Mass Transfer on a Single Sieve Plate Column Operated With Periodic Cycling

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A single sieve plate column operated with periodic cycling was mathematically modeled using the assumption of zero weeping rate and plug flow of liquid during the liquid drain period. Zero weeping rates could only be obtained by introducing baffles to control fluid oscillations on the plate. Experimental measurements of the ratio of the slopes of the equilibrium and operating lines λ, the Murphree plate efficiency ϵ, the fraction of the liquid holdup transferred per cycle η, and the separation factor X on a computer controlled column provided the first reliable confirmation of the improvements in separating ability, predicted by the theory.

SCOPE

The mathematical modeling of the unsteady state mass transfer processes on a sieve plate column demonstrates that larger mass transfer driving forces are available than with steady state operation. These larger driving forces result in improved rates of mass transfer and superior overall column performance. The magnitude of the improvements can be quantitatively measured by comparing the number of plates required in the unsteady state case with the steady state case. For normal operating flow conditions, the theory predicts that the steady state case will require twice the number of plates. The theoretical advantages of using unsteady state conditions lead to columns containing 50% of the expected number of plates. These major improvements in reducing column height and capital expenditure exceed those available by improved plate contacting efficiency or modifications to the plate geometry.

Mass balances satisfying a list of simple assumptions are used to describe the unsteady state processes occurring during the vapor-liquid contacting time. However, to achieve liquid flow through the column, it is necessary to reduce the vapor flow rate to zero for short periods to allow liquid to transfer to the plate below. The movement of liquid in the column during this liquid drain period can be described by the discrete residence time distribution (DRTD). The most desirable DRTD is for ideal plug flow, when all of the liquid holdup on a plate is transferred to the plate below, with no mixing with any other liquid stream. Multiple sieve plate columns have been shown to have a spread about the mean time in the DRTD and in an unmodified form do not satisfy the plug flow assumption required by the theory during the liquid drain period. The spread in the DRTD provides a quantitative measure of departures from plug flow and accounts for the inability of previous experimental results on distillation columns to achieve the separation predicted by the theory.

The only column configuration which provides zero spread in the DRTD and satisfies the plug flow assumption is a single sieve plate column. In addition to this advantage, the single sieve plate can be used to obtain the plate efficiency, an unknown parameter in the theoretical solution. The single sieve plate geometry provides the basic experimental equipment for the testing of the theory of periodic cycling of plate columns.

CONCLUSIONS AND SIGNIFICANCE

The theory of periodic cycling of plate columns has been developed for the linear equilibrium case using a number of assumptions, the most important being plug flow transfer of liquid during the LDP and no weeping during the VFP. A single sieve plate is the only column with a DRTD that has plug flow transfer of liquid under all operating conditions. A periodically cycled single sieve plate requires flow stabilization to control oscillations of the vapor-liquid mixtures on the plate to ensure the absence of weeping. A parallel plate baffle was found to be very effective in maintaining zero weeping rates with large liquid holdups. A baffled single sieve plate satisfies these two most important assumptions in the theory.

An on-line computer controlled the operation of the periodically cycled column by accurate regulation of the timing and valve switching sequences. However, its most important control action was to maintain constant liquid holdups at the start of each cycle through a level control algorithm. Preliminary experiments were made to obtain the parameters in the theory, and the plate efficiency ϵ was measured by an unsteady state mass transfer experiment. Values of λ and η were measured during the periodic cycling of a baffled single plate column for the stripping of ammonia from an aqueous ammonia solution with air. The separation factor X was calculated from the measured liquid composition for η ranging from 0.208 to 1.

The theory of a periodically cycled single plate column provides the equivalent number of conventional plates to achieve the same separation:

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$$\lim_{\lambda \to 1} N_{SS} = \frac{1 - e^{-\epsilon\eta}}{\epsilon\eta(e^{-\epsilon\eta})}$$

$$\lim_{\lambda \to 1} N_{SS} = N_{SS}(\epsilon\eta)$$

For values of ϵ and η measured in the experiment, values of N_{SS} (theory) range from 1.081 to 1.481. Thus, a single periodically cycled plate of efficiency ϵ could be expected to perform as 1.481 conventional plates. The experimental measurements of X lead to the experimental value of N_{SS}, that is, N_{SS} (Expt), and values of 1.481 were obtained. The good agreement between theory and experiment was maintained over fourteen runs when η varied from 0.208 to 1. This is the first reliable confirmation of the improvements in the separating ability of plate columns operated with periodic cycling.

In these gas stripping experiments, plate efficiencies of 74% were measured at large liquid holdups. However, for distillation plate, efficiencies approaching 100% could be expected. With $\epsilon = 1$, the baffled single sieve plate could be expected to perform at 1.7183 conventional plates for $\lambda = 1$ and $\eta = 1$.

Confirmation of the theory for a baffled single sieve plate can be extended to multiple sieve plates, provided weeping is negligible during the VFP and the spread in the DRTD can be reduced and approaches plug flow.

The application of on/off control of plate columns in distillation and gas absorption has produced a number of conflicting views as to whether there are separation improvements as predicted by the theory. McWhirter and Lloyd (1963) distilled mixtures in a 150 mm diameter column containing five plates. Each plate consisted of 75 mm of protruded metal packing held between screens. An overall column efficiency of 200% was measured under certain operating conditions, which indicated a good agreement with the theory. However, to confirm the theory, values of the plate efficiency ϵ and the fraction of the plate holdup η transferred per cycle needed to be measured. This early encouraging experiment was followed by Schrodt et al. (1967) who distilled acetone-water mixtures in a 300 mm diameter column with fifteen sieve plates. Their results indicated low average plate efficiencies. Neither ϵ nor η were measured in these experiments. Gerster and Scull (1970) stripped an aqueous ammonia solution with air in a 150 mm diameter column fitted with four sieve plates. The experimental procedure included preliminary experiments to obtain ϵ and the determination of η from the total liquid holdup and the mass of liquid admitted to the column per cycle. Plate efficiencies of 52% were measured, and η was varied from 0.3 to 2.0 with columns containing two and four plates. A considerable improvement in column separating performance was measured, but it was lower than the theoretical values. A parameter derived from an ideally mixed tank concept had to be added to supply a closer agreement with the theory. It is doubtful whether this type of mixing model is appropriate to liquid flow in the liquid drain period.

We can summarize the literature by stating that the high overall column efficiencies predicted by the theory have not been obtained in carefully controlled experiments where λ, ϵ, and η have all been measured. The departures from the theory can be described as departures from liquid plug flow, an assumption in the theory. The early experiments of McWhirter and Lloyd (1963) using an unusual packed plate design must have reduced the spread in the DRTD to approach plug flow in order to achieve their high overall column efficiencies. There is a need for a careful test of the theory of periodic cycling, and this can be achieved by measurements on a single sieve plate. Once the theory has been confirmed for this ideal case when the DRTD is always plug flow, then the theory can be extended to multiple sieve plates where there is a spread in the DRTD.

THEORY

Chien et al. (1966) used vector-matrix and Laplace transform techniques to derive analytical expressions for the composition vector in a periodically cycled distillation column. In a gas absorption or gas stripping column, a number of assumptions can be made which considerably simplify the analysis. The most important is the use of an equilibrium line of constant slope which is valid under isothermal conditions and the absence of mass balance equations for the reboiler and accumulator.

The theory of periodic cycling of plate columns is based on unsteady state mass balances during the vapor flow period (VFP), mass balances during the liquid drain period (LDP), and an equilibrium relationship. To obtain an analytical solution of these equations, for a linear equilibrium relationship the following occur:

1. Constant vapor flow rate V during the VFP.
2. Constant vapor inlet composition y_{n+1} during the VFP.
3. Negligible vapor holdup.
4. Constant plate efficiency ϵ.
5. Mass transfer between the phases only during the VFP.
6. Negligible entrainment or weeping.
7. Constant liquid inlet composition x_o.
8. Constant liquid holdups from cycle to cycle.
9. Plug flow of liquid during the LDP.

The equations can be written in a vector-matrix form for the VFP as

$$\dot{\mathbf{x}}(\theta) = \mathbf{A}\mathbf{x}(\theta) + \mathbf{b}u \tag{1}$$

where

$$\dot{\mathbf{x}}^T(\theta) = [\dot{x}_1(\theta) \quad \dot{x}_2(\theta) \quad \ldots \quad \dot{x}_n(\theta)]$$

$$\mathbf{x}^T(\theta) = [x_1(\theta) \quad x_2(\theta) \quad \ldots \quad x_n(\theta)]$$

$$\mathbf{b}^T = [(1-\epsilon)^{n-1} \quad (1-\epsilon)^{n-2} \quad \ldots \quad (1-\epsilon) \quad 1]$$

$$u = x_{n+1}$$

and

$$\mathbf{A} = \begin{bmatrix} -1 & \epsilon & \epsilon(1-\epsilon) & \cdots & \epsilon(1-\epsilon)^{n-3} & \epsilon(1-\epsilon)^{n-2} \\ 0 & -1 & \epsilon & \cdots & \epsilon(1-\epsilon)^{n-4} & \epsilon(1-\epsilon)^{n-3} \\ \cdot & \cdot & -1 & \cdots & \cdot & \cdot \\ \cdot & \cdot & & \cdots & \cdot & \cdot \\ \cdot & \cdot & & \cdots & \cdot & \cdot \\ 0 & \cdot & & \cdots & -1 & \epsilon \\ 0 & 0 & \cdot & \cdots & 0 & -1 \end{bmatrix}$$

The equations during the LDP can be written as

$$\mathbf{x}'(0) = \mathbf{D}\mathbf{x}'(\theta_v) \tag{2}$$

where

$$\mathbf{D} = \begin{bmatrix} 1 & 0 & \cdot & \cdot & \cdot & \cdot & \cdot \\ \eta & 1-\eta & 0 & \cdot & & & \cdot \\ 0 & \eta & 1-\eta & \cdot & & & \cdot \\ \cdot & \cdot & \cdot & \cdot & & & \cdot \\ \cdot & & & \cdot & 1-\eta & 0 & \cdot \\ \cdot & & & & \eta & 1-\eta & 0 \\ \cdot & \cdot & \cdot & \cdot & \cdot & \eta & 1-\eta \end{bmatrix}$$

The solution of Equations (1) and (2) by the use of Laplace transforms, matrix manipulation, and inversion of the resulting Laplace transforms has been given by Furzer and Duffy (1974). The result can be expressed as the ratio of two determinants which can be evaluated directly without iteration. The most useful form of the solution is an expression for the separation factor X given by

$$X = 1 - x_n'(\theta_v)/x_o'(0) \tag{3}$$

The experimental verification of this theoretical solution requires a good agreement with the listed assumptions. While most of the assumptions can be satisfied with adequate equipment design, the final assumption of plug flow of liquid during the LDP requires special analysis and experimental verification. This has been provided by the discrete residence time distribution (DRTD) analysis of Furzer and Duffy (1976). Measurements of departures from plug flow on a five-plate column were observed by the step response of an input of tracer. It can be shown that departures from plug flow lead to reductions in the separation factor X and are detrimental to the separating ability of the column.

Mathematical models of the liquid mixing during the LDP allow for the generation of the DRTD for parameter sets in the model. For multiple sieve plates, the (2S) model in which fractions a and b of the liquid holdup are transferred to the next two stages below has been successful in describing departures from ideal plug flow. For a single sieve plate, the fractions a and b, instead of falling to the next two stages below, simply fall to the base of the column and are removed to storage. We can accurately model a single sieve plate with a single parameter η, which is defined as the fraction of the liquid holdup leaving the plate per cycle. The DRTD of this model for a single sieve plate does satisfy the plug flow assumption. The single sieve plate requires special theoretical analysis and experimental testing, since it alone satisfies this plug flow assumption, whereas all multiple sieve plates could be expected to have a spread in their DRTD, indicating departures from plug flow.

The mass transfer theory of a single sieve plate is given by Equations (1) and (2) with $n = 1$. The equations for gas stripping reduce to

$$\text{VFP} \quad \frac{dx_1}{d\theta} = -x_1$$

$$\text{LDP} \quad x_1(0) = \eta x_o + (1-\eta)x(\theta_v)$$

where

$$\theta = \lambda\epsilon\eta t/t_v$$

The solution is given by

$$X = \frac{1 - e^{-\theta_v}}{1 - (1-\eta)e^{-\theta_v}} \tag{4}$$

where $\theta_v = \lambda\epsilon\eta$.

The separation factor X for a single sieve plate given by Equation (4) is a function of λ, ϵ, and η; that is, $X = X(\lambda, \epsilon, \eta)$. It is useful to compare a single periodically cycled plate with a single plate operated with continuous flows of liquid and vapor in the conventional manner. The comparison is made with equal time averaged flow rates, equal values of λ, and also with equal plate efficiencies ϵ. The conventional single plate reaches a steady state condition given by

$$X_{SS} = \frac{\lambda\epsilon}{1 + \lambda\epsilon} \tag{5}$$

The magnitude of the value of X for a single periodically cycled plate X_{pc} is greater than X_{SS}, or

$$X_{pc} > X_{SS}$$

A conventional column would require more than one plate to achieve an equal separation factor X, where $X_{pc} = X_{SS}$. The number of plates in this conventional column is given by

$$N_{SS} = \frac{\ln\left\{\dfrac{\lambda - X_{pc}}{1 - X_{pc}}\right\} - \ln\lambda}{\ln[1 + \epsilon(\lambda - 1)]} \tag{6}$$

The ratio of the number of conventional plates to the number of periodically cycled plates is a measure of the separation improvements attainable by periodic cycling. For a single sieve plate with $N_{pc} = 1$, this ratio reduces to

$$\frac{N_{SS}}{N_{pc}} = \frac{\ln\left\{\dfrac{\lambda - X_{pc}}{1 - X_{pc}}\right\} - \ln\lambda}{\ln[1 + \epsilon(\lambda - 1)]} \tag{7a}$$

TABLE 1. PERIODIC CYCLING OF A SINGLE PLATE COLUMN VALUES OF N_{SS} $(\lambda, \epsilon, \eta)$

$\lambda = 0.8$
Plate efficiency

ETA	0.2	0.4	0.6	0.8	1.0
0.1000	1.0082	1.0169	1.0261	1.0358	1.0463
0.2000	1.0165	1.0341	1.0531	1.0735	1.0956
0.3000	1.0249	1.0518	1.0810	1.1130	1.1483
0.4000	1.0834	1.0699	1.1101	1.1546	1.2046
0.5000	1.0420	1.0884	1.1401	1.1984	1.2649
0.6000	1.0507	1.1073	1.1713	1.2445	1.3294
0.7000	1.0594	1.1267	1.2037	1.2931	1.3987
0.8000	1.0683	1.1465	1.2373	1.3443	1.4731
0.9000	1.0773	1.1668	1.2722	1.3984	1.5532
1.0000	1.0863	1.1876	1.3084	1.4554	1.6395

$\lambda = 1.0$
Plate efficiency

ETA	0.2	0.4	0.6	0.8	1.0
0.1000	1.0101	1.0203	1.0306	1.0411	1.0517
0.2000	1.0203	1.0411	1.0625	1.0844	1.1070
0.3000	1.0306	1.0625	1.0957	1.1302	1.1662
0.4000	1.0411	1.0844	1.1302	1.1785	1.2296
0.5000	1.0517	1.1070	1.1662	1.2296	1.2974
0.6000	1.0625	1.1302	1.2037	1.2835	1.3702
0.7000	1.0734	1.1540	1.2428	1.3405	1.4482
0.8000	1.0844	1.1785	1.2835	1.4008	1.5319
0.9000	1.0957	1.2037	1.3259	1.4645	1.6218
1.0000	1.1070	1.2296	1.3702	1.5319	1.7183

$\lambda = 1.2$
Plate efficiency

ETA	0.2	0.4	0.6	0.8	1.0
0.1000	1.0119	1.0235	1.0348	1.0459	1.0568
0.2000	1.0239	1.0476	1.0712	1.0945	1.1178
0.3000	1.0361	1.0725	1.1092	1.1461	1.1831
0.4000	1.0486	1.0982	1.1489	1.2006	1.2532
0.5000	1.0612	1.1247	1.1905	1.2584	1.3284
0.6000	1.0740	1.1520	1.2340	1.3197	1.4090
0.7000	1.0870	1.1802	1.2794	1.3845	1.4954
0.8000	1.1003	1.2092	1.3269	1.4532	1.5879
0.9000	1.1137	1.2392	1.3766	1.5260	1.6870
1.0000	1.1274	1.2700	1.4286	1.6031	1.7931

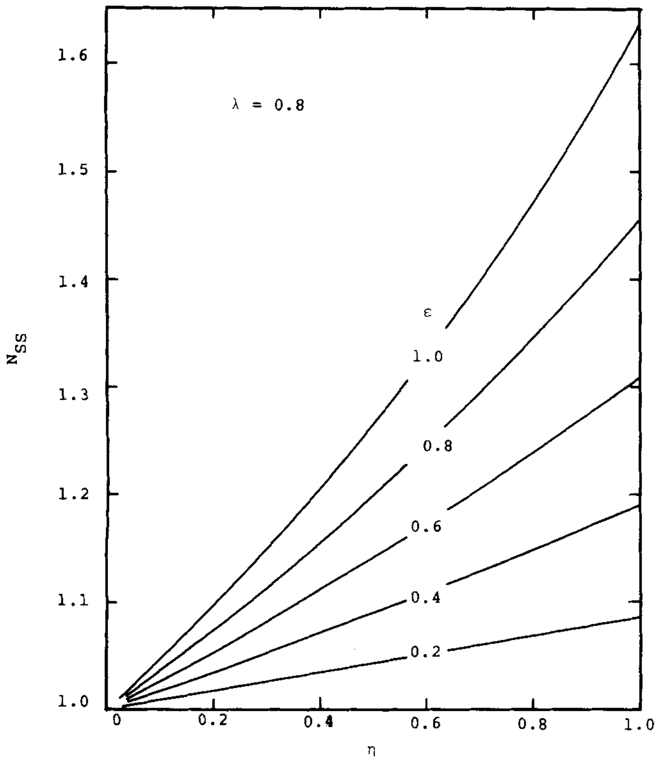

Fig. 1. Number of equivalent steady state plates, single-plate column operated with periodic cycling.

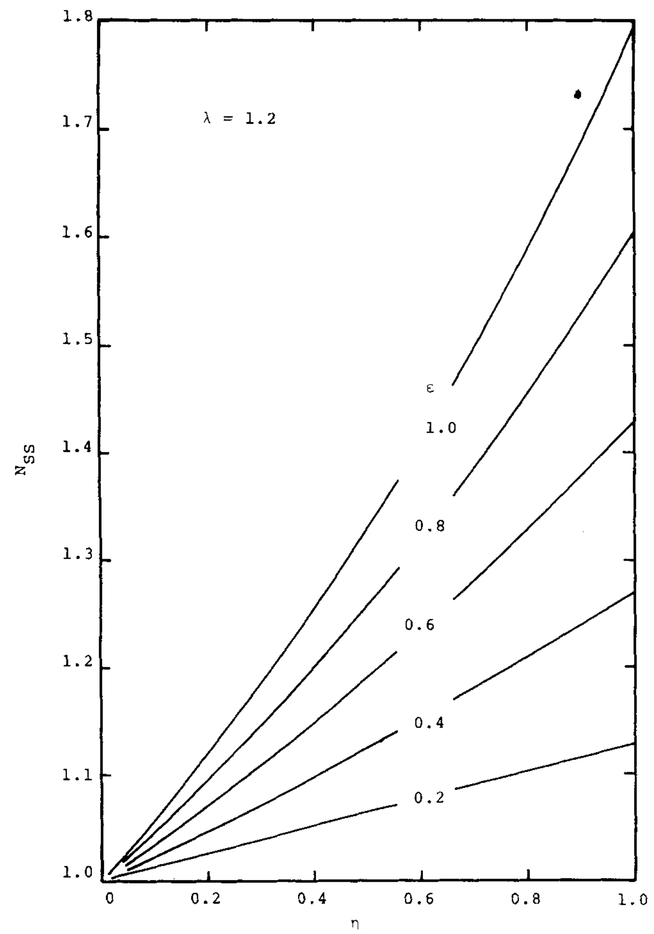

Fig. 2. Number of equivalent steady state plates, single-plate column operated with periodic cycling.

where

$$X_{pc} = \frac{1 - e^{-\lambda\epsilon\eta}}{1 - (1-\eta)e^{-\lambda\epsilon\eta}} \qquad (7b)$$

or

$$N_{SS} = N_{SS}(\lambda, \epsilon, \eta) \qquad (7c)$$

The function $N_{SS}(\lambda, \epsilon, \eta)$ for a single sieve plate is tabulated for $\lambda = 0.8$, 1.0, and 1.2 in Table 1. Figures 1 and 2 show the function for $\lambda = 0.8$ and 1.2, respectively. Figure 3 shows values of $N_{SS}(\lambda, \epsilon, 1)$.

Several limiting solutions are important as $\lambda \rightarrow 1$:

$$\lim_{\lambda \rightarrow 1} N_{SS} = \frac{1 - e^{-\epsilon\eta}}{\epsilon\eta(e^{-\epsilon\eta})} \qquad (8a)$$

$$\lim_{\lambda \rightarrow 1} N_{SS} = N_{SS}(\epsilon\eta) \qquad (8b)$$

Note that ϵ and η appear as product terms, and the solution is given on Figure 4 for the single variable $\epsilon\eta$. For an ideal plate ($\epsilon = 1$) with ideal plug flow of liquid ($\eta = 1$), as $\lambda \rightarrow 1$ we have

$$\lim_{\epsilon\eta \rightarrow 1} \lim_{\lambda \rightarrow 1} N_{SS} = e - 1 = 1.7183 \qquad (9)$$

The theory of the periodic cycling of a single plate with 100% contacting efficiency ($\epsilon = 1$) and ideal plug flow of liquid ($\eta = 1$) during the LDP results in a plate separation that is equivalent to 1.7183 plates in a conventional column when $\lambda = 1$. For values of ϵ and η that differ from 1, the solution is given by Equation (8) which satisfies all the assumptions listed. For values of λ that differ from 1, the solution can be obtained from Equation (7).

One of the other assumptions in the theory requiring confirmation during an experimental program is the absence of weeping. The open geometry of the sieve plate

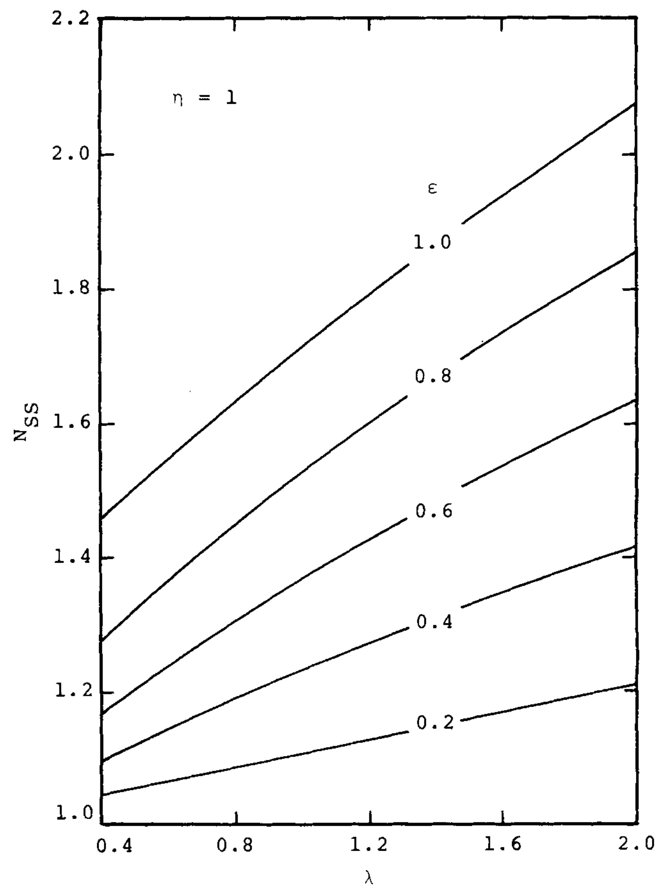

Fig. 3. Number of equivalent steady state plates, single-plate column operated with periodic cycling.

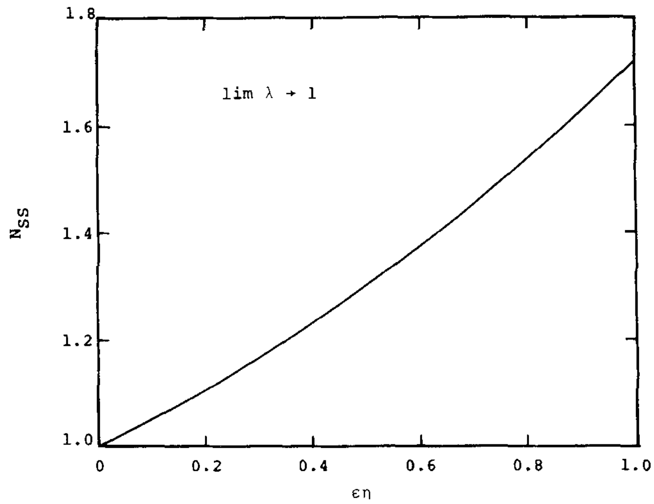

Fig. 4. Number of equivalent steady state plates, limiting case as $\lambda \to 1$.

without downcomers and the absence of a directed liquid flow across the plate allows the froth to exhibit its natural oscillation modes. The resulting wave action and sloshing in this axial symmetric case leads to an early weeping condition, which must be eliminated to satisfy the nonweeping assumption in the theory.

FLUID OSCILLATION

Fluid flow phenomena in conventional sieve plates with downcomers and weirs have been extensively investigated and include studies on pressure drop characteristics, seal and weep points, fluid oscillations on the plate, and transition from froth to spray regimes. Many of the useful results from these studies can be adopted to sieve plates without downcomers as used in columns under periodic cycling. Of particular importance are the fluid oscillations on the plate which can precipitate premature weeping. Previous studies of fluid oscillations on conventional plates began with McAllister et al. (1958) who found fluid oscillations perpendicular to the direction of the liquid flow path across the plate. The frequency of these oscillations was approximately 1 cycle/s and was dependent on the vapor and liquid flow rate. Similar frequencies have been reported by Zanelli and Del Bianco (1973). Biddulph and Stephens (1974) identified three regimes at increasing gas velocities. At low gas velocities, small wavelength oscillations were observed which had an insignificant effect on the froth. At higher gas velocities, the wavelength increased until it was equal to the column diameter at frequencies of 1.2 cycles/s. Still higher gas velocities lead to wavelengths equal to half the column diameter, and the resulting violent sloshing had a frequency of 0.67 cycles/s. Hinze (1965) was able to derive a theoretical expression for the wavelength of stable oscillations on the plate by combining the equations for mass and momentum with sinusoidal variations in froth height and pressure. The increased static head under the crest of these waves can result in an early weeping condition, rather than the condition predicted by bubble formation and pressure fluctuations at an orifice as reported by McCann and Prince (1969). Gerster and Scull (1970) operated a 150 mm diameter column fitted with sieve plates without downcomers and weirs and reported that the fluid oscillation and premature weeping could be eliminated by the introduction of an eggcrate type of baffle 38 mm high.

Preliminary experiments on a 600 mm diameter column with sieve plates without downcomers demonstrated violent sloshing, liquid dumping, and an audible fluid oscillation. Weeping was observed over wide ranges of liquid holdup and gas velocity under these unstable fluid conditions. Obviously, baffles would have to be inserted above the plates to control and dampen the fluid oscillations so as to eliminate weeping and satisfy one of the assumptions in the theory of periodic cycling of plate columns.

Stabilized Baffled Sieve Plates

There are a large number of variables that must be investigated to evaluate the effect of different baffles in reducing fluid oscillation and eliminating premature weeping. The principal variables that have been investigated include baffle types and configuration, types of liquid distributors, free area of the sieve plates, and approaching air velocity distribution. The experimental method consisted of the measurement of the pressure drop across the plate, noting that a falling time-dependent pressure drop is a measure of the weeping rate through the sieve plate. An effective baffle configuration gives a steady pressure drop when weeping is absent.

Baffle Types

A large number of baffle types were investigated, which included parallel plate baffles, two sets of parallel plate baffles (eggcrate pattern), and a layer of 25 mm ceramic lessing rings. The parallel baffles had a height *h*, a separation distance *s*, and the top edge of the baffle was bent through an angle θ, as listed below:

Baffle type number	h(mm)	s(mm)	θ
IV	76	19	0
V	76	19	45 deg
VI	76	19	±45 deg
IX	150	51	0

The square eggcrate pattern baffles had a height *h*, a square dimension *s*, and the top edge bent through an angle θ, as listed below:

Baffle type number	h(mm)	s(mm)	θ
I	76	150	0
II	76	150	45 deg
III	76	150	45 deg (plus 12 mesh wire screen)

Other parallel/eggcrate patterns involved adding *n* cross members at a separation distance *d* to a parallel baffle pattern:

Baffle type number	h(mm)	s(mm)	θ	n	d(mm)
VII	76	19	0	9	38
VIII	76	19	0	2	270
X	150	51	0	4	150

The liquid distributors to the plate included the following types: horizontal entry, 50 mm diam; vertical upwards entry, 50 mm diam; and vertical downwards entry with a deflector plate, 50 mm diam. The free area of the sieve plate was varied from 5.4 to 11.8%.

The gas velocity distribution below the plate varied from a uniform distribution with a variation about the mean of ±5% to a nonlinear distribution with a low gas velocity at the center of the plate.

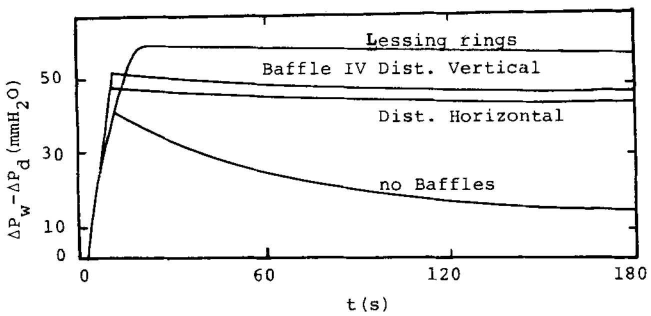

Fig. 5. Weeping characteristics of a baffled sieve plate.

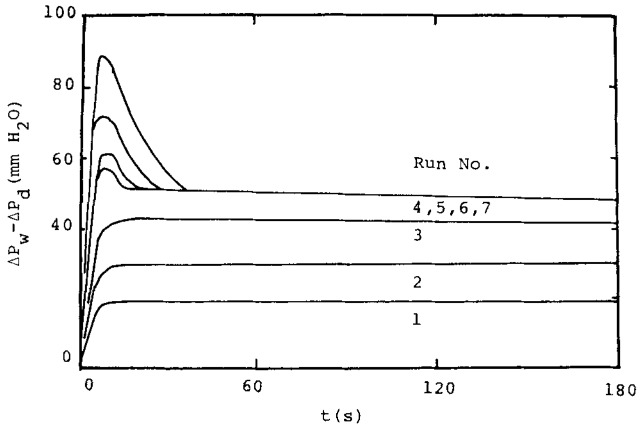

Fig. 6. Weeping characteristics of a baffled sieve plate.

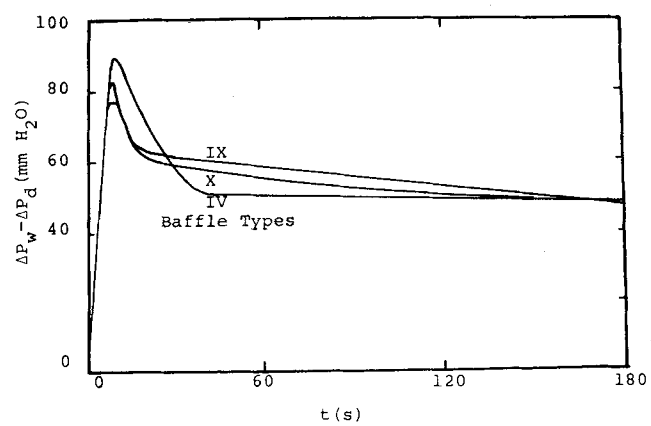

Fig. 7. Weeping characteristics of a baffled sieve plate.

PRESSURE DROP CHARACTERISTICS

The oscillation of the froth was particularly stable, and over thirty different combinations of baffle types, distributors, sieve plates, and approaching air velocity distributions were investigated, as described in detail by Duffy (1976). The time dependent pressure drop characteristics were obtained by the rapid delivery of the initial liquid holdup onto the plate and recording the wet plate pressure drop ΔP_w. The pressure drop of the wet drained plate ΔP_d was subtracted from ΔP_w to provide a measure of the falling liquid holdup and weeping rate.

Baffle types I, II, III, V, and VI all displayed weeping rates in excess of baffle type IV. Figure 5 shows the pressure drop characteristics for an 11.8% free area sieve plate with 6.3 mm diam holes. The baffle free plate has a continuous declining pressure drop characteristic due to continuous weeping. Baffle IV with the liquid distributor pointed vertically down or horizontally demonstrates a small but positive weeping rate. A layer of lessing rings had a similar small weeping rate. Baffle types VII and VIII have similar characteristics to baffle IV.

Baffle IV, being a parallel plate baffle of height 76 mm with a plate spacing of 19 mm, was simple and relatively effective and was a considerable improvement over the baffle free plate.

Increasing depths of liquid are known to promote weeping on conventional plates, so a series of experiments on the effect of liquid holdup was completed. The holdup was varied from 4.53 to 31.41 corresponding to clear liquid depths of 15.5 and 107.6 mm, and the values are listed below for an air flow rate of 3.141 kg/m^2 s:

Run number	1	2	3	4	5	6	7
Initial liquid holdup (1)	4.53	9.63	14.33	18.73	21.30	25.99	31.41

The pressure drop characteristics are shown on Figure 6.

With small initial liquid loads, the weeping rate was effectively zero, as shown by runs 1, 2, and 3. At higher liquid loads, violent sloshing and periodic dumping of liquid occurred, producing a rapid decrease in the pressure drop curves as shown for runs 4 to 7. This decrease terminated after approximately 30 s, and a stable liquid holdup was retained on the plate with an effectively zero weeping rate.

These results show that baffle IV can effectively reduce the weeping rate and retain 14 l of liquid on the plate.

The air velocity distribution approaching the sieve plate was distorted to a nonuniform value by the positioning of other sieve plates below the test plate. Wide departures from the uniform air velocity distribution had a negligible effect on weeping.

Baffle types IX and X with 150 mm deep plates and 51 mm separation distance were found to have significant weeping rates and are compared with baffle IV on Figure 7.

The effect of reducing the free area from 11.8 to 5.4% had a remarkable effect on stabilizing the fluid oscillations with a type IV baffle in position. Large liquid holdups could be supported without any weeping being observed over considerable ranges of gas velocity. A comprehensive pressure drop characteristic of this baffled sieve plate and the points of incipient weeping are shown on Figure 8.

The baffled single sieve plate has now satisfied two important assumptions in the theory, namely

(6) Negligible weeping
(9) Plug flow of liquid during the LDP

To proceed further with testing of the periodic cycling theory, values for the parameters λ, ϵ, and η are required.

POINT EFFICIENCY OF THE SIEVE PLATE

An unsteady state method was used to measure the point efficiency of a downcomerless sieve plate at various liquid holdups. The unsteady state mass balance is given by

$$H \frac{dx}{dt} = V(y - y_{in})$$

The point efficiency is given by

$$\epsilon = \frac{y - y_{in}}{y^* - y_{in}}$$

Solving the differential equation for constant y_{in}, we get

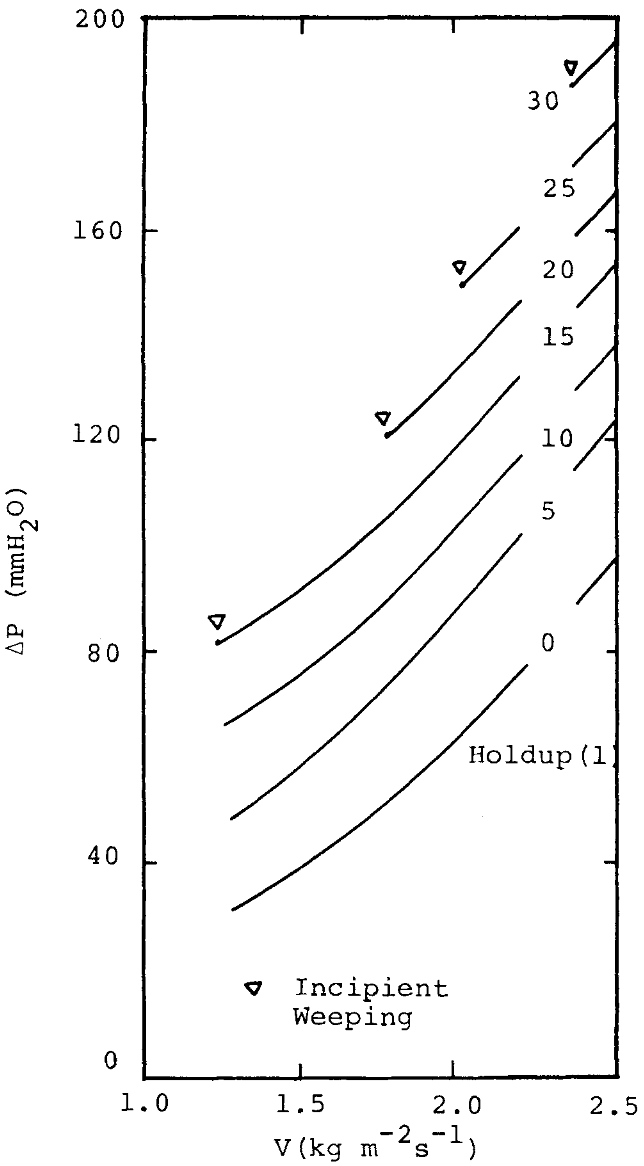

Fig. 8. Pressure drop characteristics of a 5.4% free area sieve plate with type IV baffles.

$$\epsilon = \frac{H}{mVt} \ln \left\{ \frac{mx(o) - y_{\text{in}}}{mx(t) - y_{\text{in}}} \right\}$$

If y_{in} is zero, the equation can be rearranged to give

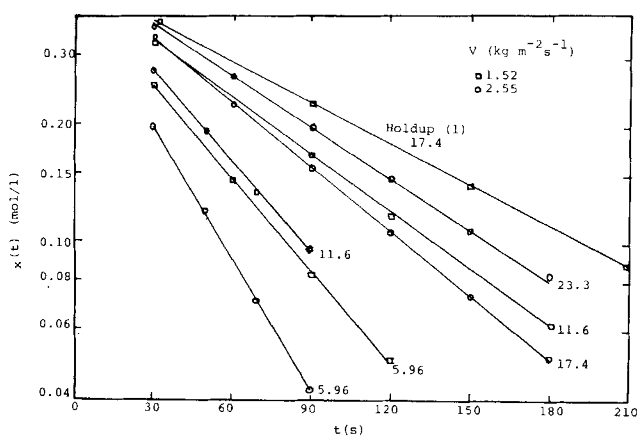

Fig. 10. Plate efficiency measurement, unsteady state mass transfer on a sieve plate.

TABLE 2. EXPERIMENTAL TRANSIENT LIQUID COMPOSITIONS
Vapor flowrate = 2.000 kg/m²s

Run number	Contact time (s)	Final liquid composition (mole/l)
B11	30.0	0.232
B12	30.0	0.226
B13	30.0	0.225
B14	60.0	0.159
B15	60.0	0.156
B16	60.0	0.156
B17	90.0	0.110
B18	90.0	0.108
B19	90.0	0.107

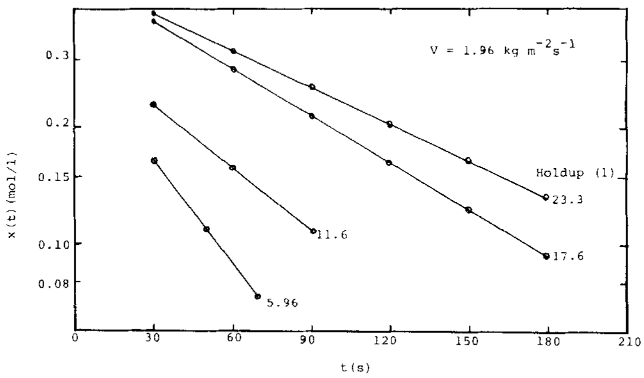

Fig. 9. Plate efficiency measurement, unsteady state mass transfer on a sieve plate.

$$\ln x(t) = -\left(\frac{mV\epsilon}{H}\right) t + \ln x(o)$$

Thus, the point efficiency can be extracted from the $mV\epsilon/H$ term, corresponding to the slope of the line of a $\ln x(t) v\, t$ plot. All three quantities, m, V, and H, must be accurately known for reliable values of the point efficiency ϵ. Values of m were obtained by measuring the temperature of the liquid holdup by a thermocouple and using the equilibrium relationships given by the A.I.Ch.E. Research Committee (1958):

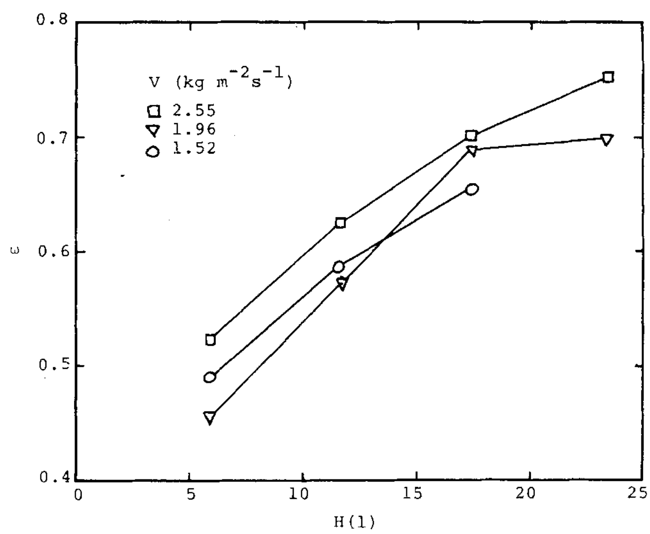

Fig. 11. Plate efficiency and liquid holdup on a sieve plate.

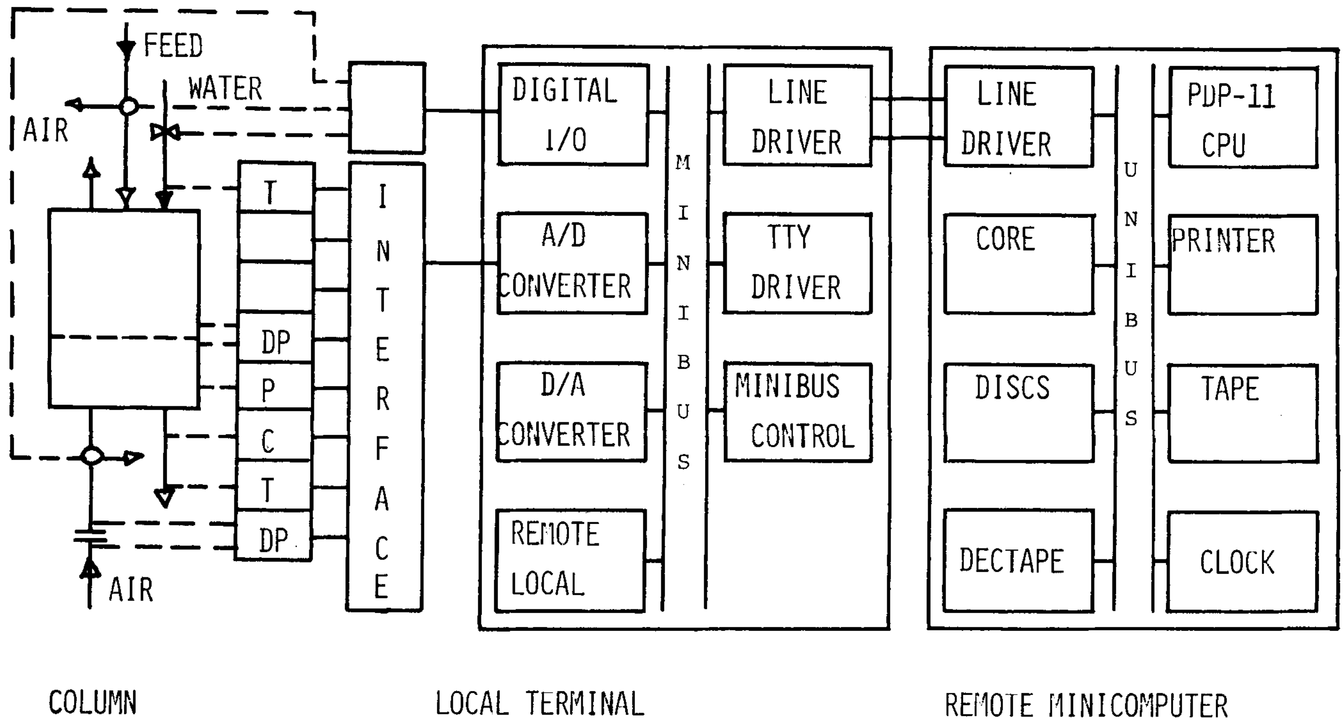

Fig. 12. Schematic diagram of the modified column layout.

$$\log_{10}K = -1\,665/T + 6.695$$

$$m = K\left(\frac{55.56}{P}\right)$$

The vapor flow rate V was obtained by differential pressure measurements over a calibrated orifice meter. An accurately calibrated delivery tank was used to rapidly place a known liquid holdup H on the plate.

The experimental procedure involved the rapid delivery of a known quantity of liquid of a given composition to the plate, where it was contacted with vapor for a time t. The vapor flow rate was then reduced to zero and the draining liquid collected and analyzed by a standard titration technique. This procedure was highly reproducible and was repeated over a range of contact times t for a given holdup H, thus avoiding the sampling of liquid from the active froth regime on the plate. Table 2 shows triplicate runs at each contact time for an equivalent liquid depth of 40 mm.

A comprehensive series of runs with liquid holdups ranging from an equivalent 20 to 80 mm of liquid was completed. These results were plotted on a log-linear plot as shown on Figures 9 and 10 to extract the point efficiencies which are listed in Table 3 and shown in Figure 11. Triplicate runs were used to ensure the position of point efficiencies shown on Figure 11.

The experimental point efficiency results show that the unsteady state modeling of the mass transfer processes is excellent and that the point efficiencies of 70% obtained with large liquid holdups are suitable for testing plates operated with periodic cycling.

PERIODIC CYCLING OF A SINGLE-PLATE COLUMN

The preceding experiments provide an accurate method of obtaining the plate efficiency ϵ under stable hydrodynamic conditions in the absence of weeping. Particular attention has been given to the design of the following experiments to ensure the accurate measurement of λ, the ratio of the slopes of the equilibrium and operating lines.

Equipment

A PDP 11 computer was directly connected to a 600 mm diam mild steel column fitted with a downcomerless sieve plate containing 6.3 mm diameter holes as shown on Figure 12. The plate was supported on 19 mm wide flanges and occupied the full cross-sectional area of the column. Viewing ports 76 mm diam were placed on diagonally opposite sides of the column and positioned under the plate to view all weeping phenomena. An efficient liquid deentrainer was fitted above the plate and returned any liquid back to the plate. The air supply consisted of a 20 kW centrifugal fan, delivering 70 m^3/min of air at a maximum head of 940 mm water gauge. The flow rate was controlled by a manually operated 300 mm diam butterfly valve fitted with a gear reduction unit. The air leaving the fan was humidified and cooled by the addition of atomized water from a Sprayco pneumatic nozzle, model 125M. A standard orifice plate 191 mm diam was placed after a 8.5 m straight length of ducting for flow metering. The air then entered a tee section fitted with two mechanically coupled 250 mm diam butterfly valves. The coupling was directly linked to an electric solenoid delivering a 270N force which rotated both butterfly valves in 125 ms. A return spring ensured counter rotation of the valves when the solenoid was deactivated. Air could be diverted to and from the column by solenoid activation. Air entered the base of the column through a 250 mm diameter pipe covered with a liquid collection cone to prevent falling water from entering the air supply system.

The liquid supply system consisted of a 900 l feed tank containing an aqueous ammonia solution which was pumped through a rotameter to a pneumatically operated three-way valve. Liquid could be recirculated to the feed tank or pumped through a nonreturn valve to the liquid distributor on the column. Liquid leaving the sieve plate during the LDP was collected in the

TABLE 3. POINT EFFICIENCY RESULTS

Set	Vapor flow rate (kg/m^2s)	$H(1)$	Point efficiency
A	2.032	5.96	0.453
B	2.019	11.59	0.573
C	1.899	17.42	0.690
D	1.893	23.35	0.700
E	2.535	17.42	0.703
F	2.517	23.35	0.752
G	2.567	11.59	0.628
H	2.641	5.96	0.523
I	1.558	17.42	0.655
J	1.540	11.59	0.589
K	1.537	5.96	0.490

TABLE 4. LEVEL CONTROL ON A SINGLE-PLATE COLUMN

Cycle No.	Time	Switch DP	Mean DP	No. (READINGS)
1	7.28	2.183	2.280	169
2	5.50	2.180	2.276	176
3	4.54	2.180	2.274	179
4	4.44	2.180	2.279	180
5	4.60	2.189	2.282	179
6	4.44	2.182	2.282	180
7	4.42	2.181	2.275	180
8	4.50	2.190	2.282	180
9	4.28	2.186	2.267	180
10	4.46	2.181	2.272	180
11	4.54	2.181	2.281	179
12	4.34	2.181	2.266	180
13	4.38	2.182	2.272	180
14	4.64	2.183	2.282	179
15	4.40	2.184	2.277	180
16	4.44	2.182	2.281	180
17	4.52	2.181	2.286	180

base of the column below the air inlet and removed by a pumping system. Sampling positions were provided in the inlet and exit liquid lines.

A Foxboro electronic differential pressure cell was mounted across the sieve plate, and a copper-constantan thermocouple was used to measure liquid temperature on the plate. The electrical conductivity of the liquid leaving the column was measured by a conductivity cell connected to a Philips PR9501 conductivity bridge.

The computer system as shown on Figure 12 consisted of a PDP11/20 linked through the UNIBUS to 16K of core, 2 × 256K disks, 2 DEC tape units, teletype, paper tape reader, and punch plus a MINIBUS line driver and receiver. The MINIBUS allows for multiple remote use of the PDP 11/20 through a local terminal which contains a MINIBUS line driver and receiver, teletype, digital output and display, and an A/D converter with a multiplexer to accept an input into the system. The digital outputs on two channel operated relays which activated solenoids in the main air supply system to the column and the compressed air supply to the pneumatic three-way valve in the liquid supply system.

The computer control strategy was developed by Dale (1976) and consisted of a level control on the sieve plate plus accurate timing of the VFP and the LDP. Programs residing in the PDP11/20 switched on liquid to fill the plate at the start of the VFP, and the level was rapidly monitored by accepting a voltage signal from the electronic d/p cell through the input channels. When the mean of sixteen voltage readings exceeded a set point value, entered through the teletype, liquid was diverted from the column. At the end of the VFP, air was diverted from the column until the end of the set cycle time when the strategy was repeated. This program provided a stable and reproducible liquid holdup on the plate from cycle to cycle and ensured accurate timing of the LDP. The program was highly flexible, and only the major details have been described. It allowed for the collection, storage, and printing of the cycle number, the time of rapid liquid entry to the plate, the set point voltage for a liquid level setting, the mean pressure drop across the plate during the VFP after level control was complete, and the results of readings used to obtain the above mean pressure drop.

A typical computer printout showing the start-up of a single plate column is listed in Table 4. Note the rapid approach to equilibrium in three to four cycles.

EXPERIMENTAL PROCEDURE AND RESULTS

The air flow rate was kept constant for all runs at 1.987 kg/m^2 s, and the depth of equivalent clear liquid was kept at 80 mm to ensure a constant plate efficiency of 74%. The value of η, the fraction of the plate holdup transferred per cycle, was measured from the relationship $\eta = M/H$, where M is the mass of liquid pumped on per cycle and H is the plate holdup. When a steady state has been established, M is measured from the change in feed tank level over a large number of cycles, while H is measured by the same level changes when $\eta = 1$.

The temperature on the plate provided values of m, and with the column operated near a $\lambda = 1$ value, the length of the VFP was calculated from

$$t_v = \frac{\lambda \eta H}{m\overline{V}}$$

The length of the LDP was kept as a variable so that η covered the range from 0.2 to 1.0. The total cycle time is given by $t_c = t_v + t_d$. An aqueous ammonia solution was used in the feed tank, and both inlet and outlet ammonia compositions were determined by titration with standard sodium hydroxide, after excess hydrochloric acid was added. The outlet ammonia composition was continuously monitored by measurement of electrical conductivity, and samples were only removed for titration when a steady state had been established. The air flow rate was calculated from the pressure drop over the orifice plate, the wet and dry bulb temperature of the entry air, and the total atmospheric pressure. Full details of the procedures are given by Duffy (1976).

The experimental results on liquid holdup and liquid compositions plus calculated values of X, λ, η, and N, the equivalent number of theoretical plates, are given in Table 5. The table contains experimental results which cover an η range from 0.208 to 1, resulting in N varying from 0.824 to 1.098. These values of N were all measured at λ values within ±5% of 1, and for ease of graphical presentation, the N values were corrected to a λ of 1, N_1, as shown in Table 6 and Figure 13. Also shown in Table 6 are values of $\epsilon\eta$ at $\lambda = 1$ and values of N_{SS} (experimental) based on the point efficiency of $\epsilon = 0.74$.

The experimental results for mass transfer on a baffled single sieve plate are shown on Figure 14 as a plot of N_{SS} (expt) against $\epsilon\eta$, while the solid line is the theoretical result given by Equation (8).

Run No.	Liquid holdup, l	Liquid compositions (mole/l) Inlet	Outlet	X	λ	η	N
1	23.12	0.2091	0.1145	0.4524	1.004	0.208	0.824
2	22.74	0.2389	0.1304	0.4542	0.973	0.310	0.854
3	23.05	0.2386	0.1233	0.4832	1.075	0.372	0.874
4	23.17	0.2004	0.1037	0.4825	1.094	0.372	0.858
5	23.47	0.2004	0.1076	0.4631	0.973	0.457	0.886
6	23.05	0.2386	0.1205	0.4958	1.068	0.543	0.923
7	23.17	0.2004	0.1030	0.4860	1.033	0.544	0.917
8	23.17	0.2686	0.1377	0.4873	0.993	0.624	0.957
9	23.06	0.2113	0.1066	0.4955	1.008	0.720	0.975
10	22.74	0.2389	0.1189	0.5023	0.990	0.815	1.022
11	23.17	0.2686	0.1371	0.4896	0.957	0.868	1.003
12	23.06	0.2130	0.1039	0.5122	0.995	0.958	1.055
13		0.1826	0.0877	0.5197	1.022	1.000	1.058
14		0.2091	0.996	0.5237	1.002	1.000	1.098

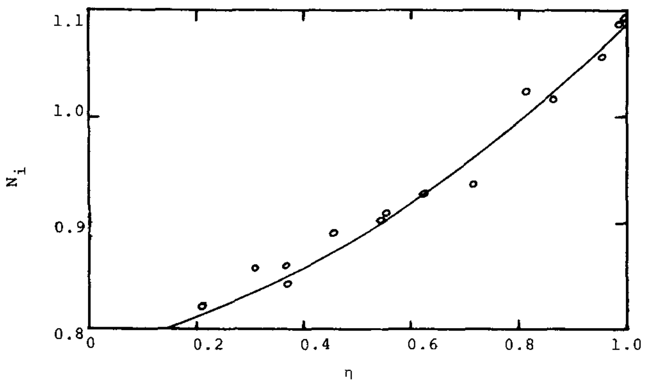

Fig. 13. Number of theoretical plates and the fraction of the plate holdup drained per cycle for a single sieve plate.

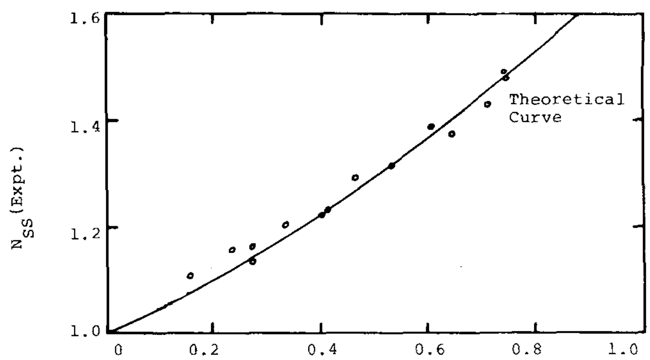

Fig. 14. Number of experimentally measured equivalent steady state plates as a function of $\epsilon\eta$.

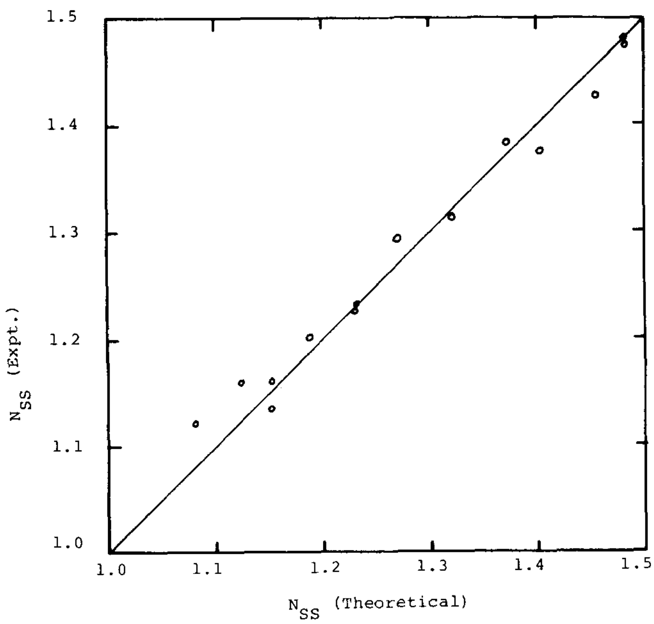

Fig. 15. Comparison of theoretical and experimental equivalent steady state plates for a single sieve plate operated with periodic cycling.

The theoretical values of N_{SS} (theory) calculated from the measured values of $\epsilon\eta$ are shown on Figure 15. The experimental results for mass transfer on a single periodically cycled plate provide the first reliable confirmation on the improvements that are theoretically expected under unsteady state operating conditions.

TABLE 6. PERIODIC CYCLING OF A SINGLE SIEVE PLATE COLUMN

$\epsilon = 0.74$ $\lambda = 1.000$

Run No.	η	N_1	$\epsilon\eta$	N_{SS} (expt)	N_{SS} (theory)
1	0.208	0.823	0.154	1.112	1.081
2	0.310	0.858	0.229	1.159	1.124
3	0.372	0.860	0.275	1.136	1.151
4	0.372	0.841	0.275	1.162	1.151
5	0.457	0.891	0.338	1.204	1.190
6	0.543	0.908	0.402	1.227	1.231
7	0.544	0.909	0.403	1.228	1.231
8	0.624	0.959	0.462	1.296	1.271
9	0.720	0.972	0.533	1.314	1.321
10	0.815	1.025	0.603	1.385	1.373
11	0.868	1.018	0.642	1.376	1.403
12	0.958	1.057	0.709	1.428	1.455
13	1.000	1.049	0.740	1.418	1.481
14	1.000	1.097	0.740	1.482	1.481

ACKNOWLEDGMENT

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NOTATION

A = coefficient matrix
b = column vector
D = matrix containing η
H = liquid holdup
m = slope of equilibrium line

K = equilibrium constant
L = liquid flow rate
N = number of theoretical plates
N_{SS} = number of actual plates in SS condition
P = pressure
T = temperature
t = time
t_v = vapor flow time
u = input
V = vapor flow rate
X = dimensionless liquid composition
x = liquid composition
x' = liquid composition difference
y = vapor composition
ϵ = plate efficiency
θ = dimensionless time
λ = mV/L
η = fraction of liquid holdup transferred per cycle

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A Design Oriented Model of Fines Dissolving

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Mass and population balances, together with nucleation kinetics of the potassium chloride system, were combined to present a design oriented model which describes the behavior of a fines destruction system (FDS) implemented in an MSMPR crystallizer. This model was tested in a well-documented, bench scale, potassium chloride crystallizer and was found to be adequate to predict the effect of fines dissolving. Product size improvement was related to incremental cost incurred with an FDS.

SCOPE

An industrially used technique for making a larger sized crystal product from a mixed magma crystallizer is to segregate fine crystals from the magma, dissolve them, and recycle the dissolved solute for further growth on existing crystals. This technique permits the same production to be made on fewer numbers of crystals of larger average size. The growth rate is forced to a higher level to produce the same mass on a smaller crystal surface area. As with most process advances, the industrial practice of fines dissolving predates the rigorous design and analysis of such techniques.

The present work presents an experimental study of a mixed magma potassium chloride crystallizer equipped with a fines segregation and destruction system. The objectives of the study were to demonstrate in a well-documented, bench scale apparatus the utility and applicability of the R parameter fines dissolving model as well as to present a design oriented technique for solution of the model equations for rapid design and analysis calculations of fines removal systems. A final objective was to relate the relative size improvement of crystal product with incremental costs incurred with a fines dissolving system.

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