Mass Transfer Inside Drops

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The transfer rates of *n*-butanol, cyclohexanol, and ethyl acetate into water drops have been measured for circulating and oscillating drops.

Methods of allowing for the end-effect transfer have been extended and measured values compared with predictions of a model.

Various mechanisms for transfer inside drops have been examined in terms of a correlation factor.

Many recent studies on heat and mass transfer rates to and from bubbles and drops as they pass through continuous media have been aimed at elucidating the transfer mechanisms during formation, rise or fall, and coalescence or bursting of the drops or bubbles. In particular, the transfer mechanisms during the steady rise or fall period have been analyzed for a variety of situations. It is apparent that if proposed mechanisms are to be checked experimentally, corrections for transfer during formation and coalescence must be made, or there must be some device for contacting two phases without these end effects.

This paper discusses methods of correcting for transfer in end effects. The experimental work reported is concerned with mass transfer inside liquid drops, and for convenience in discussing the development of the equations used drops heavier than the continuous phase will be discussed. The methods may be extended to other situations.

Possible mechanisms describing the steady-fall period will also be examined.

PREVIOUS WORK

In a recent paper (*15*) the authors discussed end-effect measurements and techniques previously reported in the literature. Attempts to measure end effects in an extraction column were made by Johnson and Bliss (*14*); Sherwood, Evans, and Longcor (*20*); Treybal (*21*) and West *et al.* (*23*). Among workers on single drops Licht and Conway (*17*) and Licht and Pansing (*18*) attempted to determine end effects separately from the steady fall transfer. Coulson and Skinner (*3*) injected and rapidly withdrew drops to study the mass transferred during formation.

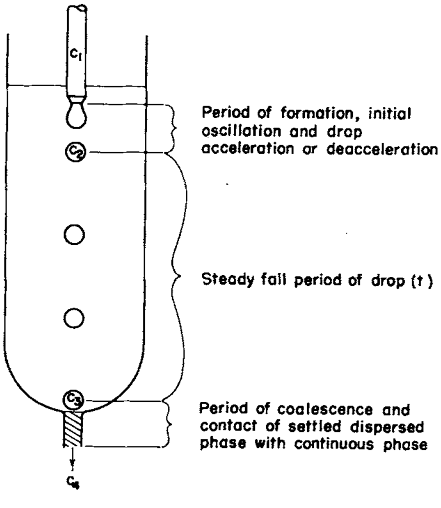

Fig. 1. Definitions of the concentrations and the periods of drop life used in this research.

Other end-effect measurements, by Geankoplis and Hixson (*5*); Geankoplis, Wells, and Hawk (*6*); and Gier and Hougen (*7*), were made by probing the continuous phase to determine abnormal transfer in the end zones.

The present authors have employed a simple mathematical analysis of end effects, introducing efficiencies for these periods, and have developed techniques to be used for plotting data both to check the mechanism and to obtain numerical values of a combined end effect. These methods will be expanded in the theoretical section of this paper.

For heat and mass transfer rates inside drops recent studies (*2, 4, 11*) have centered on the application of theory for a stagnant (noncirculating) sphere or for a drop whose circulation pattern was assumed to be that developed by Hadamard (*9*). The latter situation was analyzed by Kronig and Brink (*16*). Although the Kronig and Brink theory should apply only to slowly moving drops (Reynolds number below unity), it has been used at higher Reynolds numbers. Recently Handlos and Baron (*10*) have modified the Kronig and Brink theory, allowing for turbulent motion inside the drops.

Mass transfer studies involving two component systems have been used in this study. To the authors' knowledge, only Heertjes *et al.* (*11*), working on a spray column, have examined mechanisms for mass transfer inside drops using such systems.

THEORETICAL DISCUSSION

Transfer mechanisms

This first section will summarize the equations describing the transient mass transfer process inside a drop. For all mechanisms spherical drops, constant molecular diffusivity, uniform initial concentration, and absence of any interface resistance are assumed.

Diffusion into a stagnant drop. The fraction approach to equilibrium or the efficiency for the steady-fall period for a stagnant drop with continuous-phase resistance has been derived by Grober (*8*):

$$E_m = \frac{C_2 - C_3}{C_2 - C^*} = 1 - 6 \sum_{n=1}^{\infty} B_n \exp\left[-\lambda_n^2 \frac{Dt}{a^2} \right] \tag{1}$$

Here C_2 and C_3 are the uniformly mixed drop concentrations for the ends of the steady-fall period as indicated in Figure 1. The quantities B_n and λ_n have been tabulated for various Nusselt numbers by Grober (*8*).

For the particular case of no resistance to transfer in the continuous phase this equation reduces to the equation ascribed to Newman (*19*):

$$E_m = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp\left[\frac{-n^2 D \pi^2 t}{a^2} \right] \tag{2}$$

Diffusion into a circulating drop. For negligible interfacial tension and for a drop Reynolds number less than unity Kronig and Brink (*16*) derived the following equation for diffusion into a circulating drop with no continuous-phase resistance:

$$E_m = 1 - 3/8 \sum_{n=1}^{\infty} B_n^2 \exp\left[-\lambda_n \frac{16Dt}{a^2} \right] \tag{3}$$

Elzinga and Banchero (*4*) obtained the same equation for the case of a finite continuous-phase resistance and calculated values of the quantities B_n and λ_n for various Nusselt numbers. Table 1 summarizes values of B_n and λ_n for the case of no continuous-phase resistance (as encountered in this study).

Transfer into a drop at high Reynolds numbers. Handlos and Baron (*10*) proposed a circulation model which included radial motion. For their mechanism

$$E_m = 1 - 2 \sum_{n=1}^{\infty} B_n \exp\left[-\frac{\lambda_n 16Dt\, N_{Pe'i}}{2048 d^2} \right] \tag{4}$$

TABLE 1. SUMMARY OF VALUES OF B_n AND λ_n FOR EQUATION (3) WITH ZERO CONTINUOUS-PHASE RESISTANCE

n	B_n	λ_n	Source
1	1.29	1.656	
2	0.596	9.08	(*4*)
3	0.386	22.2	
4	0.35	38.5	
5	0.28	63.0	(*11*)
6	0.22	89.8	
7	0.16	123.8	

They claim that only one term of this series with $\lambda_1 = 2.88$ need be used. They derived an effective diffusivity as a function of radius as

$$E_{(r)} = \frac{D\, N_{Pe'_i}}{2{,}048}\,(6r^2 - 8r + 3) \qquad (5)$$

where

$$N_{Pe'_i} = \frac{N_{Pe_i}}{(1 + \mu_i/\mu_o)} \qquad (6)$$

A correlation factor for diffusion inside drops. The factor R proposed by Korchinski (*2*) and used by him with the Vermeulen equation (*22*) is used in this study as a multiplier of the molecular diffusivity to compare transfer rates with rates encountered in a stagnant drop. The following modification of Equation (1) may be considered to define R:

$$E_m = 1 - 6\sum_{n=1}^{\infty} B_n \exp\left[\frac{-\lambda_n^2 RDt}{a^2}\right] \qquad (7)$$

The Vermeulen equation is an empirical fit of Equation (1), and hence the R value used by Korchinski is essentially the same as the one defined by Equation (7).

Definition of end effects

With reference to Figure 1, the fractional approach to equilibrium in the formation, steady-fall, and coalescence periods may be defined by

$$E_{f1} = \frac{C_1 - C_2}{C_1 - C^*},\; E_m = \frac{C_2 - C_3}{C_2 - C^*},\text{ and}$$

$$E_{f2} = \frac{C_3 - C_4}{C_3 - C^*} \qquad (8a,b,c)$$

Here C_1, C_2, C_3, and C_4 are the uniformly mixed concentrations illustrated by Figure 1.

In practice only C_1 and C_4 are directly measurable, and the only accurately measured efficiency is the over-all or total efficiency:

$$E_T = \frac{C_1 - C_4}{C_1 - C^*} \qquad (9)$$

By simple algebraic manipulation of (8) and (9) the following equation results:

$$E_m = \frac{E_T - E_{f1} - E_{f2} + E_{f1} \times E_{f2}}{1 - E_{f1} - E_{f2} + E_{f1} \times E_{f2}} = \frac{E_T - E_F}{1 - E_F} \qquad (10)$$

where E_F is a combined end effect, $E_{f1} + E_{f2} - E_{f1} \times E_{f2}$.

At constant drop-formation time, drop size, coalesced dispersed-phase holdup, and continuous-phase flow rate, E_{f1} and E_{f2} may be considered constant. This will be considered more fully below.

Determination of E_F and R

Low efficiency studies. For low E_m values ($E_m < 0.5$) the following empirical equation has been found to fit Equation (7):

$$E_m = 0.905\sqrt{\frac{RD\pi^2 t}{a^2}} + 0.0189 \qquad (11)$$

This is an improvement over the Vermeulen equation as used by the authors in a previous study (*15*).

Combining (10) and (11) results in

$$E_T = 0.905\,(1 - E_F)\sqrt{\frac{RD\pi^2 t}{a^2}} + (0.0189 + 0.981\,E_F) \qquad (12)$$

At low efficiencies and for a constant R a plot of E_T against the square root of contact time should yield a straight line the intercept of which may be used to yield E_F. Knowing E_F, one can use the slope of the line to obtain the correlation factor R.

High efficiency studies. At higher E_m values all but the first term of the series on the right side of Equation (7) are negligible. Equation (7) reduces to

$$\ln(1 - E_m) = -\lambda_1^2\frac{RDt}{a^2} + \ln 6B_1 \qquad (13)$$

Combining (13) and (10) results in

$$\ln(1 - E_T) = -\lambda_1^2\frac{RDt}{a^2} + \ln 6B_1(1 - E_F) \qquad (14)$$

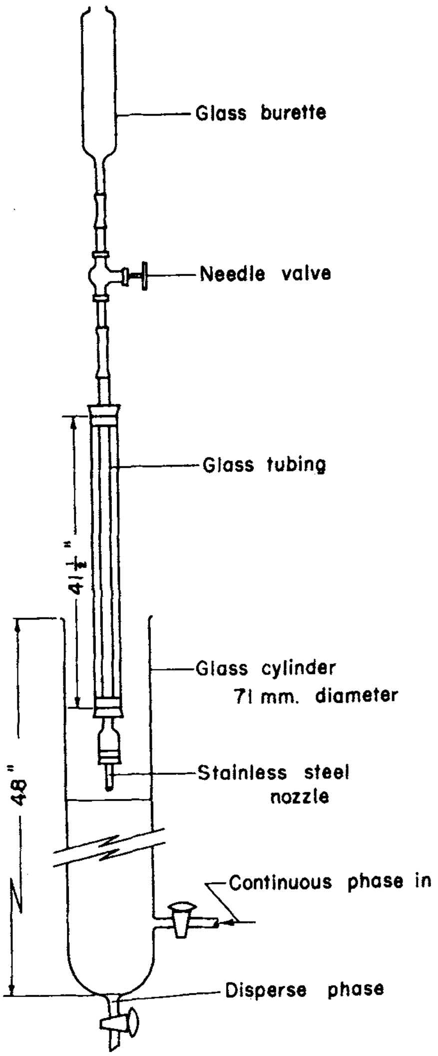

Fig. 2. Apparatus for studying mass transfer into circulating and oscillating drops.

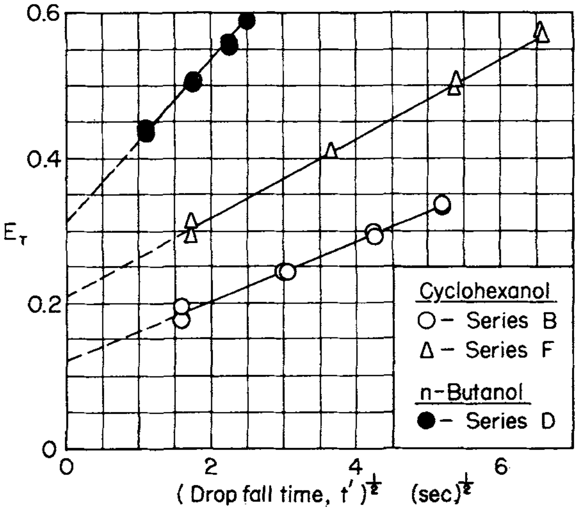

Fig. 3. Typical plot of data in accordance with Equation (12).

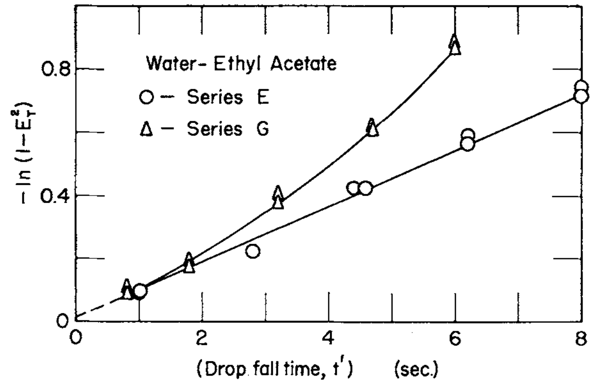

Fig. 4. Plot of data for ethylacetate diffusing into water, in accordance with modified Vermeulen equation.

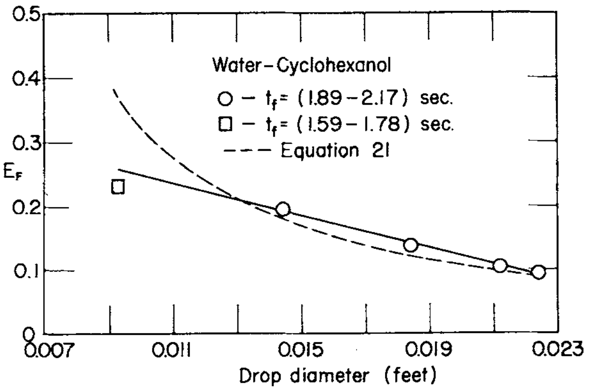

Fig. 5. Experimental E_F for water-cyclohexanol compared with a theoretical equation.

For high-efficiency data for a constant R, Equation (14) dictates that one should plot ln $(1-E_T)$ against time of contact to obtain E_F from the intercept and R from the slope. A knowledge of B_1 and λ_1 is necessary.

Prediction of R for various mechanisms

It is apparent from the above discussion that for data for noncirculation drops application of either Equation (12) or (14) should yield $R = 1$. For a circulating drop under the conditions of the Kronig and Brink model R should be about 3.

For drops with turbulent circulation R should be larger. When one uses the Handlos and Baron model for comparison purposes,

$$R = \frac{N_{Pe'_i}}{2{,}048}\int_0^1 (6r^3 - 8r + 3)\,dr$$

$$= \frac{N_{Pe'_i}}{2{,}048} \qquad (15)$$

Hence this model predicts that R is directly proportional to the modified Peclet number.

For any mechanism it is apparent from Equation (10) that

$$\ln(1-E_m) = \ln(1-E_T) - \ln(1-E_F) \qquad (16)$$

whence

$$\frac{d\ln(1-E_m)}{dt} = \frac{d\ln(1-E_T)}{dt} \qquad (17)$$

for E_F constant

It follows from equation (17) that on a plot of ln $(1-E)$ against contact time the slope of the experimental curve is independent of the magnitude of the combined end effect. This is a necessary condition that a transfer mechanism for the steady-fall period holds.

Transfer mechanisms during formation and coalescence

In order that the method discussed above for the calculation of the combined end effect be applicable it is necessary that E_{f_1} and particularly E_{f_2} be independent of concentration.

By assuming Higbie's equation (*13*) for transient diffusion into the surface of a drop as it is formed, Heertjes *et al.* (*11*) developed the following equation for the formation efficiency:

$$E_{f_1} = \frac{20.6}{d}\sqrt{\frac{D\theta}{\pi}} \qquad (18)$$

For the coalescence end it was assumed that as each drop settled it spread as a layer of initial uniform concentration C_s across the previously settled phase and that transient mass transfer occurred until the next drop arrived to cover the surface.

If the interface area is A_i and the instantaneous rate is again given by Higbie's equation, the amount transferred is that which occurs in time θ and is equal to the mass gained by the drop in the coalescence zone:

$$\int_0^\theta A_i(C^* - C_s)\sqrt{\frac{D}{\pi t}}\,dt$$

$$= 2A_i(C^* - C_s)\sqrt{\frac{D}{\pi}}\,\theta^{1/2}$$

$$= v(C_4 - C_s) \qquad (19)$$

whence

$$E_{f_2} = \frac{2A_i}{v}\sqrt{\frac{D\theta}{\pi}} \qquad (20)$$

By these mechanisms it is apparent that E_{f_1} and E_{f_2} should be functions of the drop size and time of formation. These variables and, no doubt, the continuous-phase velocity must be kept constant during a given series of runs in which the contact time is varied. It is particularly important to argue that E_{f_2} is independent of the concentration C_s, since the latter varies with the column height during a particular study for a given drop size and drop rate.

Combining Equations (18) and (20) yields the following estimate of the combined end effect:

$$E_F = E_{f_1} + E_{f_2} - E_{f_1} \times E_{f_2} =$$

$$\left(\frac{20.6}{d} + \frac{2A_i}{v}\right)\sqrt{\frac{D\theta}{\pi}} - \left(\frac{41.2A_iD\theta}{dv\pi}\right) \qquad (21)$$

Experimental details

The data reported and discussed in this paper are for the transfer of cyclohexanol, *n*-butanol, and ethyl acetate into water drops falling through these phases previously saturated with water. The relevant physical properties for these compounds are listed in Table 2.

Figure 2 shows the simple apparatus used in this study. The continuous phase was contained in a column 71 mm. I.D. This column was equipped with a drain cock for continuous removal of the settled dispersed phase and also with a side arm near the bottom for adjustment of the level of the continuous phase in the column.

TABLE 2. RELEVANT PHYSICAL PROPERTIES FOR THE SYSTEMS USED IN THIS STUDY AT 24.5° ± 0.5°C.

	Cyclohexanol	*n*-Butanol	Ethyl acetate
ρ_o (lb./cu. ft.)	59.5	52.4	55.8
μ_o [(lb./(ft.)(sec.)]	0.0120	0.00191	0.000336
σ [lb./(sec.)^2]†	0.00716	0.00330	0.0113
C^* (wt. %)	3.90	7.35	8.73
D (sq. ft./sec.)‡ 10^8	0.910	0.957	0.998

† *D was determined with water as solvent by the use of the Wilke correlation (*24*).
‡ σ was determined by the pendant drop method (*1*).

Table 3. Nozzles Used in This Study

Series	Nozzle type	Size, in. I.D.	O.D.
C	45 deg., beveled end	9/30	1/2
B	45 deg., beveled end	7/30	1/2
A	45 deg., beveled end	1/6	1/4
D	45 deg., beveled end	3/20	1/4
E	45 deg., beveled end	1/10	1/4
F	45 deg., beveled end	1/12	1/4
G	plane-end, thin-wall tube	3/60	
H	45 deg., beveled-end glass tube	1/30	1/4
J	plane-end, hypodermic needle	B-D 20	
K	plane-end, hypodermic needle	VIM 23	

The holdup of coalesced dispersed phase and the diameter of the interface at coalescence were kept constant for all runs at approximately 0.75 cc. and ⅜ in. respectively.

The water was admitted from a burette through a long jacketed nozzle with interchangeable tips of various diameters. For the most part, sharp-edged nozzles of the type used by Hayworth and Treybal (*12*) were used; details are listed in Table 3. All drops except for the series *A*, *B*, *C*, and *D* for the water-butanol system were formed in the continuous phase. For the series noted, the drops were formed about one drop diameter above the continuous phase to prevent the continuous phase from running up the nozzle.

The drop rate and the drop volume were determined by counting the drops formed from a given volume of water over a time period. The drop contact time was obtained by timing the fall of drops by a stop watch, the watch being started as a drop detached itself from the nozzle and stopped when the drop struck the coalesced dispersed phase.

Drops of each size and for the various drop-formation times indicated in Table 4 were allowed to fall through various depths of the continuous phase. The first settled dispersed phase was discarded; then the settled aqueous phase leaving the column was sampled until a steady final concentration (C_4) was obtained.

All experimental work was carried out in a constant-temperature room at 24.5° ± 0.5°C. The dispersed phase leaving the apparatus was analyzed with a Bausch and Lomb dipping refractometer which had been previously calibrated with known solutions.

For series *A* and *B* (where $E_F = 0.100$) for the water–*n*-butanol system a layer of linseed oil was used to separate the continuous phase from the coalesced dispersed phase in an effort to lower E_{f_2}. However for series *B* without linseed oil as a separating layer E_F was found to be less than it was when the separating layer was used. This indicated that the linseed oil separating layer served no useful purpose and was abandoned in later work.

DISCUSSION OF DATA OBTAINED

Use of the proposed plotting techniques

Figure 3 shows typical data plotted in the manner indicated by Equation (12), that is as E_T directly against $t^{1/2}$. Intercept values on such graphs yielded E_F values, and the slopes of the lines combined with these E_F numbers gave numerical values of the correlation factor. This method was found suitable for *n*-butanol and for cyclohexanol diffusing into water.

Figure 4 indicates the method of plotting efficiency data over a large range indicated by the Vermeulen equation (22). These data were for ethyl acetate diffusing into water, and these drops in all cases were oscillating. It appears that the high transfer rates encountered in the fall period of these drops obscure the end-effect transfer efficiency. The slight curvature of the upper line on Figure 4 is not explained. Values of *R* were obtained for this system with no end effect assumed and the straight-line portion of the curves at low contact times used.

Combined end effects

A comparison of the experimental combined end effect with that predicted by Equation (21) for cyclohexanol diffusing into water is shown as Figure 5. It is seen that the trend of E_F with diameter is as predicted.

Studies of the correlation factor R

Figure 6 indicates plots of the correlation factor of this study plotted against the modified Peclet number by Handlos and Baron. This was chosen as a correlation group to permit comparison of data with their model and also because this group includes the product of the drop velocity and diameter, both of which are significant variables in discussing the flow characteristics of a drop.

This figure shows the data for cyclohexanol and for *n*-butanol diffusing into

Table 4. Summary of Experimental Data for Water Drops in Organic Liquid Saturated with Water at 24.5° ± 0.5°C.

Series	Drop diameter d, ft.	Drop formation time, sec.	Drop velocity V, ft./sec.	Drop Reynolds number N_{Re}	Modified Peclet number $(N_{Pe}'{}_4)$ 10^{-5}	Combined end effect E_F	Experimental R	R_{KB}	R_{HB}	
C	0.0224	1.89-2.17	0.142	15.7	3.32	0.0947	3.8	3.7	160	Cyclohexanol
B	0.0212	1.89-2.17	0.139	14.6	3.07	0.103	3.2	3.7	150	
A	0.0184	1.89-2.17	0.121	11.0	2.32	0.137	2.3	3.7	110	
F	0.0142	1.89-2.17	0.0863	6.06	1.28	0.194	3.2	3.4	63	
J	0.00924	1.59-1.78	0.0482	2.20	0.464	0.232	2.2	3.0	23	
A	0.0114	3.03-3.45	0.258	80.8	2.33	0.224	15	3.2	110	*n*-Butanol
D	0.00982	1.10-1.21	0.260	70.3	2.02	0.290	7.8	3.2	99	
E	0.00804	1.00-1.11	0.232	51.2	1.48	0.311	3.8	3.2	72	
F	0.00722	1.00-1.11	0.212	42.0	1.21	0.303	2.7	3.2	59	
B	0.0121	1.20-1.49	0.240	79.8	2.30	0.040	35	2.8	110	
B	0.0124	1.49-1.69	0.240	81.8	2.36	0.100	34	2.8	110	
C	0.0133	3.00-4.00	0.238	87.0	2.51	0.070	33	2.8	120	
E	0.0149	1.30-1.40	0.283	699	1.51		52	2.8	74	Ethyl acetate
F	0.0131	1.20-1.39	0.289	628	1.36		39	2.8	66	
G	0.0116	1.20-1.39	0.303	583	1.26		42	2.8	61	
J	0.00992	1.00-1.21	0.319	526	1.14		32	2.8	56	
K	0.00845	1.00-1.21	0.305	429	0.926		29	2.8	45	

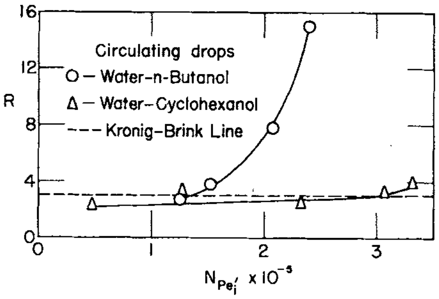

Fig. 6. Experimental *R* compared with a theoretical model.

water. All drops in this study were observed to be circulating, with Hadamard type of streamlines, when aluminum powder was suspended in the water.

It would appear that the R values of the Kronig and Brink equation are approached at low drop Reynolds numbers but that R increases with the product of drop diameter and velocity to values several times the value of the Kronig-Brink correlation.

Figure 7 shows similar R values of ethyl acetate diffusing into water drops. Here all drops were oscillating, and aluminum powder inside the drops showed no regular circulation pattern. Values of R in the range 29 to 52 have been observed. The trend is similar to that predicted by the Handlos and Baron correlation, but the observed values are somewhat lower.

Time of contact

It may be noted in the nomenclature that the contact time in the steady-fall–period equations may be slightly less (by Δt) than the observed time t^1. In practice in this paper Δt was assumed to be negligible.

CONCLUDING REMARKS

This paper has reported data on mass transfer rates inside circulating and oscillating drops. By use of two component systems with the continuous phase saturated with the dispersed phase, the transfer rates inside the drops could be studied without the necessity for correcting for an outside resistance.

The choice of the system and the use of end-effect efficiencies have permitted the comparison of the magnitude both of the combined end effect and of the correlation factor with values predicted by various mechanisms in the literature.

The Kronig and Brink mechanism applies well for low drop Reynolds numbers, but it has been shown that the correlation factor may rise above that predicted by Kronig and Brink even though a drop is apparently circulating.

For oscillating drops the Handlos and Baron model shows considerable promise.

A model for transfer at the settling end has been proposed. Combined with the Heertjes model for drop formation, it predicted the combined end effect fairly well. However this may have been accidental.

NOTATION

A_i = interfacial area at coalescence
a = drop radius
B = coefficient
C = concentration of dispersed phase (uniformly mixed)
d = drop diameter
D = molecular diffusivity
E = effective diffusivity
E = dimensionless number given by $(E_m \leqq E \leqq E_T)$
E = transfer efficiency
N_{Re} = Reynolds number, $(dV\rho_o)/(\mu_o)$
$N_{Pe'i}$ = modified inside Peclet number, $(dV)/[D(1 + \mu_i/\mu_o)]$
R = dimensionless correlation factor
r = $\rho/(d/4)$
Δt = time from the point of drop release from the nozzle to the point when transfer mechanism of the steady-fall period holds
t^1 = time taken by drop to strike the coalesced dispersed phase from the point of drop release from the nozzle
t = $(t^1 - \Delta t)$
v = drop volume
V = drop velocity

Greek letters

λ = eigenvalue
σ = interfacial tension
ρ = density, radius of circulation patterns
θ = drop formation time
μ = viscosity

Subscripts

1 = in the nozzle
2 = beginning of transfer mechanism of the steady-fall period
3 = point at which drop strikes the coalesced dispersed phase
4 = at column outlet
T = over-all, based on points 1, 4
o = continuous phase
i = drop or dispersed phase
m = steady-fall period
n = term number in series sum
f_1 = end effect at entry end for dispersed phase
f_2 = end effect at exit end for dispersed phase
KB = predicted by Kronig and Brink [Equation (3)]
HB = predicted by Handlos and Baron [Equation (4)]
$*$ = at equilibrium with respect to transfer of solute to or from dispersed phase

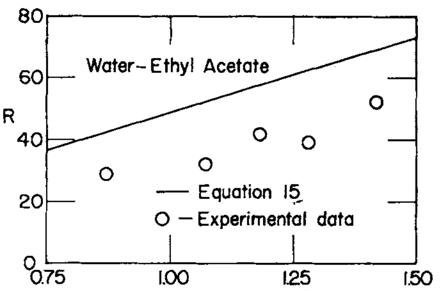

Fig. 7. Experimental *R* for oscillating drops compared with a theoretical model.

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