Incipient Three Phase Distillation: Experimental Tray Composition Profiles

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The AIChE tray design procedures for binary mixtures involves the point or local efficiency, E_{OG}, which is related to the two film coefficients in the film model. These are functions of the diffusion coefficients in the liquid and vapor phases. A large number of correlations exist for the variation of E_{OG} with flow parameter. A comprehensive description of these details is given by Lockett (1986). Biddulph and Dribika (1986) provide a table of the extensive data on binary sieve tray efficiencies with column diameters in excess of 0.45 m. These authors have measured the composition profiles along a rectangular tray, 1.067 m × 89 mm, at various F factors for the methanol-water system. The measured Peclet number was about 39, indicating an approach to plug flow of fluid across the tray. An excellent agreement between the predicted and experimental composition and temperature profiles along the tray was obtained by fitting for the best value of E_{OG}. This point efficiency is higher than values predicted by other correlations, including those in the *AIChE Bubble Tray Design Manual.* Other efficiencies, including a thermodynamic efficiency as described by Furzer (1986), can be valuable for consideration of the enthalpy balance equation.

Herron et al. (1988) have reviewed the limited literature information on fluid flow and tray efficiency in three-phase distillation given by Goodliffe (1934), Guinot and Clark (1938), Schoenborn et al. (1941), Prattle (1950), Bickerman (1973), Schenewerk (1975), Ross and Nishioka (1975), Kloubak (1975), Shinsky (1977), and Davies et al. (1987). Herron et al. (1988) have used a water-oil-air simulator to measure the stripping of acetone with two liquid phases present, at various F numbers. They claim that with the small size of the simulator, 0.090 m^2 total column cross section ($D = 0.339$ m), a well-mixed fluid exists and the overall measured compositions provide E_{OG}, the point efficiency. Values of E_{OG} were slightly dependent on the volume oil fraction on a tray but varied smoothly from 0% oil, through to 100% oil. These encouraging results point to a similarity between one-and two-liquid-phase point efficiencies, no doubt due to the excellent mixing by the vapor, and the formation of a near homogeneous tray mixture. The first detailed experimental investigation of column efficiency under distillation conditions with three phases was given by Furzer (1985) for the system, ethanol-water-224 trimethylpentane. Reasonable overall column efficiencies and the complete column composition profile indicated normal point efficiency values on the trays. A comprehensive data set for a 12-component mixture exhibiting three phase phenomena in a distillation column has been given by Cairns and Furzer (1987a, 1987b). This data set provides the composition of all components at the center of a well-mixed tray in a 150 mm diam. column, for all nine trays plus condenser and reboiler. This heteroazeotropic system of ethanol-water-hydrocarbons can produce widely different product distributions, depending on the feed composition. Some authors have indicated that foaming problems may exist as the composition profile crosses the boundary between the one- and two-liquid phase regions. An incipient condition applies when the composition is adjacent to this boundary, and small changes in composition will result in the formation of a second liquid phase.

Multicomponent Tray Composition Profiles

A 13-component mixture consisting of ethanol, water, and 11 hydrocarbons, was distilled in a 610 mm dia. column fitted with four sieve trays of 10% free area, downcomers, and weirs. The hydrocarbon analysis is given in Table 1. The fluid-phase equilibria for this system leads to a two-liquid phase region requiring VLLE data for a part of the composition space. In these experiments, the ethanol, water content was adjusted to lie just beyond the expected two-liquid phase region leading to a single liquid phase on a tray. As the composition changes across a tray from weir to weir, there is always the possibility of entering the two-liquid phase region at an intermediate point between weirs. There is also the possibility of stagnant liquid zones, due to liquid maldistribution, which could have different compositions

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Table 1. Hydrocarbon Analysis

No.	Component	Mole Fraction
1	2,4 Dimethylpentane	0.00873
2	2,3 Dimethylpentane	0.01724
3	2,2,4 Trimethylpentane	0.68245
4	2,5 Dimethylhexane	0.02546
5	2,4 Dimethylhexane	0.04241
6	2,2,3 Trimethylpentane	0.00782
7	2,3,4 Trimethylpentane	0.12054
8	2,3,3 Trimethylpentane	0.03191
9	2,3 Dimethylhexane	0.05808
10	3,4 Dimethylhexane	0.00324
11	2,2,5 Trimethylhexane	0.00211

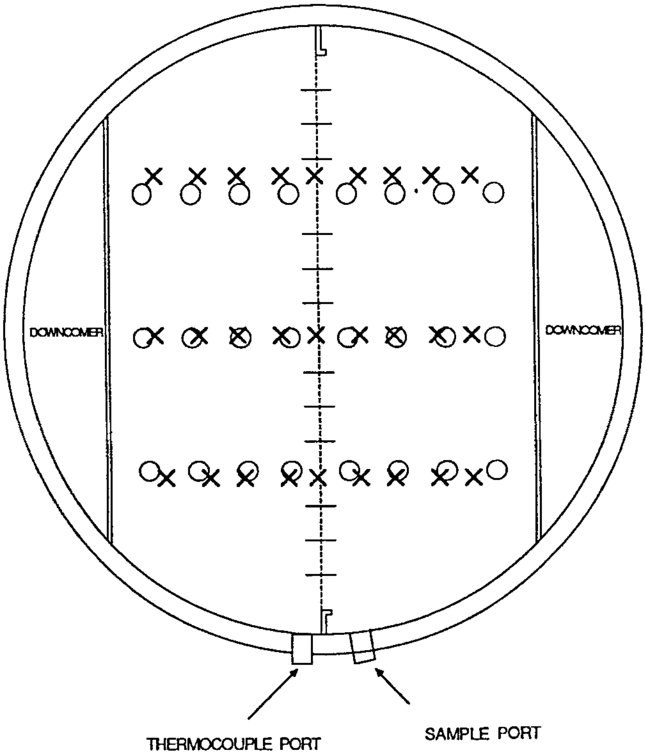

Figure 1. Tray layout and traverse positions.

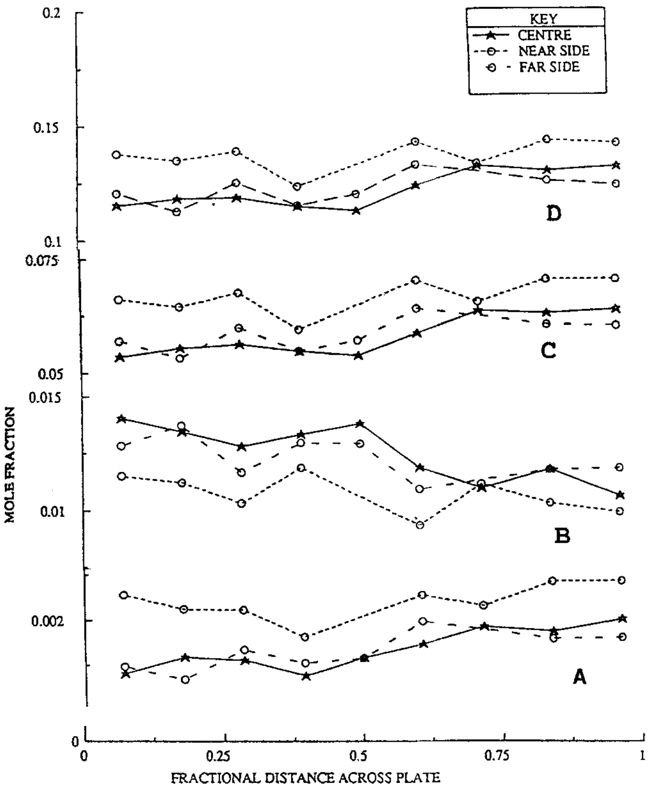

Figure 2. Composition profiles along tray.

A = 225 Trimethylpentane
B = 23 Dimethylpentane
C = 23 Dimethylhexane
D = 234 Trimethylpentane

leading to two-liquid phase regions. The tray layout shown in Figure 1 has a large weir length to minimize any stagnant perimeter regions. An entry port was provided for a calibrated thermocouple, positioned at the midpoint of the tray. A second sample port permitted the movement of a sampling needle to all points on the tray. No two-liquid phase regions were observed in these experiments, but all samples had incipient compositions close to this region. The calibrated needle position allowed for the weir to weir sampling along a center line and on two other lines, as shown on Figure 1. Samples withdrawn from the tray

Table 2. Liquid Compositions (Mole Fraction)*

Component	Fractional Distance Across Plate								
	0.071	0.179	0.286	0.393	0.500	0.607	0.714	0.839	0.964
Water	0.00126	0.00063	0.00069	0.00106	0.00106	0.00094	0.00107	0.00057	0.00088
Ethanol	0.00127	0.0	0.00806	0.01671	0.02230	0.01574	0.01568	0.00153	0.01381
2,4 DMC5	0.00453	0.00430	0.00380	0.00409	0.00428	0.00351	0.00290	0.00315	0.00265
2,3 DMC5	0.01405	0.01347	0.01285	0.01335	0.01384	0.01191	0.01100	0.01183	0.01068
2,2,4 TMC5	0.69898	0.69402	0.68414	0.68372	0.68088	0.66538	0.64621	0.66393	0.64785
2,5 DMC6	0.02647	0.02693	0.02712	0.02597	0.02584	0.02791	0.02882	0.02832	0.02872
2,4 DMC6	0.04337	0.04413	0.04446	0.04361	0.04277	0.04549	0.04757	0.04759	0.04817
2,2,3 TMC5	0.00810	0.00820	0.00822	0.00806	0.00804	0.00850	0.00867	0.00852	0.00858
2,3,4 TMC5	0.11541	0.11872	0.11952	0.11567	0.11393	0.12517	0.13390	0.13213	0.13452
2,3,3 TMC5	0.02873	0.02962	0.03036	0.02882	0.02904	0.03207	0.03548	0.03441	0.03503
2,3 DMC6	0.05366	0.05550	0.05634	0.05479	0.05371	0.05861	0.06344	0.06293	0.06375
3,4 DMC6	0.00273	0.00288	0.00291	0.00276	0.00273	0.00307	0.00338	0.00326	0.00340
2,2,5 TMC6	0.00141	0.00158	0.00154	0.00137	0.00156	0.00170	0.00189	0.00183	0.00196

*Center line traverse.

were analysed for water on a Mettler DL18 water analyser, and for the other components, on a glass capillary column as described by Cairns et al. (1987). Excellent reproduction could be obtained by duplicate and triplicate samples. A complete data set for all 13 components at 9 points on the center line between weirs, is given in Table 2. This table shows the low ethanol and water contents placing all point samples in a hydrocarbon rich region.

Figures 2A and 2D show the composition profiles across the tray for 225 trimethylpentane and 234 trimethylhexane, respectively, along three traverse lines between weirs. The center and far side traverses show similar composition values and gradients, however, there are distinct higher composition values at the near side of the tray. Figures 2B and 2C give the profiles for 23 dimethylpentane and 23 dimethylhexane, for the three traverse lines. In Figure 2B, the near side compositions are distinctly lower, while in Figure 2C, the near side compositions are distinctly higher. These results are indicative of liquid maldistribution at one side of the sieve tray. The water and ethanol profiles are shown in Figures 3A and 3B, with more composition variation from point to point between weirs, and trends difficult to identify.

Multicomponent Point Efficiencies

The use of a Peclet number, a point efficiency, E_{OG}, and λ, to characterize a tray with a dispersion model, is restricted to binary mixtures. The rate processes taking place in the two-phase mixture at a point on the tray are coupled, due to the coupling of the component diffusion coefficients. Unless this coupling is well defined and known, it is not possible to calculate the composition profiles along the tray, for multicomponent mixtures. Some earlier treatments have considered only the two key components in the mixture and then used the known binary solutions. While this may be satisfactory for a homologous series with adjacent key components, additional difficulties may arise with highly nonideal liquid mixtures like alcohol-water-hydrocarbons. For example, the local slope, m, of the equilibrium curve must be known, to evaluate λ, before a solution of the dispersion model can be obtained for a binary mixture. An equivalent m was obtained for each component at each point on the tray profiles by use of the UNIFAC method to account for the nonideal liquid phase behavior.

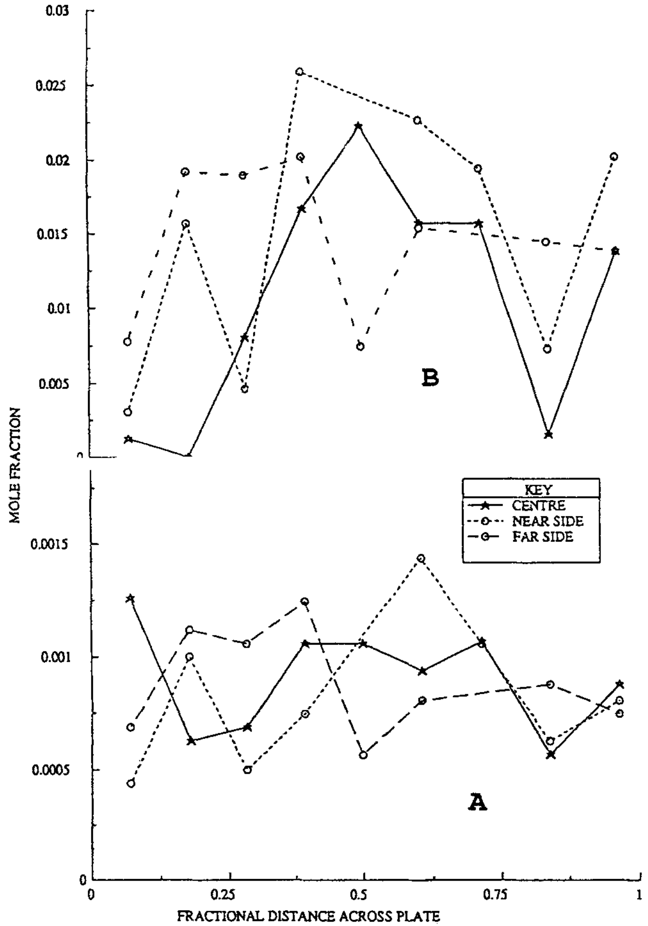

Figure 3. Composition profiles along tray.
A – water; B – ethanol

An optimization technique was used to find the optimal value of Pe and E_{OG} that would minimize a least square error function between the experimental and calculated composition profiles. Figure 4B shows the fit for a large component, 224 trimethylpentane. A Peclet number of 5 might be a suitable value to describe the flow across the tray layout, shown in Figure 1. The point efficiency is 84% for this component. Figure 4A shows a lesser component with a small composition change across the tray. The Peclet number and point efficiency are similar at 5 and 72%, respectively.

Other components can show more variation in E_{OG}, confirming its value for binary systems and its limited role in multicomponent systems.

These detailed experimental results of the composition profile on a tray for incipient three-phase distillation, indicate that normal design procedures should be followed.

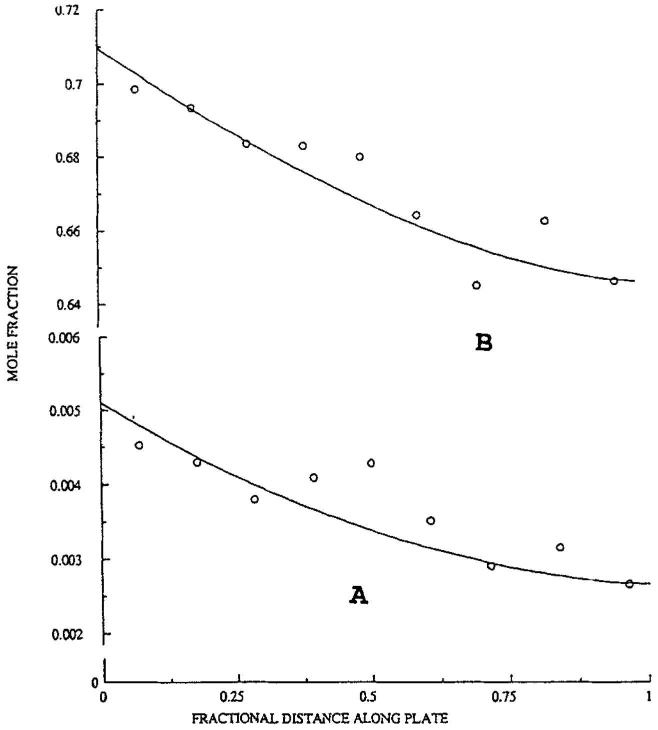

Figure 4. Backmixing model component.
central Traverse; B – 224 Trimethylpentane (Pe – 5, E_{OG} – 0.84, λ – 0.93); A – 24 Dimethylpentane (Pe – 5, E_{OG} – 0.72, λ – 2.2)

Notation

D = column internal diam., m
E_{OG} = point vapor efficiency
E_{MV} = Murphree vapor efficiency
F = F number $(v[p]^{1/2})$, $ms^{-1}\,(kg \cdot m^{-3})^{1/2}$
Pe = Peclet number
v = average vapor velocity, ms^{-1}
λ = slope ratio, equilibrium line vs. operating line
ρ = vapor density, $kg \cdot m^{-3}$

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