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Axial Mixing and Extraction in a Mechanically Agitated Liquid Extraction Tower

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Axial mixing was measured in both phases for a countercurrently operated, mechanically agitated extractor of the Oldshue-Rushton design for water continuous and toluene and kerosene dispersed. Results in terms of an eddy axial diffusivity were correlated in terms of the variables studied. These correlations were then applied to column performance during extraction of n-butyl amine from kerosene into water, and the true mass transfer coefficients, corrected for axial mixing, were determined. These are shown to be predictable for droplets of dispersed phase considered to be rigid spheres.

Axial mixing in liquid-extraction towers is the combined effect of two phenomena: that due to turbulent and molecular diffusion in the axial direction and that resulting from a nonuniform velocity across the cross section of the tower. It exists in both the continuous and dispersed liquids. It is undesirable, since the difference between solute concentrations in the two contacted liquids is thereby reduced, in turn resulting in lower rates of extraction than would prevail under plug-flow conditions.

Atttention was first called to the results of axial mixing in extractors around 1950 (*6, 8, 20*) when a substantial *end effect*, in reality an alteration of the plug-flow countercurrent concentration profile attributable to axial mixing, was noted in spray towers. Since then, methods of taking it into account in the design of extractors have been developed (*17, 24*) but these depend upon having at hand experimental measurements of the axial mixing effect, or means of predicting it. Methods of measurement have been reviewed (*15*). Despite the great importance of the phenomenon in liquid extraction, there have been relatively few measurements in equipment of interest under typical extraction conditions, that is, with two liquid phases flowing countercurrently: for the continuous phase in a small column (*11*); the continuous (*15, 23*), dispersed (*2*), and both phases (*18*) in small pulsed columns; both phases in packed columns (*13, 14*); the continuous phase in a reciprocating plate column (*7*); and both phases in rotating disk contactors (RDC) (*25*). There are no published data for two-phase flow in the types of towers used in the present work, although a report was made (*9*) of measurements taken during the flow of water alone.

EDDY DIFFUSIVITY OF AXIAL MIXING

For devices wherein the concentrations of dissolved solute change continuously with axial distance, on the assumption that the axial mixing can be adequately described by an eddy diffusivity E, the material balances over a differential height of the extractor for the raffinate liquid are

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$$\frac{\partial x}{\partial t} = -\frac{K_x a}{\phi_R}(x - y/m) - \frac{V_R}{\phi_R}\frac{\partial x}{\partial z} + E_R \frac{\partial^2 x}{\partial z^2} \quad (1)$$

and for the extract liquid

$$\frac{\partial y}{\partial t} = \frac{K_x a}{\phi_E}(x - y/m) + \frac{V_E}{\phi_E}\frac{\partial y}{\partial z} + E_E \frac{\partial^2 y}{\partial z^2} \quad (2)$$

In the normal operation of an extractor under steady state conditions, $\partial x/\partial t = \partial y/\partial t = 0$, and these become

$$-\frac{K_x a}{\phi_R}(x - y/m) - \frac{V_R}{\phi_R}\frac{dx}{dz} + E_R \frac{d^2 x}{dz^2} = 0 \quad (3)$$

$$\frac{K_x a}{\phi_E}(x - y/m) + \frac{V_E}{\phi_E}\frac{dy}{dz} + E_E \frac{d^2 y}{dz^2} = 0 \quad (4)$$

The boundary conditions, obtained by material balances for each liquid at each end of the column, are

$$\text{at} \quad z = 0, \quad V_R x_i = V_R x - \phi_R E_R \, dx/dz \quad (5)$$

$$dy/dz = 0 \quad (6)$$

$$\text{at} \quad z = L, \quad V_E y_i = V_E y + \phi_E E_E \, dy/dz \quad (7)$$

$$dx/dz = 0 \quad (8)$$

Tables giving solutions to Equations (3) and (4), assuming E_E and E_R to be constant, have been published (*17, 24*) in terms of the extraction factor $m\ V_E/V_R$, the eddy Peclet numbers $V_R\ L/\phi_R\ E_R$ and $V_E\ L/\phi_E\ E_E$, and the number of overall raffinate transfer units $N_{tOR} = K_x a\ L/V_R$. Also, Sleicher (*24*) has developed an empirical equation from these results which relates the number of transfer units required under axial mixing conditions N_{tOR} to the number N_{tORP} required to accomplish the same extraction under plug flow (that is, no axial mixing) conditions. These latter are given under conditions of constant extraction factor, for example, by the familiar

$$N_{tORP} = \frac{\ln\left[\left(\frac{x_i - y_o/m}{x_1 - y_o/m}\right)\left(1 - \frac{V_R}{mV_E}\right) + \frac{V_R}{mV_E}\right]}{1 - V_R/m\,V_E} \quad (9)$$

In this equation the distribution coefficient is defined in the usual manner, $m = y/x$ at equilibrium, and the extraction factor is $m\ V_E/V_R$. These are the inverse of these terms as used by Sleicher. Thus, if the axial mixing in each liquid is known in terms of the eddy diffusivities E_E and E_R, and if K_x, a, and ϕ_R can be estimated, then the number of transfer units N_{tOR} and hence L, the required length of the extractor, can be calculated by reference to Sleicher's equation.

Alternatively, for truly stage types of devices, where concentrations change in a stepwise manner from stage to stage and are uniform throughout each stage, Gutoff (*9*) has suggested that axial mixing may be characterized in terms of the actual volumetric rate of liquid backflow, and has shown the relationship between this and eddy diffusivity. As he points out, devices of the sort used in this investigation are neither truly differential nor truly stagewise in their behavior, and neither the concept of eddy diffusivity nor liquid backflow will apply perfectly. As the number of stages, or compartments in the case of the present extractor, is increased, the concept of an eddy diffusivity becomes more likely applicable, however. The excellent manner in which the data for the continuous phase particularly follow Equation (11), developed below in terms of eddy diffusivities, indicates that a sufficient number of compartments is evidently present in this work. And since for a related extractor, the RDC, the eddy diffusivity concept has been successfully applied (25), it is this which has been used here.

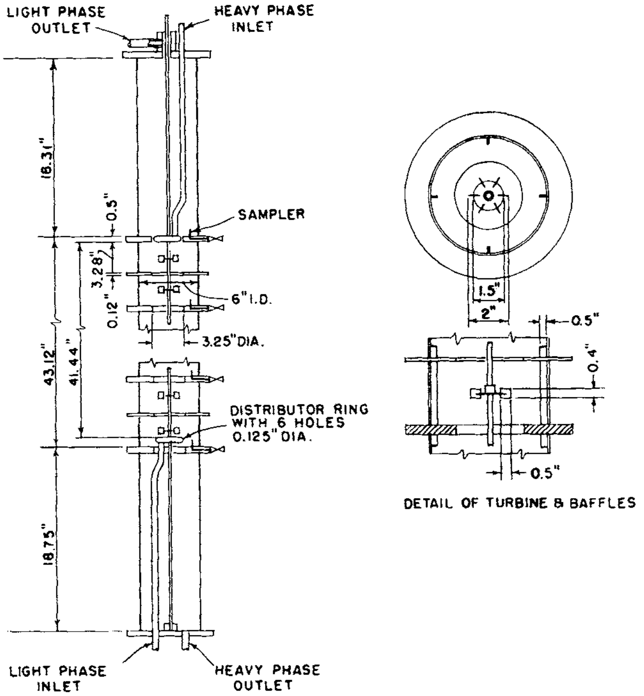

Fig. 1. Mechanically agitated extractor.

MECHANICALLY AGITATED EXTRACTOR

The extractor studied here (Figure 1) is of the Oldshue-Rushton design (*21*). The walls are of Pyrex glass, 6-in. I.D., and the twelve agitated compartments, each 3.25 in. high, are separated by horizontal, annular ring baffles having a central opening 3.25 in. in diameter. Four radial wall baffles, 0.5 in. wide, arranged at 90-deg. intervals, are provided in each compartment. The 2-in. diameter, six-bladed, flat-blade turbine agitators, one centrally located in each compartment, are on a common shaft turned by a variable speed drive at the top (not shown). The settling zones at either end of the tower are each 18 in. long. Small diameter tubes lead from alternate compartments through the horizontal baffles to the outside, and these may be used for injecting liquids into the extractor or for sampling the compartment contents. The extractor is arranged with the usual supply and receiver tanks, pumps, and rotameters for flow rate measurements.

DETERMINATION OF AXIAL MIXING DIFFUSIVITIES

Axial mixing in each of the two phases flowing countercurrently was determined experimentally by pulse injection of a tracer. Such a technique measures the axial mixing resulting from both the contributory effects mentioned earlier. The tracer, a dye solution, that is soluble in only one of the liquids is chosen. The column is placed in operation without transfer of solute between the liquids, and a pulse of tracer is suddenly injected into the column through one of the sampling connections. Samples withdrawn from a position downstream from the point of injection at timed intervals are then analyzed for tracer content. These data provide a curve of c/c_i vs. time, such as those in Figure 2.

Equation (1), applied to the tracer, is simplified since there is no interphase mass transfer. The replacing of x by c, and of V_R/ϕ_R by u, the linear velocity of the liquid under study, produces

$$\frac{\partial c}{\partial t} = -u\frac{\partial c}{\partial z} + E\frac{\partial^2 c}{\partial z^2} \quad (10)$$

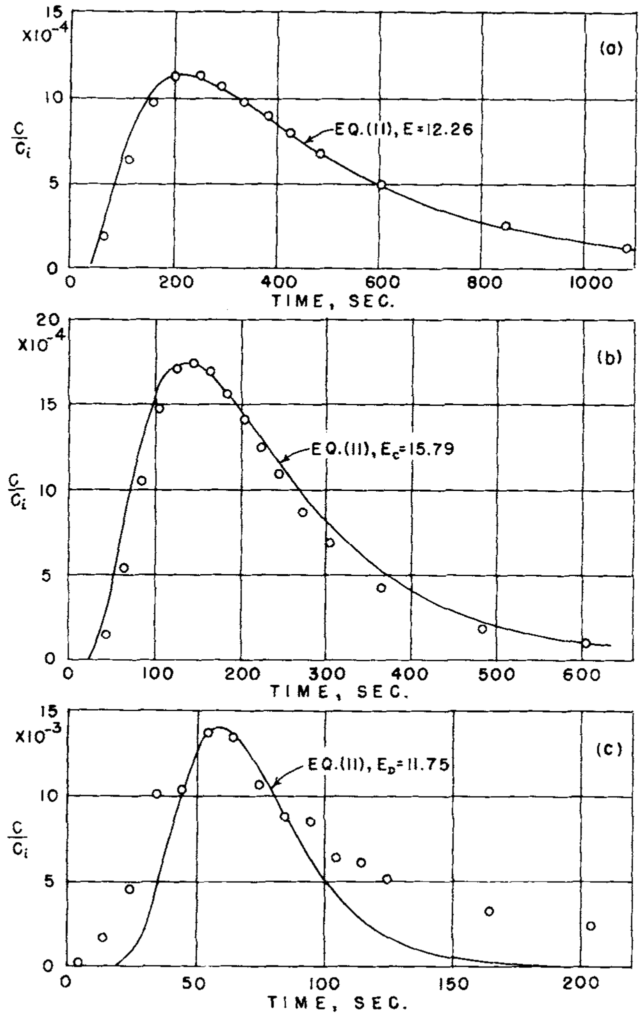

Fig. 2. Tracer concentrations: (*a*) water alone, $V = 22.73$, 300 rev./min. (*b*) water continuous, $V_C = 30.88$, toluene dispersed, $V_D = 34.58$, 200 rev./min. (c) water continuous, $V_C = 27.72$, toluene dispersed, $V_D = 25.84$, 226 rev./min.

which is applicable to study of either phase. Since it was inconvenient to inject the tracer at the point where one of the liquids entered the column, the integration of Equation (10) based on a column extending to infinity was used (5):

$$\frac{c}{c_i} = \frac{Q}{\phi A(4\pi E t)^{0.5}} \exp\left[-(z' - ut)^2/4\,Et\right] \quad (11)$$

where ϕA is the net mean flow area for the liquid being studied. This is the equation of the curves shown in Figure 2. The value of E may be found for a given experiment by trial, choosing the value producing a curve which best follows the experimental points. Since the point on the curve most influenced by E is the peak, it is essential that the calculated curve match the experimental data at that point. It was difficult to obtain truly representative values of ϕ, to which E is sensitive. Consequently, the value of u was determined from the observed time t_m corresponding to the peak c/c_i by differentiating Equation (11) with respect to time and setting the result equal to zero:

$$u^2 = \frac{E}{t_m}\left(-1 + \sqrt{1 + \left(\frac{z'u}{E}\right)^2}\right) \quad (12)$$

The group ϕA was then computed as q/u, and E determined by simultaneous solution of Equations (11) and (12).

Measurements were made with water flowing alone, and also in both liquids when water (continuous) was contacted with toluene and with kerosene (dispersed). Table 1 lists the range of variables studied. Preliminary experiments showed that some relatively small variation in values of E developed from measurements made with different numbers of compartments between points of tracer injection and sampling. This is believed to be the result of the impossibility of realizing experimentally the idealized conditions implied in Equation (11), such as injection of tracer over a zero time period, and instantaneous spreading of the injected tracer throughout the tower cross section. The largest value of E developed when compartments 4 and 10 from the top were used, and since for design purposes such values would be conservative, these compartments were then used for the final measurements. In each case, after 1 hr. of operation to establish steady state, 30 cc. of dye solution (methylene blue for water, Oil-Red-O for organic liquid) were injected at the sampling point by means of a hypodermic syringe. Samples were analyzed for dye concentration with either a model B or DU Beckmann spectrophotometer. Figure 2 shows typical data, and detailed tables of data are available (3).* Better fits of Equation (11) to the data were always obtained for either water alone or as the continuous phase in two-liquid runs. The injected tracer never mixed perfectly with the dispersed phase, and a mixture of colored and clear drops was always seen

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* Data tables have been deposited as document 8766 with the American Documentation Institute, Photoduplication Service, Library of Congress, Washington 25, D. C., and may be obtained for $1.25 for photoprints or 35-mm. microfilm.

Table 1. Range of Variables Studied

	Axial mixing diffusivities		Extraction of butyl amine
	Water alone	Two liquids	
Agitator speed, rev./min.	150 to 401	150 to 300	200 to 275
V_C, ft./hr.	14 to 63	14 to 39	14 to 31
V_D, ft./hr.	—	16 to 43	16 to 35
μ_C, lb./(ft.)(hr.)	1.955 to 2.35	1.855 to 2.66	2.03 to 2.48
μ_D, lb./(ft.)(hr.)	—	1.28 to 3.59	2.66 to 3.04
ρ_C, lb./cu. ft.	62.1 to 62.2	62.0 to 62.4	62.17 to 62.31
$\Delta\rho$, lb./cu. ft.	—	8.30 to 11.70	11.67 to 12.12
ϕ_D, vol. fraction	—	0.05 to 0.36	0.035 to 0.436
σ, (lb.$_m$/hr.^{2}) 10^{-5}	—	7.25 to 9.71	6.48 to 7.11
Calculated d_p, cm.	—	—	0.0496 to 0.0693* 0.0635 to 0.0925†

* Based on Thornton and Bouyatiotis (26).
† Twenty-five percent of Hinze's maximum (12).

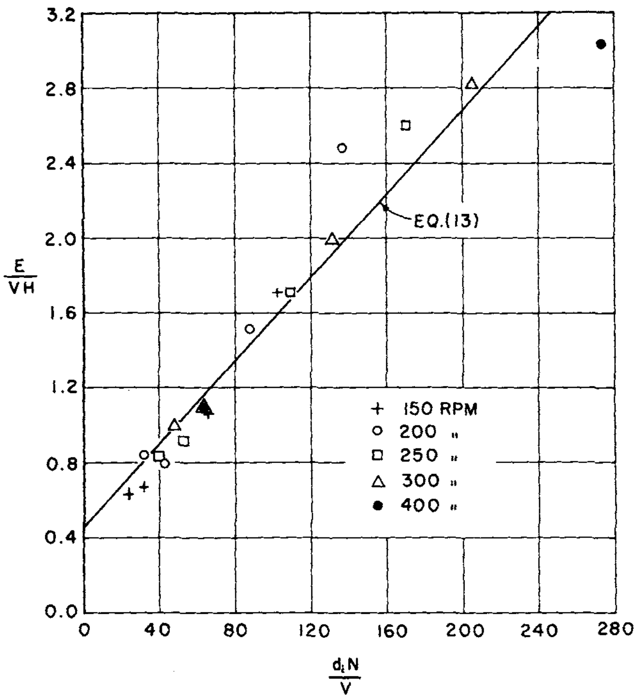

Fig. 3. Single liquid eddy diffusivities.

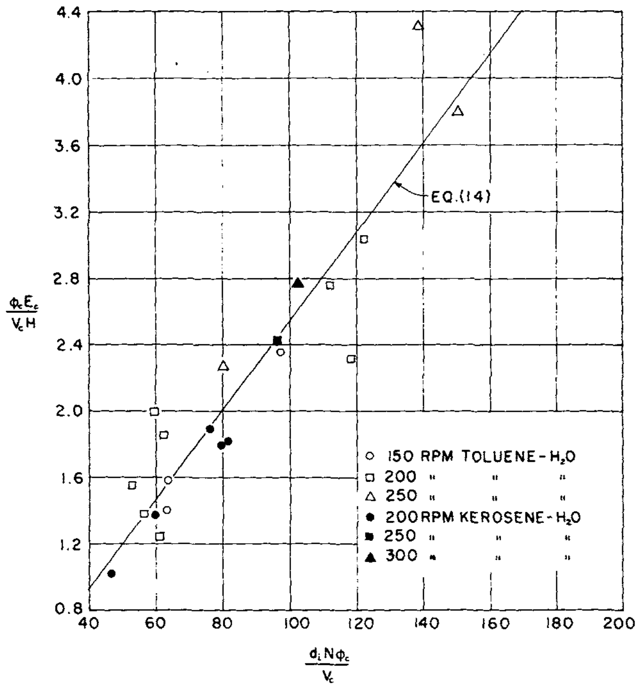

Fig. 4. Continuous phase eddy diffusivities.

in the rising stream. On occasion large colored drops lingered for a long time beneath the horizontal separating baffles, particularly at low agitator speeds. Truly representative samples of the dispersed phase were difficult to obtain. These difficulties account for the greater scatter of points and the longer tails on the experimental curves for the dispersed liquid as compared with Equation (11), but the intermittently upward motion of the drops caught under the horizontal baffles makes it possible that an eddy diffusivity alone may be inadequate to describe the dispersed phase effect.

The data for water alone are empirically correlated by

$$\frac{E}{V\,H} = 0.449 + 0.01118\,\frac{d_i\,N}{V} \tag{13}$$

with maximum and average error of 21 and 9%, respectively, as shown in Figure 3. Those for water in either two-phase systems are described by

$$\frac{\phi_C\,E_C}{V_C\,H} = -\,0.1400 + 0.0268\,\frac{d_i\,N\,\phi_C}{V_C} \tag{14}$$

with maximum and average error of 31 and 10%, respectively, as shown in Figure 4. Neither H nor d_i were varied, and their inclusion in Equations (13) and (14) was suggested by the work of Strand et al. (25), whose results for RDC extractors follow the same form. In the case of Equation (14), the effect of V_D on E_C is taken care of by ϕ_C, which decreases as V_D increases. Unlike the experiences of Strand, however, the data for the dispersed phase could not be described by expressions of this form. Instead, the best relationship found is

$$\frac{d_i^2\,N}{E_D} = 3.93(10^{-8})\left(\frac{d_i^3\,N^2\,\rho_C}{\sigma}\right)^{1.54}\left(\frac{\rho_C}{\Delta\rho}\right)^{4.18}\left(\frac{d_i^2\,N\,\rho_C}{\mu_C}\right)^{0.61} \tag{15}$$

where the groups shown were developed by dimensional analysis, with maximum and average errors of 52 and 18%, respectively, as shown in Figure 5. The impeller diameter d_i was not varied; variations in μ_C and ρ_C occurred only by virtue of the temperature range of the experiments, 15° to 25°C. The group d_i^2N/E_D is a Peclet number formed from the eddy Schmidt number and impeller Reynolds number. There seems to be no important effect of flow rate of either phase.

Equation (15) shows E_D to decrease with increased speed, which seems surprising. It was noted during the experiments, however, that at increased agitator speed stagnant pockets of dispersed liquid which had a tendency to collect under the horizontal annular baffles were more readily swept away, and also more uniformly sized drops, which would have a more uniform rising velocity, were produced. *In this manner, two of the factors contributing to dispersed phase axial mixing are reduced by*

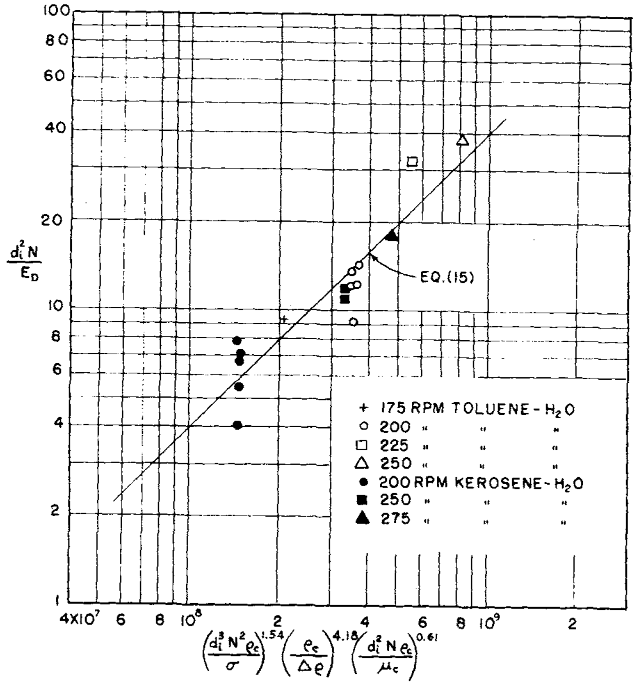

Fig. 5. Dispersed phase eddy diffusivities.

increased agitation. Nevertheless, the net effect of increased agitator speed is to increase the axial mixing effect in the dispersed phase on ultimate column performance, through increased dispersed phase holdup and correspondingly decreased Peclet number $V_D L/\phi_D E_D$.

INFLUENCE OF AXIAL MIXING ON EXTRACTION

In order to demonstrate the utility of the axial mixing measurements, a series of extractions of *n*-butyl amine from kerosene (dispersed) into water was run in the column. The range of conditions covered is shown in Table 1. Amine material balances closed within 3.55 (maximum) and 1.59 (average) %. Detailed data are available (*3*).*

For each run, the axial mixing Peclet numbers were computed through Equations (14) and (15), N_{tORP} through Equation (9), and the true number of transfer units N_{tOR} through Sleicher's results (*24*). From the last, the overall volumetric mass transfer coefficient may be computed: $K_x a = N_{tOR} V_R / L$. The fact that the ratio N_{tOR}/N_{tORP} varied from 1.14 to 3.70 emphasizes the importance of axial mixing.

It is of interest to compare the observed overall mass transfer coefficients with those predictable from available correlations. This requires an estimate of the droplet size, and with this and dispersed phase holdup, the interfacial area: $a = 6\phi_D/d_p$. Average droplet size was first estimated from the correlation of Thornton (*26*), and the observed $K_x a$ converted to K_x, to be compared with a predicted K_x.

Predicted values of K_x depend upon what the nature of the dispersed phase droplets is assumed to be, that is, whether they are assumed to be like rigid spheres or if circulation within the droplet is allowed for. Both types were tried. For rigid spheres, the computations were made through (*1*, *19*):

$$\log k_C = 0.85\, v^{0.03} (\log N_{Re} - 3.504) - (0.550\, v^{1/3} + 0.7605) \qquad (16)$$

and

$$\exp(-k_D a\, t/\phi_D) = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp(-4\,\pi^2 n^2 D_D\, t/d_p^2) \qquad (17)$$

where $t = L\phi_D/V_D$. For circulating spheres (*4*)

$$k_C = \left(\frac{4\, D_C V_S}{\pi\, d_p} \right)^{0.5} \qquad (18)$$

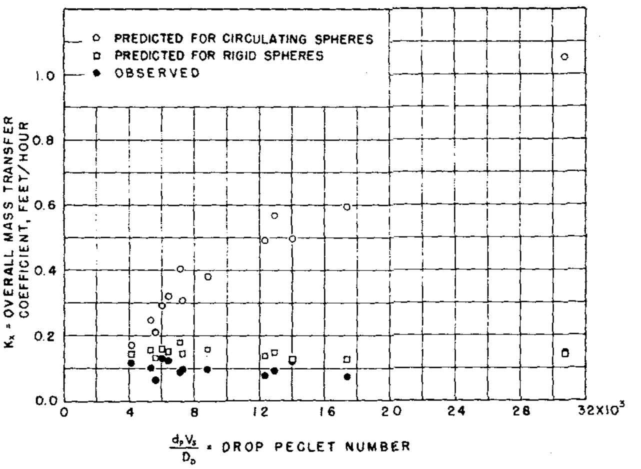

Fig. 6. Overall mass transfer coefficients.

where the slip velocity V_S is

$$V_S = \frac{V_D}{\phi_D} + \frac{V_C}{\phi_C} \qquad (19)$$

and (*10*)

$$k_D = \frac{0.00375\, V_S}{1 + \mu_D/\mu_C} \qquad (20)$$

For either type, the predicted overall coefficient is then given by

$$\frac{1}{K_x} = \frac{1}{k_D} + \frac{1}{m k_C} \qquad (21)$$

K_x's calculated for each type of droplet for each run are compared with the observed values in Figure 6. The observed K_x's fall just a bit below those calculated for rigid spheres. It was noted that drop sizes calculated from Thornton's correlation were on the average 18% of the maximum values predicted by Hinze (*12*), and by raising these to 25% of the maximum, agreement with rigid spheres was excellent.

Agreement of observed coefficients with those for rigid spheres was also the experience of Strand et al. (*25*) with RDC extractors. It is noteworthy that, had the experimental mass transfer rates not been corrected for axial mixing, the resulting overall mass transfer coefficients could only have been predicted by assuming k_D's and k_C's very much smaller than those for rigid spheres, an

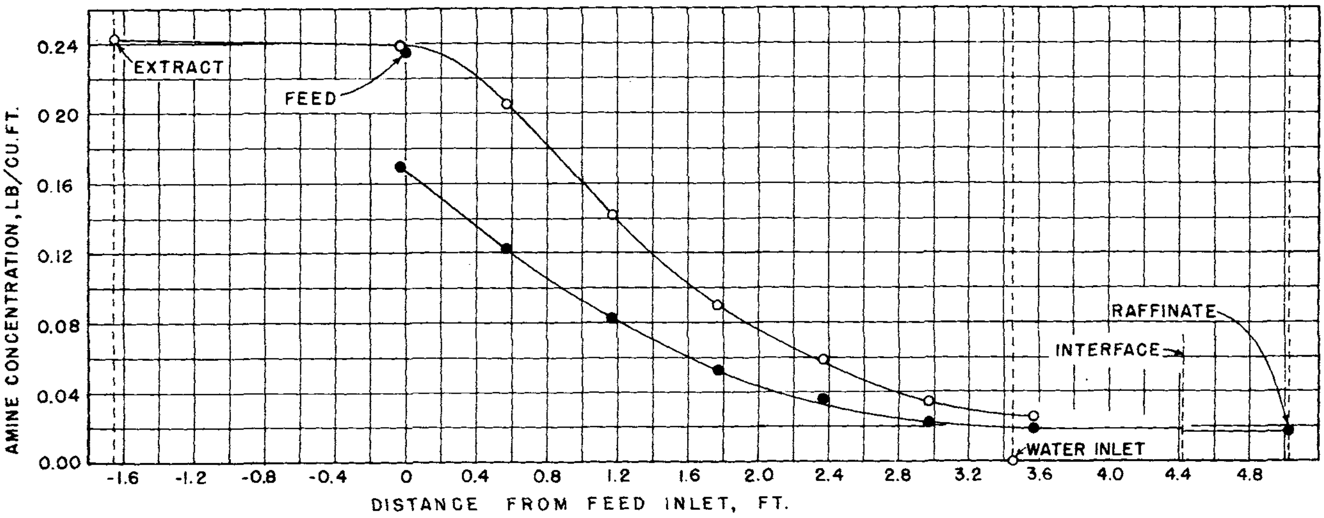

Fig. 7. Concentration profiles, $V_C = 22.8$, $V_D = 25.6$, 275 rev./min.

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* See footnote on page 474.

unlikely situation. This emphasizes the necessity of accounting for axial mixing in the study of such extractors, and points up the likelihood that regularizing of experimental data will not otherwise be possible.

In some of the extraction runs, samples of the column contents were obtained at seven points along the column length. These were two-phase samples, and they were brought to equilibrium before analysis. To calculate the compositions as they existed at the instant of sampling requires that the operating line be located (*27*). This was done by Rod's method (*22*) to account for axial mixing. Concentration profiles within the column were then prepared. Figure 7 is typical; it shows clearly the large concentration changes at both feed and solvent inlets, and the zero slope of the profile curves at the liquid outlets, as required by Equations (6) and (8).

ADDENDUM

The holdups ϕ_D and $\phi_C = 1 - \phi_D$ were determined experimentally for each case in the extraction runs reported here. In order to make Equation (14) more generally useful, we quote here from the work of Wong (*28*), who measured holdup with the systems toluene and kerosene with water (continuous) in the same extractor. The characteristic velocity V_K is defined as

$$V_K = V_S\,(1 - \phi_D) \qquad (22)$$

and V_K is given for this extractor by

$$\frac{V_K\,\mu_C}{\sigma} = 1.77(10^{-4})\left(\frac{g}{d_i\,N^2}\right)\left(\frac{\Delta\rho}{\rho_C}\right)^{0.9} \qquad (23)$$

Holdup ϕ_D may then be estimated through Equations (19), (22), and (23).

NOTATION

A = mean flow area of the extractor = volumetric capacity/length, sq. ft. (0.1822 sq. ft. in this case)
a = specific interfacial surface, sq. ft./cu. ft.
c = tracer concentration, lb./cu. ft.
D = molecular diffusivity, sq. ft./hr.
d_i = impeller diameter, ft.
d_p = drop diameter, ft.
E = eddy axial diffusivity, sq. ft./hr.
g = acceleration due to gravity, ft./hr.^{2}
H = compartment height, nominal (exclusive of gasket thickness), ft. (0.271 ft.)
K_x = overall raffinate mass transfer coefficient, ft./hr.
k = individual phase mass transfer coefficients, ft./hr.
L = distance between liquid-inlet distributors, ft. (3.45 ft.)
m = distribution coefficient = y/x at equilibrium
N = agitator speed, rev./hr.
N_{Re} = impeller Reynolds number = $d_i^2 N\,\rho_C/\mu_C$, dimensionless
N_{tOR} = true number of overall raffinate transfer units, dimensionless
N_{tORP} = number of overall raffinate transfer units for plug flow, dimensionless
Q = volume of tracer of concentration c_i, cu. ft.
q = volumetric flow rate, cu. ft./hr.
t = time, hr.
t_m = time for maximum c/c_i at sampling point, hr.
u = true linear velocity, ft./hr.
V = mean superficial velocity based on mean flow area = q/A, ft./hr.
V_K = characteristic velocity, ft./hr.
V_S = slip velocity, ft./hr.
v = volume of an agitated compartment, cu. ft.
x = concentration of solute in the raffinate liquid, lb./cu. ft.
y = concentration of solute in the extract liquid, lb./cu. ft.
z = distance along the mean flow path from feed inlet, ft.
z' = distance between tracer injection and sampling points, ft.

Greek Letters

$\Delta\rho$ = difference in density, lb./cu. ft.
μ = viscosity, lb./(ft.)(hr.)
ρ = density, lb./cu. ft.
σ = interfacial tension, lb.$_m$/hr.^{2}
ϕ = fractional volume of the contact zone (between liquid-inlet distributors) occupied by a phase, dimensionless

Subscripts

C = continuous liquid
D = dispersed liquid
E = extract
i = initial, outside the extractor
R = raffinate
1 = inside the extractor at raffinate outlet
0 = inside the extractor at feed inlet

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