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Analysis and Evaluation of Periodic Separations Using COPS Trays

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Stripping of dilute aqueous ammonia solutions with air, using an in-house developed cyclically operated column, has been explored in the past by our group [Toftegård B. et al., 2016. Ind. Eng. Chem. Res., 55 (6): 1720]. The column uses novel so-called cyclically operated perforated sheet (COPS) trays with a Sulzer Mellapak^{TM} layer on top to prevent oscillations and enhance point efficiency. The column operates by the principle of sequential liquid draining. This enables continuous vapor flow, thereby avoiding interrupting the vapor flow during the liquid flow period, and allows the feed streams for distillation systems to be two-phased. It also means that, when applied to distillation operations with reboilers, one can avoid the necessity of special precautions (such as large control valves) against pressure dynamics possibly resulting from having to interrupt the vapor flow rate, during liquid flow. In this paper, an extended range of operation is explored with the realization mentioned above. The proposed periodic trays are operated – without weeping problems - at higher holdups with column efficiencies approaching 200%. This exceeds greatly past results obtained with other internals, such as conventional sieve trays. Also point efficiencies were determined for a series of combinations of liquid hold up and gas flow with the objective of validating the Lewis case 2, which forms the basis of periodic separation process. The COPS tray efficiency was almost twice as large as those of conventional sieve trays.

1. Introduction

Research communities and practicing engineers have for long paid attention to the costs of separation, and the influence these costs have on process economy. Developing trays of higher efficiency has significant scope in this context. Trays with greater efficiency allow the construction of less tall towers. This is advantageous in its own right, since not only the additional material costs extend the expenses associated with a taller tower. Gains from tray efficiency enhancements can also be invested in decreasing operational expenses. To accomplish the same separation, lower internal flows should be required, with the same number of trays, when these are more efficient. Thus, there are several incentives to develop high-efficiency trays for fluid separations. Particularly, trays that compared to conventional trays, are not much more expensive in terms of material and development costs. Impacts on ease-of-operation, maintenance requirements and capacity are also important considerations.

1.1 Lewis Cases

Lewis (1936) analyzed relationships among (tray and point) efficiencies of bubble cap trays based on models of cross flow situations. Since the length of the liquid flow path across a tray is usually much greater than the weir height, it is much more difficult to achieve full liquid mixing in the liquid flow direction than it is to achieve it in the vapor flow direction. As a consequence, it is commonly assumed that liquid is fully mixed in the direction of vapor flow, but not in the direction of liquid flow. Vapor entering an intermediate tray is probably neither fully mixed (as in Case 1), nor completely unmixed (Cases 2-3) in the liquid flow direction. Nevertheless, these were the limiting situations analyzed by Lewis. He found that the overall tray efficiency was higher than the local point efficiency (compositions exiting and entering at a single point) in conventional distillation equipment. Overall tray efficiencies for various tray configurations could in addition be expressed as functions

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of point efficiency, E_P, and the ratio, λ (dimensionless), of the slopes of the equilibrium and operating lines. An important conclusion was that if downcomers could be developed to realize parallel liquid flow patterns on trays (corresponding to the Lewis Case 2), it would lead to higher tray efficiencies, than the conventional counter-current flow patterns (Lewis Case 3). As noted, three decades later, by Sommerfeld et al. (1966) the Case 3 is probably most common because of designers' understandable reluctance to install downcomers in the configuration of Case 2'.

1.2 Cyclic Mode of Operation

Material balance principles applied to a tray with perforations (no downcomers) as the only avenue for passing of vapors, leads to an ordinary differential equation in time. That ordinary differential equation is identical to the one obtained for the liquid spatial concentration profile on a conventional tray, if time and horizontal position are interchanged as independent variables. As a consequence, periodic separation becomes a means of realizing the Lewis Case 2. The realization of this principle has been explored by several investigators during 1960-1990, when many programs were terminated. The challenge in addition to demonstrating theoretical performance, is to realize a device which is convenient to operate and not excessively more expensive than conventional means.

2. Background

2.1 Previous Efforts

Cannon (1961) has often been given credit for the first experimental results on periodic separation. His realization formed the basis of later generations. A problem with early implementations was non-uniform draining of trays during the liquid flow period. Thus, the second generation (published during the 1980s) mainly focused on improving the control of liquid flow, to avoid back-mixing and oscillations on trays. Another problem was pressure oscillations, resulting from interrupting the vapor flow from a reboiler. When interuputions happen, presumably huge investments are required to ensure pressure control. Toftegaard (2016) proposed to solve this problem by avoiding the interruption of the vapor flow during the liquid flow period. This is made possible by inventing the principle of sequential draining, as suggested by Toftegaard and Jorgensen (1988) – as opposed to simultaneous draining which is the conventional approach to liquid flow control. The present paper continues the previous experimental efforts of studying periodic stripping of aqueous ammonia solutions. The advancement is a better exposition of the relationship between point and tray efficiencies.

2.2 Experimental Setup

Our experimental setup has been described previously (Toftegård et al., 2016), in great detail, so only the most essential elements are repeated here. The column is 3500 mm tall. It is made in stainless steel, with a diameter of 470 mm. Feed mixtures are prepared in a 400 L tank, by mixing one liter of 25% ammonia solution into 400 L distilled water, under stirring. The result is a feed concentration near 0.03 M. The column may be equipped with either three sieve trays or three periodic trays. In both cases, the spacing is 500 mm. The volumetric rate of liquid may be varied up to about 40 L/min. Air flow is supplied by an 11 kW centrifugal blower. This produces a maximum volumetric flow of 0.4 m^3/s. The maximum air velocity is 3.2 m/s. A frequency converter is located on the blower to control the gas flow rate. The pressure drop across an orifice is used to measure of the gas flow rate. The pressure drop is input to a PI-control loop manipulating the frequency of the frequency converter.

2.3 Measurements

Measurements are logged by a computer, which also is used for controlling the process. The essential measurements are the air flow rate, the liquid hold up and liquid concentrations. Measurements are sampled through each experiment such that variances can be determined. Liquid hold ups are determined by collecting outlet liquid in buckets. When the column operates with periodic trays, the liquid flow rates are determined by weighing the holdup and dividing the weighed amount by the period time. Concentrations of ammonia are measured for feed and product streams by hydrogen chloride titration using methyl red as indicator.

2.4 Trays

The conventional sieve trays employed have hole diameters of 2 mm, a spacing of 500 mm and a weir height of 5 cm. The periodic cycling trays employed also have hole diameters of 2 mm. A 100 mm layer of Mellapak packing with a porosity of 92% and a specific area of 500 m^2/m^3 is located above each periodic tray. The Mellapak packing layer serves two purposes: One is to avoid oscillations during the vapour flow period. This

could also be achieved with simpler baffles (eight-arm asterisk baffles were explored). However, Toftegård (1989) observed that adding the Mellapak packing enhanced the point efficiency of the trays over baffles, so even though this choice is undoubtedly more expensive, it is preferred here. Later, packing layers located above tray decks, below tray decks, on tray decks, and in the downcomers of trays were explored by Chuang of University of Alberta in the mid-to late-1990's (see Xu et al., 1996) into the early 2000's (Urbanski, 2016). Our trays differ from those of Maleta et al. (2011) and BASF (Buetehorn et al., 2015; Knoesche et al., 2016), since no sluice chambers are used. To avoid back mixing, it is advantageous to enforce sequential tray draining, where trays are emptied sequentially rather than simultaneously, while the vapor flow is maintained throughout. The combination of these trays with the sequential draining principle has a set of advantages (Toftegård, 1989).

3. Experimental Work and Data Analysis

3.1 Column Experiments

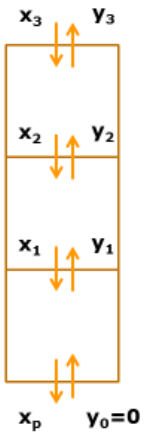
We have combined full column experiments (3 trays) reported by Toftegård (2016) with a set of new column experiments at higher hold up to gas flow ratios. Tray efficiency, E_T, has been determined, by solving the equation set summarized in Figure 1. We assume straight operating and equilibrium lines, which is valid since the feed solutions are quite dilute. It has been assumed that all three trays have the same efficiency. This may not be correct, since the bottom tray is in fact a Lewis Case 1 tray, whereas the other trays are Lewis Case 2 trays. Combining the equations in Figure 1 gives,

$$\frac{x_f}{x_p} = \left[\lambda E_T (1 + \lambda E_T - E_T)^2 + \lambda E_T (1 + \lambda E_T - E_T) + \lambda E_T + 1 \right] \quad (1)$$

To facilitate calculations, the following quantities are then calculated (*e*, *u* and *w* are all dimensionless),

$$e = E_T \quad , \quad u = \lambda E_T \quad , \quad w = 1 + \lambda E_T - E_T = 1 + u - E_T \quad , \quad \frac{x_f}{x_p} = uw^2 + uw + u + 1 \quad (2)$$

Above here, λ, is the ratio of slopes of operating and equilibrium curves.



(1) $y_1 - y_0 = (K \cdot x_p - y_0) \cdot E_T$
(2) $x_1 = (G/L) \cdot y_1 + x_p$
(3) $y_2 - y_1 = (K \cdot x_1 - y_1) \cdot E_T$
(4) $x_2 = (G/L) \cdot y_2 + x_p$
(5) $y_3 - y_2 = (K \cdot x_2 - y_2) \cdot E_T$
(6) $x_3 = (G/L) \cdot y_3 + x_p$
(7) $x_3 = x_f$

Specify:

K, x_p, x_f, G, L

Variables:

$x_1, x_2, x_3, y_1, y_2, y_3, E_T$

Figure 1: Tray efficiency, E_T, model to obtain data from column experiment.

3.2 Tray Experiments

Point efficiencies are obtained from a single tray experiment, as done by Gerster and Scull (1970). The point efficiency, E_P (dimensionless), is:

$$y_{out} = E_p \cdot y_{eq} + (1 - E_p) \cdot y_{in} \quad , \quad y_{eq} = m \cdot x \quad (3)$$

y is the mole fraction in the air and x in liquid. Subscript 'out' refers to air leaving the tray, 'in' to air entering the tray, and 'eq' to equilibrium. The separation factor, m (near unity), depends upon temperature, pressure and composition. However, for the present system, only the temperature dependence is significant. m is calculated as done by Toftegård et al. (2016). Since the incoming air has no ammonia, a tray material balance is

$$H\frac{dx}{dt} = -G \cdot y_{out} \tag{4}$$

Here H is the hold up and x is the mole fraction of ammonia. G is the air flow. Introduce the point efficiency,

$$H\frac{dx}{dt} = -G \cdot y_{out} = -E_p \cdot G \cdot m \cdot x \quad \Rightarrow \quad \frac{d\ln x}{dt} = -\frac{E_p \cdot G \cdot m}{H} \tag{5}$$

This shows that the point efficiency can be determined from the slope of a plot of ln x as a function of time, t, without determining vapor concentrations. Here we have sampled at various time intervals and determined the concentrations by titration. The mass transfer may be expressed in terms of $(NTU)_{OG}$ (Number of transfer units). It is related to the point efficiency as follows (King, 1980, p. 613):

$$E_p = 1 - e^{-(NTU)_{OG}} \quad , \quad (NTU)_{OG} = \frac{h}{G} \cdot K_G \cdot P \cdot a \cdot D^2 = \frac{h}{G} \cdot \beta \tag{6}$$

Here h is the liquid height (m). P is pressure, a (m^2/m^3) is the specific phase contact area and D^2 (m^2) is a tray area. K_G is a mass transfer coefficient (consistent units). We need not know specific values of K_G (sor a), since only the beta parameter is determined in what follows. The procedure is to initiate air flow, fill in an appropriate amount of feed solution on the tray. Then, at different time intervals, samples of liquid are taken from the tray and corked. After the experiment the samples are titrated. Various air flow to liquid holdup ratios are examined.

4. Results

4.1 COPS Tray Point Efficiency

The single tray experimental results are summarized in Table 1. The point efficiencies are obtained from slopes of ln x versus time, based on samples taken at different points of time with concentrations determined by titration.

Table 1: Single Tray Experimental Results

Experiment	H/G (s^{-1})	±	E_p	h/G (s·m/mol)
S1	104.12	2.57	0.92	1.30E-02
S2	86.05	1.95	0.79	1.07E-02
S3	84.05	1.5	0.94	1.05E-02
S4	77.57	1.56	0.81	9.65E-03
S5	73.44	0.78	0.79	9.14E-03
S6	68.24	1.18	0.59	8.49E-03

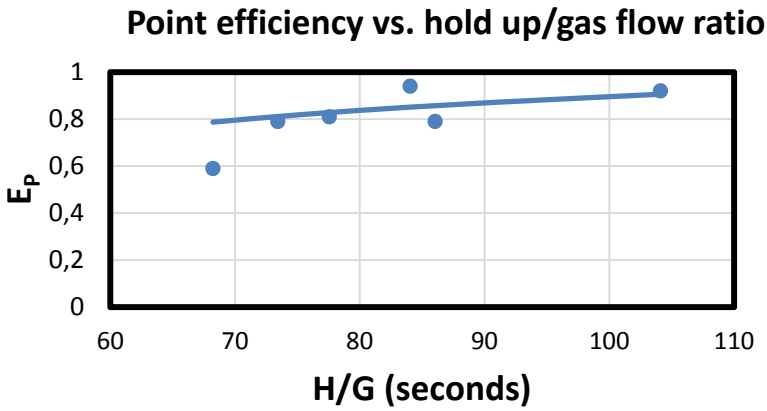


Figure 2: Point Efficiency as function of tray loading. Symbols are data from Table 1. The curve is the model.

The data were interpreted using the expression relating the point efficiency to the number of transfer units:

$$\underbrace{-\ln(1-E_p)}_{y} = \underbrace{\frac{h}{G}}_{x} \cdot \underbrace{K_G \cdot P \cdot a \cdot D^2}_{\beta} \tag{7}$$

While assuming this linear relationship is a simplification, scatter in the data (Figure 2) suggests that it may suffice for the present purpose. The measured point efficiencies increase with loading, as theory suggests, but only qualitative model representation is possible. We have tried to detect and delete outliers based on analysis of Studentized residuals. However, that does not substantially change the model parameter, β (mol/m·s).

4.2 COPS Tray Efficiency from Column Operation Results

Table 2: Sieve Tray Experiments (Toftegård et al., 2016).

Experiment	T (°C)	L (mol/s)	G (mol/s)	m	x_f	x_p	E_T
C1	22.5	12.2	8.2	0.84	0.0007	0.0004	0.81
C2	22.5	7.2	8.2	0.84	0.0007	0.0003	0.74
C3	22.7	21.4	8.2	0.85	0.0007	0.0005	0.88
C4	24.5	4.4	8.2	0.92	0.0005	0.0001	0.56
C5	25.2	3.4	8.2	0.95	0.0005	9E-05	0.53
C6	25.5	2.3	8.1	0.96	0.0005	6E-05	0.46
C7	25.5	2.6	6.2	0.96	0.0005	8E-05	0.59
C8	25.1	17.6	8.2	0.95	0.0006	0.0004	0.79
C9	24.7	27.1	8.2	0.93	0.0006	0.0005	0.84
C10	25	17.3	6.2	0.94	0.0006	0.0004	0.84
C11	24.9	8.7	6.2	0.94	0.0006	0.0003	0.73
C12	24.6	4.8	6.2	0.93	0.0006	0.0002	0.62
C13	24.7	2.6	6.2	0.93	0.0006	9E-05	0.62

The column experimental results of Toftegård et al. (2016) for sieve trays are summarized in Table 2. For the periodic 3-tray column, we combine the data of Toftegård with newly obtained data in Table 3. These have λ-values not far from unity. Tray efficiencies are obtained from the linear equation systems of Figure 1, with the product/feed stream purities and λ-values as input. The first striking observation is how much higher the periodic tray efficiencies are compared to those for sieve trays. E_P is determined from the tray hold up and gas flow.

Table 3: Periodic Tray Experiments.

TP (s)	L (mol/s)	G (mol/s)	x_f	x_p	E_P	H (mol)	h (m)	λ	E_T/E_P	E_T
90	4.2	4.2	2.70E-03	7.03E-04	0.88	396.7	0.049	0.80	1.94	1.71
40	8.8	8.3	5.09E-04	1.70E-04	0.66	396.0	0.049	0.83	1.42	0.94
80	3.9	4.2	2.70E-03	6.49E-04	0.84	335.3	0.042	0.84	1.94	1.62
90	4.2	4.9	2.70E-03	4.68E-04	0.84	398.3	0.050	0.94	2.26	1.90
80	4.1	4.9	2.70E-03	4.50E-04	0.80	349.4	0.043	0.96	2.35	1.88
90	4.3	5.3	2.70E-03	4.68E-04	0.83	409.4	0.051	0.98	2.04	1.69
70	7.4	8.3	5.94E-04	1.31E-04	0.78	552.8	0.069	1.01	1.48	1.15
80	4.1	5.3	2.70E-03	4.68E-04	0.77	345.0	0.043	1.04	1.87	1.45
70	3.2	4.2	2.70E-03	4.50E-04	0.72	236.9	0.029	1.05	2.01	1.46
55	5.3	7.0	6.30E-04	1.38E-04	0.64	317.4	0.039	1.07	1.62	1.04
47	5.9	7.0	6.30E-04	1.48E-04	0.63	308.9	0.038	1.08	1.47	0.93
70	3.9	5.3	2.70E-03	4.68E-04	0.71	289.4	0.036	1.09	1.82	1.29
70	4.8	7.0	6.30E-04	1.12E-04	0.69	361.5	0.045	1.13	1.71	1.18
70	3.4	4.9	2.70E-03	4.50E-04	0.69	256.1	0.032	1.15	1.74	1.20
60	5.9	8.4	6.22E-04	8.05E-05	0.65	383.5	0.048	1.38	1.70	1.10
100	4.4	6.9	6.22E-04	7.19E-05	0.78	465.2	0.058	1.39	1.53	1.20
70	5.3	8.3	6.22E-04	6.47E-05	0.66	394.5	0.049	1.46	1.81	1.19
100	4.3	6.9	6.22E-04	6.28E-05	0.77	447.3	0.056	1.50	1.52	1.17
100	3.5	7.0	6.30E-04	6.17E-05	0.70	367.5	0.046	1.52	1.68	1.17
85	3.7	7.0	6.30E-04	5.34E-05	0.66	329.4	0.041	1.61	1.80	1.18
80	4.4	8.4	5.09E-04	3.74E-05	0.64	373.2	0.046	1.63	2.00	1.27
70	4.2	8.3	5.94E-04	3.87E-05	0.57	312.0	0.039	1.83	2.02	1.15

Figure 3 shows that the λ-values cover ranges from 0.8 to 2. The point efficiencies are in general less than unity. The ratios of tray to point efficiencies are located in the range from 1.5 to 2 (in a few cases exceeding 2).

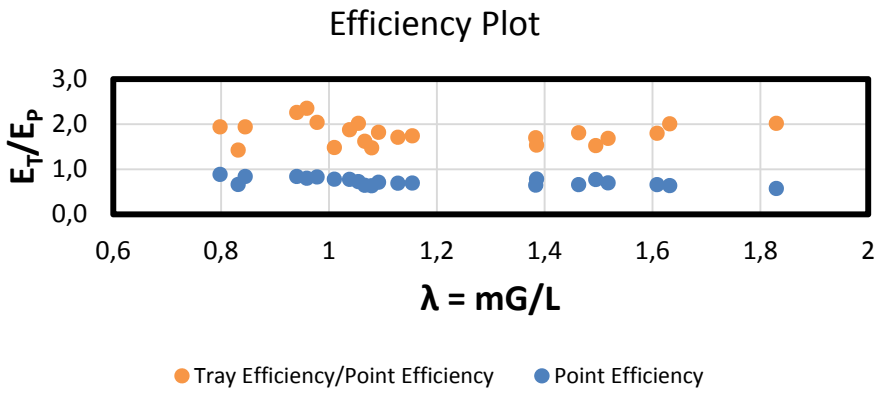


Figure 3: Ratio of tray to point efficiency (yellow) and point efficiency (blue) as function of tray loading.

4.3 Discussion

The result of the Lewis Case 2 analysis is not in general that the ratio of tray to point efficiency must be near 2. In fact, this conclusion is restricted to the situation where $E_P = \lambda = 1$. In the present cases, the point efficiencies are less than unity. Therefore, the ratio should also be less than unity. This tendency is seen in the data. When λ exceeds unity the (E_T/E_P)-ratio should increase. This may be possible to validate, but further investigations are needed to do so. However, based on the data in Figure 3, the theoretical performance is within reach.

5. Conclusions

We have explored the relation between point efficiency and tray efficiencies of periodic COPS trays to demonstrate the theoretical performance predicted from the Lewis Case 2 analysis. The analysis is qualitatively confirmed, though less noisy data would be desirable. Current efforts are directed towards establishing improved instrumentation, temperature control, mixing of feed solutions and data logging. A thorough determination of operating windows of periodic and sieve trays is planned.

References

Buetehorn S., Paschold J., Andres T., Shilkin A., Knoesche C., 2015. "Impact of the duration of the vapor flow period on the performance of a cyclic distillation", Chemie-ingenieur-technik, 87(8), 1070.

Cannon M.R., 1961, Controlled Cycling Improves Various Processes, Ind. Eng. Chem., 53, 629.

Gerster J.A., Scull H.M., 1970, Performance of Tray Columns Operated in the Cycling Mode, AIChE J., 16, 108.

King C.J., 1980. Separation Processes, Wiley.

Knoesche C., Paschold J., Buetehorn S., 2016. “Cyclic Operation of Distillation Columns”, Paper 87b, AIChE Spring Meeting, Houston, Texas, USA.

Lewis Jr. W.K., 1936, Rectification of Binary Mixtures, Ind. Eng. Chem., 28, 399.

Maleta V.N., Kiss A.A., Taran V.M., Maleta B.V., 2011, "Understanding process intensification in cyclic distillation systems", Chemical Engineering and Processing 50 (2011) 655–664.

Sommerfeld J.T., Schrodt V.N., Parisot P.E., Chien H.H., 1966, Studies of Controlled Cyclic Distillation: I. Computer Simulations and the Analogy with Conventional Operation, Sep. Sci., 1, 245.

Toftegård, B.; Jørgensen, S.B., 1988. Operational Principles for Periodic Cycled Separation. In Distillation and Absorption 87 (Brighton, UK); Haselden, G.G., Ed.; Symposium Series 104; IChemE: Rugby, pp A473−A482.

Toftegård B., Clausen C.H., Bay Jørgensen S., Abildskov J., 2016, New Realization of Periodic Cycled Separation, Ind. Eng. Chem. Res., 55(6), 1720.

Toftegård B., 1989. Periodic Cycled Separation, PhD thesis, Technical University of Denmark Department of Chemical and Biochemical Engineering, Kgs. Lyngby, Denmark.

Urbanski N., 2016. Personal Communication.

Xu Z.P., Afacan A., Chuang K.T., 1996, Prediction Of Packed Sieve Tray Efficiency In Distillation, Trans IChemE, Vol 74, Part A, November 1996.