Hydrodynamics and Mass Transfer on Three-Phase Distillation Trays

Several oil-water mixtures were contacted by air in a sieve tray distillation column simulator. The oil was mineral spirits with 183–206°C boiling range and 51.8° API gravity. Two sieve tray designs were used, and both hydraulic and mass transfer measurements were made, the latter utilizing acetone as the transferring solute. It was found that gas agitation caused the mixtures to behave as a homogeneous liquid that followed general correlations for pressure drop, liquid holdup, froth height, downcomer liquid level, and fractional entrainment. No unusual problems with foaming or entrainment were observed. Mass transfer efficiency was influenced by oil-water ratio, with mild minima occurring at about 50 vol % mixtures. It was concluded that, when the immiscible liquids are both present in significant proportions, designers need not avoid formation of a second liquid phase for fear of unusual hydraulic or mass transfer behavior.

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Introduction

In certain distillation or stripping operations, it is possible for a second liquid phase to form in one or more parts of the contacting column. Example situations include distillation of heterogeneous azeotropes and steam stripping of organics from water. There has been much speculation regarding the behavior of contacting devices under such three-phase conditions, and the fear of unusual hydraulic or mass transfer behavior has guided many process engineers away from a three-phase involvement. To "design around" the formation of a second liquid phase often results in contrived and nonoptimum conditions.

The purpose of this work was to make experimental and correlational studies of three-phase contacting on crossflow sieve trays. Such studies have not been reported previously. The results of the work would aid designers in their analyses of the advantages and disadvantages of specifying trays to handle two liquid phases.

Previous Work

Very little experimental work has been conducted on three-phase contacting. The most extensive study was made by Schoenborn et al. (1941), who conducted laboratory distillations of organics with an immiscible phase of water present. They used both packed and tray columns. The packed column was 0.127-m diameter and contained 1.6 to 2.8 m of 1.27-cm carbon Raschig rings. The tray column was constructed of glass and contained fifteen 0.203-m-diameter trays, each with two Badger VIX bubble caps. These experiments were concerned only with the efficiency of the columns, and no hydraulic measurements were made. The authors observed that the phases were mixed well on the active portions of the trays and concluded that the presence of the water phase had no adverse effects on column performance.

Goodliffe (1934) presented some data from a column for fractionating roughly-topped crude oil. The column contained 22 1.26-m-diameter bubble-cap trays and used both a reboiler and live steam injection. The author calculated an overall tray efficiency of 18.5% and concluded that this low efficiency was partially caused by "... the hindering effect of the water flooding each tray in preventing contact between the hydrocarbon vapors and liquid."

Schenewerk (1975) investigated immiscible three-phase distillation in a 7.62-cm glass column equipped with ten Teflon sieve trays. The primary purpose of the work was to ascertain reboiler duty for the concentration of phosphoric acid in the presence of *n*-heptane. A three-ideal-stage separation was observed, but no comments were made on the behavior of the column or the reasons for the low (30%) tray efficiency.

Speculations have been made on the effect of the second liquid phase on the behavior of crossflow trays. Shinskey (1977)

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presumed that a second and heavier phase would tend to accumulate on the trays with the lighter liquid either being vaporized or flowing preferentially over the outlet weir. For this reason, he advised that the weirs be designed to permit both overflow and underflow. Guinot and Clark (1938) concluded that "the efficiency of the plates was apparently undiminished by the heterogeneity of the boiling liquid and that was undoubtedly due to the violent agitation produced on the plates by the rapid bubbling of the vapors through the liquid."

Several computer simulation programs have been developed for equilibrium stage calculations involving three phases. For these calculations the equilibria are usually obtained from the NRTL equations of Renon and Prausnitz (1968) or the UNIFAC system (Fredenslund et al., 1977). Examples of such work may be found in the papers of Block and Hegner (1976), Niedzwiecki et al. (1980), Mauri (1980), Austin and Crico (1981), and Prokopakis et al. (1981).

Foaming problems are often associated with the formation of a second liquid phase. Ross and Nishioka (1975), on the basis of experimental work, concluded that foamability increases as the plait point is approached, but in the two-liquid phase region proper, "one of the liquid phases acts as a defoamer for the other liquid phase." For this work, however, the rate of vapor agitation was quite low, as was the case for related work by Bikerman (1973) and Pattle (1950). Davies et al. (1987) presented qualitative information indicating that in a small-diameter (30-mm) Oldershaw column foaming occurred when the immiscible liquid-phase boundary was crossed. Kloubek (1975), in a series of experiments to relate foaming power to physical properties, concluded that only the dynamic surface tension affected foamability. Methods for measuring this property, however, apparently do not produce consistent results.

In a related area of three-phase contacting, Priestley and Ellis (1978) studied gas-agitated liquid extraction columns. Air was sparged into the column to increase the rate of mass transfer between the two liquid phases. Even at a very low air rate (0.30 cm/s superficial velocity), a substantial improvement of transfer rate was observed, with a 60–70% reduction in the height of an equilibrium stage. This is a key point to consider when evaluating the approach to liquid-liquid equilibrium on a tray through which vapor passes at 100 times or more the rate used by Priestley and Ellis.

On the basis of the available literature, it does not appear that a controlled study of the hydraulics and mass transfer on crossflow trays of any size has been reported previously.

Experimental Work

Tests were carried out in a three-tray simulator using air as the gas and water plus a light oil as the immiscible liquid system. Properties of the oil (mineral spirits) are given in Table 1. A flow diagram of the simulator system is shown in Figure 1. The liquid phases were fed to the simulator on a once-through basis; it was found that a recirculating pump created a stable, milky emulsion thought not to be typical of the mixture created by gas agitation on a sieve tray. For hydraulic studies, the liquids were introduced separately to the inlet downcomer. For mass transfer studies, the liquids were mixed in a 0.254-m length of 3.18-cm-i.d. pipe containing several 2.54-cm ceramic Raschig rings. A clear plastic front on the simulator permitted close observation of the three-phase mixture on the tray.

Table 1. Properties of Mineral Spirits

Boiling Range, °C		
IBP	183	
50%	189	
End	206	
Flash point, °C		60.6
Specific gravity (21°C)		0.772
API gravity, degrees		51.8
Viscosity, mPA · s (21°C)		1.66
Surface tension at 21°C, mN/m		24.75
Interf. tension with water at 21°C, mN/m		11.3
Vapor pressure at 37.8°C, kPa		1.01

The column had a 0.30 m^2 inside cross section. The area between the downcomer baffle and the overflow weir was 0.0447 m^2. Spacing between trays was 0.457 m. Two tray designs were used, Table 2. For each design, perforated sections were used for the top and middle trays, with the former being used to collect liquid entrainment and the latter being used for hydraulic and mass transfer measurements.

For hydraulic studies, the air rate from the blower was measured by a calibrated orifice meter, and the liquid flows were measured separately with calibrated rotameters. The following measurements were made:

- *Tray Pressure Drop.* A U-tube manometer filled with Meriam red oil was connected to the vapor spaces above and below the middle tray. For dry tray pressure drop measurements, the inlet and outlet downcomers were sealed off.
- *Liquid Holdup.* A manometer was connected to the middle tray floor to measure hydraulic head, and correction was made for momentum loss of the gas as it passed through the aerated liquid.
- *Liquid Entrainment.* The amount of entrainment was measured by collecting liquid from the top tray. Special screens above this tray were used to remove (and return) any liquid reentrained from the top tray.

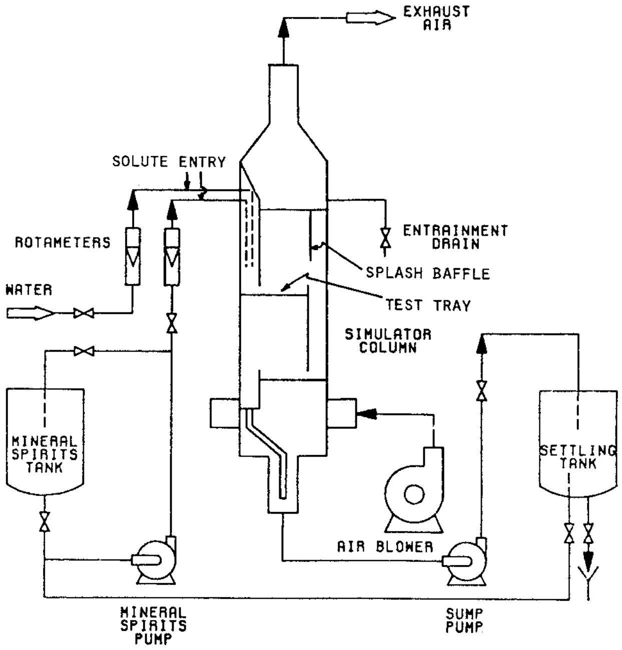

Figure 1. Flow diagram of sieve tray simulator.

Table 2. Dimensions of Trays Tested*

Hole diameter, m × 10^{-3}	6.35	3.18
Number of holes	71	310
Total hole area, m^2	0.00225	0.00247
Total active area, m^2	0.0448	0.0448
Area of perforated field, m^2	0.0245	0.0245
Net area**, m^2	0.0505	0.0505
Total column cross section, m^2	0.090	0.090
Tray thickness, m × 10^{-3}	1.5	1.5
Outlet weir height, m × 10^{-3}	26.9	26.9
Outlet weir length, m × 10^{-2}	30.3	30.3
Downcomer clearance, m × 10^{-2}	12.7	12.7

*Holes were punched and trays were oriented with punch direction upward. Hole pattern was equilateral triangular.

**Area on which velocity of gas approaching the entrainment tray above is based. This takes into account the use of a splash baffle above the overflow weir. (See Figure 1.)

- *Other Measurements.* Froth height was observed visually, and measured at the center of the tray. Downcomer backup was measured directly by visual observation. Temperature was measured by I-C thermocouples located in downcomer bases, tray center (aerated liquid zone), and vapor space above bottom and middle trays.

For the mass transfer studies, liquid acetone (99%+ purity) was added to the inlet liquid through a calibrated rotameter. Addition could be to either liquid phase separately or the mixed liquid phases.

Samples of the gas flowing from the aerated mixture were taken with gas-tight hypodermic syringes inserted well into the flowing stream through sample ports. Liquid samples were taken at the middle tray inlet, center and outlet; the liquid flows were directed into glass vials that were stoppered immediately. The organic and aqueous phases were then separated using a hypodermic syringe, placed in stoppered vials, and later analyzed for acetone with a Varian Model 3700 gas chromatograph.

Additional details on the equipment and experimental method may be found in the graduate theses of Kruelskie (1983) and Herron (1983).

Background

The hydraulic parameters to be investigated were tray pressure drop, tray liquid holdup, height of aerated mixture (froth), downcomer liquid level, and entrainment rate. A number of correlations are available for predicting these parameters. For tray pressure drop, the method of Bennett et al. (1983) was selected as reliable based on a comprehensive database. An alternate method, found by the Bennett group to be reasonably reliable and much simpler to use, was the earlier method of Fair (1963). Both methods utilize the basic pressure drop relationship:

$$h_T = h_D + h_\sigma + h_L \tag{1}$$

where the total drop h_T is presumed to have components of dry tray drop h_D, drop due to surface tension h_σ, and liquid holdup h_L.

The dry drop is obtained by the method of Leibson et al. (1957). The Bennett model utilizes the following equations:

$$h_D = 0.0508(U_o/C_o)^2(\rho_g/\rho_L) \tag{2}$$

$$h_L = \phi_e[h_w + C(L_w/\phi_e)^{2/3}] \tag{3}$$

$$C = 0.0327 + 0.0286 \exp[-137.8h_w] \tag{4}$$

$$\phi_e = \exp[-12.55K_s^{0.91}] \tag{5}$$

$$K_s = U_a[\rho_g/(\rho_L - \rho_g)]^{0.5} \tag{6}$$

$$h_\sigma = (6\sigma)/(g\rho_L D_{B,\max}) \tag{7}$$

$$D_{B,\max} = 1.27[(D_H\sigma)/(g(\rho_L - \rho_g))]^{1/3} \tag{8}$$

The Fair model utilizes a simpler set of equations:

$$h_\sigma + h_L = \beta(h_w + h_{ow}) \tag{9}$$

$$h_{ow} = 0.227(L_w)^{2/3} \tag{10}$$

$$\beta = 0.58 + 0.42 \exp(-1.62F_a) \tag{11}$$

For liquid holdup prediction, Eq. 2 or 9 may be used. For the visual height of froth, the AIChE method (1958), based on observations on small bubble-cap trays, is useful:

$$Z_F = 0.0432F_a^2 + 1.89h_w - 0.0406 \tag{12}$$

The equivalent clear level of liquid in the downcomer is normally obtained by a simple pressure balance on the tray; in the present work, no attempt was made at a generalized correlation of the level, since the primary interest was in observing whether there would be difficulties in phase separation in the downcomer. Finally, there appears to be no generally reliable correlation for the estimation of rate of liquid entrainment. The method of Fair (1961) is useful in showing how the level of entrainment affects the mass transfer efficiency on the tray.

For mass transfer, a material balance across a differential slice of aerated mixture gives

$$G dy_A = K_{OG_1}a_1P(y^*_{A_1} - y_A)dZ_1 + K_{OG_2}a_2P(y^*_{A_2} - y_A)dZ_2 \tag{13}$$

where subscripts 1 and 2 refer to liquids 1 and 2 and *A* refers to species *A*, the solute in the present case. Equation 13 is based on a two-layer model, in which the vapor is assumed to move through the oil and water phases successively. If the liquid phases are in equilibrium,

$$x_{A_2} = \alpha x_{A_1} \tag{14}$$

Where α is the liquid-liquid phase distribution coefficient and

$$y^*_{A_2} = y^*_{A_1} = y^*_A \tag{15}$$

Equation 13 then becomes

$$G dy_A = K_{OG_1}a_1P(y^*_A - y_A)dZ_1 + K_{OG_2}a_2P(y^*_A - y_A)dZ_2 \tag{16}$$

$$\frac{d_{y_A}}{y^*_A - y_A} = P/G[(K_{OG}adZ)_1 + K_{OG}adZ)_2] \tag{17}$$

Integrating between the initial and final gas-phase mole fractions Y_{Ai} and Y_{Af},

$$\frac{y_A^* - y_{Af}}{y_A^* - y_{Ai}} = \exp - [P/G\{(K_{OG}aZ)_1 + (K_{OG}aZ)_2\}] \quad (18)$$

Accepting the conventional definition of numbers of overall gas-phase transfer units, and since

$$E_{OG} = \frac{y_{Af} - y_{Ai}}{y_A^* - y_{Ai}} \quad (19)$$

Equation 18 then becomes

$$E_{OG} = 1 - \exp - [(N_{OG_1}) + (N_{OG_2})] \quad (20)$$

The efficiency term in Eqs. 19 and 20 is a local efficiency, appropriate for the small size of simulator used in the present work.

Experimental Results

Typical measurements of pressure drop are shown in Figure 2. Discharge coefficients were found to be 0.73 for the 0.00635-m holes and 0.75 for the 0.00318-m holes. The relatively small effect of oil/water ratio on tray pressure drop is influenced by a fairly high ratio of dry tray drop to total tray drop. However, liquid holdup data, Figure 3, do not show any unusual effects of oil/water ratio. Observed heights of froth, Figure 4, show definite increases in height with increasing proportion of oil. Since only minor effects of oil/water ratio on liquid holdup were found, this would indicate relatively low froth densities ϕ in the high oil regions, but not necessarily low *effective* froth densities ϕ_e as defined by Bennett et al. (Eq. 4). It would appear that the higher froth heights for oil would lead to lower flooding velocities, and this would be expected for lower surface tension systems (Fair, 1961).

Observed heights of two-phase mixture in the downcomers are shown in Figure 5. While 100% oil shows a relatively stable froth in the downcomer, only 5% water causes a significant drop in mixture level. There was a tendency toward phase separation at liquid compositions of 20% oil and greater, but downcomer velocities were low (0.0091 to 0.0183 m/s) and higher velocities

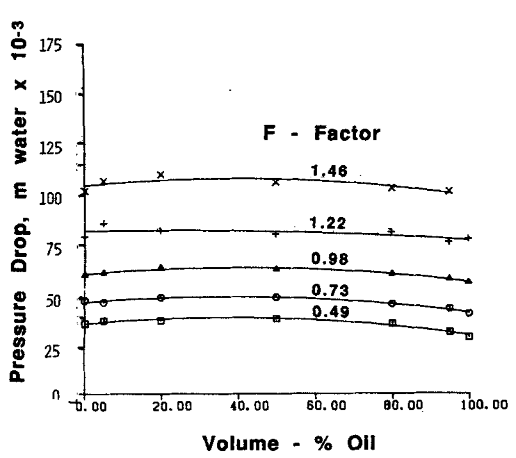

Figure 2. Tray pressure drop as a function of oil-water ratio.
Hole size – 6.35 mm; liquid rate – 0.0744 m³/min · m weir.

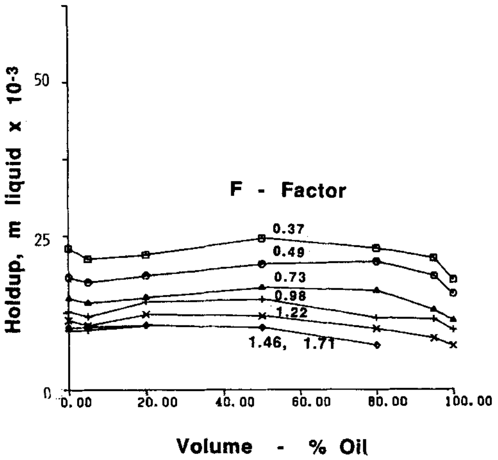

Figure 3. Liquid holdup on tray as a function of oil-water ratio.
Hole size – 6.35 mm; liquid rate – 0.0744 m³/min · m weir.

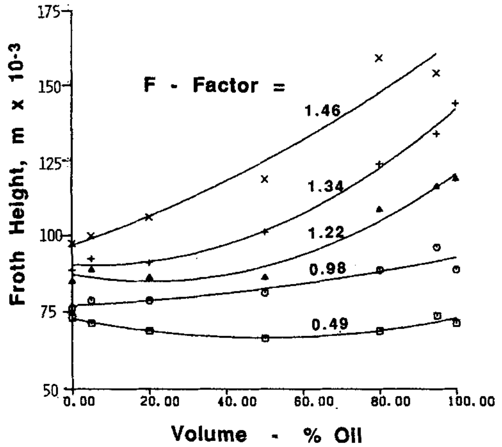

Figure 4. Tray froth height as a function of oil-water ratio.
Hole size – 6.35 mm; liquid rate – 0.0744 m³/min · m weir.

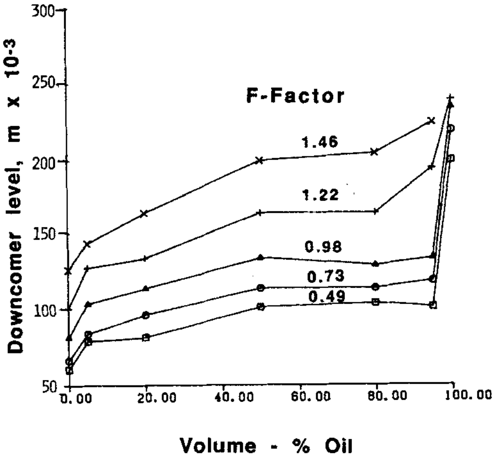

Figure 5. Downcomer level as a function of oil-water ratio.
Hole size – 6.35 mm; liquid rate – 0.0744 m³/min · m weir.

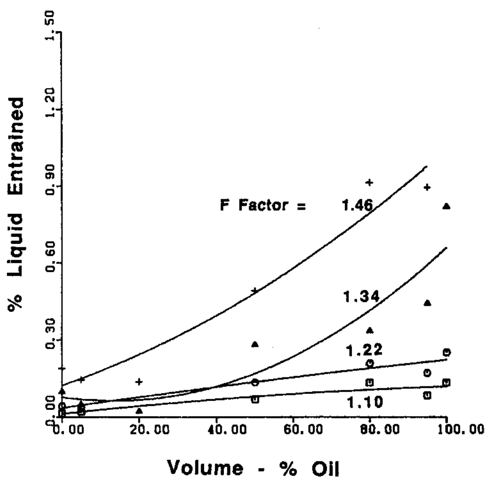

Figure 6. Percent of entering liquid entrained as a function of oil-water ratio
Hole size – 6.35 mm; liquid rate – 0.0744 weir.

would create a greater degree of mixing. Even so, no operating difficulties were evident at the lower rates.

Liquid entrainment measurements are shown in Figure 6. As expected, higher oil content results in a higher fraction of the entering liquid that is entrained. At lower gas rates oil was entrained preferentially, but at higher gas rates the composition of the entrained material approximated that of the tray mixture. Typical data are shown in Figure 7 where it may be observed that at high *F*-factors the composition of the entrained liquid is trending toward the composition of the tray mixture.

The mass transfer studies involved the stripping of acetone from one or both of the liquid phases. Tests with single phases were made first, Figure 8. Although there is some experimental scatter of the data points, it is clear that the water efficiency is higher than the oil efficiency. The water efficiencies compare very closely with those reported by Zuiderweg and van der Meer (1972) for the same system and for sieve trays. The efficiencies of Strand (1963) for the same system, but for bubble-cap trays, are some 20% lower.

Efficiency as a function of oil fraction is shown in Figure 9. While there appears to be a minimum efficiency at about 50% oil, the data scatter do not permit this to be confirmed. Importantly, no unusual trends are noted for the intermediate cases.

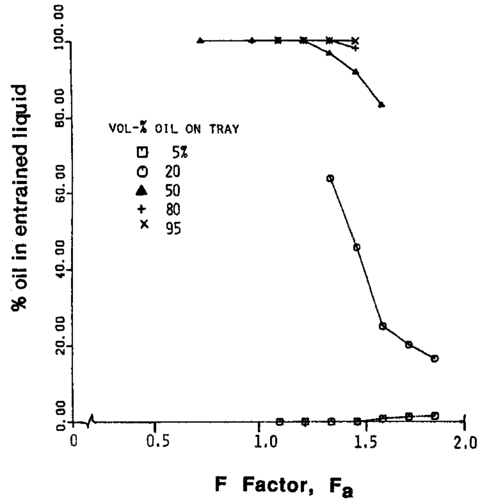

Figure 7. Entrained liquid composition as a function of gas rate.
Hole size – 6.35 mm; liquid rate – 0.0744 weir.

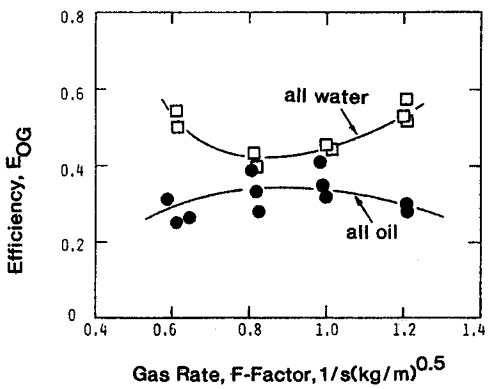

Figure 8. Efficiencies for all-water and all-oil runs.
Hole size – 6.35 mm; liquid rate – 0.0744 weir.

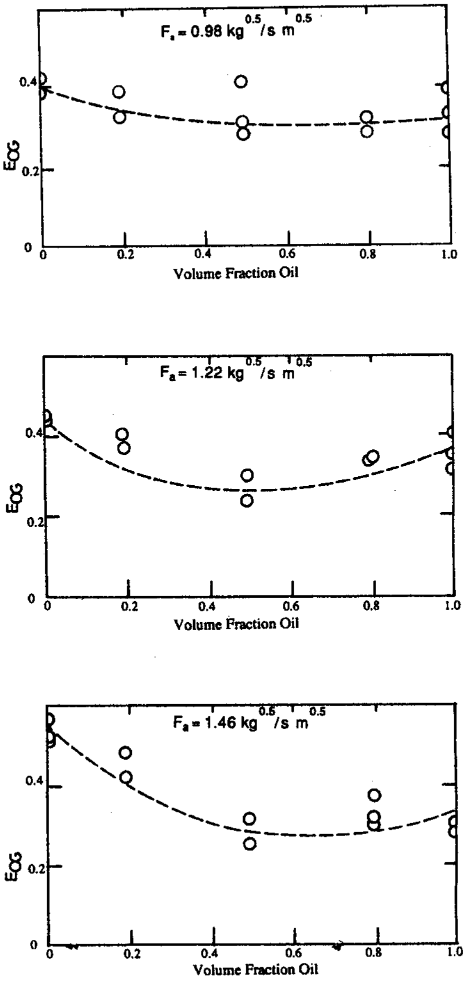

Figure 9. Efficiency as a function of oil volume fraction, for three different gas rates.
Hole size – 6.35 mm; liquid rate – 0.0744 weir.

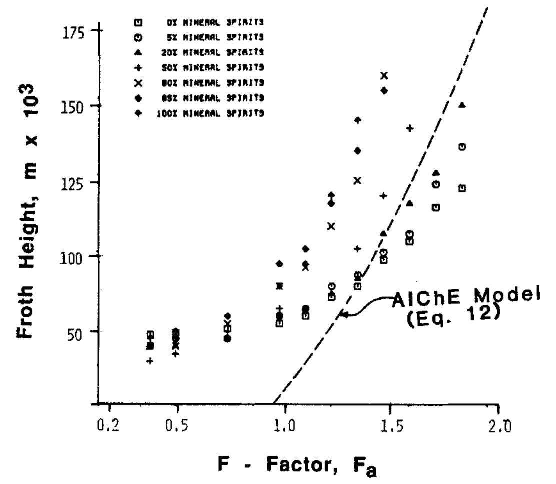

Figure 10. Froth height vs. gas rate.

Hole size = 6.35 mm; liquid rate = 0.0744 weir.

Discussion of Results

Measured tray pressure drops were correlated very well by the Bennett et al. model (Eqs. 1–7), with an average absolute error of 7.1%. A somewhat better fit was obtained with the Fair model (Eqs. 1, 8–11), the average absolute error being 4.9%. It would appear that standard estimating methods can be used when two liquid phases are present. For liquid holdup, neither the Bennett nor the Fair model gave good fits with experimental results, both overpredicting the holdup values by about 40%. This lack of agreement was very likely due to the fairly simplistic approach of measuring dynamic holdup with a manometer system.

The observed froth heights were correlated as follows:

0.0064-m holes:

$$Z_F = 0.0237F_a^2 + 0.252L_w + 0.021\Phi + 0.0366 \quad (21)$$

0.0032-m holes:

$$Z_F = 0.0135F_a^2 + 0.270L_w + 0.0053\Phi + 0.0472 \quad (22)$$

The AIChE model for froth height (Eq. 12) did not predict well the heights observed in this study. A typical comparison is shown in Figure 10. The model does not account for liquid properties and also does not take into account liquid flow rate, both variables found to be significant in this work. The relationships shown in Eqs. 21 and 22 are only indicative of effects and should not be considered general for all systems and tray geometries.

Fractional liquid entrainment may be predicted by the method of Fair (1961) and for a two-liquid phase system this requires some average value of the surface tension. With the assumption of a linear variation of surface tension with volume fraction of oil, values of φ (= fraction of entering liquid that is entrained) were predicted and compared with those measured, Figure 11. Since the maximum value of φ was about 0.01, no great significance should be placed on the general agreement at that entrainment level.

For the mass transfer runs, the question of whether acetone equilibrated between the two liquid phases arose early in the present work. Values of the distribution coefficient (Eq. 14) were measured carefully in the laboratory and found to be constant at $\alpha = 0.885$. That equilibrium was reached in the simulator runs was verified by sampling the tray mixture, quickly segregating the phases and then determining the acetone content of each phase. A histogram of all of the runs is shown in Figure 12. There is reasonable agreement between the distribution coefficient determined in the laboratory and the calculated coefficient from the simulator runs. This supports the use of the mass transfer model outlined in Eqs. 13–20.

Overall efficiencies were estimated by the method of Chan and Fair (1984) which was developed for single liquid phase systems in a distillation mode. A comparison between estimated and measured overall efficiencies is shown in Figure 13. The fit is reasonable for all but the 100% oil case. The reason for this discrepancy is not apparent but could be due to a composition effect on the liquid diffusion coefficient.

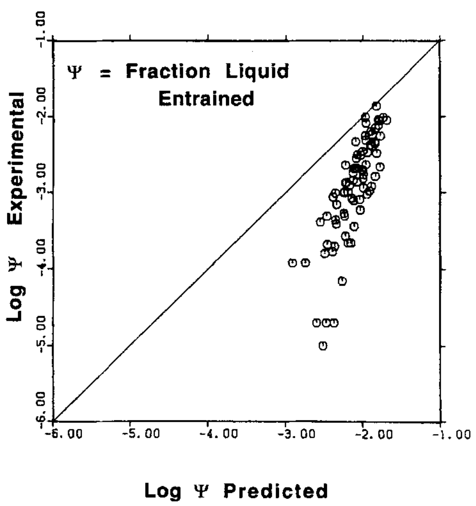

Figure 11. Fractional entrainment for operation with two liquid phases: experimental vs. predicted by method of Fair (1961).

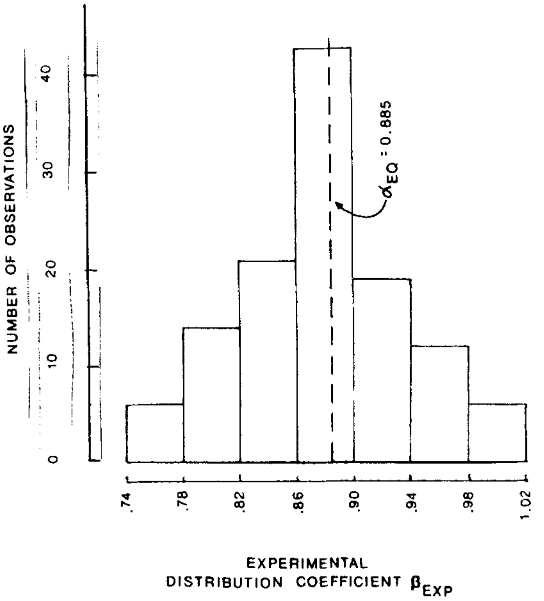

Figure 12. Histogram of distribution coefficients obtained from simulator experiments.

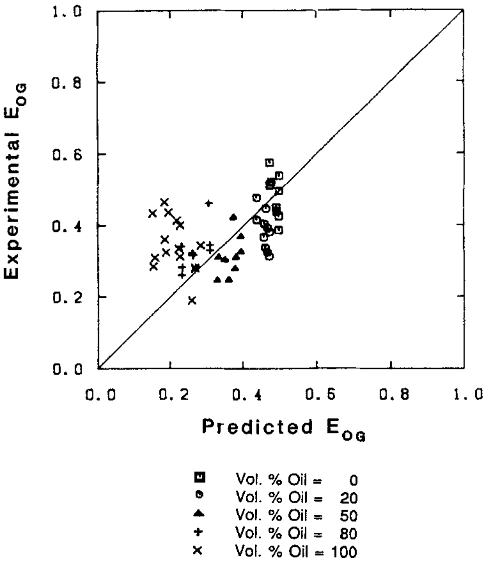

Figure 13. Local efficiencies: experimental vs. predicted by method of Fair and Chan (1984).

Conclusions

This exploratory research project was initiated to determine whether unusual or unexpected phenomena might be present when a second liquid phase occurs on a distillation tray. Pertinent hydraulic parameters were observed under conditions simulating two liquid phase contacting in distillation, and no unusual phenomena were observed. It was found also that values of these parameters could be predicted adequately by standard methods for single liquid phase situations. The effect of a second liquid phase on mass transfer efficiency was also measured, and again no unusual effects were observed. The reliability of efficiency prediction under two-liquid-phase conditions appears to be no worse than that for the single-liquid-phase case. The two-resistance model appears to be relevant, and controlled experiments will be necessary for the determination of individual liquid-phase mass transfer coefficients.

Thus, it appears that designers can permit second liquid-phase operation without undue concern. Whether these observations will apply to a wide range of immiscible liquid systems may be determined by further experimental work.

Acknowledgment

The sieve trays used in this work were generously donated by Glitsch, Inc. of Dallas, Texas.

Notation

a = interfacial area, m^{-1}
C = constant in Eq. 3
C_o = discharge coefficient, Eq. 2
D_H = hole diameter, mm
E_{OG} = local efficiency, fractional
F_a = gas F factor, $= U_a\rho_g^{1/2}$, kg$^{1/2}$/s · m$^{1/2}$
g = acceleration due to gravity, m/s^2
G = molar flow of gas, kg · mol/s
h_D = dry tray pressure drop, m liquid
h_L = liquid holdup, m liquid
h_{ow} = weir crest, m clear liquid
h_T = total pressure drop across tray, m liquid
h_w = weir height, m
h_σ = pressure drop due to surface tension, m of liquid
Ks = $U_a[\rho_g/(\rho_L - \rho_g)]^{1/2}$, m/s
L_w = liquid rate, m^3/min · m weir
U_a = vapor velocity based on active area of tray, m/s
U_o = vapor velocity based on hole area of tray, m/s
x = mole fraction in liquid
y = mole fraction in vapor
y^* = mole fraction of vapor in equilibrium with liquid
Z = height, m
Z_F = froth height, m

Greek letters

α = liquid-liquid distribution coefficient
β = aeration factor
ϕ = relative froth density
ϕ_e = effective relative froth density
Φ = volume fraction of oil in liquid phase
ρ_L = liquid density, kg/m^3
ρ_g = gas density, kg/m^3
σ = surface tension, mN/m
Ψ = fractional entrainment, kg/kg entering liquid

Subscripts

a = active (bubbling) area of tray
A = species A
i = initial or entering
f = final or exiting

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Manuscript received Jan. 13, 1988, and revision received Apr. 11, 1988.