Spray-to-Bubbly Transition for Distillation Systems Containing Two Liquid Phases

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The tray hydrodynamics were determined for a system containing water and kerosene as immiscible liquids. Plates with hole diameters ranging from 3.18 to 12.70 mm were used in a 44.5-cm^2 perspex air-water-kerosene simulator. Experiments were also carried out in a 50-mm-ID column using different depths of oil and water mixtures to study the drop and bubble mechanisms. A spray-to-bubble transition occurred for the two liquid-phase system experiments. The liquid holdup at the transition increased directly with gas velocity and hole diameter, and decreased with increasing free area. At the same hole velocity, the presence of two liquid phases caused the transition to occur at different liquid holdups than for the single pure liquid. Two different modes of coalescence were observed in the small column work. New correlations have been proposed for the liquid holdup at the transition which allow for the presence of two liquid phases.

Introduction

Foaming in tray columns is a common problem, which was reported by Bolles (1967) and van der Meer (1971a,b). Foaming on trays leads to reduce throughputs, and to avoid excessive entrainment, foaming factors are used in the design method leading to the column being derated to a larger diameter. It has been shown by Davies et al. (1987a) that severe foaming occurs on trays when two liquid phases are present (usually created by a mixture of an organic liquid and water), and little is understood about foaming caused by the presence of two liquid phases on a tray.

Very little information is available in the open literature concerning the behavior of a dispersion when two liquid phases are present on a tray and, therefore, there is a need to determine the behavior of a two liquid-phase dispersion on an operating tray.

In this article, the behavior of a two-liquid phase dispersion will be investigated for a range of trays and gas and liquid-liquid loadings, and the transition between any regime will be determined and quantified. The results for any regimes which are found for trays operating with two liquid phases will be correlated in terms of the parameters investigated such as the liquid holdup at the transition (i.e., the height of clear liquid on the tray). This is an important parameter in tray correlations and design methods.

Previous experience has shown that the spray regime represents a stable mode of operation at high column throughputs for single liquid phases, and thus it is thought that foaming associated with the presence of two liquid phases may be avoided by operating the tray in or close to the spray regime. Thus, this article will investigate whether the spray regime exists in two liquid-phase distillation systems and future work will be carried out to show whether foaming can be minimized by operating in such a regime.

The experimental program has shown that there is a transition between the spray and bubble regimes for systems containing two liquid phases. The liquid holdup at the transition was found to increase with gas hole velocity and hole diameter and to decrease with tray free area. Two different modes of coalescence were observed in the small column work where air was passed through mixtures containing different amounts of kerosene and water. New correlations have been proposed for the liquid holdup at the transition allowing for the presence of two liquid phases.

Air-Water Perspex Simulator Program

Previous work

The study of the thermodynamics of the phase equilibria in three-phase distillation has received some attention, while a number of researchers have simulated the behavior of distil-

Table 1. Details of Sieve Plates Used in Experiments

Tray No.	Hole Size d_h (mm)	No. of Holes	Triangular Pitch P (mm)	% Free Area	Tray Material
1	3.18	1,415	12.70	5.50	perspex
2	4.76	607	19.05	5.32	perspex
3	6.35	313	25.40	4.87	perspex
4	3.18	2,613	9.50	10.18	metal
5	4.76	1,267	14.4	11.10	metal
6	6.35	635	19.1	9.89	metal
7	12.7	149	38.1	9.29	metal
8	3.18	1,415	12.7	5.50	perspex
9	4.76	1,267	14.4	11.10	perspex
10	6.35	635	19.1	9.89	perspex
11	12.7	149	38.1	9.29	perspex
12	4.76	1,267	14.4	11.10	perspex
13	6.35	607	19.66	9.46	perspex
14	12.7	149	38.1	9.29	perspex

lation towers containing two liquid phases in terms of the concentration profile and the number of theoretical stages required for a given separation. Herron et al. (1988) have reviewed the limited information on fluid flow and tray efficiency in three-phase distillation. Recently, Stevens and Furzer (1989) have obtained experimental tray composition profiles for a 13-component mixture consisting of ethanol, water and 11 hydrocarbons in a 610-mm-dia. column fitted with four 10% free area sieve trays, downcomers and weirs.

Some preliminary observations regarding the tray hydrodynamics of a water-kerosene-air system in a perspex simulator were published by Davies et al. (1987c). The hydraulics of a two-liquid-phase system have been investigated by Ashton et al. (1987) who studied the motion of gas bubbling from a single orifice through the interface between two immiscible liquids. The behavior of air bubbling through the water—kerosene mixture was observed over a range of operating conditions, and six different flow regimes were identified. The authors found that these regimes depended on liquid depth, the ratio of the depth of water to oil and the gas velocity through the orifice. Herron et al. (1988) have carried out a series of investigations where several oil-water mixtures were contacted by air in a sieve-tray distillation column simulator. Both hydraulic and mass transfer measurements were made, the latter utilizing acetone as the transferring solute. No unusual problems with foaming or entrainment were observed, and they concluded that the mass transfer efficiency was influenced by the oil-water ratio.

Experimentally, the spray-to-bubble transition for a single liquid phase has been studied by relatively few workers (Fane and Sawistowski 1973; Hofhuis and Zuiderweg, 1979; Payne et al., 1979; Payne and Prince, 1975; Pinczewski and Fell, 1972; Riley 1984; more extensively by Porter and Wong, 1969, and correlations have been proposed by the previous authors and Davies et al. (1987b), Wong and Kwan (1979), Prince et al. (1979), Barber and Wijn (1979), and Lockett (1981). These correlations do not agree with one another. More recently Davies et al. (1987b) found that the tray thickness and the ratio of the hole diameter to the tray thickness have an effect on the transition—an effect which had not been considered by previous researchers. Their experimental results showed that the liquid holdup at the transition for a given hole diameter increased with increasing plate thickness and decreased with increasing ratio of hole diameter to plate thickness. It is apparent from these studies that the gas superficial velocity, free area, hole diameter, gas and liquid densities, plate thickness, and the ratio of the plate thickness to the hole diameter would

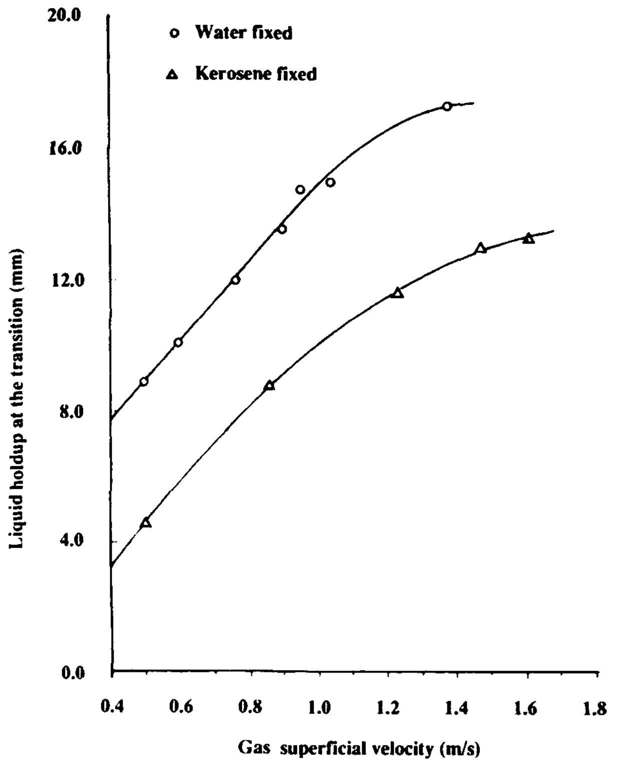

Figure 1. Effect of gas superficial velocity on transition: air-water-kerosene, tray 3.

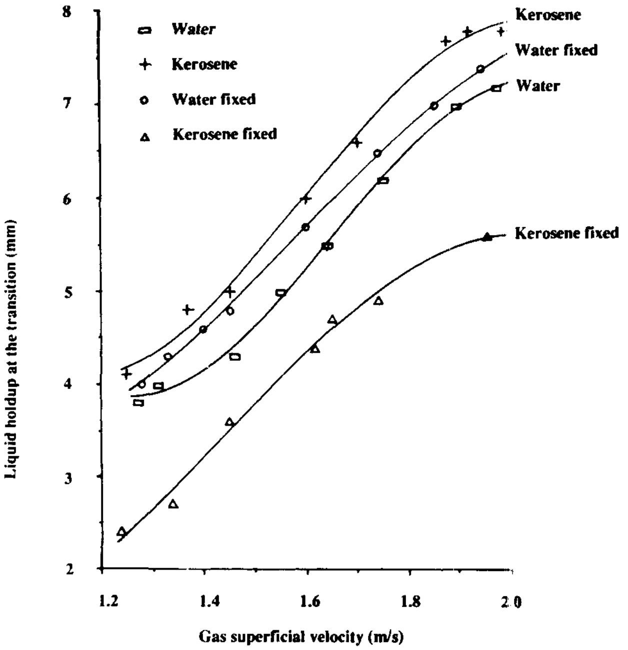

Figure 2. Effect of gas superficial velocity on transition: tray 5.

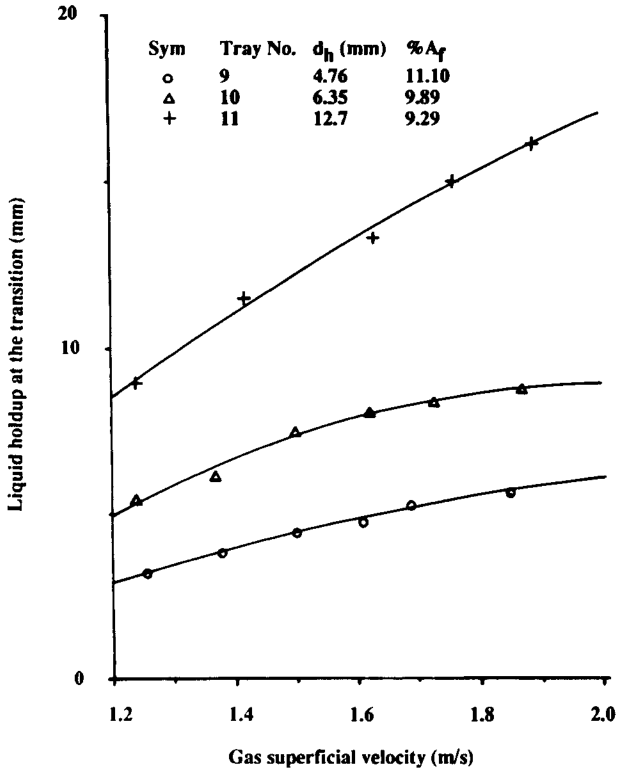

Figure 3. Effect of hole diameter on transition: water-fixed system.

be important parameters in the investigation of the interregime dispersions.

Experimental details

A perspex simulator (45.1 cm by 45.1 cm), similar to that of Porter and Wong (1969) with no downcomers or liquid crossflow, was used for a range of mixtures of water and kerosene. Air was supplied by a blower, the flow rate was varied by a butterfly valve and measured by a manometer connected to a Dall tube. The air blower was started and set to give a flow rate such that weeping would not occur. The liquids were injected from calibrated reservoirs connected to the center of the tray. A T-piece was inserted in the liquid line. One arm of this led to the liquid reservoir, while the other arm led to a manometer so that when the liquid injection was momentarily stopped, the manometer reading gave a measure for the liquid holdup at the center of the sieve plate.

The spray-to-bubble transition was determined using the light transmission technique where a beam of light (located at a known height above the tray floor) was shone across the simulator to a photocell. For a set gas flow rate, liquid was slowly introduced onto the sieve plate. The voltage measured across the photocell represented the light transmitted and was recorded for the known liquid holdups on the sieve plate. This procedure was carried out well past the transition and the actual transition was later determined graphically.

The experimental details and equipment have been described by Ali (1988). Details of the different types of sieve plates used in the experimental program are given in Table 1.

The experimental procedure for the spray-to-bubble transition for two liquid phases was carried out in two different ways:

- A given volume of kerosene was introduced onto the plate so that initially spray was observed on the tray. Water was added onto the tray until a change in light intensity was observed indicating that the transition had been achieved (kerosene fixed).
- A given volume of water was introduced onto the plate so that initially spray was observed on the tray. Kerosene was added onto the tray until a change in light intensity was observed indicating that the transition had been achieved (water-fixed).

Column Modifications. In the initial experimental work, it was difficult to observe the behavior of the liquid dispersion on the tray because of the formation of an emulsion on the inside surface of the perspex walls of the simulator. The formation of an emulsion was also thought to interfere with the transmission of the light beam used in the transition detection technique. Two "port holes" were incorporated in the opposite sides of the perspex walls so that the light beam would pass through an area of perspex which was relatively free from any liquid emulsion film. In addition, a windscreen wiper was fitted to one of the adjacent walls to clean the wall and a small window (15.24 cm^2) was inserted into the opposite wall for photographic and video recording studies.

Experimental results

The preliminary experiments indicated that the tray dispersion behavior did change broadly in the same way as the single liquid phase. Thus, for example, it was possible to observe a bubbly regime and a spray regime, and there was a definable transition between these operating regimes. Therefore, as one long-term objective of this research program was to prevent the formation of foam associated with two liquid phases by deliberately choosing to operate in the spray regime, the remaining experimental work was concentrated on the detemination of the spray-to-bubble transition.

The liquid holdup at the transition increased with increasing gas velocity (Figure 1). The rate of increase of the liquid holdup decreased at higher gas velocities, and the rate of increase was marginally less for the kerosene-fixed system than for the water-fixed system. The data given in Figure 2 for a tray with 6.35-mm-dia. holes were typical of the data found for the remaining trays quoted in Table 1, and all the experimental results for these trays are given by Ali (1988). The liquid holdup at the transition increased with increasing tray hole diameter (Figures 3 and 4), and thus for a given gas superficial velocity small-hole diameter trays favored a bubble dispersion while larger hole diameter trays favored the spray regime. The liquid holdup increased with decreasing free areas Figures 5 and 6 and hence trays with low free areas would tend to favour the spray regime. The liquid holdup at the transition increased directly with increasing liquid density for the kerosene-fixed system only. The opposite was found to occur in the case of the water-fixed system (Figures 2 and 7).

In addition, the following points were noted for the two systems:

Water-Fixed System:

- As the gas superficial velocity increased, the volume fraction of the organic phase at the transition (VFK) increased,

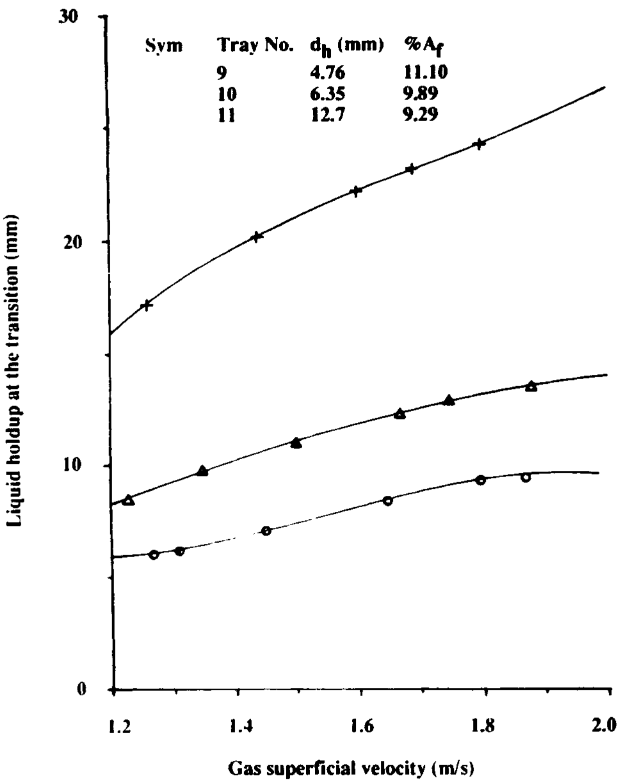

Figure 4. Effect of hole diameter on transition: kerosene-fixed system.

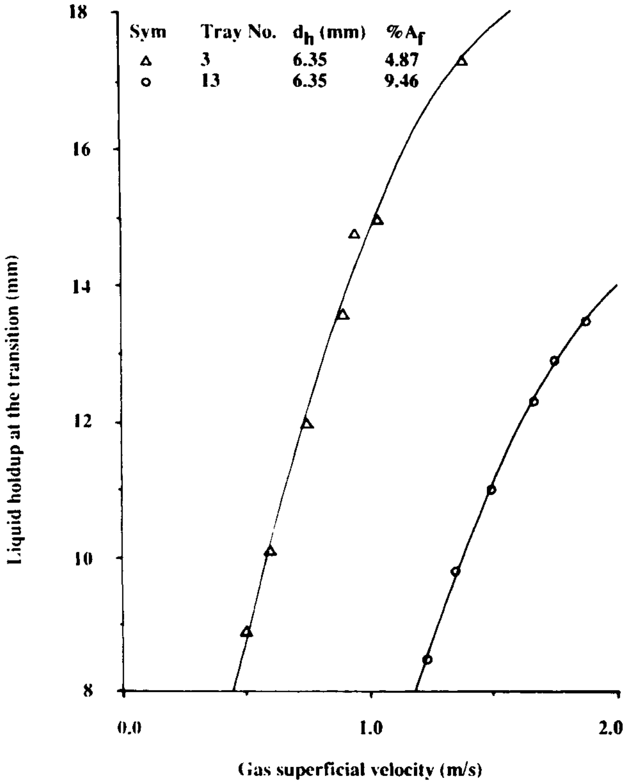

Figure 5. Effect of free area on transition: water-fixed system.

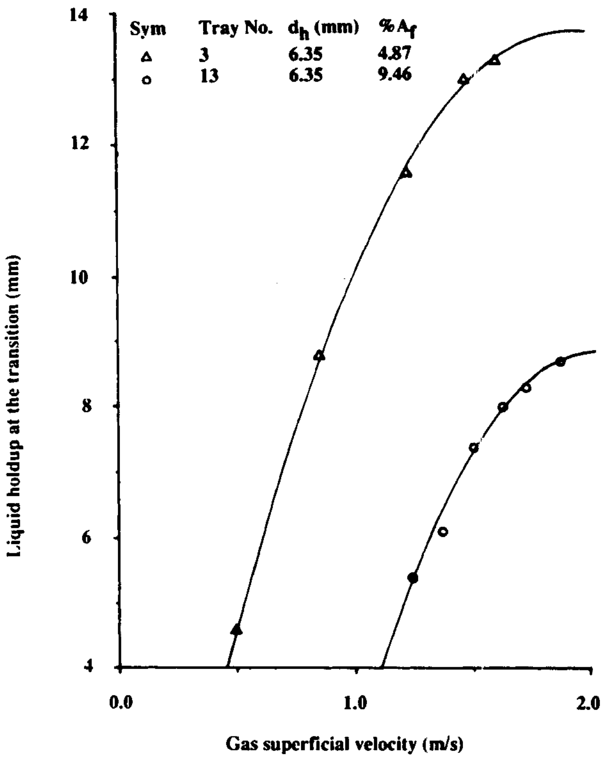

Figure 6. Effect of free area on transition: kerosene-fixed system.

and similarly the volume fraction at the transition of the aqueous phase (VFW) decreased (Figure 8).

- As the gas superficial velocity increased, the ratio of the aqueous phase to the organic phase at the transition decreased (Figure 9).
- As the gas superficial velocity increased, the density of the mixture decreased (Figure 10).

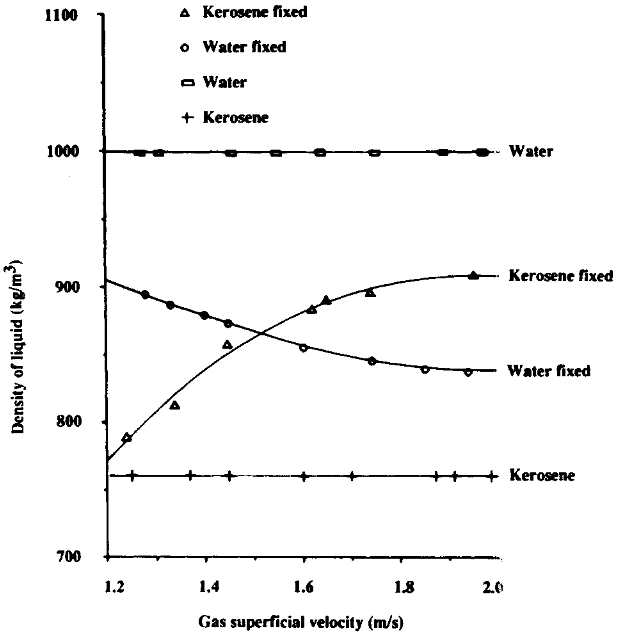

Figure 7. Effect of liquid density on transition: tray 5.

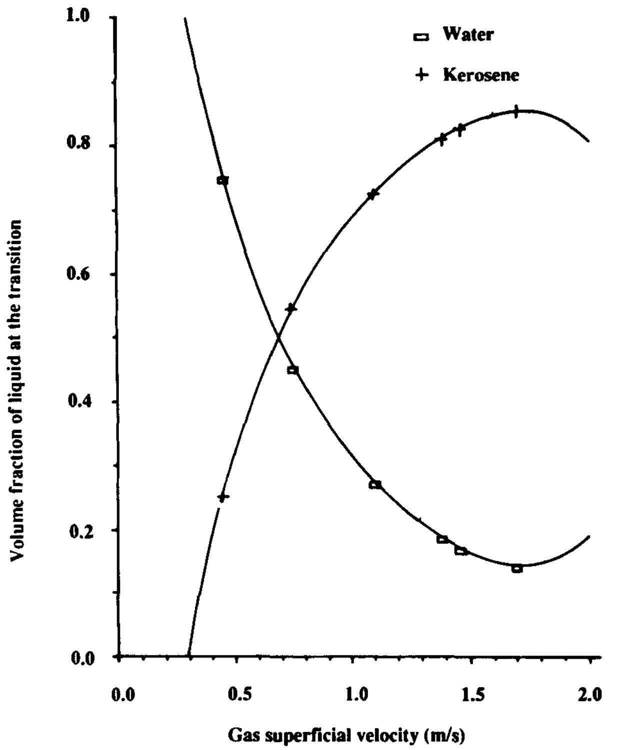

Figure 8. Variation of volume fraction of liquid at the transition with gas velocity: water-fixed system, tray 8.

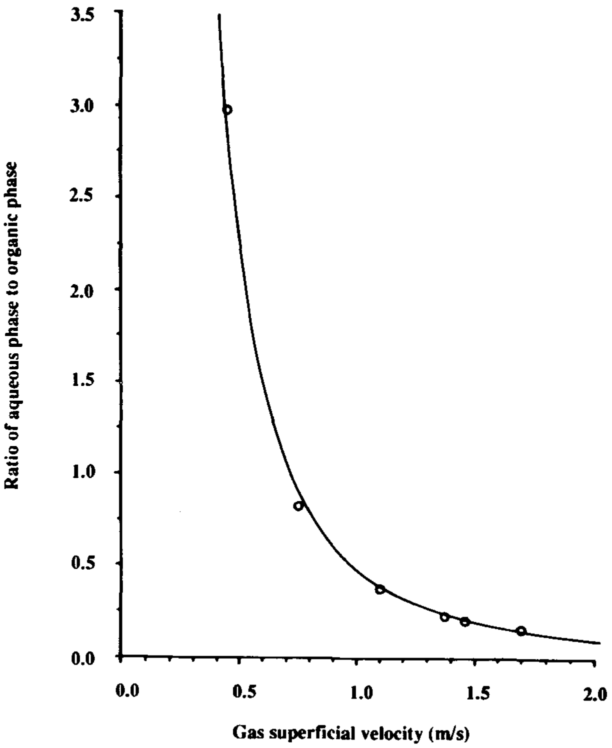

Figure 9. Variation of water/kerosene ratio at transition with gas velocity: water-fixed system, tray 8.

- As the ratio of the aqueous phase to the organic phase increased, the density of the mixture increased (Figure 11).
- There was a linear increase in the density of the mixture with the VFW, and similarly there was a linear decrease in the density with the VFK (Figure 12).
- As the VFK increased the ratio of the aqueous phase to the organic phase decreased, and similarly as the VFW increased the ratio increased (Figure 13).

Kerosene-Fixed System:

- As the gas superficial velocity increased, the volume fraction of the organic phase at the transition (VFK) decreased, and similarly the volume fraction at the transition of the aqueous phase (VFW) increased (Figure 14).
- As the gas superficial velocity increased the ratio of the aqueous phase to the organic phase increased (Figure 15).
- As the gas superficial velocity increased, the density of the mixture increased (Figure 16).
- As the ratio of the aqueous phase to the organic phase increased, the density of the mixture increased (Figure 17).
- The density increased linearly with VFW and decreased linearly with VFK, or an increase in VFM resulted in an increase in density and an increase in VFK resulted in a decrease in density (Figure 18).
- As the VFW increased the ratio of the aqueous phase to the organic phase increased, and similarly as the VFK increased the ratio decreased (Figure 19).

Two important points emerged from the experimental data. In all the transition curves (Figures 1 to 6), the data below the solid line indicated that the column was operating in the spray regime while data above the line would indicate that the column was operating in the bubbly regime. Also for both two liquid-phase examples, the transition curves lay below the corresponding pure-liquid transition curves; e.g., as more kerosene was added to the water-fixed system, the liquid holdup at the

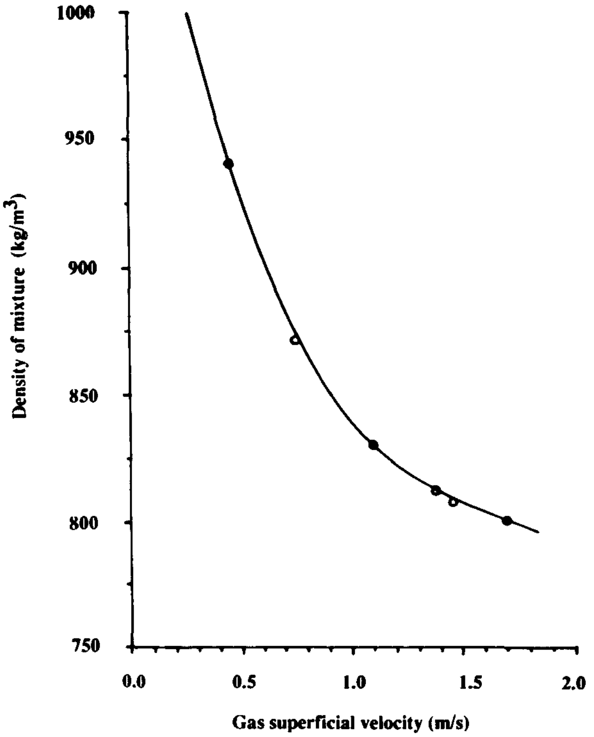

Figure 10. Variation of mixture density with gas velocity: water-fixed system, tray 8.

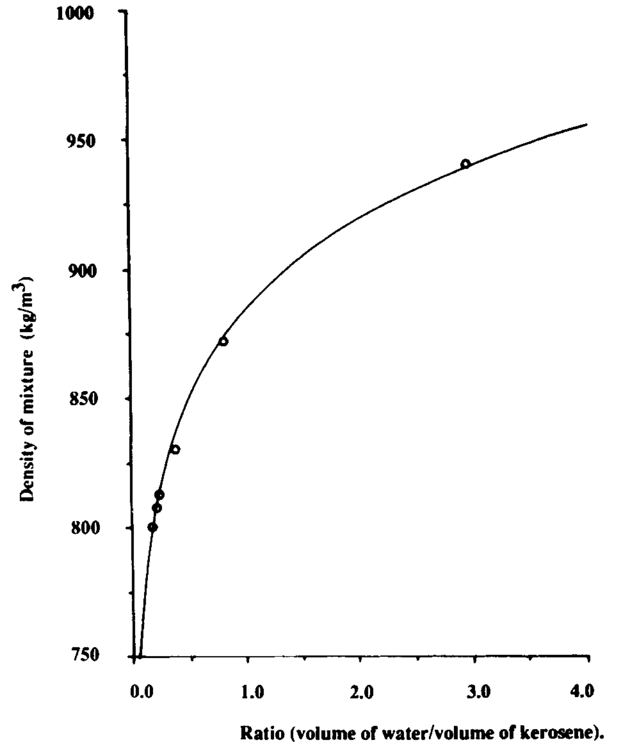

Figure 11. Variation of liquid density with water/kerosene ratio: water-fixed system, tray 8.

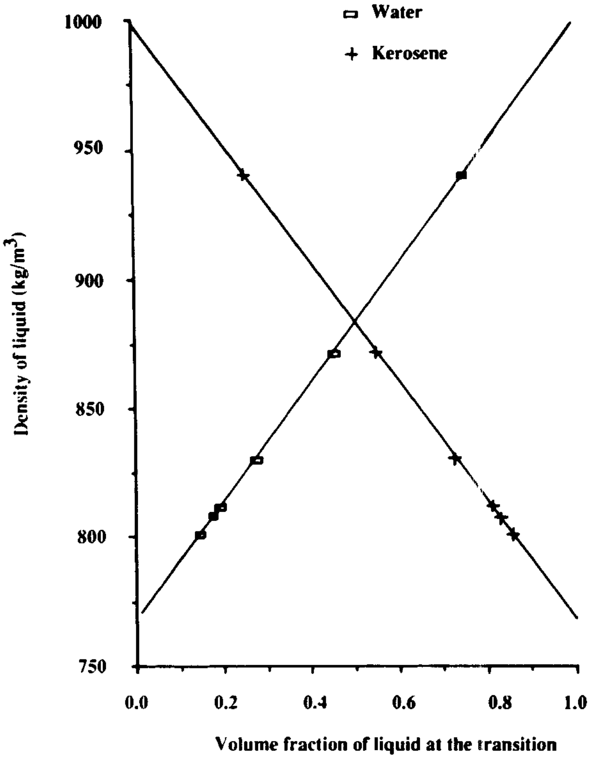

Figure 12. Variation of liquid density with volume fraction of liquid: water-fixed system, tray 8.

transition was always less than that for pure kerosene at the same superficial gas velocity. Thus, a dispersion containing two liquid phases would tend to move into the bubble regime more quickly than the pure liquid operating at the same conditions of gas or hole velocity.

Consequently, the liquid holdup at the spray-to-bubble transition for a system containing two liquid phases may depend on the manner in which the two liquid phases are formed and also the relative amounts of the aqueous and organic liquids in the two phase mixture. Thus, to understand the differences between the water-fixed and the kerosene-fixed systems at the spray-to-bubble transition, a further series of experiments were carried out in a small column.

Small-Column Experimental Program

Previous work

Ashton et al. (1987) found that the operating regimes in a two-phase dispersion above a single-hole plate depended on the liquid depth of each phase (ratio of heavy phase to light phase) and the gas hole velocity. It was decided to repeat these experiments to relate the observations to the results obtained in the perspex simulator. The experiments were carried out in a small glass column using an orifice plate with 1-, 2- and 3-mm-dia. holes.

Experimental details

An air flow rate of 12 L/min was passed through a sintered plate/small glass column (50-mm-dia.) on which 5 cm^3 of water were added from a calibrated liquid reservoir (water-fixed). Then, 15 cm^3 of kerosene was added and observations made

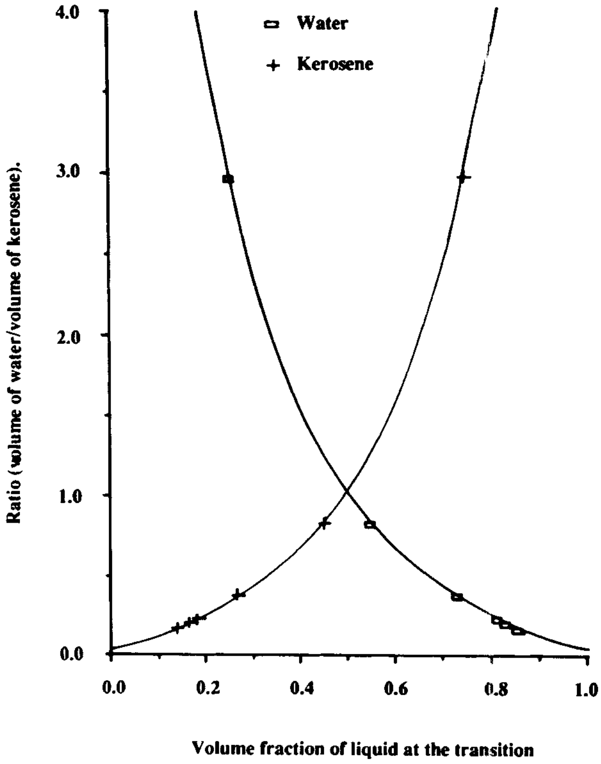

Figure 13. Variation of water/kerosene ratio with volume fraction of liquid: water-fixed system, tray 8.

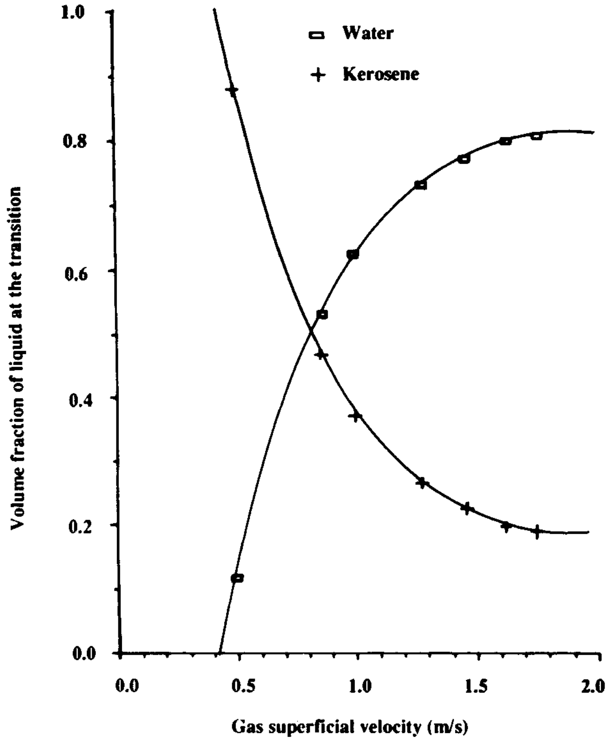

Figure 14. Variation of liquid volume fraction with gas superficial velocity: kerosene-fixed, tray 8.

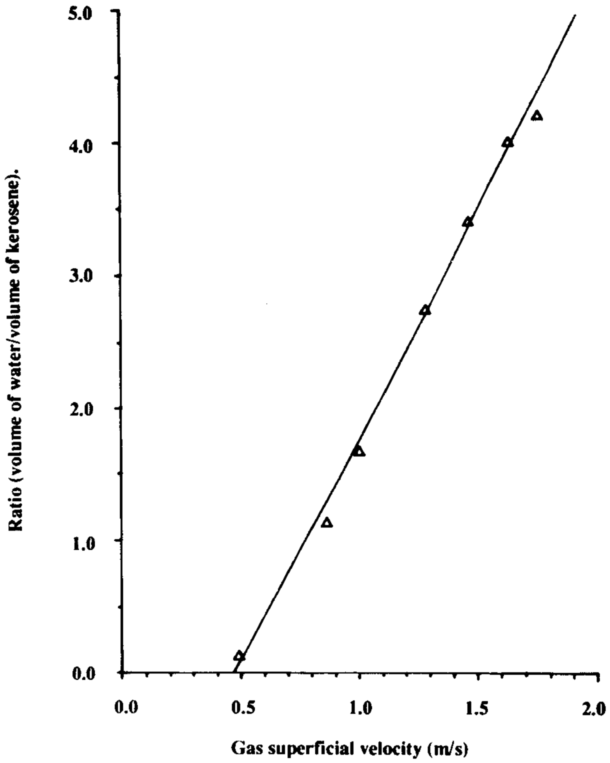

Figure 15. Variation of volume ratio of water/volume of kerosene with gas superficial velocity: kerosene-fixed, tray 8.

on the behavior of the dispersion of the two liquids on the plate. The procedure was repeated using 5 cm^3 of kerosene and 15 cm^3 of water (kerosene-fixed).

Experimental results

At gas hole velocities of less than 0.01 m/s, it was observed that as the air bubble rose through the water layer, some water was entrained with it. As the bubble with a water film surface rose into the kerosene, some water detached from the bubble and fell back onto the liquid-liquid interface. The water drop stayed at the interface until it collapsed into the aqueous phase. The bubble rose up to the liquid kerosene air interface and remained until some more bubbles arrived and accumulated at the interface with subsequent rupture of the bubbles.

The following stages were observed for a drop arriving and coalescing at a plane liquid interface:

- The approach of the drop to the interface and subsequent deformation of the drop and interface profile.
- The damping oscillations caused by the impact of the drop at the interface
- The formation and drainage of a continuous film between the drop and its bulk interface
- Rupture of the film
- The drop contents are deposited onto the interface.

When the water layer is small (less than 0.5 cm), all the water is consumed by entrainment and kerosene is in contact with the tray floor. Thus, the bubbles are formed in the kerosene phase at the orifice and a kerosene film surrounds the bubbles. When the bubbles collapse at the surface, the water droplets fall back onto the tray floor where they are quickly

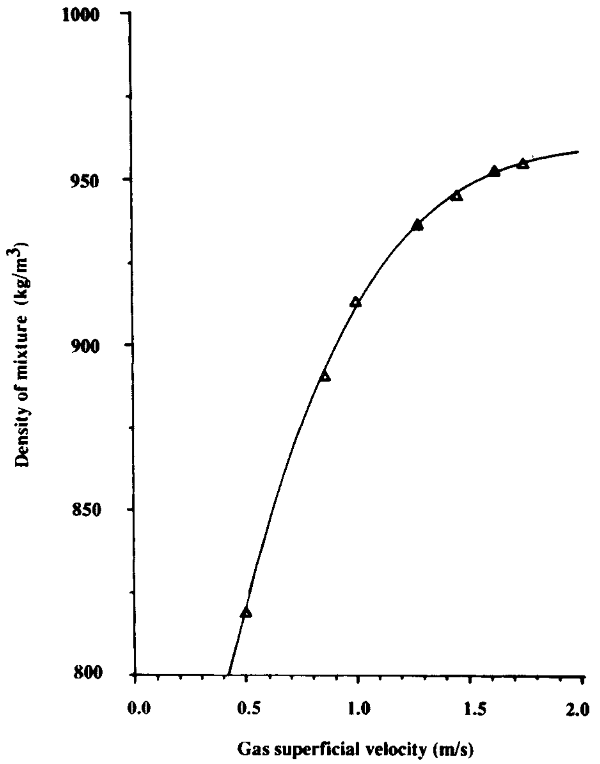

Figure 16. Variation of mixture density with gas superficial velocity: kerosene-fixed system, tray 8.

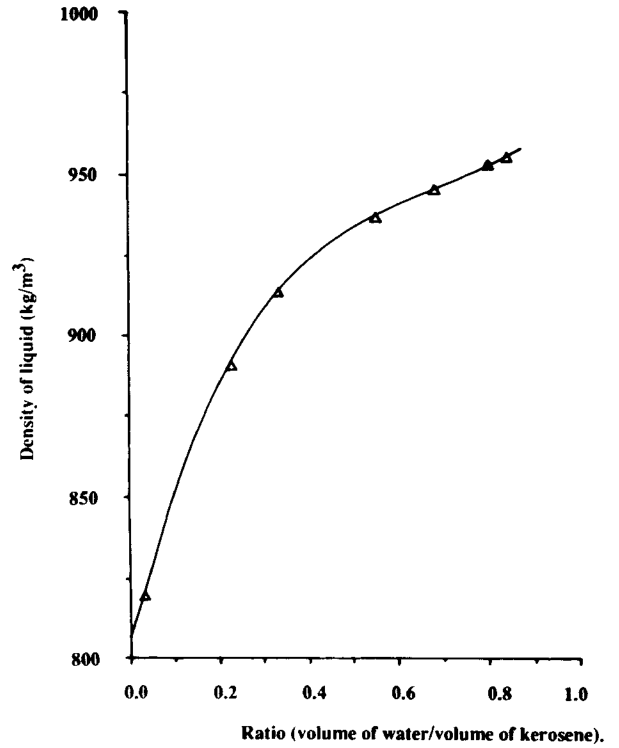

Figure 17. Variation of liquid density with water/kerosene ratio: kerosene-fixed system, tray 8.

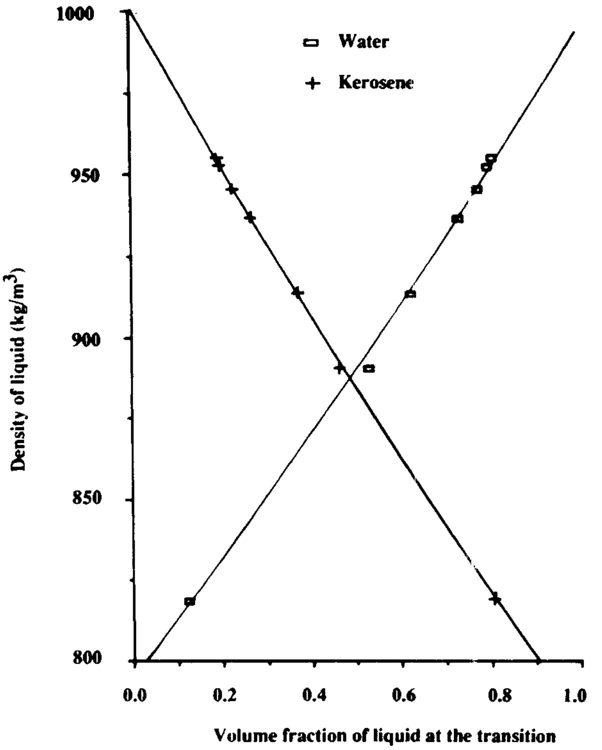

Figure 18. Variation of liquid density with volume fraction of liquid: kerosene-fixed, tray 8.

removed by entrainment after the clean water surface has been formed.

When the depth of kerosene is very small, the bubble reaches the top of the liquid surface without entrainment. When the bubble frequency is large, the bubbles form a continuous channel and there is no entrainment into the kerosene layer. If the volume ratio of kerosene to water is greater than 1.0, with an increase in gas flow rate, kerosene is broken into droplets and forms a well mixed dispersion of kerosene in water. A drop-drop coalescence mechanism could then exist and the possible steps are:

- A binary collison of the drops
- Drainage of the continuous film between the drops at a critical thickness
- Rupture of the film continuous phase
- Consolidation of new drops.

Drop size increased with an increasing number of dispersed-phase drops, probably as a result of more frequent drop collisions. Thus, drop-drop coalescence studies necessitate consideration of collision theory and coalescence processes. With these simple experiments, however, it appeared that when the dynamic balance between droplet breakup and coalescence in the perspex simulator and the orifice plate column was disturbed by an increase in coalescence, phase inversion may occur. In addition, it appeared that at high dispersion concentrations the process of drop coalescence and breakup resulted in the entrainment of the continuous phase by the dispersed phase.

Close to the inversion point, drops of the continuous phase were observed contained within drops of the dispersed phase. It appeared that the continuous process of drop coalescence

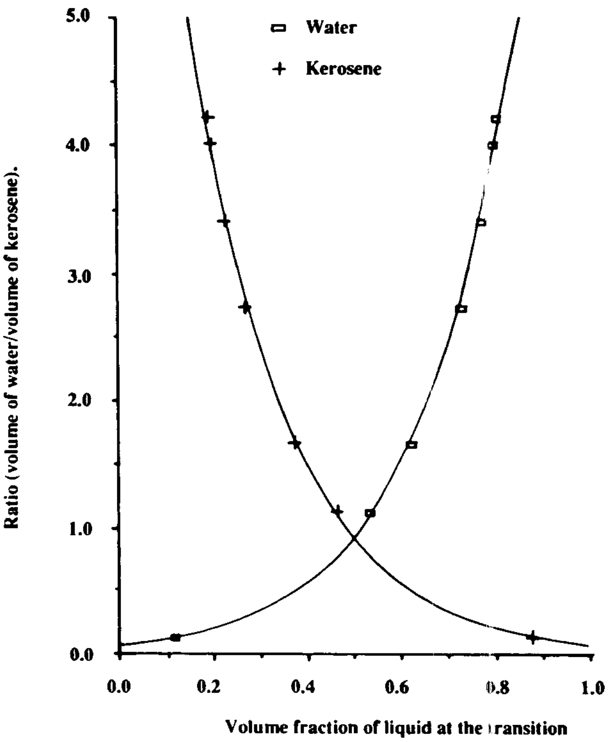

Figure 19. Variation of water/kerosene ratio with volume fraction of liquid: kerosene-fixed system, tray 8.

and breakup resulted in the entrainment of the continuous phase by the dispersed phase.

Discussion

The type of dispersion formed on a sieve plate where two immiscible liquids were present, depended on the volume fraction of the two liquids and their physical properties. Because of the presence of immiscible liquids, coalescence phenomena could be an important mechanism in the behavior of the dispersion on a sieve plate. It is known that drop coalescence followed by breakup affects the mass transfer characteristics of a separation process.

There are two distinct modes of coalescence: drop-interface coalescence and drop-drop coalescence. In both cases, the basic mechanism involved is the approach of a drop to an interface, trapping a film of the continuous phase between the drop and the interface. This film drains away and ruptures at some critical thickness, and subsequently fusion of the drop with the interfacial liquid occurs. Hence, it was concluded that any direct collison will change the hydrodynamics of the coalescing drop. Also, the surface force ($\sigma_i/d_{\text{bubble}}$) arising from the interfacial tension (σ_i) which resists drop formation is smaller for a continuous water phase affecting the tray behavior in both the perspex simulator and the orifice plate column.

The approach of the spray-to-bubble transition in the perspex simulator from the two different directions (i.e., water-fixed and kerosene-fixed) and the different behavior of the two cases may give some insight into the foaming behavior of the systems containing two liquid phases. For the kerosene-fixed system, the low density and low surface tension organic layer on the tray will affect the surface behavior of the bubbles as water is introduced onto the tray causing the surface of the air bubbles to be reinforced by a water film forming around the air bubble. Small pockets of water may be created, as water is entrained from the tray floor by the rising gas bubbles, subsequently this falls through the liquid dispersion as the bubbles collapse at or near the surface. Such pockets of water will be associated with a larger surface tension and hence, the pockets will contract forcing the bubbles apart and creating a bubbly dispersion.

New Correlations for Spray/Bubble Transition for Two Liquid Phases

Although several correlations exist in the literature to predict the spray-to-bubble transition for a single liquid phase, none of these can be applied satisfactorily to a system containing two liquid phases and no correlations have been found in the literature which predict the clear liquid holdup at the spray-to-bubble transition for a system containing two liquid phases. It has been observed from the experimental program that the spray-to-bubble transition for a system containing two liquid phases does depend on the manner in which the two liquid phases were created and therefore two different correlations will be required for the water-fixed and the kerosene-fixed systems.

The following correlations have been proposed for the two systems using the experimental data obtained in this study.

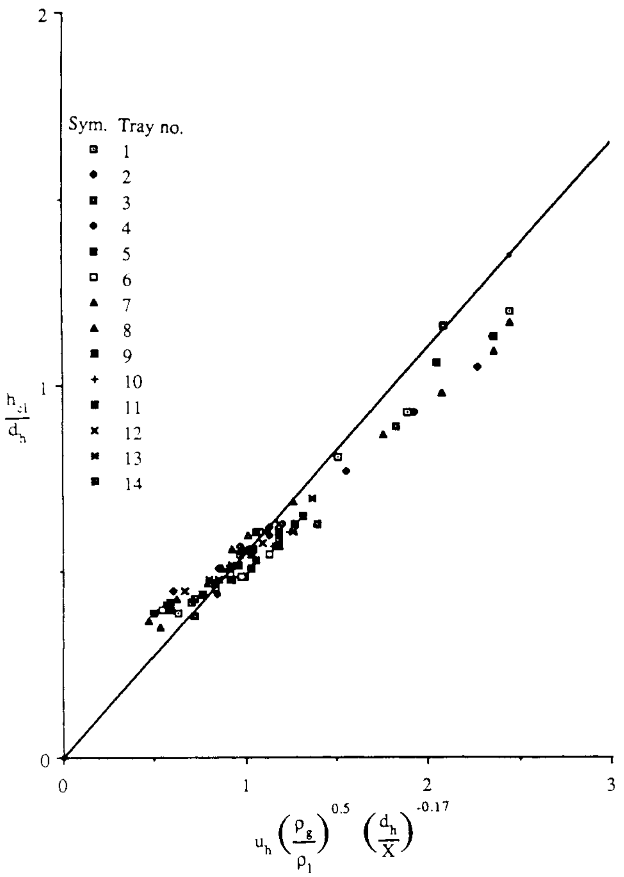

Figure 20. New correlation for spray-to-bubbly transition: kerosene-fixed system.

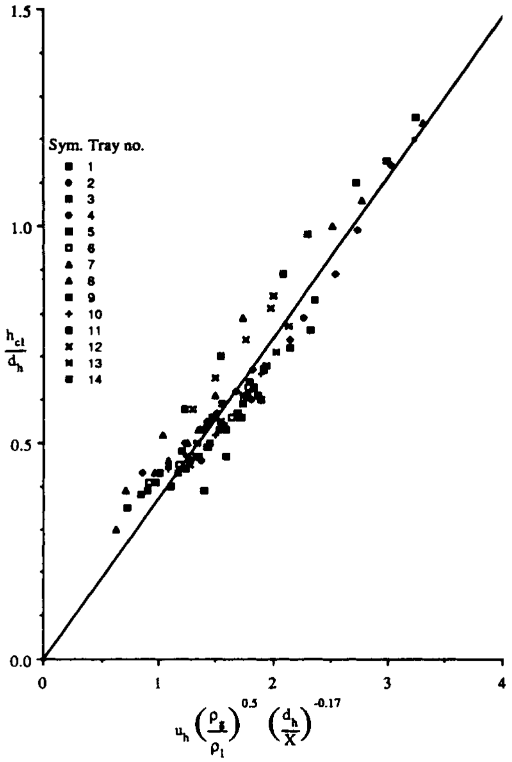

Figure 21. New correlation for spray-to-bubbly transition: water-fixed system.

Water-Fixed System:

$$\frac{h_{cl}}{d_h}=2.64\,u_h\left(\frac{\rho_g}{\rho_1}\right)^{0.5}\left(\frac{d_h}{X}\right)^{-0.17}$$

Kerosene-Fixed System:

$$\frac{h_{cl}}{d_h}=1.86\,u_h\left(\frac{\rho_g}{\rho_1}\right)^{0.5}\left(\frac{d_h}{X}\right)^{-0.17}$$

It can be seen from Figures 20 and 21 that both of these correlations, although empirical, correlate the experimental data well.

Conclusions

- Systems containing two liquid phases, as represented by the water-kerosene-air system, do exhibit both bubble and spray regimes.
- The transition was approached from two different directions. The tray was loaded with a fixed amount of water, and kerosene was added until spray-bubble transition was reached or *vice versa*. The behavior of the system in the water-fixed experiment differed from that of the kerosene-fixed system in that at the same gas hole velocity, the kerosene-fixed system would form a bubble regime at lower liquid holdups.
- At the same gas hole velocity, a two liquid-phase mixture would change into the bubble regime at a lower liquid holdup than for the same conditions as the single liquid phase.
- The liquid holdup at the transition for both the kerosene-fixed and water-fixed systems was found to be a function of the gas hole velocity, hole diameter, the free area of the tray, and the volume fraction of the water and the kerosene on the tray.
- The dispersion formation mechanism on an orifice plate was studied. It was found that the mechanism depended on the volume fraction of the two liquids on the tray and the gas velocity.
- Two correlations are required for the kerosene-fixed and the water-fixed systems to predict the clear liquid holdup at the spray to bubbly transition for two liquid-phase systems.

Notation

A_f = free area of plate (= actual area/bubbling area)
d_h = hole diameter, m
g = acceleration due to gravity, m/s^2
h_{cl} = clear liquid height, m
P = triangular pitch
U_h = gas hole velocity, m/s
X = tray thickness, m

Greek letters

ϵ = gas volume fraction in the liquid phase
ρ_g = gas density, kg/m^3
ρ_1 = liquid density, kg/m^3)

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