exp} - E_{mV}^{exp})_j^2\right)/6\right]^{1/2} = \pm 0.0057 \ . \qquad (2)$$

Figure 2 shows the effect of superficial vapour velocity w_V on the Murphree efficiency E_{mV}^{exp} of the small-size Teflon plate investigated. The scatter in these results is caused by the influence of varying mixture composition and weir height. The experimental points 8 and 16 are not included in Fig. 2 because the low values of the plate efficiency are obtained at quite high downcomers and high vapour velocities. It can be seen that in limits of vapour velocity w_V from 0.08 to 0.27 m s^{-1}, where the plate is in regime of stable operation (above the weep point and below the entrainment point), the efficiency E_{mV}^{exp} varies within the range of 0.6–0.8. The experimentally recorded decrease of the plate efficiency at vapour velocities above 0.15 m s^{-1} may be explained by an influence of the drop entrainment between trays.

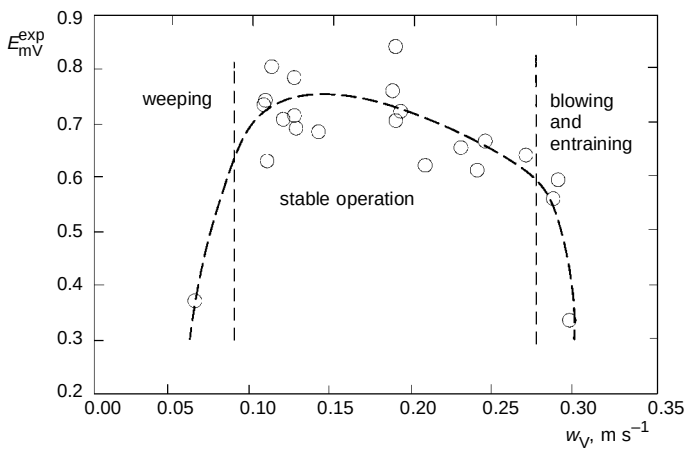

FIG. 2
Effect of superficial vapour velocity w_V on the plate efficiency E_{mV}^{exp}

The interpretation of the effect of drop entrainment on mass-transfer efficiency is also supported by the experimental results shown in Fig. 3. Tracing different ranges of superficial vapour velocity, the effect of weir height h_w on the measured plate effi-

TABLE I
Experimental conditions and Murphree vapour plate efficiency values

j	x_f mole fraction	x_p mole fraction	w_V, m s^{-1}	h_w, mm	E_{mV}^{exp}
1[a]	0.231	0.293	0.111	10.0	0.803
2[a]	0.231	0.298	0.125	10.0	0.783
3[a]	0.240	0.314	0.125	8.8	0.713
4[a]	0.231	0.298	0.268	10.0	0.640
5	0.231	0.297	0.285	10.0	0.559
6	0.240	0.297	0.288	8.8	0.594
7[a]	0.240	0.330	0.126	17.2	0.690
8	0.240	0.301	0.247	17.2	0.326
9[a]	0.744	0.781	0.106	10.0	0.733
10[a]	0.742	0.782	0.108	8.8	0.629
11[a]	0.747	0.776	0.243	10.0	0.666
12[a]	0.747	0.777	0.228	10.0	0.654
13[a]	0.742	0.775	0.238	8.9	0.612
14[a]	0.742	0.775	0.107	17.1	0.741
15[a]	0.742	0.775	0.118	17.1	0.706
16	0.742	0.778	0.231	17.1	0.360
17	0.490	0.552	0.295	13.2	0.335
18	0.490	0.575	0.063	13.2	0.371
19[a]	0.500	0.554	0.186	20.4	0.759
20[a]	0.492	0.547	0.188	6.0	0.704
21[a]	0.894	0.908	0.188	13.0	0.840
22[a]	0.894	0.906	0.140	13.0	0.683
23[a]	0.062	0.101	0.206	13.0	0.621
24[a]	0.492	0.554	0.191	13.2	0.721
25[b]	0.492	0.554	0.192	13.2	0.718
26[b]	0.492	0.553	0.190	13.2	0.732
27[b]	0.492	0.554	0.190	13.2	0.724
28[b]	0.492	0.553	0.190	13.2	0.714
29[b]	0.492	0.553	0.192	13.2	0.719

[a] Experimental points in the region of stable operation. [b] Repeated runs.

ciency E_{mV}^{exp} is shown. It is observed that the increase in the weir height for vapour velocities between 0.1 and 0.2 m s^{-1} leads to a slight enhancement of mass-transfer plate efficiency. In contrast, while considering vapour velocities approaching the upper limit (0.3 m s^{-1}), a similar trend to those shown in Fig. 2 is observed, that is, a decrease in the plate efficiency at increasing weir height. This trend could be again explained by an increased amount of the liquid entrained by the vapour flow at elevated vapour velocities and higher downcomers.

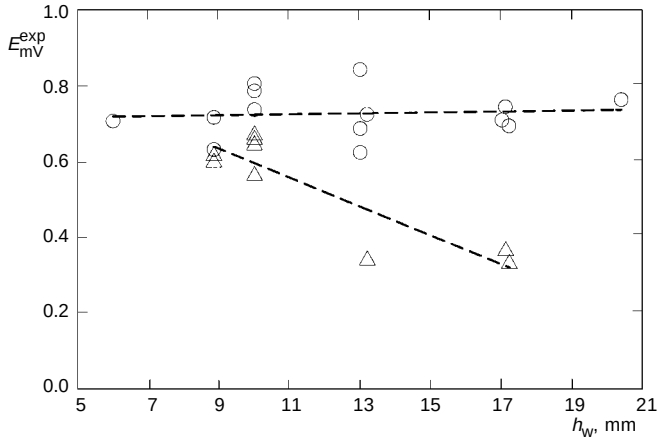

FIG. 3
Effect of weir height h_w on the experimental plate efficiency E_{mV}^{exp} for different ranges of superficial vapour velocity. Δ $w_V \in$ (0.1–0.2 m s^{-1}), ❍ $w_V \in$ (0.2–0.3 m s^{-1})

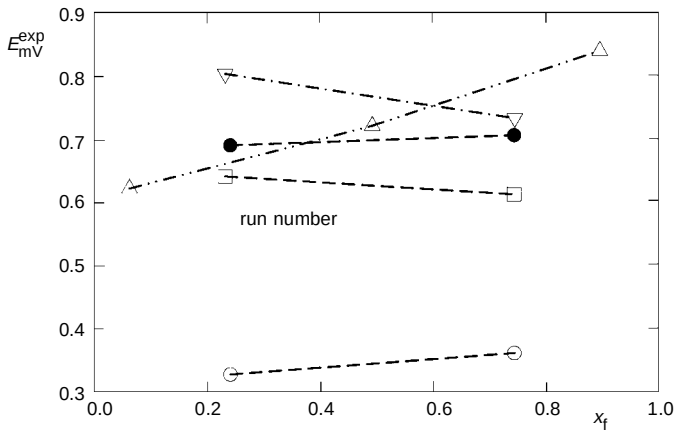

FIG. 4
Effect of the concentration of more volatile component x_f on the plate efficiency E_{mV}^{exp}. Run numbers: ● 7, 15; ❍ 8, 16; ❒ 4, 13; ∇ 1, 9 and Δ 21, 23, 27

The attempts to use the Hunt correlation[5] for drop entrainment calculations at runs 8, 16 and 17 (in Table I) did not yield the expected results. The calculated values of liquid entrained net fractions are quite small – 0.018–0.029 kg liquid per kg vapour.

Figure 4 compares the experimental values of the plate efficiency E_{mV}^{exp} in experiments where w_V and h_w are similar to each other – runs 1 and 9; 4 and 13; 7 and 15; 8 and 16; 21, 23 and 27 (see Table I). It can be seen that an increase in C_6H_{14} mole fraction in mixture studied (which decreases mixture viscosity, density and increases liquid surface tension) leads to different trends of change in the plate efficiency – increasing (runs 21, 23 and 27), decreasing (runs 1 and 9) or fairly constant (runs 4 and 13; 7 and 15; 8 and 16).

These contradictory results could be explained in terms of a prevailing influence of the flow conditions and tray geometry over the effect of physical properties on the efficiency of the plate investigated.

Various efficiency prediction methods summarized by Vital *et al.*[6] were tested against experimental values of the plate efficiency for the small-size Teflon bubble-cap tray proposed. The experimental plate efficiencies and those calculated by the equations are compared for the experimental points falling within the range of stable tray operation (denoted with asterisks in Table I).

The MacFarland equation[7] and the point AIChE method[8] without correction for the imperfectness of liquid phase mixing, both accounting for the combined effects of the mixture physical properties, the tray geometry, and the flow conditions, are most reliable in predicting the plate efficiency of the small-size Teflon bubble-cap tray.

Much lower values of plate efficiency predicted and great deviations with respect to the experimental values are obtained by using the equations of Walter and Sherwood[9], and Bakowski[10].

CONCLUSIONS

On the basis of the present investigation, the following may be concluded:

– The experimental analysis indicates that, at vapour superficial velocties of 0.08 to 0.27 m s^{-1}, the values of the Murphree plate efficiency E_{mV}^{exp} are relatively high and vary within the range of 0.6–0.8 for the small-size Teflon bubble-cap tray (D = 0.08 m) investigated.

– The tendency of decreasing the plate efficiency for this small-size Teflon tray with a tray spacing 0.04 m at superficial vapour velocities above 0.15 m s^{-1} may be caused by the influence of drops entrainment between plates. The effect of the flow conditions and the tray geometry upon the plate efficiency is much greater than the influence of the mixture physical properties, at least for the system studied.

– The plate efficiency is predicted most reliably by the equation of MacFarland[7] and the point AIChE method[8]. The equations by Walter and Sherwood[9], and Bakowski[10] are most unsuitable.

SYMBOLS

A	cross-sectional area of column, m^2
D	tray diameter, m
E_{mv}	Murphree vapour point efficiency
E_{mV}	Murphree vapour plate efficiency
$\overline{E}_{mV}$	average Murphree vapour plate value of the repeated runs
E_0	overall column (plate) efficiency
h_w	weir height, m
k_b, k_p	equilibrium constants for more volatile component
L	liquid molar flow-rate, mole s^{-1}
Q_L	liquid volumetric flow-rate, $m^3\ s^{-1}$
Q_V	vapour volumetric flow-rate, $m^3\ s^{-1}$
RMSD	Root-Mean-Square Deviation, Eq. (*2*)
V	vapour molar flow-rate, mole s^{-1}
w_V	superficial vapour velocity, m s^{-1}
x	mole fraction of more volatile component in the liquid phase
y	mole fraction of more volatile component in the vapour phase
y^*	mole fraction of more volatile component in the vapour phase in equilibrium with liquid x
α	relative volatility of more volatile component
μ_L	liquid viscosity, kg $m^{-1}\ s^{-1}$
μ_V	vapour viscosity, kg $m^{-1}\ s^{-1}$
ρ_L	liquid density, kg m^{-3}
ρ_V	vapour density, kg m^{-3}
σ_L	liquid surface tension, kg s^{-2}
Subscripts	
b	bottom
d	distillate
f	feed
p	plate under investigation
Superscript	
exp	experimental value

REFERENCES

1. Krell E.: *Handbuch der Laboratoriumdestillation*, 3rd ed. VEB Deutscher Verlag der Wissenschaften, Berlin 1976.
2. Reid R. C., Prausnitz J. M., Sherwood T. S.: *The Properties of Gases and Liquids*, 3rd ed. McGraw–Hill, New York 1977.
3. Nikolskii B. P. (Ed.): *Spravochnik Khimika*, Vol. I. Khimiya, Moscow 1963.
4. Rodger A. J., Hsu C. C., Furter W. F.: *J. Chem. Eng. Data* **1969**, *14*, 362.
5. Hunt C., Hanson D. N., Wilke C. R.: *AIChE J.* **1955**, *1*, 441.
6. Vital T. J., Grossel S. S., Olsen P. I.: *Hydrocarbon Process.* **1984**, *63* (11), 147.
7. MacFarland A., Sigmund P. M., van Winkle M.: *Hydrocarbon Process.* **1972**, *51*, 111.
8. *Bubble Tray Design Manual*. American Institute of Chemical Engineers, New York 1958.
9. Walter F., Sherwood T. K.: *Ind. Eng. Chem.* **1941**, *33*, 493.
10. a) Bakowski S.: *Br. Chem. Eng.* **1963**, *6*, 384; b) Bakowski S.: *Br. Chem. Eng.* **1963**, *7*, 472.