Translation of Continuous Phase in the Wakes of Single Rising Drops

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Volumes translated in wakes of single drops of organic liquids rising through aqueous media have been measured. Organic drops studied comprised methyl isobutyl ketone, toluene, and toluene-carbon tetrachloride mixtures. Aqueous media comprised water and water-sugar solution. Distance of drop rise varied from 10 to 20 in. Drop size was varied such that Reynolds numbers of 40 to 800 were obtained. Volumes translated were nearly independent of distance for nonoscillating drops and decreased rapidly with distance for oscillating drops. Transition from nonoscillating to oscillating drops occurred at Reynolds number of 200. Measurements of drop velocities were also made.

In analysis of continuous countercurrent liquid-liquid exchange operations, it is frequently assumed that perfect countercurrent action or slug flow of both phases exists. However in both spray and packed extraction towers the concentration of solute in the continuous phase next to the dispersed phase-continuous phase interface has been reported as differing from that of the fresh entering continuous phase (1, 3, 4, 6, 9, 14). This concentration discontinuity, frequently called *end effect*, cannot be accounted for by postulating an abnormally high rate of exchange at the continuous phase entrance end of the column, because the dispersed phase does not appear to show a corresponding discontinuity in the same region of the column.

Newman (10) suggested that the observed end effect could well be attributed to vertical or backmixing in the continuous phase. Kisliak et al. (7) reported concentration gradients in both continuous and dispersed phases of spray and packed columns. They observed the continuous phase concentration discontinuity as previously reported but they also observed that no corresponding dispersed phase discontinuity occurred; thus experimental support of Newman's back mixing idea was provided.

Patton (12) studied back mixing in a spray column using a technique of injecting water-soluble dye into the continuous, aqueous phase. The extent of backmixing decreased with increase in flow rate of either phase but the effect was most pronounced for the dispersed phase. In fact, at extremely high dispersed phase flow rates backmixing essentially disappeared. Pierce et al. (13) observed temperature end effects in a mercury-water spray column where heat was being transferred from falling mercury drops to the water.

Garner and Tayeban (2) studied a number of aspects of the role of drop wakes in mass transfer. Mass transfer from a drop was greater for a circulating drop interior than for a stagnant interior. The transition from stagnant to circulating drop interior for at least one system occurred at a Reynolds number of approximately 160. Transition from nonoscillating to oscillating was reported as occurring at Reynolds numbers as low as 200 and

high as 460. It thus appears that there may be a connection between internal drop circulation and drop oscillation. Garner and Tayeban also discuss the role of the wake in behaving as sort of a solute reservoir attached to the drop.

Klee and Treybal (8), Null (11), and Hu and Kintner (5) have reported studies of velocity of drop rise or fall. The last suggest that drop oscillation appears to set in at a Reynolds number where drag coefficient change with respect to Reynolds number becomes zero. Torobin and Gauvin (15) review experimental and theoretical studies of the wake formed behind a sphere moving steadily through a turbulence-free field. Wake size is seen to grow with increase in Reynolds number but the onset of drop oscillation or what happens to the wake at this condition is not discussed.

In the present study a dispersed phase drop is formed in a dyed continuous phase. The drop, after reaching a hydrodynamic steady state, emerges from the dyed region into clear continuous phase carrying a dyed wake and rises through a fixed height after which it is trapped in a sampling cup and withdrawn together with an excess of continuous phase. From this experiment it is possible to compute the volume Q of original wake that is translated through the clear continuous phase.

The following development is intended to clarify the concept of volume of translated continuous phase. Define:

Q_i = true volume of drop wave, assumed unchanged with distance of drop travel; also initial volume of DCP in drop wake.

C_i = concentration of dye in dyed continuous phase (DCP); also initial concentration of dye in drop wake when drop emerges from DCP into clear continuous phase (CCP).

C = concentration of dye in drop wake, may change with distance of drop travel.

Q may now be defined in terms of the solute balance equation:

$$Q = \frac{C}{C_i} Q_i \qquad (1)$$

It is thus seen that Q may be regarded as the volume of DCP that is translated in the drop wake. If none of the drop wake is shed to the CCP during the journey of the drop, $C = C_i$ and by Equation (1), $Q = Q_i$. If some of

the drop wake is shed to the CCP, C will be less than C_i and by Equation (1), Q will be less than Q_i. An attempt is made here to measure Q as a function of distance of drop travel, drop size, and system properties. Any understanding which is gained about the behavior of Q should throw light on one possible mechanism of backmixing in spray columns.

THEORY

Volumes of continuous phases translated in the wakes of dispersed drops were determined by use of dye solutions and colorimetric analysis. The dye used, Amaranth red, was shown to obey Beer's law,

$$\ln \frac{T_o}{T} = eC \qquad (2)$$

Aramanth red had a minimum light transmission at a wavelength of 525 millimicrons; consequently this wavelength was used in all colorimetric analyses so as to obtain maximum sensitivity.

In the sampling procedure one may visualize a dispersed phase drop carrying a wake of volume Q and concentration C_i being removed with some additional continuous phase from the region of the sampling cup. The total volume of the withdrawn continuous phase S_b is measured. Let C_b be the concentration of the total sample, C_a the concentration of the continuous phase in the region of the sampling cup, and S_a the volume of the latter material in the sample. An overall volume balance gives

$$Q + S_a = S_b$$

and a solute balance gives

$$QC_i + S_aC_a = S_bC_b$$

Elimination of S_a between these two equations yields

$$Q = \frac{S_b(C_b - C_a)}{C_i - C_a} = \frac{S_b(C_b - C_a)}{kC_d - C_a} \qquad (3)$$

where k is a dilution factor S_d/S_i and S_d is the volume to which a sample of the dyed continuous phase S_i is diluted. Dilution of the dyed continuous phase was usually used because of the relatively high concentration of dye. C_a was usually negligible relative to C_i and so Equation (3) can be closely approximated by

$$Q = \frac{S_bS_i(C_b - C_a)}{S_dC_d} \qquad (4)$$

Applying Beer's law to Equation (4) where T is transmission and its subscripts correspond to those of concentration, we obtain

$$Q = \frac{S_bS_i \ln \dfrac{T_a}{T_b}}{S_d \ln \dfrac{T_o}{T_d}} \qquad (5)$$

It is seen from Equation (5) that absolute concentrations are not needed to compute Q. One needs the above-mentioned sample volumes, and light transmissions of three samples plus that of the reference liquid, distilled water in this case.

EXPERIMENTAL PROCEDURE

Equipment and Materials

All construction materials were glass, stainless steel, Teflon, or polyethylene. A diagram of the apparatus is presented in Figure 1. The column, 3-in. Pyrex glass pipe, was divided
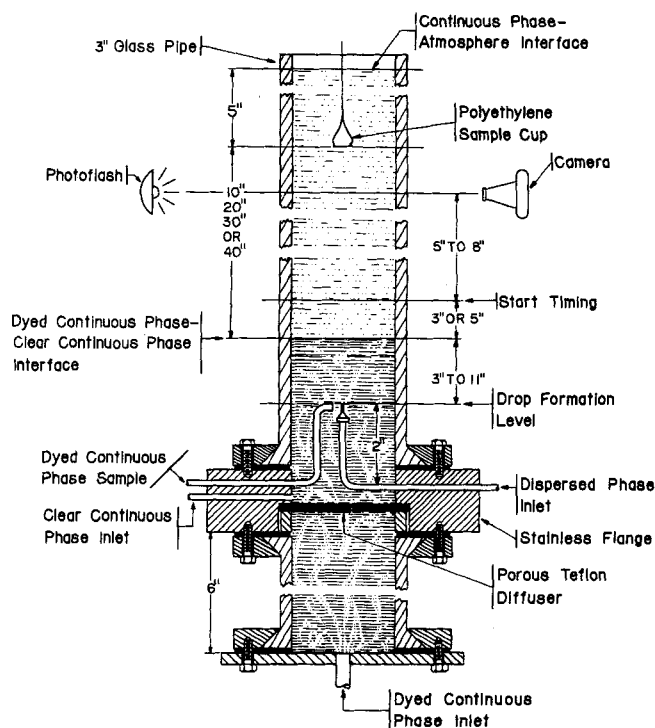

Fig. 1. Column design.

into upper and lower sections by a porous plastic diffuser. The lower section consisted of 6 in. of pipe closed at the bottom by a stainless steel plate and a polyethylene gasket. The upper section consisted of 60 in. of pipe open to the atmosphere at the top. The bottom of the upper section and the top of the lower section were connected to a stainless steel flange which supported the porous plastic diffuser.

A 3/16-in. stainless tube passed horizontally through the flange into the column upper section, turned through 90 deg. to a vertical position near the center of the column, and terminated about 2 in. above the diffuser. To this end of the tube a selected hypodermic needle was mounted and to the other end outside the flange was connected a screw-driven 10-ml. hypodermic syringe by means of polyethylene tubing. This arrangement provided a means of forming single drops of dispersed phase below the dyed continuous phase (DCP)-clear continuous phase (CCP) interface, which was adjusted at a height of 3 to 11 in. above the hypodermic needle. A second 3/16-in. stainless tube through the flange served as a means of sampling the DCP at a point within ¼ in. of the point of drop formation.

A horizontal ¼-in. hole through the flange was fitted with a valve arrangement that permitted filling, flushing, and draining of the upper section.

The CCP was stored in a stainless 10-gal. tank for several hours before use to permit release of dissolved air and to bring the CCP to ambient temperature; it was filtered through 3 in. of glass wool enroute to the upper section. The DCP, stored in 1-gal. glass bottles, was fed to the lower section through polyethylene tubing and a stainless needle valve. Continuous phases were fed to the column under gravity head.

The upper section of column was jacketed with a water-filled 5-in. by 5-in. Plexiglas box whose purpose was to minimize distortion of the photographed drops and to stabilize the column temperature.

Specially fabricated polyethylene sampling cups were made to catch the drops and their translated continuous phase. These cups were approximately cone shaped, with bases of about ¾-in. and heights of 1½-in. Hypodermic syringes of 10 ml. capacity were used to withdraw the translated continuous phase from the sample cups. The cups and syringes were connected with polyethylene tubing.

Fine wires were mounted horizontally on the water-jacket to mark the DCP-CCP interface level, the level for starting

of drop velocity timing, and levels at which the drops were to be caught. The DCP-CCP interface level, 3 to 11 in. above the point of drop formation, was taken as the reference level for translation measurement purposes. Drop velocity timing was started at a level above the DCP-CCP interface and was stopped as the drops were caught at either 10, 20, 30, or 40 in. above the interface. The drops were photographed 11 in. or more above the DCP-CCP interface.

The photographic system consisted of a high-speed photoflash and camera mounted on opposite sides of the water jacket. This method of illumination refracted light away from the drop periphery and produced a sharp contrast between the drop and the surrounding continuous phase. The drop images on the negatives were approximately actual drop size. Three equally spaced fine wires were mounted vertically on each side face of the water jacket and served as means of observing the drop-to-lens distance. The drop diameter measurements were then corrected for variations in the object-to-lens distance. The photographic system was calibrated by suspending a brass rod of known diameter in the water-filled column and photographing it at each of the three reference positions marked by the vertical wires on the water jacket. The calibration was performed each time the column was assembled.

Filtered water was used as continuous phase in most runs, and a 10% aqueous sugar solution was used in the others. Methyl isobutyl ketone, toluene, and mixtures of toluene-carbon tetrachloride were employed as dispersed phases. The toluene-carbon tetrachloride mixtures were formulated to obtain dispersed phases having high surface tensions with densities near that of water so that nearly spherical drops moving at very low velocities could be included in this study. The aqueous and organic phases were mutually saturated before determination of physical properties and before introduction to the column. See Table 1 for physical properties of the systems employed.

Procedure

The 0.5% dye solution (DCP) was fed to the column lower section under gravity head. The flow was stopped when the DCP began to pass through the diffuser. The column upper section was then flushed with CCP until the effluent was dye free. The dispersed phase system was flushed with fresh dispersed phase.

When the upper section was dye free, it was filled with CCP. When convection currents had subsided, a dyed continuous phase layer was formed above the diffuser by passing the DCP into the lower section under gravity head. The DCP was thus forced through the diffuser and into the column upper section. The time required to raise the DCP-CCP interface to the desired level was about 2 min. The interface formed in this manner was quite sharp.

The sample cup was adjusted to the desired height above the DCP-CCP interface (10, 20, 30, or 40 in.). The CCP-air interface was held about 5 in. above the cup mouth. An initial sample of CCP was withdrawn from the cup into a hypodermic syringe before each run. An initial sample of
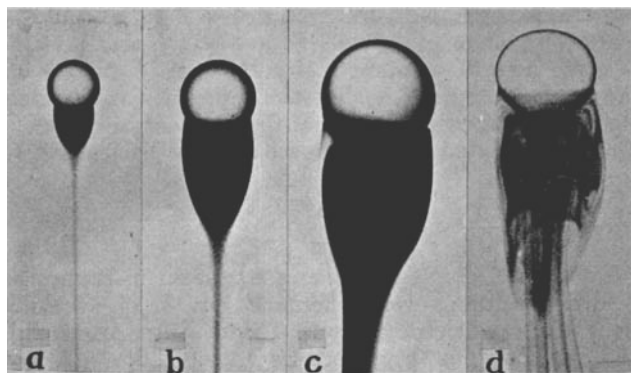

Fig. 2. (a) Tol·CCl₄–H₂O(II) system, nonoscillating behavior, d = 1.62 mm., N_{Re} = 80. (b) Tol·CCl₄–H₂O(II) system, nonoscillating behavior, d = 2.32 mm., N_{Re} = 159. (c) Tol–CCl₄–H₂O(I) system, transitional behavior, d = 3.45 mm., N_{Re} = 215. (d) MIK–H₂O system, oscillating behavior, d = 2.75 mm., N_{Re} = 283.

DCP was withdrawn from a region within ¼ in. of the point of drop formation before most runs.

A single drop was formed and released from the tip of the hypodermic needle below the DCP-CCP interface by turning slowly the driving screw of the hypodermic syringe. Drop rise was timed and the drop was photographed as it passed the camera. The distance of the drop from the camera was estimated from observation of drop position relative to the vertical wires on the water jacket. Timing was stopped as the drop entered the mouth of the sample cup. A sample of CCP was immediately withdrawn from the sample cup into a tared 10-ml. hypodermic syringe. Care was taken to insure that all visible DCP dragged into the cup was drawn into the syringe.

Several runs could be made after each interface preparation. The passage of drops through the DCP-CCP interface disturbed the interface only slightly. The limit on the number of runs for a given column preparation was the build-up of dye at the top of CCP phase rather than disturbance of the DCP-CCP interface.

The volume of translated continuous phase was computed from knowledge of the dye concentration in the DCP (as determined by a Beckman model DU spectrophotometer) and the dye concentration and mass of the captured sample.

The drop photographic negatives and the calibration negatives were enlarged to about 12X object diameter. The actual drop diameters were then computed from knowledge of the calibration object diameter.

RESULTS

Two general patterns of drop behavior were observed visually. The first pattern consisted of a nonoscillating, steady behavior. The drops were spherical or very nearly so in shape and they rose in smooth, vertical paths. For the lower Reynolds number drops, little or no trails of dye were left behind (Figure 2a), but for the high Reynolds number, nonoscillating drops (Figure 2b) faint lines of dye were left behind in the clear continuous phase. The second behavior pattern was characterized by a side-to-side oscillating motion. For the oscillating drops the motion also included a periodic change in shape back and forth between oblate and prolate ellipsoids. Oscillating drops left a trail of dye along their paths in the clear continuous phase. Thus it was apparent that such drops would translate less and less of the original dyed continuous phase in their wakes as the distance of travel progressed. Figure 2d depicts a typical oscillating drop and shows clearly the trail of dye being left behind in the drop path. Figure 2c shows a drop whose behavior is transitional. It did not actually oscillate but it rose diagonally toward the wall of the column in a smooth path and it left a significant trail of dye behind.

TABLE 1. PHYSICAL PROPERTIES OF SYSTEMS STUDIED

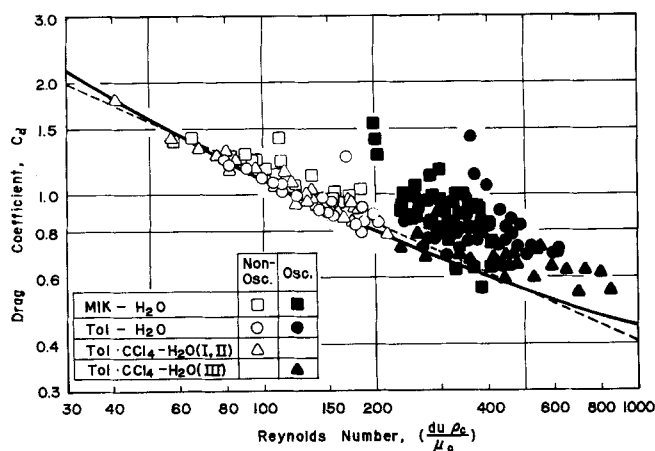|	Density, g./ml.		Continuous phase viscosity, centipoise	Dispersed phase viscosity, centipoise
	Continuous phase ρ_c	Dispersed phase ρ_d	μ_c	μ_d
MIK — H₂O	0.996	0.800	0.873	.570
Tol — H₂O	0.997	0.861	0.880	.546
Tol · CCl₄ — H₂O (I)	0.997	0.945	0.860	.576
Tol · CCl₄ — H₂O (II)	0.996	0.897	0.830	.560
Tol CCl₄ — H₂O (III)	0.994	0.918	0.870	.564
Tol · CCl₄ — 10% Aq. Sugar (II-S)	1.033	0.896	1.05	.560

Fig. 3. Drag coefficient vs. Reynolds number.

Experimentally obtained drop velocity data are presented in Figure 3 in the form of drag coefficient vs. Reynolds number on log-log coordinates. The solid line represents behavior of solid spheres, the open point symbols represent velocity data for nonoscillating drops, and the blackened point symbols denote oscillating drops. It is seen that data for nonoscillating cases scatter less than those for oscillating cases and that the former agree reasonably well with solid sphere behavior. Oscillating drop data are very erratic and lie above the solid sphere line. It is apparent from Figure 3 that for the systems studied, the transition from nonoscillating to oscillating behavior occurs at a Reynolds number of approximately 200. Hu and Kintner (5) and Klee and Treybal (8) indicate that such a simple Reynolds number criterion for transitional behavior would not be expected to be generally valid for all systems. The fact that drag coefficients for oscillating drops become higher than those for solid spheres causes the drag coefficients to level off or perhaps start increasing with increase in Reynolds number. This behavior tends to support the postulation of Hu and Kintner that transitional behavior occurs when drag coefficient change with respect to Reynolds number becomes zero.

Assuming drop velocity to be a power function of drop diameter (d), viscosity (μ_c), and density difference $(\Delta\rho)$, we get, upon application of the method of least squares to the logarithmic form of the function for nonoscillating drop velocity data only

$$v = 10.0 \ d^{0.917} \ \mu_c^{-0.395} \ \Delta\rho^{0.601} \qquad (6)$$

The standard deviation of a single observation of v is about 3% of the true value.

Reference to Table 1 will show that μ_d and ρ_c did not vary significantly among the systems studied. Taking $\rho_c = 1$ g./cc. and $\mu_d = 0.563$ centipoise, we may put Equation (6) into the dimensionless form:

$$C_D = 8.70 \ (N_{Re})^{-0.435} \left(\frac{\mu_c}{\mu_d}\right)^{0.184} \left(\frac{\Delta\rho}{\rho_c}\right)^{0.060} \qquad (7)$$

In the systems studied, $(\mu_c/\mu_d)^{0.184}$ varied from 1.08 to 1.11 and $(\Delta\rho/\rho_c)^{0.060}$ from 0.86 to 0.91. Taking values of these groups as 1.1 and 0.89, respectively, we may approximate Equation (7) by

$$C_D = 8.50 \ (N_{Re})^{-0.435} \qquad (8)$$

and this is represented by straight dotted line of Figure 3.

Experimental results on volumes of continuous phase translated by single dispersed drops (Q) are presented in Figures 4, 5, and 6. Data for both nonoscillating and for oscillating drops are included in the plots. Again it is seen

that the transition from nonoscillating to oscillating behavior occurs at Reynolds numbers of approximately 200. For nonoscillating drops log Q increases linearly with log (drop diameter) and Q appears to be independent of the distance of drop travel; thus Q for this case is the same as Q_i. For oscillating drops the behavior of Q is erratic but it generally decreases rapidly with increase in distance of drop travel.

The plots of Q_i (same as Q) vs. drop diameter for non-oscillating drops suggest that Q_i may be represented by a power function of diameter. One might also assume that variation of Q_i vs. diameter from one system to another should be accounted for by physical properties of the systems. By taking Q_i to be a power function of diameter (d), density difference $(\Delta\rho)$ and viscosity of the continuous phase (μ_c), and applying the method of least squares to the logarithmic form, we obtain the following equation:

$$Q_i = 1.88 \times 10^{-4} \ d^{6.65} \ \Delta\rho^{1.08} \ \mu_c^{-0.71} \qquad (9)$$

The standard deviation of a single observation of Q is about 17% of the true value. Making use of Equation (6) and taking μ_d as constant at 0.563 centipoise, we may put Equation (9) into a dimensionless form:

$$q = 1.86 \times 10^{-5} \ (N_{Re})^{1.90} \left(\frac{\Delta\rho}{\rho_c}\right)^{-0.06} \left(\frac{\mu_c}{\mu_d}\right)^{1.94} \qquad (10)$$

The solid lines of Figures 4, 5 and 6 represent Equations (9) and (10) for the various systems. It is seen that excellent fits are obtained.

Attempts were made to estimate wake volumes for some of the nonoscillating drops from photographs of the drops and their wakes. In all cases the latter method gave volumes somewhat higher, 40 to 65%, than Q's. This may mean that even though little or no exchange appears to occur between drop wakes and continuous phase during the main portion of travel of the drop, some exchange might take place when the drop passes through the DCP-CCP interface. Such exchange could be associated with very slight discontinuities in the physical properties between the dyed region and the continuous phase.

An unsuccessful attempt was made to analyze the Q data for oscillating drops. If one postulates an exchange mechanism operating between the drop wake and the continuous phase in which rate of exchange is proportional to difference in solute concentration, he finds that for a single
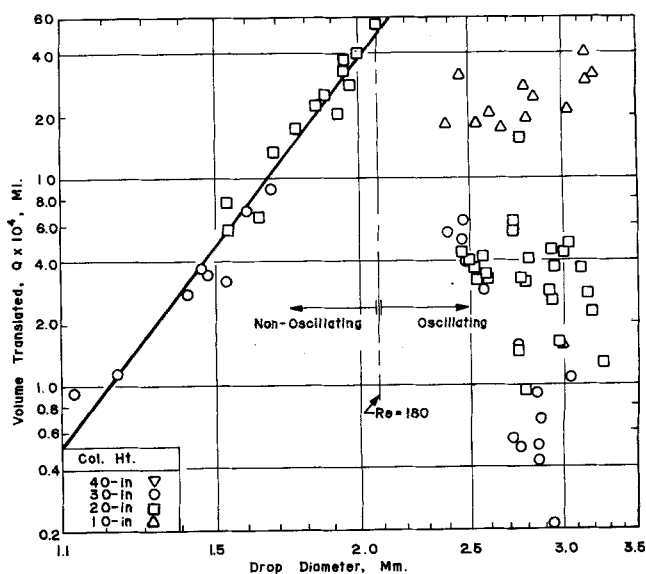

Fig. 4. Volume translated in drop wake as function of drop diameter; methyl isobutyl ketone-water system.
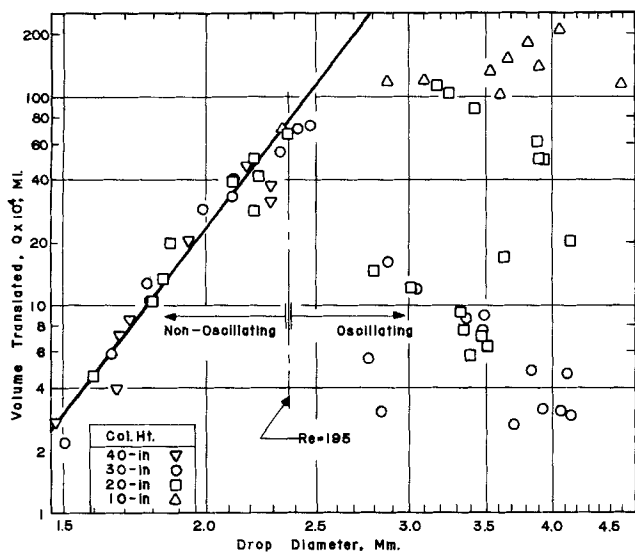
Fig. 5. Volume translated in drop wake as function of drop diameter; toluene-water system.

drop, the logarithm of Q should be linear with distance of travel. Data from Figures 4, 5, and 6 were plotted in this manner and only in the case of plots from Figure 6 was anything remotely resembling a straight line obtained. Data from Figures 4 and 5, particularly for small drop diameters, scattered considerably and not only were not linear but actually showed Q increasing with distance of travel which is physically impossible. Two factors probably account largely for poor results for oscillating drops: Q at small values is extremely difficult to measure because of the very low dye concentrations involved, and the exchange of solute between drop wake and continuous phase takes place in a stepwise and erratic fashion because it is closely associated with the oscillatory motion of the drops.

One possible explanation for experimentally observed end effects in countercurrent liquid-liquid extraction columns is that the dispersed phase carries continuous phase in the wakes of drops from the continuous phase exit end of the column back to the continuous phase inlet end. The worst case would be that where no interchange takes place between the drop wakes and the continuous phase and hence some continuous phase, of exit concentration, would be taken back and mixed with entering continuous phase. Using this assumption, we used the data of the present study to make rough calculations of end effects expected in countercurrent spray columns operating with MIK-pro-

pionic acid-water and with toluene-acetic acid-water systems. The calculated results were compared with experimental observations of Geankoplis et al. (4, 9). In general, order-of-magnitude agreement (predicted end effect ± 50% of experimental) was found, but comparisons were erratic, such that no conclusions regarding mechanism could be drawn. Certainly the backmixing phenomenon is more complex than the simple transport of solute in the wakes of drops.

CONCLUSIONS

The technique using aqueous-soluble dye reported here for measuring volume of continuous phase Q translated by a single drop in its wake appears to give reproducible and reasonable results for nonoscillating drops. For this case Q is independent of distance of drop travel and Equation (9) correlates the Q_i satisfactorily. Equation (6) correlates drop velocity. Also for the nonoscillating drops the drag coefficient vs. Reynolds number plot agrees well with that for solid spheres.

For oscillating drops, Q results were erratic and non-reproducible. Q did appear to diminish rapidly with distance of drop travel, which means that drop wake is shed continually to the surrounding continuous phase as the drop travels its course.

Transition of drop behavior from nonoscillating to oscillating behavior took place at a Reynolds number of approximately 200 for the systems studied here. Drag coefficients for oscillating drops generally were higher than those for solid spheres.

An explanation for the erratic behavior of the oscillating drops is probably partially due to the oscillatory and consequently erratic motion itself. As far as Q is concerned, the wakes also shed dye into the continuous phases intermittently and erratically. Q's for drops having traveled a long distance (particularly the 30-in. case) were very small and consequently measurement of very low dye concentration was involved; here relative errors are high.

Future work with oscillating drops should include wake measurements at smaller increments of drop travel distance and measurements at essentially zero distance from the DCP-CCP. The latter would give values of Q_i and would enable one to extend the range of the correlation of Q_i vs. Reynolds number beyond a Reynolds number of 200.

NOTATION

C = dye concentration, moles/ml.
CCP = clear continuous phase
C_d = drag coefficient, $4\Delta\rho dg/3\rho_c v^2$
d = drop diameter (for ellipsoidal drops, diameter of sphere of same volume), mm. (cm. in Reynolds number)
DCP = dyed continuous phase
e = light adsorption coefficient, ml./mole
g = acceleration due to gravity, cm./sec.^{2}
k = dilution factor, dimensionless
L = continuous phase superficial flow rate in extraction column, cu.ft./(sq.ft.)(hr.)
MIK = methyl isobutyl ketone
N_{Re} = Reynolds number, $dv\rho_c/\mu_c$, dimensionless
Q = volume of translated wake, ml.
q = ratio of translated wake volume to drop volume, dimensionless
S = sugar contained in aqueous phase solution
S = sample volume, ml.
T = light transmission, %
T_o = transmission of a reference or blank sample
Tol = toluene
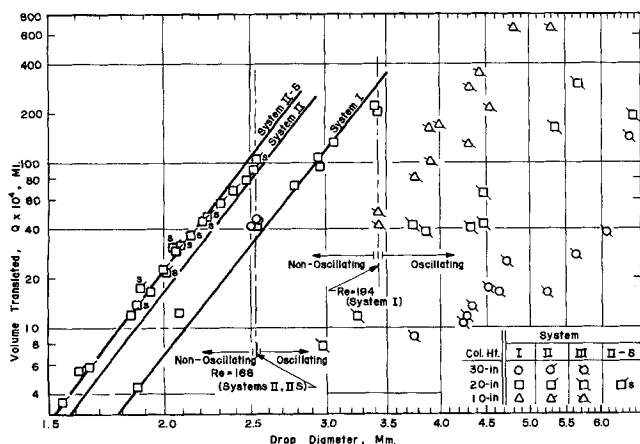Tol · Cl_4 = toluene-carbon tetrachloride solution

Fig. 6. Volume translated in drop wake as function of drop diameter; toluene·CCl₄–water and toluene·CCl₄–aqueous sugar solution systems.

V = dispersed phase superficial flow rate in extraction column, cu.ft./(sq.ft.) (hr.)

v = drop velocity, cm./sec.

x = solute concentration in continuous phase of extraction column, lb.-moles/cu.ft.

y = solute concentration in dispersed phase of extraction column, lb.-moles/cu.ft.

$\Delta\rho$ = density difference between phases, g./ml.

μ_c = viscosity of continuous phase, centipoises [g./(cm.)(sec.) in Reynolds number]

Subscripts

a = CCP sample

b = combined sample of CCP and wake

d = diluted DCP sample

i = wake just after drop formation or to DCP

o = reference solution

1 = continuous phase inlet end of extraction column

2 = continuous phase exit end of extraction column

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Continuous Flow in Packed Thermal Diffusion Columns

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A parallel-plate thermal diffusion column packed with glass wool was operated in continuous flow. The standard equation used for continuous flow through nonpacked columns satisfactorily correlates separation and flow rate. The packed-column theory correctly gives the effects on the regression coefficients of plate spacing and packing permeability, the two variables unique in the packed column. The theory gives poor approximations of the absolute magnitudes of the coefficients, however, which is the same situation found in results from nonpacked columns.

The ordinary open (or nonpacked) thermal diffusion column is difficult to construct and very difficult to use in theoretical research because the wall spacing must be very small, on the order of 0.02 in. or less. However, the same mass separations may be obtained with wall spacings as large as ¼ in. if some packing material fills the working space. Debye and Bueche (1) first reported this advantage in 1948, and some work on the operating variables was conducted by Sullivan et al. in 1957 (2).

Lorenz and Emery (3) developed the theory for packed thermal diffusion columns, and showed that in batch operation this theory is as successful with packed columns as the classical theory is with open columns (4). The

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