Intraparticle Mass Transport in Slurries by Dynamic Adsorption Studies

Rates were measured at 25°C for the adsorption of benzaldehyde from an aqueous solution into particles of Amberlite, or activated carbon, suspended in the solution. The data for monodisperse, Amberlite particles (mean pore radius = 50Å) showed significant intraparticle diffusion resistance over the entire particle size range 200 to 900 microns (diameter). Intraparticle diffusivities were larger (tortuosity factor ~ 0.35) than expected from pore-volume diffusion in the liquid-filled pores.

For the activated carbon particles (of the same size) which have a bidisperse pore-volume distribution (pore diameter range 15 to 10^4Å), the effect of intraparticle diffusion was much less important and separation of the external diffusion resistance to obtain a precise value of the intraparticle diffusivity was not possible. However, D_e values are larger than for Amberlite and much greater than the molecular diffusivity of benzaldehyde in liquid-filled pores. The unusually high intraparticle diffusivity seems most likely to be due to interpreting dynamic adsorption data for a bidisperse porous particle with a theory involving but one diffusivity.

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SCOPE

One of the potential advantages of slurry reactors is reduction of intraparticle mass transport resistances in comparison with those in fixed-bed and trickle-bed reactors. Retardation of rate processes due to fluid-to-particle mass transfer resistance is known to be significant in slurries (Brian et al., 1969; Furusawa and Smith, 1973; Levins and Glastonbury, 1972), but no thorough studies have been made concerning intraparticle diffusion. On the one hand, the small particle size in slurries reduces the effect of intraparticle transport, but low diffusivities in liquid filled pores increases the effect. Two investigations (Satterfield et al., 1968; Kenney and Sedriks, 1972) of catalytic reactions with liquid filled pores and large (~ 0.6 cm) particles have been made. The results showed that normal (~ 4) values of the tortuosity, along with liquid phase molecular diffusivities, could be used to predict an appropriate effective intraparticle diffusivity. A preliminary finding, in a paper whose main objective was fluid-particle mass transfer in slurries (Furusawa and Smith, 1973), suggested that unusually high diffusivities were required to explain intraparticle diffusion. These results were obtained from dynamic adsorption experiments with a slurry of activated carbon particles which had a bidisperse pore-size distribution.

In view of this state of the problem it was decided to make a careful study of the significance of intraparticle diffusion in slurries, to evaluate intraparticle diffusivities experimentally, and to compare the results with values predicted from normal tortuosity factors.

CONCLUSIONS AND SIGNIFICANCE

Dynamic measurements were made for the rate of adsorption of benzaldehyde on Amberlite and on activated carbon particles in aqueous slurries. Over the particle size range studied (200 to 900 microns) intraparticle diffusion significantly retarded the rate for the Amberlite particles. The effective intraparticle diffusivity D_e was independent of particle size and was somewhat higher than the molecular diffusivity, corresponding to a tortuosity factor of about 0.35.

Intraparticle diffusion was much less significant for activated carbon particles in the same size range and of about the same surface area. Estimated values of D_e were about one-half order of magnitude greater than for Amberlite. The carbon particles (Filtrasorb 200) have a broad and bidisperse pore size range.

It is shown that comparison of the experimental data with results predicted when there is no intraparticle diffusion resistance gives a time-dependent effectiveness factor. This measure of the relative importance of fluid-particle and intraparticle mass transfer resistances, when applied to the Amberlite data, indicated a rate reduction as high as 63% due to intraparticle diffusion.

The practical significance of these results is that they explain, in part, why activated carbon is an effective adsorbent for purifying gases and liquids. The bidisperse pore structure reduces intraparticle diffusion resistance (high D_e) and thus makes more of the surface available for adsorption.

Slurry reactors have features (isothermal operation, reaction at constant composition, simple construction, and stable operating characteristics) which may be advantageous in comparison with other reactor forms. However, retardation of the rate due to several mass transfer resistances can occur. In recent years considerable information has been obtained about gas bubble-to-bulk liquid transport (for example, Misic and Smith, 1971; Satterfield, 1970) and about bulk liquid-to-particle transport (Brian, et al., 1969; Furusawa and Smith, 1973). Similar results are not available for intraparticle diffusion. With the small particles (normally $\leqq$ 1000 microns) used in slurries, intraparticle-diffusion path lengths are reduced, but the molecular diffusivity in liquids is low with respect to that in gases. Preliminary measurements (Furusawa and Smith, 1973) suggested that intraparticle diffusion resistances could be significant. Hence, the work reported here was done in order to evaluate intraparticle diffusion resistances and effective diffusivities in slurries. Data were obtained for the transport of benzaldehyde in liquid-filled pores of Amberlite and activated carbon, two materials with distinctly different pore-size distributions and adsorption capacities (see Table 1 and Figure 1). Measurements were made at 25°C and 1 atmosphere with no gas phase present. The primary variables were particle size d_p, solid concentration m_s, and initial concentration of benzaldehyde in the aqueous slurry. The ranges of these variables are given in Table 2.

The method consisted of injecting rapidly a 5 cm^3 aqueous solution of benzaldehyde into the aqueous slurry and measuring continuously the benzaldehyde concentration in the bulk liquid. These concentration versus time curves were analyzed to determine the intraparticle diffusivity after accounting for fluid-to-particle transport resistance. Therefore, it was necessary to know values of k_s as accurately as possible. For this reason, the adsorber used here was identical to that employed earlier (Furusawa and Smith, 1973) for measuring k_s for carbon-particle slurries. For this design the power number is 9.5, and at the impeller speed of 400 rev./min. (used for all the runs) the energy dissipation rate ϵ is 1.21×10^4 ergs/(s)(g). Knowing ϵ, the correlation of Brian et al., 1969, based upon slurries of spherical particles (data of Harriott, 1962), also could be used to determine k_s for Amberlite particles (also spherical). The intrinsic rate for physical adsorption of liquid benzaldehyde on either adsorbent is likely to be very large. Hence, instantaneous equilibrium was assumed at the interior adsorption site.

EXPERIMENT

The Pyrex adsorber (1.6-cm I.D., 1.7 cm high) was equipped with 8 equally spaced, stationary baffles attached along the circumference. The 8-bladed impeller was positioned so that the center line of the blades was 0.8 cm above the bottom of the adsorber. This construction, which is described in detail elsewhere (Furusawa and Smith, 1973), was chosen so that the relationship between energy input to the vessel and k_s could be correlated by previously tested methods.

Benzaldehyde loss from the covered vessel by evaporation was measured to be about 2%/hour. Since the time required for the runs was less than one-half hour, this error was not significant. The impeller speed of 400 rev./min. was chosen to maintain the particles in a well-suspended condition. Preliminary tests indicated that the time required to obtain complete mixing of the injected sample of benzaldehyde was negligible with respect to the time of the runs.

The benzaldehyde concentration was measured by withdrawing a small quantity of liquid (through a 80 × 700 mesh screen) from the adsorber through a 0.08-cm I.D. tube and into an ultraviolet photometer (cell volume = 0.02 cm^3) operating with 2570Å radiation. At this wave length benzaldehyde has a very high absorbance so that low concentrations could be accurately measured. The photometer reading was recorded, providing a continuous record of the concentration. The stream from the photometer was pumped back into the adsorber. The

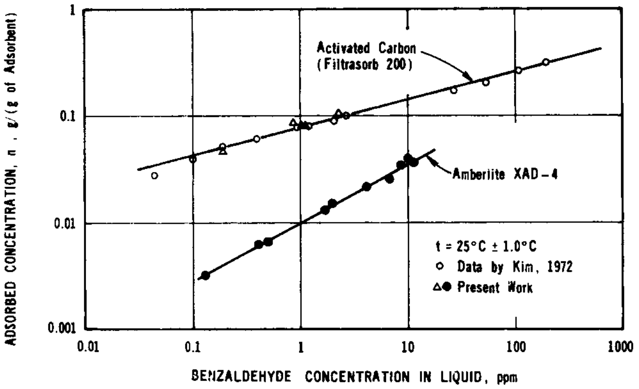

Fig. 1. Adsorption isotherms for benzaldehyde on activated carbon and on Amberlite XAD-4.

TABLE 1. PROPERTIES OF ADSORBENTS

	Activated carbon[a] filtrasorb 200	Amberlite[b] XAD-4
1. Solid phase density, g/cm^3	2.04	1.08
2. Particle density, g/cm^3	0.956	0.529
3. Surface area, m^2/g	800-900	750
4. Porosity, ϵ_p	0.53	0.51
5. Pore sizes		
1) Nominal diameter	micropores 15-20Å macropores $2 \times 10^3 - 10^4$	50
2) Cumulative pore volumes[c], cm^3/g		
Pore diam., Å		
15	0.005	
20	0.265	
50	0.325	0.04
100	0.34	0.148
500	0.39	0.602
1,000	0.40	0.807
5,000	0.50	0.934
10,000	0.55	
100,000	0.59	

[a] From Calgon Corporation, Filtrasorb Technical Bulletin.

[b] From Rohm and Haas Summary Bulletin on Amberlite Polymeric Adsorbents.

[c] Data for Amberlite were measured in a dry state.

TABLE 2. SCOPE OF DATA

	Amberlite	Activated carbon
d_p, microns	228 to 912	192 to 912
m_s, g/cm^3	0.634×10^{-3} to 1.714×10^{-3}	0.588×10^{-3} to 1.714×10^{-3}
Initial conc., C_o, ppm	2.78 to 12.2	6.11

Average particle size d_p, microns	Range of sieve openings microns
192	175-210
228	210-246
271	246-295
542	495-589
767	701-833
912	833-991

experimentally measured average response time in the line from the adsorber through the photometer was about 1 min. Since, the half-life of the concentration versus time curves were about 15 min., the error in concentration versus time results due to holdup and dispersion in the line from the adsorber to the cell was not large. For example, for 15-min. half-life, the predicted error in concentration at 10 min. due to dispersion is 2.0%. At very small times (0 to 2 min.) the error would be significant so this portion of the concentration versus time curve was not accurate. At higher times this error would deflect the curves upward while the evaporation error, mentioned earlier, and any error introduced due to distribution of particle sizes, would be in opposite direction.

The properties of the adsorbents given in Table 1 show that both kinds of particles had approximately the same surface area. However, the carbon particles exhibited a definite bidisperse pore size distribution while the Amberlite showed a broad, monodisperse pore structure. These properties were supplied by the two manufacturers, Calgon Corporation and Rohm and Haas, respectively. The Amberlite XAD 4 particles are hard, insoluble, relatively nonpolar, beads of polystyrene. They appear to be agglomerates of randomly packed microspheres. Adsorption occurs by van der Waals forces between the microspheres and the hydrophobic portion of the adsorbate molecule. These Amberlites are particularly recommended for adsorption of organic substances from aqueous solution.

Before use, the carbon particles were ground, sieved, dried in air at 110°C until constant weight was attained, and boiled in distilled water for 30 min. For the hydrophobic Amberlite, the particles were partially dried in methanol and then sieved. Prior to use the particles were first soaked in methanol for 15 min., and then the methanol was replaced with water. The range of sieve openings and the corresponding average particle sizes are given in Table 2.

ADSORPTION ISOTHERMS

Adsorption isotherms were measured by introducing known samples of benzaldehyde solution into a flask containing the slurry and operating until equilibrium was achieved. The results are plotted, according to the Freundlich equation, in Figure 1. For Filtrasorb 200, earlier measurements by Kim (1972) obtained in different equipment were available for comparison. At liquid concentrations less than 0.4 ppm the Langmuir equation fit the data. It was necessary to use both equations in quantitative analysis of the adsorption data since the Freundlich equation is not convenient to use at near-zero concentrations. The numerical expressions for the isotherms are:

Freundlich Equation
Amberlite

$$n = 18.6\, C^{0.545} \qquad (1)$$

Langmuir ($C < 0.4$ ppm)

$$n = \frac{3.45 \times 10^4 C}{1 + 2.90 \times 10^6 C} \qquad (2)$$

Activated Carbon

$$n = 7.82\, C^{0.328} \qquad (3)$$

$$n = \frac{9.61 \times 10^5 C}{1 + 13.3 \times 10^6 C} \qquad (4)$$

A few tests were made in which the liquid concentration was measured versus time when pure water was initially added to particles containing benzaldehyde. The results showed that desorption rates were comparable to adsorption rates, verifying that the process was reversible.

Despite similar surface areas (by nitrogen adsorption) Figure 1 shows that adsorbed concentrations at equilibrium were four to ten times larger for the carbon particles.

METHOD OF ANALYSIS

The overall adsorption is supposed to occur by three processes in series: mass transfer from bulk liquid to particle surface, intraparticle diffusion in the liquid-filled pores, and adsorption from the liquid on an interior site. It is assumed that the benzaldehyde concentration in the liquid, and the concentration m_s of adsorbent particles are uniform throughout the adsorber. Then m_s is directly calculable from the measured mass of adsorbent and volume of particle-free liquid,

$$m_s = \frac{W}{V} \qquad (5)$$

Assuming smooth spherical particles, the surface area for mass transfer to the particles can be obtained from m_s as follows:

$$S_s = \frac{6\, m_s}{d_p\, \rho_t\, (1 - \epsilon_p)} \qquad (6)$$

Conservation equations for benzaldehyde in the bulk liquid and in the particles are

$$\frac{dC}{dt} = -\, k_s\, S_s\, (C - C_s) \qquad (7)$$

$$D_e\left(\frac{\partial^2 C_r}{\partial r^2} + \frac{2}{r}\,\frac{\partial C_r}{\partial r}\right) - \frac{\rho_p}{\rho_L}\,\frac{\partial n_r}{\partial t} = \epsilon_p \frac{\partial C_r}{\partial t} \qquad (8)$$

In Equation (8) it is supposed that intraparticle diffusion can be represented by a single, constant effective diffusivity D_e regardless of the pore-size distribution.

Boundary and initial conditions corresponding to the experimental arrangements are

$$D_e\left(\frac{\partial C_r}{\partial r}\right)_{r=R} = k_s(C - C_s) \qquad (9)$$

$$\frac{\partial C_r}{\partial r} = 0 \quad \text{at} \quad r = 0 \qquad (10)$$

$$C_r = 0 \quad \text{at} \quad t = 0 \quad \text{for} \quad 0 \leq r \leq R \qquad (11)$$

$$C = C_0 \quad \text{at} \quad t = 0 \qquad (12)$$

Adsorption at an interior site is assumed to occur rapidly in comparison with mass transfer and to be reversible. Then n_r and C_r are related by the instantaneous equilibrium expression:

$$\frac{\partial n_r}{\partial t} = \frac{\partial n_r}{\partial C_r}\left(\frac{\partial C_r}{\partial t}\right) \qquad (13)$$

where $\partial n_r/\partial C_r$ is given by the derivative of the appropriate isotherm expression, Equation (1) or (2), or (3) or (4).

Since k_s is known, Equations (7) to (13) can be solved, numerically, to give predicted concentration versus time curves in terms of D_e. Then comparison with the experimental curves will give the most appropriate value of D_e.

To illustrate the effect of intraparticle diffusion in retarding the overall rate, it is desirable to compare the observed C versus t curves with those predicted when intraparticle diffusion is neglected. For this single-resistance model, $C_r = C_s$ for any r and n_r is uniform throughout the particle. Equation (8) may be replaced by

$$m_s \frac{dn}{dt} = k_s\, S_s\, (C - C_s) \qquad (14)$$

and Equation (13) becomes

$$\frac{dn}{dt} = \frac{dn}{dC_s}\left(\frac{dC_s}{dt}\right) \qquad (15)$$

The solution of Equations (7), (14) and (15), with (11)

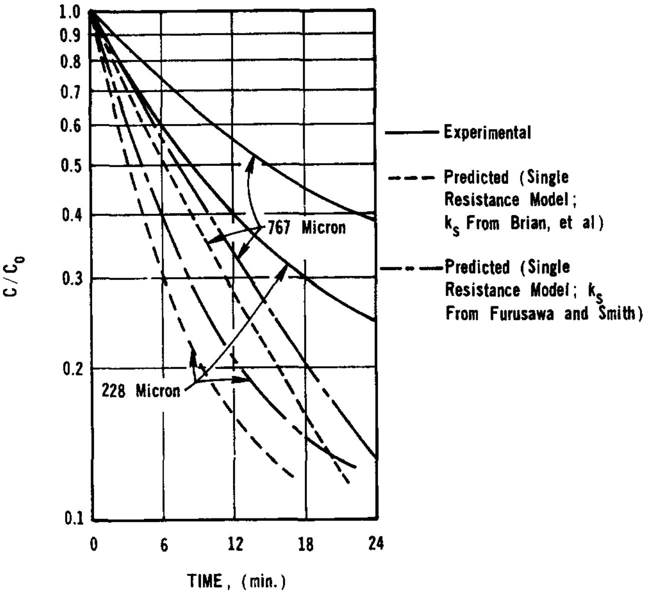

Fig. 2a. Typical concentration decay curves for Amberlite ($C_0 = 6.11$ ppm; $m_s = 1.176 \times 10^{-3}$ g/cm^3 for 767 micron particles, and $m_s = 0.588 \times 10^{-3}$ g/cm^3 for 228 micron particles.

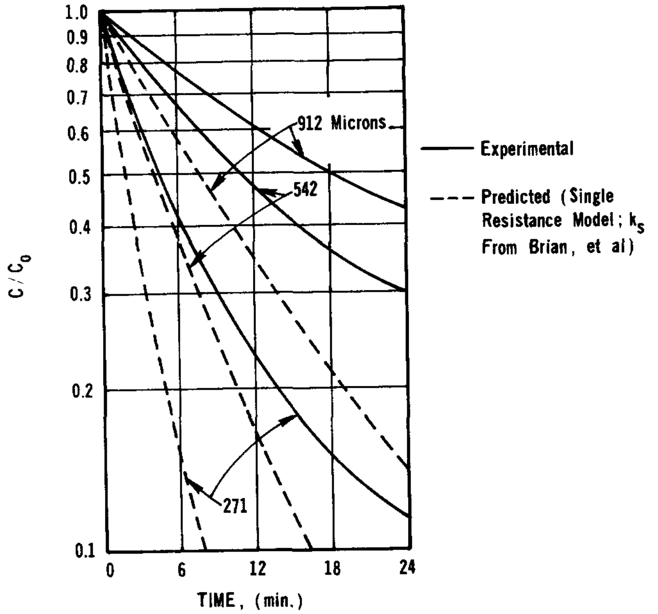

Fig. 2b. Typical concentration decay curves for Amberlite ($C_0 = 6.11$ ppm; $m_s = 1.176 \times 10^{-3}$ g/cm^3).

and (12), gives predicted C versus t curves for the single-resistance case. Again a numerical solution is required because the isotherms are nonlinear.

The molecular diffusivity of benzaldehyde in water at 25°C was calculated to be 0.913×10^{-5} cm^2/s using the Othmer-Thakar equation recommended by Reid and Sherwood (1966). The corresponding Schmidt number was 982. For the operating conditions employed, k_s values from the data of Furusawa and Smith (1973) for the various particle size are given in Table 3. Also shown are k_s estimated from the correlation of Brian et al. (1969). These latter values are 10 to 30% higher than those of Furusawa and Smith. As discussed later, this difference does not affect the conclusions regarding the significance of intraparticle diffusion for the Amberlite particles.

TABLE 3. FLUID-TO-PARTICLE TRANSPORT COEFFICIENTS k_s

Average particle size, d_p, microns	k_s, cm/s Brian et al. (1969)	 Furusawa and Smith (1973)
192	0.0120	0.00896
228	0.0117	0.00892
271	0.0115	0.00899
542	0.0108	0.0089
767	0.0105	0.0089
912	0.0102	0.0088

INTRAPARTICLE DIFFUSION RESULTS

Amberlite Particles

Figures 2a and 2b (solid lines) show typical, experimental C/C_0 versus t curves for various particle sizes but for the same C_0 and solid concentrations m_s. The dotted curves in Figure 2a were predicted by neglecting intraparticle diffusion and using both sets of k_s values given in Table 3. Regardless of the k_s employed, the predicted curves are considerably lower than the experimental result, showing that intraparticle diffusion significantly affects slurry adsorption rates at these operating conditions. Because particle shapes and fluid-solid density differences were nearly the same for the Amberlite slurries used here and the data upon which the Brian et al. correlation was based, these k_s values were believed to be most appropriate for evaluating the intraparticle diffusivity. Figure 2b shows the comparison of predicted, single-resistance curves and experimental results for three other particle sizes, using Brian et al. k_s values.

D_e was obtained by comparing numerical solutions of Equations (7 to 13), using Equations (1) to (4), with the experimental C/C_0 versus t curves. Since intraparticle diffusion becomes of negligible importance and because the data are less accurate, as $t \to 0$ more weight was given to agreement between experimental and predicted data at larger times (> 12 min.). The so-determined diffusivities are given in Table 4 for all the data. There appears to be no regular variation in D_e with particle size, C_0, or m_s, and the average deviation from the average for all the runs is about 10%. Figures 3 and 4 illustrate the agreement of the experimental and predicted C/C_0 versus t curves using the D_e values from Table 4. Agreement for other runs was similar.

To determine the effect of k_s on D_e, the fluid-to-particle coefficient of Furusawa and Smith (Table 3) was used to evaluate D_e for the 271 micron particles. The effect of k_s should be larger for the smaller particles. D_e so obtained was 1.55×10^{-5} cm^2/s in comparison with 1.4×10^{-5} shown in Table 4.

Tortuosity factors calculated from the expression

$$\delta = \epsilon_p \frac{D}{D_e} \qquad (16)$$

are given in the last column of Table 4. The lower than expected values are discussed later.

Activated Carbon Particles

Figure 5 compares experimental data and predicted curves, based upon no intraparticle diffusion resistance, for 912 activated carbon particles. Comparison with Figures 2a and 2b shows that the intraparticle effect is much less important for activated carbon than for Amberlite particles. The predicted curves are based upon k_s results

TABLE 4. EFFECTIVE INTRAPARTICLE DIFFUSIVITIES FOR AMBERLITE

d_p, microns	$m_s \times 10^3$ g/cm^3	C_o, ppm	$D_e \times 10^5$ cm^2/s	Tortuosity factor, σ
912	1.176	6.11	1.5	0.31
	1.71	6.11	1.5	0.31
767	1.176	2.78	1.6	0.28
	1.176	6.11	1.4	0.33
542	0.634	6.11	1.3	0.36
	1.176	6.11	1.2	0.40
	1.71	6.11	1.0	0.46
	1.176	3.05	1.3	0.34
	1.176	12.2	1.1	0.45
271	1.176	6.11	1.4	0.33
228	0.588	6.11	1.3	0.34
	Average		1.3	0.35

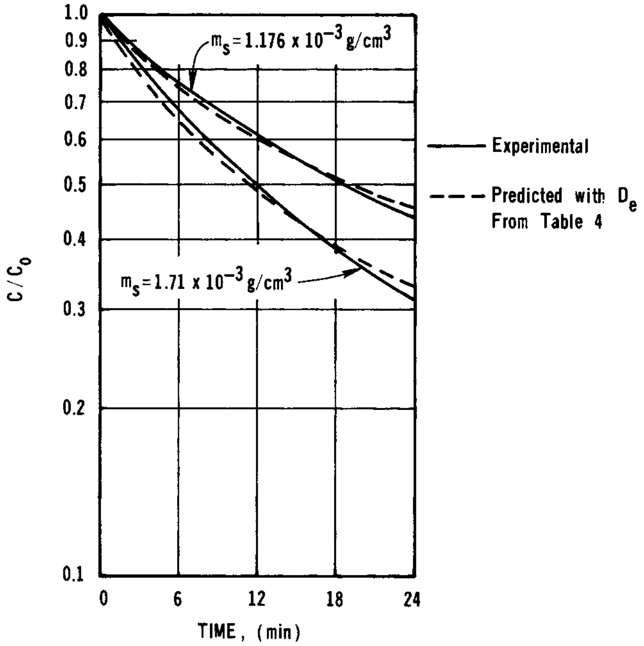

Fig. 3. Effect of solid concentration for Amberlite slurries (C_0 = 6.11 ppm, d_p = 912 microns).

(Table 3) of Furusawa and Smith which are also for the granular carbon particles. In view of the small magnitude of the effect and the uncertainty in k_s, a precise value of D_e cannot be obtained. Using the same method as used for Amberlite, agreement of the experimental and predicted curves in Figure 5 is obtained with $D_e = 5 \times 10^{-5}$ cm^2/s, a result five times as large as the molecular diffusivity benzaldehyde in water at 25°C. Such a high D_e is in agreement with the earlier preliminary find of Furusawa and Smith (1973).

The unimportance of intraparticle diffusion for carbon particles is emphasized by the results for smaller particle sizes where the intraparticle effect is inherently small. Such results are shown in Figure 6 where for 192 micron particles the predicted curve, neglecting intraparticle diffusion, is actually above the experimental result. Under these conditions, intraparticle diffusion effects are so small that uncertainty in k_s masks the results. This is emphasized in Figure 6 by including a predicted, single-resistance curve based upon k_s from Brian et al. The deviation between the two predicted curves is greater than between the experimental result and either predicted curve. Similar results were observed for other small-size particles.

DISCUSSION

The difference between experimental and predicted (single-resistance) curves in Figures 2a and 2b shows the cumulative effect of intraparticle diffusion on bulk liquid concentration. However, this difference does not give direct information about the relative importance of fluid-particle and intraparticle mass transfer resistances on the overall adsorption rate. It would be helpful in designing slurry adsorbers to be able to predict, from the properties of the system, whether or not intraparticle diffusion significantly retarded the rate. Such a criterion for transient adsorption would depend upon the time, particle size, k_s,

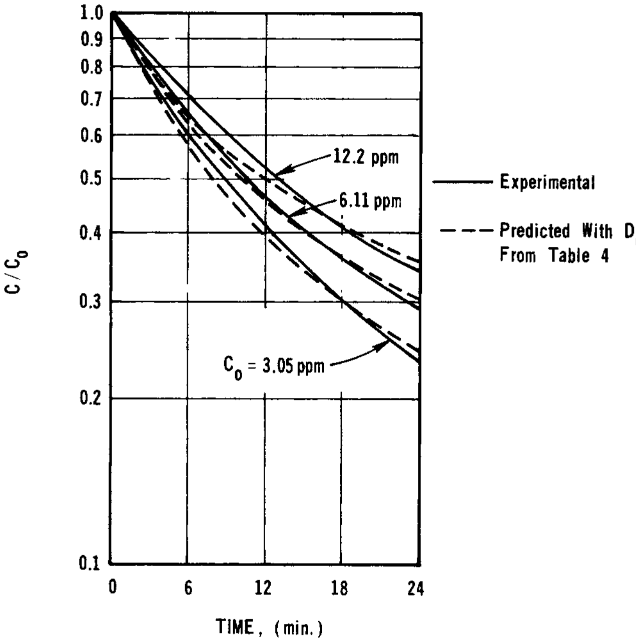

Fig. 4. Effect of initial concentration, Amberlite slurries (m_s = 1.176 × 10^{-3} g/cm^3, dp = 542 microns).

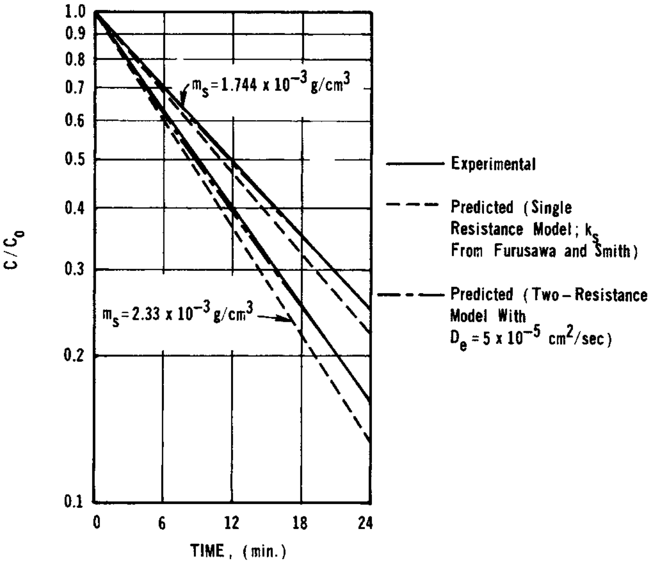

Fig. 5. Concentration decay curves for large activated carbon particles (C_0 = 6.11, d_p = 912 microns).

Table 5. Effectiveness Factors from Amberlite Slurry Data

Time, min.	Particle diam., microns	Effectiveness factor
3	271	0.81
	912	0.53
18	271	0.46
	912	0.37

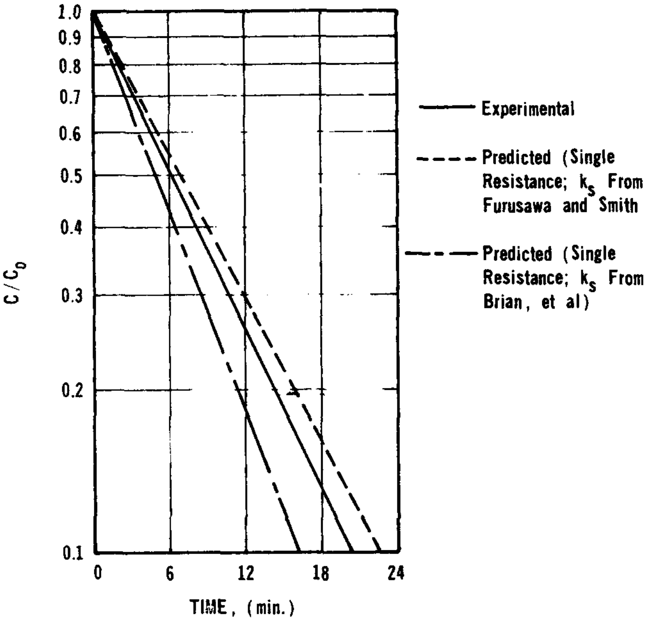

Fig. 6. Typical concentration decay curves for small activated carbon particles ($C_0 = 6.11$, $d_p = 192$ microns, $m_s = 0.588 \times 10^{-3}$ g/cm^3).

D_e, and equilibrium adsorption capacity. The results could be expressed as a time-dependent effectiveness factor, defined as the ratio of the actual rate of adsorption to the rate corresponding to no intraparticle diffusion resistance, evaluated at the same adsorbed concentration, n. The time-dependent fluid concentration and nonlinear isotherm precludes a theoretical development for such an effectiveness factor applicable to our experimental work. However, the information in Figure 2b, for example, can be used to obtain experimental values for this effectiveness factor. The slope of the experimental curve at any time is proportional to the rate of adsorption. For a given C_0 and m_s, adsorbed concentrations (n) are identical at the same C/C_0 values. Hence, the slope of the dotted line at the same C/C_0 value is proportional to the rate corresponding to no intraparticle diffusion resistance. The ratio of these slopes then gives the effectiveness factor. Table 5 shows such effectiveness factors evaluated from Figure 2b at two time levels and for two particle sizes. Since a value of unity would correspond to no intraparticle retardation, the results show that for the larger particle size, at the longer time, the rate is reduced by 100 to 37, or 63%, by intraparticle diffusion resistance. As the time is reduced the effectiveness factor increases. This trend would continue until at zero time the effectiveness factor must be unity because initially adsorption will occur at the outer surface of the particle.

For both Amberlite and carbon slurries, effective diffusivities were larger than expected for porous particles, and indeed, larger than the molecular diffusivity. Surface diffusion seems to be the most likely explanation for these results with liquid-filled pores.

ACKNOWLEDGMENT

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NOTATION

C = concentration of benzaldehyde in the bulk liquid; C_r = concentration in liquid in pores at radius r; C_0 = initial concentration in bulk liquid; C_s = concentration at outer surface of particle, g/(g water)
d_p = particle diameter, cm
D = molecular diffusivity, cm^2/s
D_e = effective diffusivity in porous particle, cm^2/s
k_s = mass transfer coefficient between bulk liquid and outer surface of particle, cm/s
m_s = mass of adsorbent particles per unit volume of particle-free slurry, g/cm^3
n = concentration of adsorbed benzaldehyde, g/(g of adsorbent); n_r = concentration at radius r
r = radial coordinate in particle (assumed spherical, cm)
S_s = outer surface area of adsorbent particles per unit volume of particle-free slurry, cm^{-1}
t = time, s
V = volume of particle-free liquid in the adsorber, cm^3
W = mass of particle-free liquid in the adsorber, g

Greek Letters

ϵ = energy dissipation rate in slurry per unit mass of particle-free liquid, ergs/g-s
ϵ_p = porosity of particles
δ = tortuosity factor, defined by Equation (16)
ρ_p = density of particle as a whole, g/cm^3

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