Distillation Efficiencies on a Large Sieve Plate with Small-Diameter Holes

M. W. Biddulph, M. M. Dribika Department of Chemical Engineering, University of Nottingham, Nottingham NG7 2RD, England

The efficiencies of large-scale conventional distillation sieve trays usually turn out to be lower than would be expected based on small-column results suitably scaled-up. One of the reasons for this is the presence of nonuniformities in the liquid as it flows across circular trays from inlet to outlet weir.

Efficiency data from commercial-scale distillation columns are less readily available than the large amount of data that have been reported from studies of small-scale laboratory columns. A survey of some of the available literature on largescale sieve tray efficiencies, where the column diameter is greater than 0.45 m, is shown in Table 1. Often the design of large columns has involved the use of predictive methods for point efficiency and tray efficiency, the most used one being the AIChE method summarized in the Bubble Tray Design Manual (AIChE, 1958). It has been observed (Lashmet and Szczepanski, 1974; Hughmark, 1971; Strand, 1963) that this method often tends to predict tray efficiencies that are higher than those found in practice. Since the AIChE method was established a number of studies have been made of the effects of flow nonuniformities across conventional trays (Bell, 1972a,b; Porter et al., 1972; Weiler et al., 1973). A considerable amount of effort has been devoted to allowing for the effects of stagnant zones (Porter, 1972; Lockett et al., 1973; Lockett and Safekouridi, 1976; Lim et al., 1974), but the problem of predicting the point efficiencies that exist in the center of a large tray has received less attention. One of the difficulties is that of measuring or inferring such efficiencies.

The study described here involved the measurement of temperature and composition profiles along a rectangular sieve tray having 1.8 mm dia. holes, the object being to calculate tray efficiencies and to infer point efficiencies. This type of sieve tray material is used commercially in low-temperature, air-separation columns; the trays used in many small-scale point efficiency columns have similar hole sizes. The system studied was methanol-water, enabling a comparison with other reported results

the tray was chosen to enable a reasonable liquid flow path length to be used, about 1 m, and to avoid any problems with stagnant zones.

covering a wide range of composition. The rectangular form of

Experimental

Equipment

A general flowsheet of the new experimental rig is shown in Figure 1. The rectangular section distillation column B has three trays with overall dimensions 1,067 × 89 mm, and 154 mm tray spacing. The sieve tray material is typical of that commonly used in the low-temperature, air-distillation industry, 2 mm thick aluminium with 1.8 mm holes; details are given in Table 2. Double-glazed glass portholes are provided above the middle tray to enable easy observation of the biphase. All the hot surfaces of the equipment are insulated with 50 mm thick glass fiber material and aluminium cladding. A detailed diagram of the test tray, the middle one of the three, is shown in Figure 2. This shows the location of the liquid sampling and temperature measuring points S. The temperatures 3 mm above the liquid sampling points were measured using fixed thermocouples giving an accuracy of 0.1°C. The liquid samples were drawn through insulated narrow sampling tubes set into the floor of the tray. The tubes, which were as short as possible, emerged from the column below the tray through valves.

The six equally spaced temperature/sample points gave good profiles across the tray. In addition temperature readings and samples were taken on the floors of the inlet and outlet downcomers to the test tray, and samples were also taken from the reflux liquid leaving the condenser and the return liquid to the reboiler.

Column Operation

The column was operated at total reflux and at atmospheric pressure, the boil-up rate being measured by a rotameter in the reflux line. The column was run for 5 h to establish steady state conditions, the temperatures being continuously monitored dur-

M. M. Dribika is a Lecturer in the Chemical Engineering Department at Al-Fateh University, Tripoli, Libya.

Table 1. Large-Scale Sieve Tray Efficiencies

System	Col. Dia. m	Hole Dia. mm	Free Area %	Weir Height mm	Path Length m	Reference
n-Propanol-sec-butanol	1.8	4.76	_	25.4	0.61	Mayfield et al. (1952)
Acetic acid-water	0.46	3.18	8.7	38.0	0.25	Jones and Pyle (1955)
Acetic acid-water	0.46	3.18	6.7	38.0		Rush and Stirba (1957)
MIK-water	0.46	3.18	6.7	14.3	_	Rush and Stirba (1957)
Ammonia-air-water	0.91	3.18	9.2	25.4	_	Johnson and Marangozis (1958)
	x0.076			-101.6		- , ,
n-Heptane-toluene	0.45	10.0	16.0	75.0		Zuiderweg et al. (1958, 1960)
n-Heptane-MCH	0.45	10.0	16.0	75.0		Zuiderweg et al. (1958, 1960)
Benzene-toluene	0.45	10.0	16.0	75.0	_	Zuiderweg et al. (1958, 1960)
Ethanol-water	0.75	2.54	9.8	20.0		Kirschbaum (1962)
Methanol-water	0.98	4.0	4.2	40.0	0.76	Kastanek and Standart (1967)
Ethylbenzene-styrene	0.80	12.5	14.8	$38.0 \\ -19.0$	0.54	Billet (1967)
Methanol-ethanol	0.61 x0.305	3.18	6.4	0 - 76.0	0.61	Shore and Haselden (1969)
Ammonia-air-water	1.22	12.7	7.9	51.0	0.81	Nutter (1971)
CO ₂ -air-water	0.91 x0.30	9.5	10.8	76.0	0.61	Thomas (1976)
O ₂ -air-glycerol	0.91 x0.30	9.5	10.8	76.0	0.61	Thomas (1976)
Benzene-n-propanol	0.46	6.35	12.8	25.4 -76.0		Anderson et al. (1976)
n-Propanol-toluene	0.46	6.35	12.8	25.4 - 76.0	_	Anderson et al. (1976)
Cyclohexane-heptane	1.2	12.7	8.0	51.0	0.76	Sakata and Yanagi (1979)
i-Butane-n-butane	1.2	12.7	8.0	51.0	0.76	Sakata and Yanagi (1979)
Cyclohexane-heptane	1.2	12.7	14.0	25.0 -51.0	0.76	Yanagi and Sakata (1981)
i-Butane-n-butane	1.2	12.7	14.0	25.0 -51.0	0.76	Yanagi and Sakata (1981)
Methanol-water	0.59	4.8	9.0	50.0	0.37	Lockett and Ahmed (1983)

ing this period. The boil-up rate, the temperatures, and the froth height were noted and then liquid samples were collected in the usual way. About 15 runs were carried out, mostly at a constant vapor phase F-factor over a range of test tray compositions. A few additional runs were made at a different F-factor but constant composition. Samples were analyzed using chromatography, giving an accuracy of ± 0.0026 mol fraction.

Theoretical Model

A number of models have been proposed to represent the behavior of the biphase on an operating tray, the object being to derive relationships between point efficiencies and tray efficiencies. The concept of eddy diffusion has been used in this study to model the observed profiles across the tray, and hence infer point efficiencies from these observations. This model has been used previously in an analysis of low-temperature air-distillation columns (Biddulph, 1975b) and a detailed analysis and derivation was given. The use of this model to infer point efficiencies by matching predicted composition profiles to those measured in the experiments requires a knowledge of the extent of mixing present in the liquid phase on the tray.

Mixing Studies

The mixing characteristic on small hole size sieve trays have been extensively studied previously (Biddulph, 1975a), but it was felt to be desirable to measure actual mixing characteristics on the present tray. This was done using the system water-steam with a sodium nitrate solution tracer injection. An injection tube was installed immediately above the liquid sampling point nearest the outlet weir, and samples below the injector and upstream were taken to establish the extent of back-mixing. The level of sodium ions in these samples was measured using atomic absorption, the background level of sodium in the inlet water also having been measured. The well-established method of Barker and Self (1962) was used to estimate that Peclet number, and hence the value of the eddy diffusivity. The variation of tracer level with position gave good linear relationships, as required by the eddy diffusion model, and the result was a measured value of 0.0013 m²/s for the eddy diffusivity under conditions of similar loading to those used in the distillation experiments. This result agrees well with earlier air-water simulation measurements, those indicating a value of about 0.0015 m²/s. Using the hydraulic conditions existing during the distillation experiments, a Peclet number of about 39 was established from the measured eddy diffusivity value. This result indicates that conditions are approaching plug flow on this tray, and a small variation in Peclet number, perhaps due to slightly different vapor velocities in different runs, will not greatly affect the predicted composition profiles.

Results

The above theoretical model was used to predict composition profiles across the test tray for a series of runs at different com-

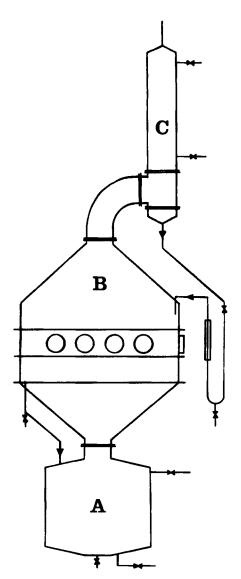


Figure 1. Schematic flowsheet of experimental equipment.

positions. The point efficiency value used in the model was varied until a good match was achieved between predicted and measured profiles, and this was then taken as the correct point efficiency operating during that run. Figure 3 shows these comparisons for eleven different runs. The equilibrium data of Maripuri and Ratcliff (1972) were used, as reported thermodynamically consistent by Gmehling and Onken (1977), together with the enthalpy data from Reid et al. (1977) and Kern (1950). It can be seen that the comparison of shape between the experimental points and the model lines is good. The composition profiles reported by Lockett and Ahmed (1983) show a similar form.

Table 2. Tray Details

Weir length	83 mm
Liquid flow path	991 mm
Tray spacing	154 mm
Hole diameter	1.8 mm
Percentage free area	8%
Outlet weir height	25 mm
Inlet weir height	4.8 mm

The tray efficiencies calculated from the experimental and the model values are shown in Figure 4. The mean liquid composition X on the try was calculated from:

$$X = \int_0^1 X_W dW \tag{1}$$

where W is the relative position on the tray. The results in Figure 4 show efficiencies at two different values of vapor phase F-factor, and no detectable variation is apparent. Most runs were carried out at values of F-factor of about 0.6. Observation of the tray behavior indicated that very little entrainment was occurring, and the predicted level of entrainment (Fair, 1963) was less than 2%. This would have a negligible effect on the efficiency (Rahman and Lockett, 1981). Furthermore there appeared to be no weeping present.

The experimental liquid temperature profiles across the tray are shown in Figure 5. These measured temperatures were compared with the bubble point temperatures, and this comparison is shown in Figure 6. The bubble point temperatures were calculated taking into account the nonidealities in the phases, the form of calculation having been described previously (Dribika et al., 1985). The comparison showed that the liquid on the tray was within ±0.2°C of the bubble point, justifying the usual assumption. The temperatures in the downcomers indicated that slight vaporization was occurring, similar to the observations of Ellis and Shelton (1960) and Lockett and Ahmed (1983), who also used methanol-water. This was due to variation of temperature from tray to tray producing heat transfer. The results shown in Figure 5 illustrate that the effect on the efficiency is very small, the average difference between measured and predicted values being about 2%. This agrees with the conclusions of Lockett and Ahmed (1983).

The tray efficiency results shown in Figure 4 confirm the predictions from the model that very high efficiencies are possible in the region of low methanol concentration. This is due to the large value of the equilibrium line slope in this region, resulting in steep concentration gradients across the tray, and consequently considerable enhancement of tray efficiency over point efficiency. Similar observations have been reported by Hubner (1972) for isopropanol-water, by Hay and Johnson (1960) and Lockett and Ahmed (1983) for methanol-water, and by Shilling and Beyer (1953) for ethanol-water. The values measured in this study are generally higher than those in earlier studies due

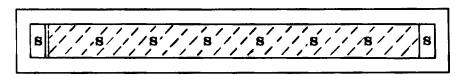


Figure 2. Detail of tray showing sample/temperature points.

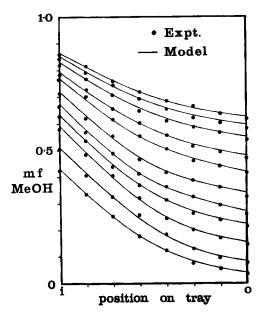


Figure 3. Liquid composition profiles across tray.

to the length of the liquid flow path and the small hole size giving higher point efficiencies. Furthermore the rectangular section used avoided any detrimental influences due to flow nonuniformities.

Point Efficiencies

The effective values of point efficiency that were deduced from matching the model to the profiles are shown in Figure 7. The efficiencies are fairly constant over most of the composition range, with a slight reduction at low concentrations of methanol.

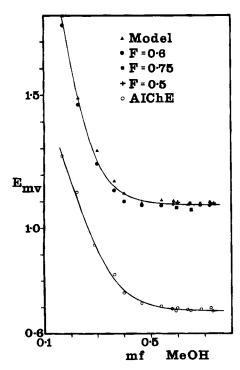


Figure 4. Tray efficiencies vs. mean liquid composition on tray.

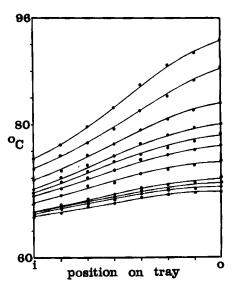


Figure 5. Temperature profiles across tray.

The very high tray efficiencies in this region are therefore due to the enhancement factors discussed earlier. Variation of vapor phase F-factor within the range studied produced no significant effect on point efficiency. Similar variations in efficiency were reported by Lockett and Ahmed (1983) and Hay and Johnson (1960), although at lower values. The higher values found here are attributed to the smaller hole size providing a greater liquid-vapor interfacial area. This is substantiated by comparing these results with those of Haselden and Thorogood (1964) for a 1 mm hole size laboratory-scale column operating under similar flow conditions on the system nitrogen-oxygen.

This comparison, made in Figure 7, shows very similar high values of point efficiency. This seems to confirm that high point efficiencies are characteristic of these small hole size trays. It therefore should be possible to achieve very high tray efficiencies, similar to those shown in Figure 4, in conventional commercial-scale columns incorporating these types of tray. However, plant experience has been that in columns operating on nitrogen-argon-oxygen at pressures around atmospheric, tray effi-

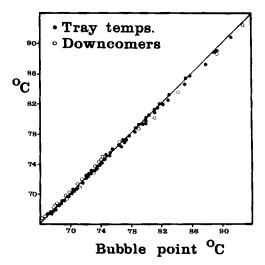


Figure 6. Comparison of measured temperatures with bubble points.

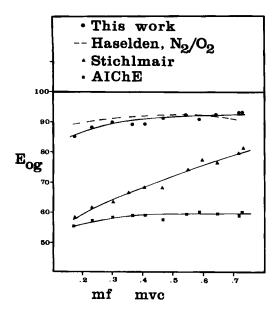


Figure 7. Point efficiency measurements and predictions.

ciencies do not achieve these very high levels, and this must be due to the other factors mentioned earlier.

The froth heights were also measured during the present experiments, and the variation with composition is shown in Figure 8. The accuracy of these measurements was only perhaps ± 1 cm, but the trend is fairly clear. This effect may have been due to increasing liquid loading necessary to maintain a constant vapor phase F-factor.

Efficiency Predictions

The AIChE method of prediction of efficiencies was applied to this situation. The point efficiencies predicted are shown in Figure 7, and it can be seen that the predicted values are very much lower than those measured. Some deficiencies in the AIChE method have been discussed by Lockett and Ahmed (1983), but an additional reason here seems to be in not having accounted for the influence of the small hole size. Stichlmair (1978) developed a predictive method for point efficiencies tak-

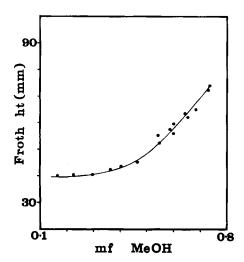


Figure 8. Froth height vs. mean liquid composition.

ing into account surface tension effects and flow regime. The predictions using this method are also shown in Figure 7. The values are higher than those from the AIChE method, but still lower than the experimental values, probably because the method also was not based on small hole size data. The enhancement factor of tray efficiency over point efficiency is very similar for predicted and measured cases, which is not surprising since the AIChE predictive method is also based on the eddy diffusion model and does not include any factor for nonuniformities in flow. The high experimental tray efficiencies are due to the high values of point efficiency. The AIChE predicted tray efficiencies, shown in Figure 4, show values which are, fortuitously, quite similar to the order of value actually experienced in large columns with trays of this type. However, these values have been arrived at by the incorrect route, and not by correctly including the contributing factors. Since the severity of the reduction in tray efficiency due to stagnant zones is a function of column size and tray configuration, this must either be included in any predictive method or, better still, the stagnant zones should be eliminated.

Conclusions

The experimental and theoretical study described here has demonstrated that sieve trays with very small holes can give very high point efficiencies. The high tray efficiencies reported here should be achievable on conventional commercial-scale trays, and the fact that they are not generally this high illustrates the detrimental effects of nonuniformities in liquid flow and stagnant zones. This gives an idea of the incentive to improve the hydraulic behavior of circular-section trays.

Literature cited

AIChE, Bubble Tray Design Manual, Am. Inst. Chem. Engrs., New York (1958).

Anderson, R. H., G. Garrett, and H. Van Winkle, "Efficiency Comparison of Valve and Sieve Trays in Distillation Columns," Ind. Eng. Chem. Process Dev., 15(1), 96 (1976).

Barker, P. E., and M F. Self, "The Evaluation of Liquid Mixing Effects on a Sieve Plate Using Unsteady and Steady State Tracer Techniques," *Chem. Eng. Sci.*, 17, 541 (1962).

Bell, R. L., "Experimental Determination of Residence Time Distributions on Commercial Scale Distillation Trays Using a Fiber-Optic Technique," AIChE J. 18, 492 (1972a).

——, "Residence Time and Fluid Mixing on Commercial-Scale Sieve Trays," AIChE J, 18, 498 (1972b).

Biddulph, M. W., "Oscillating Behavior on Distillation Trays. II," AIChE J. 21, 41 (1975a).

——, "Multicomponent Distillation Simulation—Distillation of Air," AIChE J. 21, 327 (1975b).

Billet, R., S. Conrad, and C. M. Grubb, "Some Aspects of the Choice of Distillation Equipment," Int. Chem. Eng. Symp. Ser. No. 32, 5:111 (1969).

Dribika, M. M., I. G. Rashed, and M. W. Biddulph, "Vapor-Liquid Equilibria in the Ternary System Methanol-Ethanol-Isopropanol at Atmospheric Pressure," J. Chem. Eng. Data, (1985).

Ellis, S. R. M., and J. T. Shelton, "Interstage Heat Transfer and Plate Efficiency in a Bubble-Plate Column," *Int. Chem. Eng. Symp. Ser.* No. 6, 171 (1960).

Fair, J. R., in B. D. Smith, ed., Design of Equilibrium Stage Process, McGraw-Hill, New York, pp. 545-547 (1963).

Gmehling, J., and U. Onken, "Vapor-Liquid Equilibrium Data Collection," Chemistry Data Series, Dechema, Frankfurt, 1, pt. 2a, 54 (1977).

Haselden, G. G., and R. M. Thorogood, "Point Efficiency in the Distillation of the Oxygen-Nitrogen-Argon System," *Trans. Inst. Chem. Engrs.*, 42, T81 (1964).

- Hay, J. M., and A. I. Johnson, "A Study of Sieve-Tray Efficiencies," AIChE J, 6, 373 (1960).
- Hubner, W., "Der Einflub der Konzentration Auf das Verstarkungsverhaltnis Von Rektifizierboden," Chem. Ing. Techn. 44(8), 546 (1972).
- Hughmark, G. A., "Models for Vapor-phase and Liquid-phase Mass Transfer on Distillation Trays," AIChE J, 17(6), 1,295 (1971). Johnson, A. I., and J. Marangozis, "Mixing Studies on a Perforated Dis-
- tillation Plate," Can. J. Chem. Eng., 36, 161 (1958).
- Jones, J. B., and C. Pyle, "Relative Performance of Sieve and Bubble-
- Cap Plates," Chem. Eng. Progr., 51(9), 424 (1955). Kastanek, F., and G. Standart, "Efficiency of Selected Types of Large Distillation Trays at Total Reflux," Sep. Sci., 2(4), 439 (1967).
- Kern, D. Q., Process Heat Transfer, McGraw-Hill, New York, p. 815 (1950).
- Kirschbaum, E., "Hochleistungs-Rektifizier-Siebboden," Chem. Ing. Techn., 34(4), 283 (1962).
- Lashmet, P. K., and S. Z. Szczepanski, "Efficiency Uncertainty and Distillation Column Overdesign Factors," Ind. Eng. Chem. Process Des. Dev., 13(2), 103 (1974).
- Lim, C. T., K. E. Porter, and M. J. Lockett, "The Effect of Liquid Channelling on Two-Phase Distillation Plate Efficiency," Trans. Inst. Chem. Engrs., 52, 193 (1974).
- Lockett, M. J., and I. S. Ahmed, "Tray and Point Efficiencies from a 0.6 meter Diameter Distillation Column," Chem. Eng. Res. Des., 61, 110 (1983).
- Lockett, M. J., C. T. Lim, and K. E. Porter, "The Effect of Liquid Channelling on Distillation Column Efficiency in the Absence of Vapor Mixing," Trans. Inst. Chem. Engrs., 51, 61 (1973).
- Lockett, M. J., and A. Safekourdi, "The Effect of the Liquid Flow Pattern on Distillation Plate Efficiency," Chem. Eng. J., 11, 111 (1976). Maripuri, V. O., and G. A. Ratcliff, "Measurement of Isothermal
- Vapor-Liquid Equilibria for Acetone-n-Heptane Mixtures Using Modified Gillespie Still," J. Chem. Eng. Data, 17, 366, (1972).
- Mayfield, F. D., et al., "Perforated-Plate Distillation Columns," Ind. Eng. Chem., 44, 2,238 (1952).
- Nutter, D. E., "Ammonia Stripping Efficiency Studies," AIChE Symp. Ser. No. 12, 68(124), 73 (1971).
- Porter, K. E., M. J. Lockett, and C. T. Lim, "Effect of Liquid Channell-

- ing on Distillation Plate Efficiency," Trans. Inst. Chem. Engrs., 50, 91 (1972).
- Rahman, M. A., and M. J. Lockett, "Efficiency Reduction of Large Distillation Trays by Entrainment," Int. Chem. Eng. Symp. Ser. No. 61, 111, (1981).
- Reid, R. C., J. M. Prausnitz, and T. K. Sherwood, The Properties of Gases and Liquids, 3rd ed. McGraw-Hill, New York, pp. 631, 635
- Rush, F. E., and C. Stirba, "Measured Plate Efficiencies and Values Predicted from Single-Phase Studies," AIChE J. 3(3), 336 (1957).
- Sakata, M., and T. Yanagi, "Performance of a Commercial-Scale Sieve Tray," Int. Chem. Eng. Symp. Ser. No. 56, 3.2/21 (1979).
- Shilling, G. D., and G. H. Beyer, "Bubble-Plate Efficiencies in Ethanol-Water Fractionation," Chem. Eng. Progr., 49(3), 128 (1953).
- Shore, D., and G. G. Haselden, "Liquid Mixing on Distillation Plates and Its Effect on Plate Efficiency," Int. Chem. Eng. Symp. Ser. No. 32, 2:54 (1969).
- Stichlmair, J., Grundlagen der Dimensionierung des Gas/Flussigkeit-Kontaktapparates Bodenkolonne, Weinheim: Verlag Chemie, pp. 107-110, 133-144, (1978).
- Strand, C. P., "Bubble Cap Tray Efficiencies," Chem. Eng. Prog., **59**(4), 58 (1963).
- Thomas, W. J., and M. A. Hag, "Studies of the Performance of a Sieve Tray with 3/8" Diameter Perforations," Ind. Eng. Chem. Process Des. Dev., 15(4), 509 (1976).
- Weiler, D. W., W. V. Delnicki, and B. L. England, "Flow Hydraulics of Large-Diameter Trays," Chem. Eng. Progr., 69, 67 (1973).
- Yanagi, T., and M. Sakata, "Performance of a Commercial-Scale 14% Hole Area Sieve Tray," Ind. Eng. Chem. Process Des. Dev., 21(4), 712 (1982).
- Zuiderweg, F. J., and A. Harmens, "The Influence of Surface Phenomena on the Performance of Distillation Columns," Chem. Eng. Sci., 9,
- Zuiderweg, F. J., H. Verburg, and F. A. H. Gilissen, "Comparison of Fractionating Devices," Int. Chem. Eng. Symp. Ser. No. 6, 202 (1960).

Manuscript received Apr. 8, 1985, and revision received Oct. 24, 1985.