Multicomponent Efficiencies in Two Types of Distillation Column

A study of the efficiencies of two plate distillation columns is reported. One column had three rectangular trays with a flow path length of about 1 m, the other was a modified glass Oldershaw column with one tray of 38 mm dia. Tray efficiencies were measured in both columns and point efficiencies were deduced for the rectangular column.

The systems studied were ternary and quaternary mixtures of alcohols and water. The results showed that for thermodynamically nonideal systems significant differences existed among the individual component point efficiencies. In the case of an ideal system the component point efficiencies were effectively equal, but tray efficiencies were different due to differing volatilities and composition profiles across the large trays.

This study illustrates the need to allow for the above effects in large-column designs for multicomponent systems. The possibility of using laboratory-scale point efficiencies in these designs is explored.

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Introduction

Although the majority of distillation processes separate multicomponent mixtures, relatively few efficiency data exist compared with the simpler case of binary systems. It is not surprising, therefore, that most design procedures have been based on binary systems, and it has been widely assumed that multicomponent systems would behave in the same manner.

In designing a column for a multicomponent system, it is the normal procedure to calculate the number of theoretical stages from the equilibrium data for a required separation; a constant column efficiency is then used to estimate the required number of trays. Nord (1946) and Qureshi and Smith (1958) were among the first investigators to point out that in multicomponent systems individual components may operate with different efficiencies. Toor (1957) showed theoretically that for thermodynamically nonideal multicomponent systems, there are marked differences between binary and ternary mass transfer, arising out of interactions between the diffusing species. These interactions were designated:

1. Diffusion barrier—no mass transfer occurs despite there being a driving force
2. Osmotic diffusion—mass transfer when there is no driving force
3. Reverse diffusion—mass transfer against the direction of the driving force

Toor and Burchard (1960) studied the mass transfer behavior of the nonideal system methanol-isopropanol-water to demonstrate these effects. They computed different component point efficiencies existing in this system. Haselden and Thorogood (1964), using the system nitrogen-oxygen-argon, measured equal component point efficiencies. This is the expected result since the system is thermodynamically ideal. Meanwhile, different component point efficiencies were measured for the thermodynamically nonideal systems methanol-acetone-water (Diener and Gerster, 1968; Vogelpohl, 1979), acetone-methanol-ethanol (Free and Hutchinson, 1960), methanol-isopropanol-water (Vogelpohl, 1979; Vogelpohl and Cerretto, 1972), methanol–ethanol–*n*-propanol–butanol–water (Gelbin, 1965), ethanol–tetra-butanol–water (Krishna et al., 1977), cyclohexane–toluene–*n*-heptane (Medina et al., 1979), and cyclohexane-toluene-ethanol and hexane-methylcyclopentane-ethanol-benzene (Young and Weber, 1972).

Mixing effects on the tray have an important influence on the individual component tray efficiencies. In a series of papers Biddulph (1975, 1977) and Biddulph and Ashton (1977) used an eddy diffusion model to allow for the extent of the liquid backmixing on the tray and to simulate conditions corresponding to those in large industrial applications. They computed different component tray efficiencies despite equal component point efficiencies operating across the tray.

It might be questioned whether the taking into account of differing tray efficiencies for different components actually makes any difference to the final column design. This question was addressed in an earlier paper (Biddulph, 1975), in which simulation of a large air-separation column was made, and nine different assumptions were made. These included equal and constant point efficiencies, experimental composition-dependent efficiencies from two sources, and various hydraulic conditions. The results of this comparison indicated a range of tray counts from 57 to 115 for the same product specification. Closer inspection reveals the lightest component showing tray efficiencies well over 120% in the lower part of the column, while the other components had tray efficiencies of around 80%. With similar hydraulic conditions, changing from constant point efficiency values to experimental values made a difference of about 10% in the tray count. This indicates that it is important to attempt to model the behavior of trays more accurately, especially for multicomponent systems.

The systems used in the study described here were methanol–ethanol–*n*-propanol, methanol-ethanol-water, and methanol–ethanol–*n*-propanol–water. These systems were chosen to extend data on multicomponent distillation efficiencies, since nonidealities were present and different component point efficiencies were expected to exist for thermodynamically nonideal multicomponent alcohol-water systems. Figure 1 shows comparisons among the binary gas diffusivities for this system. Large differences exist between alcohol-alcohol and alcohol-water pairs, and significant interaction effects can be expected in these systems (Toor, 1957). In addition, the point efficiencies are expected to be composition-dependent since the extent of liquid phase control is composition-dependent (Dribika, 1986; Mostafa, 1979). The middle components of such systems are known to exhibit maxima in concentration (Ciliano et al., 1974), and the adequacy of the Murphree definition of point efficiency to represent these conditions has been discussed. The results presented in this paper are among the first to illustrate the influence of liquid phase backmixing on the tray efficiencies of multicomponent systems.

Vapor-Liquid Equilibria

Vapor-liquid equilibrium (VLE) data for the quaternary system were not available in the literature, but binary measurements had been carried out and thermodynamically consistent Wilson parameters had been established and are given in Table S1 deposited as supplementary material. In order to study the feasibility of using these parameters, it was decided to measure the vapor-liquid equilibrium data for this system and to compare these with the predictions from the Wilson model using the published parameters. An Ellis-Froome (1954) still was used to carry out the VLE experiments at atmospheric pressure. A statistical test comparing the measured and predicted equilibrium data established the applicability of these parameters, Table S2 (supplementary material). A computer model taking into account the nonidealities in the phases (Prausnitz et al., 1967) was used to carry out the required computations of the activity coefficient. Further experimental details are given elsewhere (Kalbassi, 1987). The same parameters also represented the ternary systems' vapor-liquid equilibria.

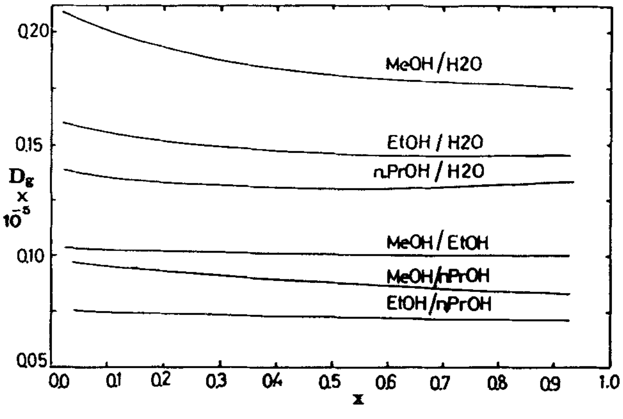

Figure 1. Binary gas diffusivities.

Equipment

Modified Oldershaw column

A small laboratory-scale glass column is used to measure point efficiencies directly. The column is especially designed to avoid wall effects due to the surface tension forces. An expansion of the column above the tray keeps the newly formed bubbles away from the glass walls and allows them to burst naturally rather than to be supported. The column, shown in Figure 2, has external downcomers. Further particulars of this column are given in Table 1, and development details are given elsewhere (Kalbassi, 1987; Kalbassi and Biddulph, 1987).

A 10 L reboiler is provided with a heating mantle and covered by a heating jacket, together providing 1,300 W heat input. The column is contained in a heated cabinet, with glass observation windows, to minimize heat losses. The vapor is condensed in two glass condensers in series, open to the atmosphere, and the

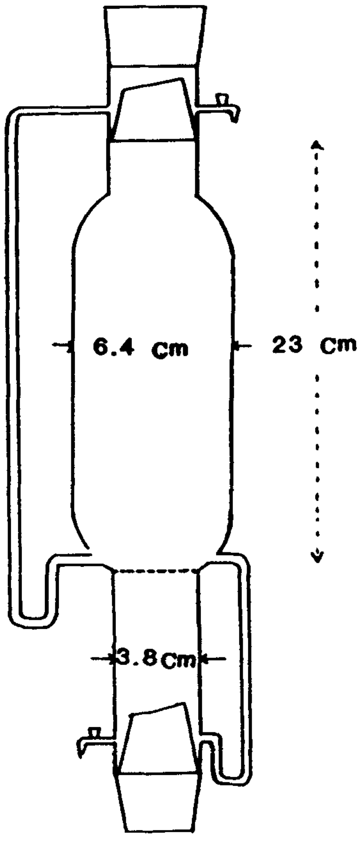

Figure 2. Modified Oldershaw column.

Table 1. Modified Oldershaw Column

Tray dia., cm	3.8
Column dia. above tray, cm	6.4
No. of holes	46
Hole dia. mm	1.1
Outlet weir height, mm	2.0
Free area, %	8

resulting reflux is returned to the column passing through a flowmeter. About 6 L of the test mixture was placed in the reboiler and a run was of sufficient duration for steady state to be established.

Rectangular column

This distillation column has been described previously by Biddulph and Dribika (1986). Briefly, it consists of a rectangular distillation column, Figure 3, having overall dimensions of 1,067 x 89 mm, a steam-heated stainless steel reboiler having a capacity of 450 L, and glass condensers providing a condensing area of 5.3 m^2. The rectangular distillation column incorporates three trays, the middle one being the test tray. This test tray has six equally spaced sample/temperature measurement points along the center line, Figure 4, and is provided with double-glazed glass portholes to enable easy observation of the biphase. The details of the column are given in Table 2.

The sieve tray material is aluminum and is typical of that commonly used in the low-temperature distillation of air. In addition, the equipment is provided with temperature/sample points in the inlet and outlet downcomers as well as in the reboiler. Two other sample points are provided for the liquid leaving the condensers and the column. All the hot surfaces of the equipment are insulated with 50 mm thick glass fiber and aluminum cladding. With a narrow tray width there is always a possibility of wall effects being present. However, the column was carefully insulated, and the comparison of temperatures with bubble-point values, and the observed composition profiles, seemed to indicate that wall effects were not significant.

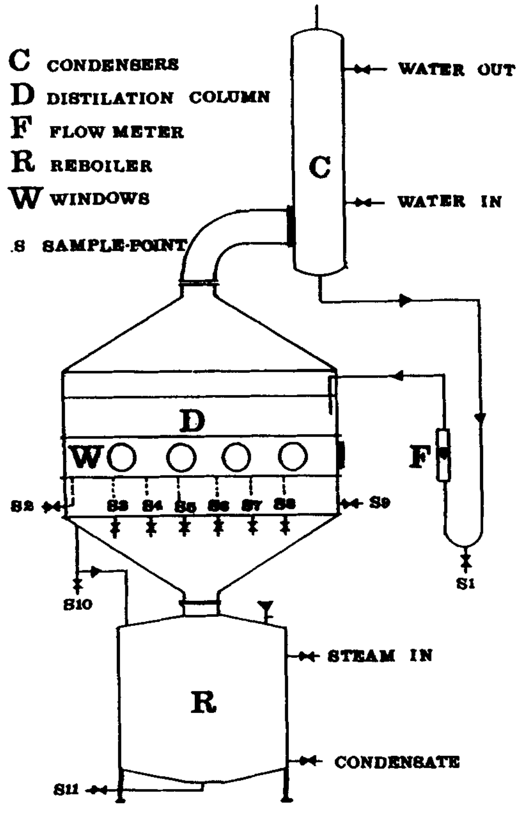

Figure 3. General set-up.

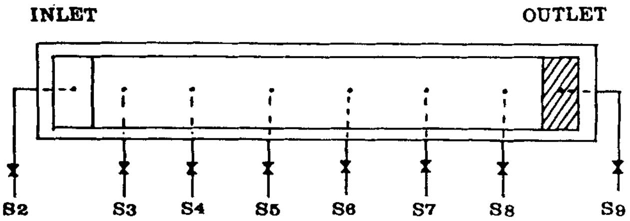

Figure 4. Sampling across the tray.

Theoretical Model

A number of models have been proposed to represent the behavior of the biphase on an operating tray in order to establish the relationships between point and tray efficiencies. The concept of eddy diffusion has been used in this study to model the observed profiles of each component across the tray, and hence to infer component point efficiencies. This concept has become accepted as providing the most realistic representation of the turbulent mixing in the liquid phase on the tray. Among many users were those developing the *AIChE Bubble Tray Design Manual* (1958), and a review has been described previously (Biddulph, 1975). This model has been developed and used previously in an analysis of a low-temperature air distillation column by Biddulph (1975). In addition, it has been applied in a study of an aromatics column (Biddulph, 1977; Biddulph and Ashton, 1977), and was recently applied in binary studies (Biddulph and Dribika, 1986; Dribika and Biddulph, 1986). Briefly, a mass and enthalpy balance is carried out over a slice through the biphase on the tray. The eddy diffusion model is used to introduce backmixing in the liquid phase and a simple partial average model is used to account for the relatively less important influence of mixing in the vapor phase (Diener, 1967). Liquid mixing was considered to be complete in the vertical direction. The resulting differential equations are solved numerically using a predictor-corrector method, stepping across the tray from the outlet weir to the inlet weir against the direction of the liquid flow. This is a stable iterative method and provides predicted component composition profiles of vapor and liquid phases across the tray for given values of component point efficiencies. The solution uses 50 steps across the tray, making the solution stable up to values of Peclet number greater than 60. The model

Table 2. Tray Details of Rectangular Column

Weir length, mm	83
Liquid flow path, mm	991
Tray spacing, mm	154
Hole dia., mm	1.8
Outlet weir height, mm	24.5
Free area, %	8
Inlet weir height, mm	4.8

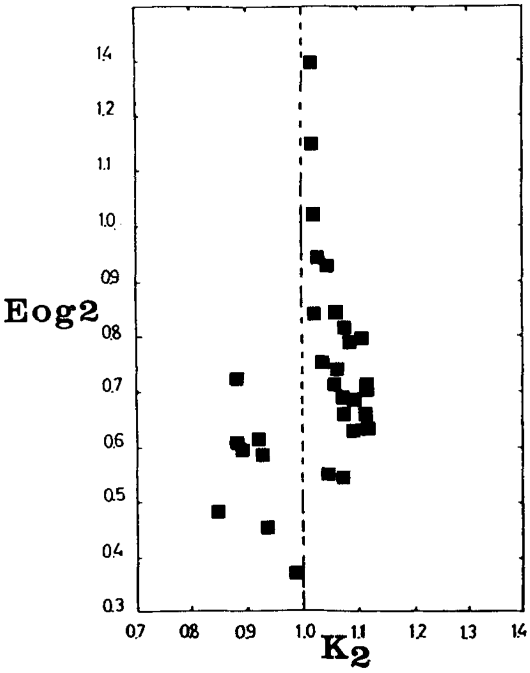

Figure 6. Point efficiencies of ethanol in quaternary system.

requires K values, and these were available from the measured vapor-liquid equilibrium data. In addition, component vapor and liquid enthalpy data were available from Chopey and Hicks (1984) and Kern (1950). Mixing studies on the 1.8 mm hole size tray using the system water-steam with a sodium nitrate tracer injection were carried out by Biddulph and Dribika (1986), and they indicated that the conditions were approaching plug flow on this tray. A Peclet number of about 39 was evaluated from the eddy diffusivity measurements. Further details of the mixing experiments and results have been published previously (Biddulph and Dribika, 1986).

Experimental Procedure

The experiments were carried out at total reflux and atmospheric pressure. Sufficient time was allowed to reach steady state conditions, the boil-up rate and temperatures being noted at regular intervals. The operation of the columns was carried out at a vapor F factor of about 0.5 m/s $(kg/m^3)^{0.5}$, which provided stable hydrodynamic conditions on the trays. A wide range of composition was covered, and samples were collected into prechilled bottles and analyzed by gas-liquid chromatography techniques, giving an average error of up to ±0.004 mol fraction.

Results

Observation of the biphase behavior on the tray indicated steady hydrodynamic operation, with negligible entrainment and weeping. The main results from the studies in the two different columns were as follows.

Modified Oldershaw column

The Murphree point efficiency, defined below, was calculated for the individual components. The alcohol-water systems were the subject of study in this column.

$$E_{og} = \frac{y_{i,n+1} - y_{i,n}}{y^*_{i,n+1} - y_{i,n}} \tag{1}$$

where $y_{i,n}$ is the vapor inlet and $y_{i,n+1}$ is the vapor outlet (mol frac.). $y^*_{i,n+1}$ is the vapor composition that would be in equilibrium with the exit liquid. The point efficiencies and the compositions are presented in Table S3 (supplementary material). A wide range of composition was investigated, and Figure 5 illustrates the variation of the point efficiency of methanol in the quaternary mixtures. These efficiencies are composition-dependent and exhibit similar trends to those shown in the binary systems methanol-water and methanol–*n*-propanol (Kalbassi, 1987; Kalbassi and Biddulph, 1987), that is, a decrease in point efficiency at low methanol composition. The scatter in these results is caused by the influence of varying composition of the other components. A middle component, ethanol, shows the largest variation in point efficiency, due largely to errors occurring in the calculation as the K value approaches unity, Figure 6.

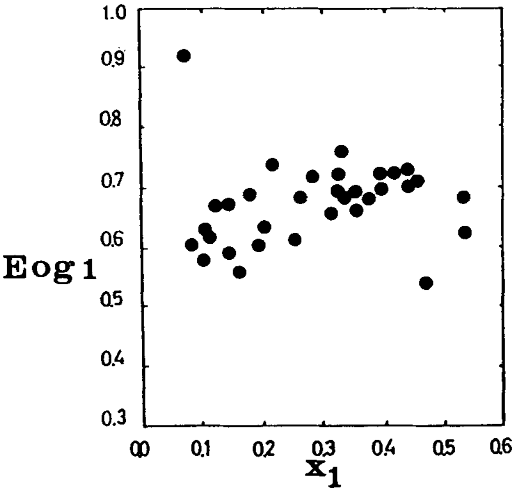

Figure 5. Point efficiencies of methanol in quaternary system.

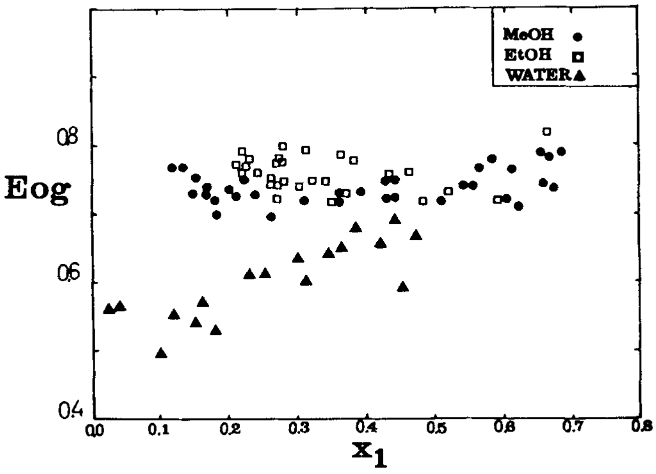

Figure 7. Modified column point efficiencies of MeOH/EtOH/H$_2$O system.

Table 3. Experimental and Predicted Results in System MeOH/EtOH/n-PrOH

Runs	Mean Liquid Composition Across Tray			Exp. Comp. Tray Effic.			Component Point Effic.	Pred. Comp. Tray Effic. Present Model		
	MeOH	EtOH	*n*-PrOH	MeOH	EtOH	*n*-PrOH	Equal	MeOH	EtOH	*n*-PrOH
A3R	0.1189	0.6705	0.2106	1.328	0.465	1.005	0.77	1.289	0.376	0.953
G3R	0.5914	0.3046	0.1040	1.044	1.116	0.973	0.81	1.059	1.167	0.950
I3R	0.6790	0.2448	0.0762	1.076	1.158	0.986	0.81	1.035	1.114	0.941
K3R	0.2243	0.4901	0.2856	1.223	−0.084	0.900	0.81	1.277	−0.195	1.025
N3R	0.0445	0.4570	0.4985	1.736	0.976	1.126	0.78	1.562	0.955	1.070
O3R	0.4270	0.2796	0.2934	1.118	1.916	1.052	0.79	1.075	2.158	0.970

Medina et al. (1979) showed that experimental errors can be very significant under these conditions, since the numerator and the denominator of Eq. 1 are of the order of the experimental error. As expected, the individual components of this nonideal system were also shown to exhibit different point efficiencies. Ethanol showed similar behavior in the ternary MeOH/EtOH/H_2O system but in general the components showed similar point efficiencies, Figure 7, for this system at high concentrations of methanol.

Rectangular column

The rectangular shape of the trays in this column should remove the uncertainties of stagnant zones and flow nonuniformities characteristic of chordal-weir circular trays. The measured concentration profiles were matched with those predicted using the eddy diffusion model to infer point efficiencies. This involved guessing and reguessing component point efficiencies until a good match was achieved. Table 3 and Tables S4 and S5 (supplementary material) summarize the results of all these measurements. Figures 8 to 11 illustrate representative composition and temperature profiles for these systems. A comparison is also made, Figure 12, between the measured temperatures and computed bubble point temperatures for these systems, showing a tendency to values slightly above or below the bubble point temperature. This is probably the result of heat transfer from the vapor phase or the liquid phase.

Discussion

The studies of these highly nonideal systems highlight the possible effects of diffusional interactions which, according to Toor (1957) and Krishna et al. (1977), arise from the presence of reverse diffusion, osmotic diffusion, or diffusion barrier effects, defined in the Introduction. It is assumed that these effects must be responsible for such large variation in individual component point efficiencies. The quaternary system studied here is particularly interesting as there are two "middle" components that can transfer from the liquid to the vapor phase or vice versa. These middle components also reach maxima in concentrations where their mass transfers reach minima. In the majority of the tests, *n*-propanol was transferred from the vapor to the liquid phase and its point efficiencies were in general higher than the other components. Ethanol, however, showed a more confused picture. As expected for the ideal alcohol system, equal component point efficiencies were operating across the tray. For the nonideal systems, maximum variation in individual component point efficiencies was obtained when one of the middle components reached its composition maximum. In this region errors become very significant. However, there were some runs where equal component point efficiencies were operating across the tray, similar to the behavior of the ideal system. Under these circumstances the interaction effects must have been minimized.

The Murphree definition of point efficiency clearly has its

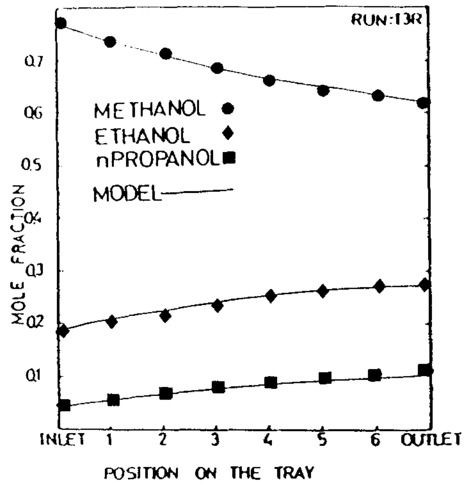

Figure 8. Typical composition profiles for MeOH/EtOH/*n*-PrOH.

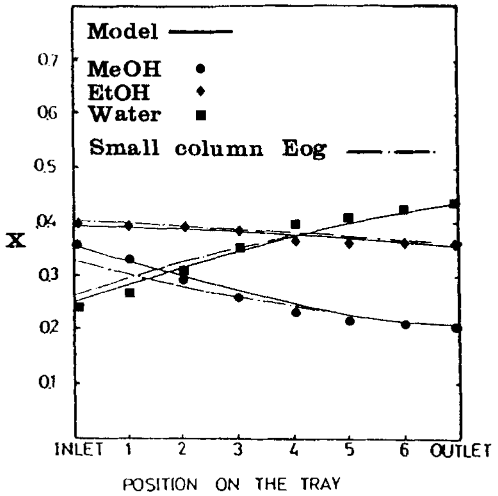

Figure 9. Typical composition profiles for MeOH/EtOH/water.

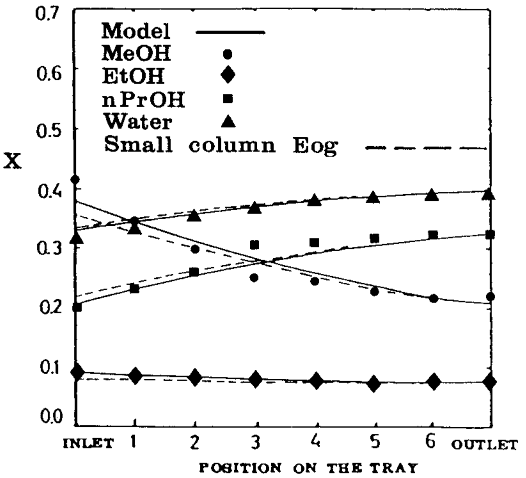

Figure 10. Typical composition profiles for quaternary system.

limitations, especially for the components reaching a maximum in concentration. Under these circumstances the magnitudes of the numerator and the denominator of Eq. 1 become comparable with the experimental errors (Medina et al., 1979), and meaningless values of point efficiency are obtained for that component. By using the mathematical model over a range of composition, more reliable point efficiencies can be obtained for the component that has reached its maximum in concentration. However, it is also important to note that when a component reaches a maximum in concentration, its vapor composition becomes independent of the point efficiency, and the efficiency obtained by Eq. 1, although possibly unusual, has little influence on the evaluation of the vapor composition.

The component tray efficiencies in these systems are significantly different from each other. There are large differences between individual component tray efficiencies even with equal component point efficiencies operating across the tray. These differences result from the effect of limited backmixing (Biddulph, 1975; Biddulph and Ashton, 1977).

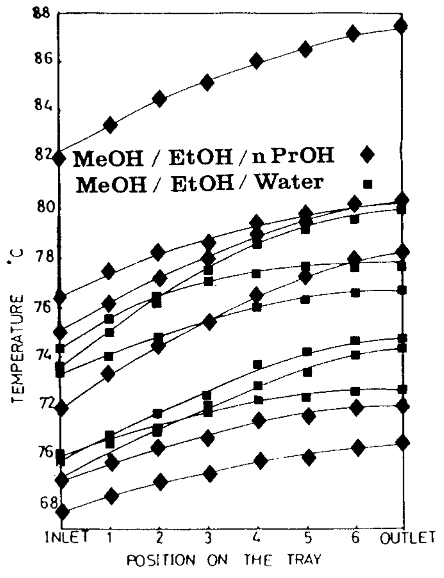

Figure 11. Typical temperature profiles across tray.

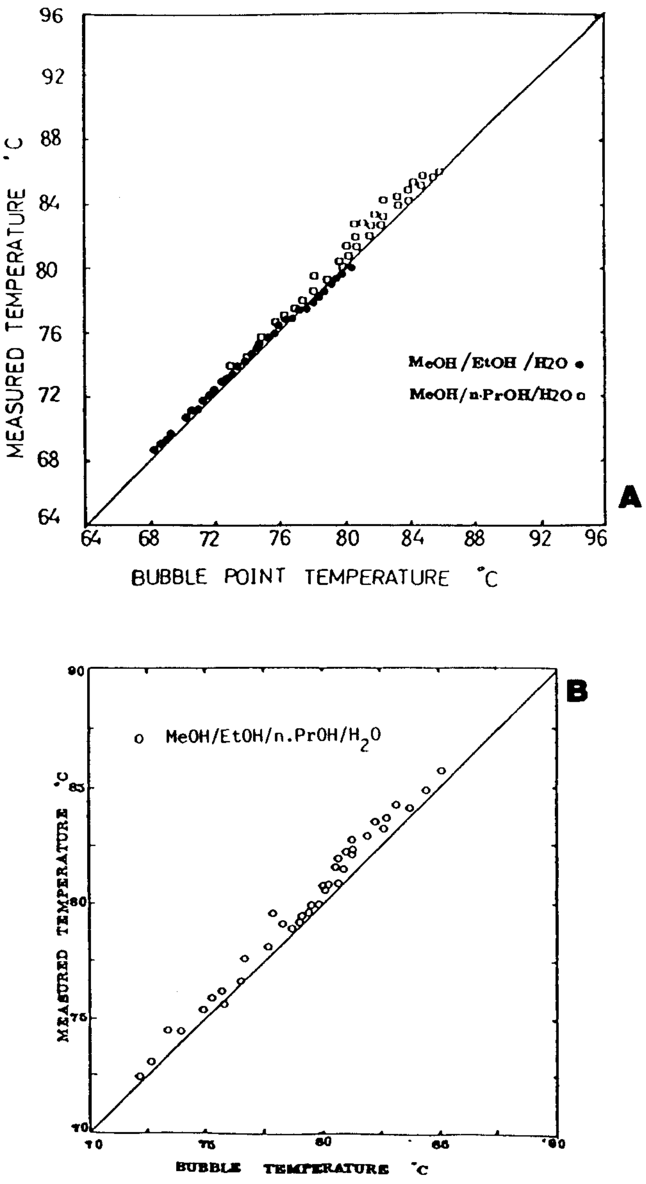

Figure 12. Comparison of measured and bubble point temperatures.

The eddy diffusion concept was found to model and predict well the profiles and the differences in component tray efficiencies. Table S6 (supplementary material) shows the mean percent deviation in measured and predicted component tray efficiencies. The efficiencies of the components that exhibited a maximum in composition were excluded from this evaluation as errors had influenced the computation of the tray efficiencies (Medina et al., 1979). It can be seen that these deviations are reasonable.

The point efficiencies of the nonideal systems were found to be composition-dependent, as in the case of some of the constituent binaries, namely ethanol-water, methanol-water, and *n*-propanol–water (Kalbassi, 1987).

The measured tray efficiencies, shown in Tables 6 and 7, indicate that high efficiencies can be obtained if the detrimental influences of flow nonuniformities and stagnant zones, which are known to reduce the tray efficiency, are eliminated. This provides further evidence for the high efficiencies available to the design engineer if these effects can be eliminated by improving the hydraulic behavior of conventional circular trays.

The point efficiencies measured in the modified Oldershaw column are lower than the point efficiencies deduced from the rectangular column experiments. This is the result of shorter contact time between the vapor and liquid in the smaller column (Kalbassi, 1987). The individual components follow similar variations in point efficiencies in both columns. Table S7 (supplementary material) compares these differences for similar composition nonideal quaternary and ternary experiments. The most and least volatile components demonstrate the least differences in point efficiencies. The middle components show larger differences, which are the result of the experimental errors in the computation of point efficiencies for these components, as they exhibit either negligible or small volatilities. If a small column is to be used for direct measurements of point efficiency for the design of a multicomponent distillation column, it will involve a number of experiments covering a wide range of composition, identifying the components exhibiting maxima in composition and measuring point efficiencies for these components on either side of the maximum. This will reduce the possibility of experimental errors in point efficiency measurements.

These point efficiencies were then incorporated into the eddy diffusion model to simulate their corresponding runs, and hence predict composition profiles across the tray. They are included in the composition profile diagrams, Figures 9 and 10, as the "Small column Eog" curves. The prediction seems very reasonable considering the low liquid holdup and hence lower point efficiencies obtained in the modified column. The tray efficiencies predicted for individual components are included in Table S8 (supplementary material) together with their deviation from the original rectangular tray measurements.

An improvement in the liquid holdup, and hence the point efficiencies, measured in the modified column (Kalbassi, 1987) may provide even better results with relative ease.

Conclusions

Two aqueous nonideal quaternary and ternary systems and an ideal ternary system were studied using two types of sieve plate distillation columns. The nonideal systems, as expected, exhibited different individual component point efficiencies and composition dependencies due to interaction effects. However, there were some experiments where equal point efficiencies were operating across the tray for these systems. The ideal system unsurprisingly showed equal individual component point efficiencies.

The Murphree definition of point efficiency, although unreliable when a component reaches its composition maximum, can still be used. This is because the vapor composition of that component becomes independent of its point efficiency, and since $N - 1$ point efficiencies are defined, this component can be excluded. N is the number of components in the system.

The prediction of the composition profiles in the rectangular column using modified column quaternary and ternary point efficiencies gave good agreement with the measurements. This is very encouraging as the prediction methods available, with a large number of restrictions, are only applicable for ternary efficiency predictions and there is no numerical method available to predict the efficiencies of systems with more than three components.

Significant differences in component tray efficiencies were also measured even when equal component point efficiencies existed across the tray. The eddy diffusion model, taking into account the extent of the liquid backmixing, simulated these differences in individual component tray efficiencies, confirming previous theoretical expectations.

The high tray efficiencies obtained in the absence of the flow nonuniformities and stagnant zones gives further encouragement to design engineers to improve the hydraulics of conventional sieve tray columns for more efficient operation.

Notation

Dg = binary gas diffusivity, m^2/s
E_{mv_i} = tray efficiency
E_{og_i} = point efficiency
F = vapor phase F factor, $V\sqrt{\rho_v}$
K = equilibrium distribution coefficient
N = number of components in system
T = measured temperature, °C
T_b = bubble point temperature, °C
V = vapor velocity, m/s
x = mole fraction of liquid
y = mole fraction of vapour
y^* = equilibrium mole fraction of liquid
ρ_v = vapor density, kg/m^3

Subscripts

1, 2, 3, . . . = component number, lightest component first
i = component i
modif = modified column
P = predicted
recta = rectangular column
n = tray number

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