waves were observed.

In closing, it is interesting to note that viscosity stratification induced long waves described by Yih (1967) are not expected for the fluid pairs used here. His analysis for plane Poiseuille flow was extended to show the density difference between the two liquid phases was high enough to stabilize long waves.

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Manuscript received August 28 and accepted August 30, 1974.

Mass Transfer in a Fixed-Bed Gas-Liquid Catalytic Reactor with Concurrent Upflow

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Mass transfer coefficients for the hydrogenation of α-methylstyrene were measured in a packed-bed reactor with concurrent gas and liquid upflow. The packing was 0.29-cm alumina spheres coated with palladium catalyst. Mass transfer coefficients increased as the ½ power of the liquid rate and increased with gas rate to a gas phase Reynolds number of about 50. The severe decrease for higher gas rates may be due to pulsing flow.

SCOPE

Gas-liquid reactions requiring a solid catalyst are generally carried out in trickle-bed or slurry reactors. Interest in the present system arose during development work at Oak Ridge National Laboratory on a reactor to reduce uranium(VI) to uranium(IV) nitrate in aqueous solution with hydrogen in the presence of a supported palladium catalyst. Higher conversions were obtained with the reactor flooded than in the trickle-bed mode, leading to a study of mass transfer characteristics in concurrent upflow.

The model reaction of hydrogen and α-methylstyrene was chosen because it is a simple reaction with known kinetic rate constants. Furthermore, determination of the degree of conversion is achieved by means of a simple analysis of the product mixture since the only reaction product is cumene. The kinetics of this reaction were studied by Babcock et al. (1957) and Satterfield et al. (1968). The reaction has been used by Satterfield et al.

(1969) to elucidate the mass transfer characteristics of trickle-bed reactors.

The authors were unable to find any previous report of a mass transfer study of concurrent upflow in a catalyst bed although the work of Mashelkar and Sharma (1970) on packed bubble columns is somewhat related. In the present study the effects of gas and liquid flow rates on mass transfer coefficients were determined.

CONCLUSIONS AND SIGNIFICANCE

Mass transfer coefficients varied from 0.006 to 0.06 cm/s over Reynolds number ranges of 20 to 250 for the liquid phase, and 2 to 75 for the hydrogen phase. The coefficients increased with liquid rate to about the ½ power, and with gas rate to about the 0.4 power, up to an Re_g of 50. Very low mass transfer coefficients and reaction rates were observed at higher gas rates. This adverse effect of high gas rates may be due to pulsing flow.

Mass transfer coefficients are much larger (by as much as a factor of 5) for this system than are those for liquid flowing in the absence of a gas phase. Presumably, the gas flow thins the liquid film and increases turbulence in the liquid, thereby promoting transport of the dissolved hydrogen.

A comparison of mass transfer coefficients in concurrent upflow with those in trickle-bed reactors would be of interest; however, measurements of the latter are not available. Satterfield et al. (1969) have made some estimates for an industrial trickle-bed hydrodesulfurization reactor which fall in about the middle of the range for concurrent upflow.

EXPERIMENT

The reactor vessel was made from sched 40, 3.81-cm pipe and was water-jacketed for temperature control. The reactor vessel and water jacket, the product sample reservoir, and the product receiver were fabricated as a unit. The sample reservoir and the product receiver were thus maintained at the operating pressure of the reactor, which was 18 atm. for most of the runs. This eliminated the need to continuously reduce the pressure of the liquid stream. The three vessels were made of 304-L stainless steel pipe.

The volume of the reactor that was not utilized for a particular catalyst loading was filled with inert glass beads of the same size as the palladium catalyst beads. The catalyst and glass beads were retained between support grids. Approximately 100 holes, 0.159 cm in diameter and located 0.318 cm apart, were drilled in each grid to permit passage of the liquid and gas phases. The top of the reactor vessel was enlarged to 10 cm above the product overflow point. This enlargement, and the use of a 7.5 cm-deep bed of Yorkmesh, satisfactorily removed all entrained liquid from the hydrogen stream exiting from the reactor; no organic film appeared in the packed humidifier column after more than 100 runs. The maximum superficial hydrogen velocity in the reactor was about 7 cm/s at reactor temperature and pressure.

Metal gaskets were used to prevent possible contamination of the α-methylstyrene-cumene mixture by the gasket material. Since only two metals, gold and tungsten, do not occlude hydrogen (Johnson et al., 1957), the choice of material was limited. Gold was selected as the gasket material because it has good malleable properties.

The liquid metering system consisted of a calibrated feed tank, a duplex positive displacement pump, and a pump pulse damper. The feed tank, which was fabricated from a 40.6-cm diam. 304-L stainless steel tube, was connected to a recirculating centrifugal pump to permit mixing of the feed. A spray nozzle was used to rinse the walls of the feed tank. A sample valve was located on the discharge line of the recirculating pump. Samples of feed were withdrawn through this valve after the contents of the feed tank were thoroughly mixed. The analyses of these samples were used to determine the composition of feed entering the catalyst bed.

A duplex positive displacement pump with Teflon bellows was used for the liquid metering pump. A pulse damper reduced the pressure variation at the reactor inlet to only 25,000 N/m^2. The estimated accuracy for the volume of liquid pumped during a given run varied from 3 to 5%; the higher number is applicable for runs in which the liquid Reynolds number was approximately 15, and the lower one for runs in which the Reynolds number was approximately 300.

Commercial hydrogen containing small amounts of water and oxygen was used for these studies. Both contaminants were undesirable. The presence of water in the organic feed causes a slimy precipitate to form, covering the catalyst particles and thus interfering with the hydrogenation reaction. The presence of oxygen increases the reaction rate of the catalyst by causing a phase transformation of the palladium (Babcock, 1955). In addition, the presence of water in the α-methylstyrene-cumene mixture changes the refractive index of the mixture. Oxygen produces the same effect by causing a polymerization reaction which, in turn, changes the refractive index of the mixture (Babcock, 1955). To ensure that no water or oxygen entered the reactor with the hydrogen stream, the stream was passed over a platinum catalyst bed and then over a silica-gel bed. The platinum catalyst formed water from any existing oxygen, and the water was subsequently removed by the silica-gel bed.

The flow of hydrogen to the reactor was measured by a high-pressure rotameter. The effluent hydrogen from the reactor was saturated with water vapor, and the volume was measured by a wet-test meter. A back pressure regulator on the effluent hydrogen controlled the reactor pressure.

Analyses for α-methylstyrene and cumene were made by measuring the refractive index of the feed. A dipping refractometer was used. Three prisms were required to span the range of refractive indexes (1.53880 to 1.49020) encountered during these studies. All refractive index measurements were made at 25 ± 0.05°C. The refractive index of each sample was measured at a wavelength of 2537 Å.

The catalyst (distributed by Englehard Industries, Inc.) consisted of alumina spheres coated with 0.5-wt % palladium. Prior to use, the spheres were passed through a U.S.S. Standard screen No. 6 and retained on a U.S. Standard screen No. 8. These screens have openings that pass particles of 0.336 and 0.238 cm, respectively. Measurements of a sample of the screened catalyst spheres showed that the average diameter was 0.29 cm, and this value was used for calculational purposes for all beds. The calculated superficial surface area per unit weight of catalyst was 14 cm^2/g.

Two catalyst beds, referred to as beds A and B, were used for this study. They were 30.48 and 45.72-cm deep, respectively, and were prepared from different batches of catalyst. The weights of catalyst were 287 g for A, and 442 g for B. The depth of glass beads (0.30-cm diam.) below the catalyst was always at least 60 cm. This depth is much greater than

Table 1. Insensitivity of Rate of Reaction to Reactant Concentration and Temperature

Concentration, mole fraction α-Methylstyrene	Temperature, °C	Re_l	Re_g	Rate of reaction, g-moles/hr (g Catalyst)
0.178	31.5	35	8	0.0127
0.426	30.0	31	7	0.0139
0.151	31.6	66	21	0.0176
0.600	31.0	58	21	0.0179
0.166	34.2	41	36	0.0207
0.376	32.6	41	42	0.0221
0.426	27.8	52	12	0.0214
0.561	38.0	46	20	0.0208
0.473	28.2	52	32	0.0236
0.560	40.1	54	33	0.0239

that required to produce a fully developed velocity profile in a packed bed (Ford and Perlmutter, 1963). Glass beads were packed above the catalyst to a depth of at least 10 cm.

REACTION RATES

About 70 runs were made at a pressure of 18 atm to explore the effects of α-methylstyrene concentration, temperature, and gas and liquid flow rates. The reactor was operated as a differential reactor, with small changes of the liquid phase composition during passage through the reactor. The maximum change in the mole fraction of α-methylstyrene during a run was about 0.04. The average composition of the α-methylstyrene-cumene mixture ranged from 0.1 to 0.6 mole fraction α-methylstyrene, and reaction temperature was varied from 24.0 to 40.5°C. Reaction rates could be correlated roughly with liquid and gas rates although the large scatter of the data shows that composition and temperature sometimes affected the rates significantly. The strong mass transfer dependence of some runs is illustrated by Table 1. The first three pairs of runs in Table 1 show that large variations in α-methylstyrene concentration had little affect on the reaction rate. The last two pairs show that temperature changes of 10 to 12°C also made little difference.

The influences of gas and liquid flow rates on reaction rate for the two catalyst beds are shown in Figures 1 and 2. The reaction rate increased with the liquid rate over the full range investigated and increased with the gas rate up to a Reynolds number of about 50. Catalyst bed A had a higher reaction rate than catalyst bed B for given values of Re_l and Re_g. The increase in reaction rate with an increase in either Re_l or Re_g was also greater for catalyst bed A.

CATALYST SURFACE CONCENTRATION OF HYDROGEN

The mass transport behavior of the liquid phase was the subject of interest in this study. The gas phase, being essentially pure hydrogen, offered negligible resistance to the transport of hydrogen. The molar rates of transfer of hydrogen and α-methylstyrene to the catalyst surface must be equal under steady state conditions by the stoichiometry of the reaction. Since the concentration of α-methylstyrene in the liquid phase is much higher than that of hydrogen, the transfer of hydrogen must be rate-limiting.

The rate of mass transfer of hydrogen in the liquid phase was represented by the equation

$$R = k_L a (C^* - C_s) \tag{1}$$

The liquid phase at the gas-liquid interface was assumed to be saturated, and C^* was obtained from the reactor pressure and the Henry's Law constant (Johnson et al., 1957). The interfacial area associated with the reaction was taken to be the external surface of the catalyst pellets, 10.6 cm^2/cm^3 of bed volume, as calculated from the pellet diameter.

Values of k_L for all 70 runs were first calculated from Equation (1) under the assumption that C_s was negligibly small with respect to C^*. Under this assumption the rates of the chemical reaction steps at the catalyst surface are taken to be very fast compared with the rate of mass transfer of hydrogen through the liquid phase. This assumption is supported by the data in Table 1. The resulting values of k_L are plotted against the liquid phase Reynolds number in Figures 3, 4, and 5 for the three ranges of gas velocities. For comparison, the correlation of Williamson et al. (1963) is shown for liquids flowing through packed beds in the absence of a gas phase. The

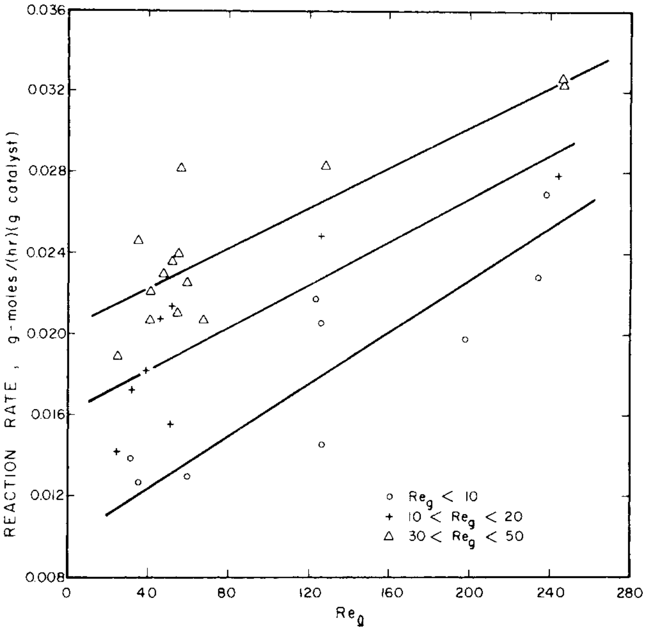

Fig. 1. Reaction rates in Catalyst Bed A.

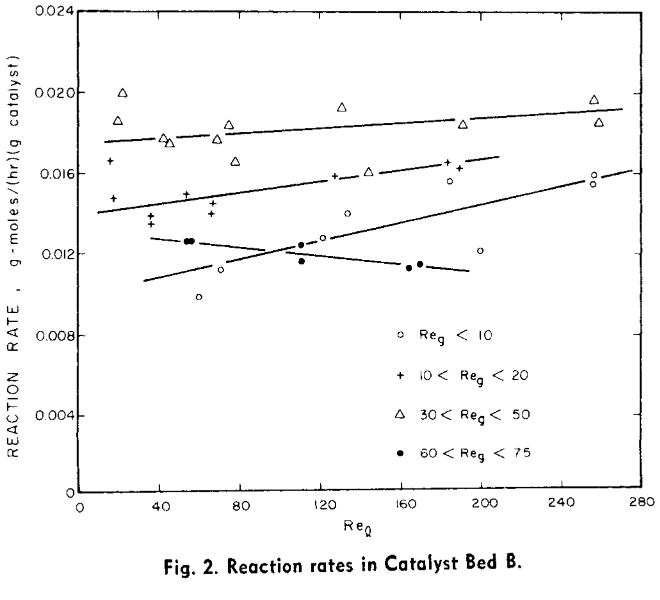

Fig. 2. Reaction rates in Catalyst Bed B.

liquid phase Reynolds number cannot be converted to superficial liquid velocity (cm/s) by use of a single factor because of the temperature and composition dependence of viscosity, but the factors (to be multiplied times the Reynolds number) range from about 0.011 to 0.024. The gas phase superficial velocity (cm/s) can be estimated fairly well by applying a factor of 0.081.

The values of k_L calculated under the assumption of negligible hydrogen concentration at the catalyst surface do not appear to be very satisfactory. Clearly the points for beds A and B must be represented by different lines in each of the Figures 3, 4, and 5. There does not seem to be any justification for different mass transfer coefficients between the two beds since they were identical with respect to hydraulic characteristics. It is also disturbing that a significant number of the points lie below the correlation of Williamson. It might be expected that moderate gas velocities would improve the mass transport properties of the liquid over those for liquid alone.

As an alternative to the assumption of negligible concentration at the catalyst surface, the concentration can be estimated by use of the rate expression for the chemical reaction. This procedure was applied and appears to yields improved values of k_L, as shown in the following sections.

THE REACTION RATE EQUATION

Babcock et al. (1957) investigated the kinetics of this reaction over a pressure range of 2 to 12 atm, a temperature range of 24 to 57°C, and a catalyst of 0.5% palladium deposited on 0.318 by 0.318-cm cylindrical alumina pellets. They proposed the following rate equation which fits their data with an average deviation of less than 3% for pressures above three atmospheres:

$$R = \frac{A C_s X}{(1 + B\sqrt{C_s})^2 (1 + C X)} \qquad (2)$$

Equation (2) was used to calculate hydrogen concentration at the catalyst surface in the present study. Values of the constants A, B, and C are presented for a range of temperatures by Babcock et al. Their values for B and C were used directly, but the constant A had to be adjusted since it depends not only on the chemical properties of the system but also the geometrical details of the catalyst surface.

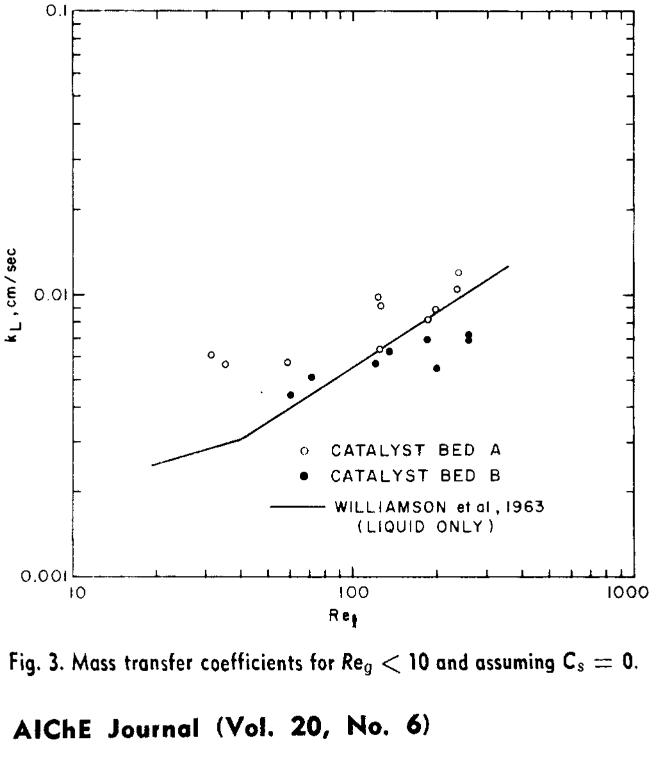

Fig. 3. Mass transfer coefficients for $Re_g < 10$ and assuming $C_s = 0$.

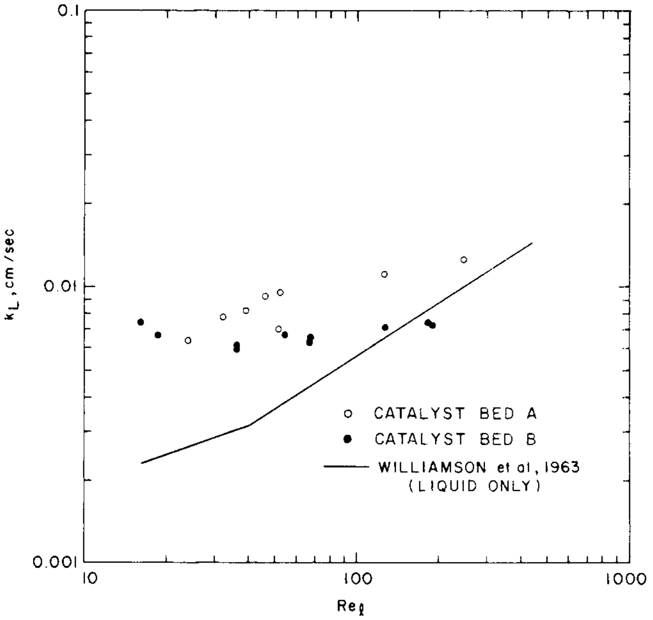

Fig. 4. Mass transfer coefficients for $10 < Re_g < 20$ and assuming $C_s = 0$.

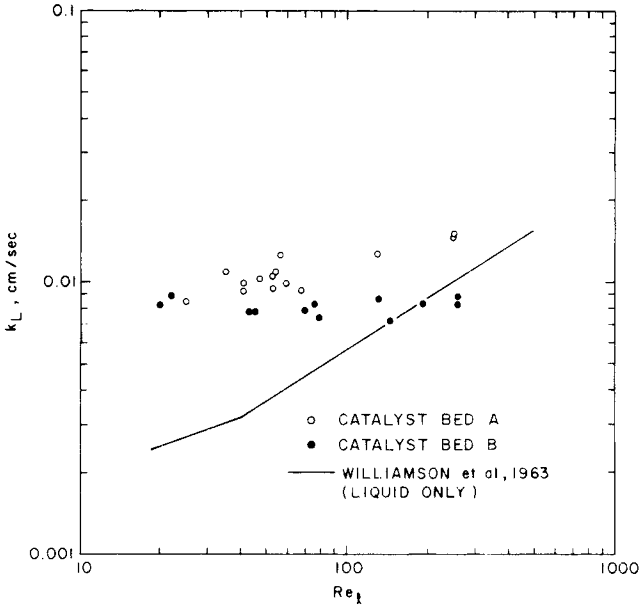

Fig. 5. Mass transfer coefficients for $30 < Re_g < 50$ and assuming $C_s = 0$.

A few of the reaction rates measured during these experiments were greater than those calculated by the kinetic rate equation, assuming the system was controlled by the kinetics and using Babcock's values for A. A new value of the constant A in Equation (2) was required to account for the higher activities of the catalyst used in this work than in Babcock's work. The maximum ratio of the measured rate to the rate calculated by the kinetic equation using Babcock's value for catalyst bed A was 2.5. Thus, a proportionality constant of 2.50 was used for catalyst bed A to ensure that the calculated concentration of hydrogen at the catalyst surface would not exceed C^*. The value of the proportionality constant for catalyst bed B was 1.40.

The proportionality constants were also estimated by another method as a check. Several runs at the lowest gas rates were selected, and liquid phase hydrogen concentrations at the catalyst surface were calculated using single fluid-phase mass transfer coefficients (Williamson et al., 1963). These were used in Equation (2), along with Babcock's values for B and C, to determine proportionality constants to be multiplied times Babcock's values for A. Maximum values by this method were 2.55 for bed A and 1.40 for bed B, which agree well with the values previously obtained.

MASS TRANSFER COEFFICIENTS

The mass transfer coefficient was defined by Equation (1). The hydrogen concentration at the catalyst surface C_s was calculated for each run with Equation (2), using the measured reaction rate and the bulk concentration of α-methylstyrene. The liquid phase at the gas-liquid interface was assumed to be saturated, and C^* was obtained from the reactor pressure and the Henry's Law constant (Johnson et al., 1957). The interfacial area associated with the reaction was taken to be the external surface of the catalyst pellets, 10.6 cm^2/cm^3 of bed volume, as calculated from the pellet diameter.

Values of k_L as influenced by gas and liquid rates are presented in Figures 6, 7, and 8. The mass transfer coefficient increased with liquid rate to about the ½ power, the slopes of the lines in the figures falling in the range of 0.47 to 0.50. The coefficients also increased with gas rate up to Re_g values of about 50. A cross plot of the data in this range shows a gas rate dependence to about the 0.4 power.

The lines through the points in Figures 6 to 8 are compared in Figure 9 and are shown to lie above the correlation for a liquid flowing through a fixed bed in the absence of a gas phase. Hence, there is a consistent picture of increasing mass transfer coefficient with increasing gas rate over the entire liquid rate range up to a gas flow Reynolds number of 50. Presumably, the gas flow thins the liquid film and increases turbulence in the film, promoting transport of the dissolved hydrogen.

The estimates of k_L in Figures 6 to 8 appear to be preferable to those made under the assumption of negligible values of C_s. The points for beds A and B were brought closer together at the higher liquid rates and the liquid rate dependence is of the same order as that for the case of liquids flowing in the absence of a gas phase. None of the points fall below the liquid only correlation.

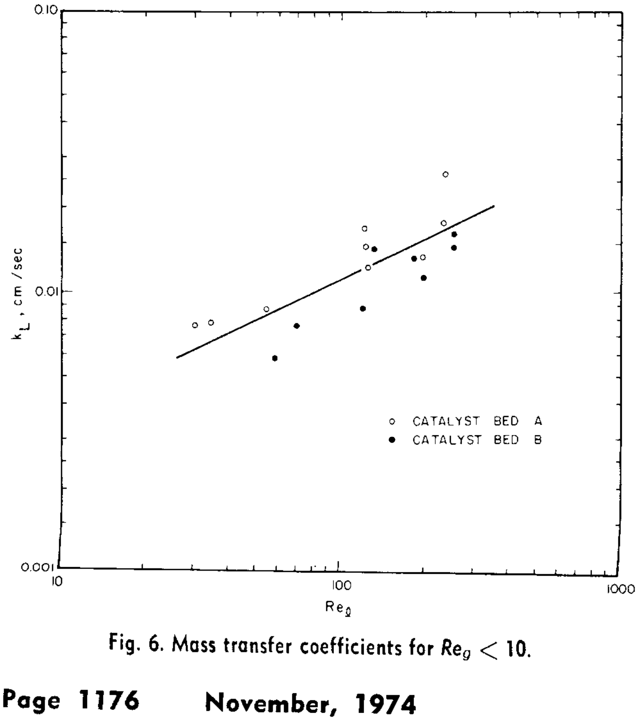

Fig. 6. Mass transfer coefficients for $Re_g < 10$.

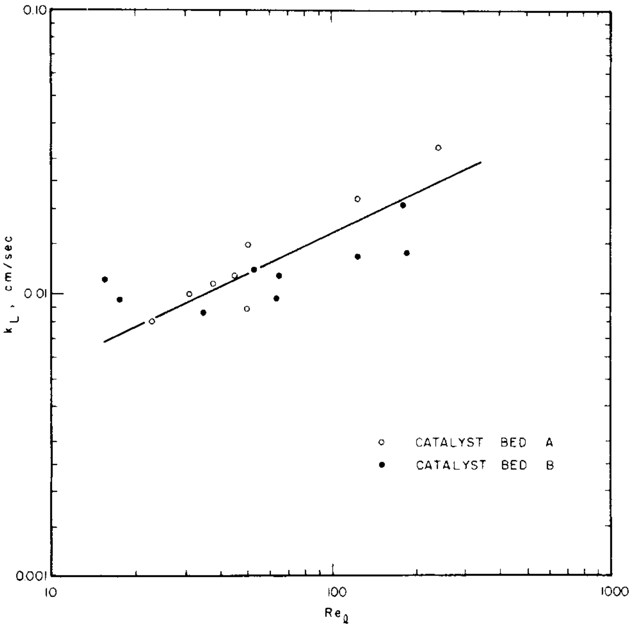

Fig. 7. Mass transfer coefficients for $10 < Re_g < 20$.

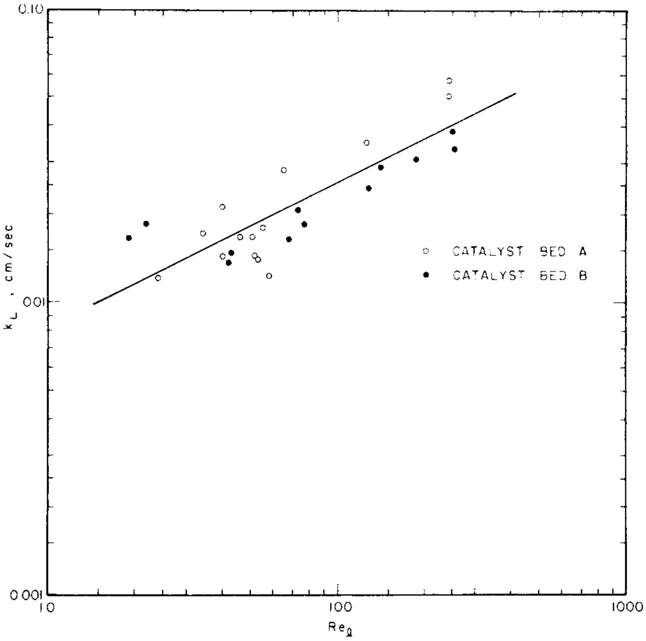

Fig. 8. Mass transfer coefficients for $30 < Re_g < 50$.

Low mass transfer coefficients were obtained at gas Reynolds numbers above 50 (Figure 9). The reason for this is not known, although we surmise that a fraction of the catalyst was not effective, or perhaps was not covered by the liquid phase. Flow and holdup studies of two-phase flow in packed beds were surveyed for a breakpoint or change of regime at these conditions.

The liquid saturation (fraction of the nonsolid volume in the bed occupied by liquid) was estimated from the correlation of Larkins et al. (1961). The data of Larkins were taken with flow in the downward concurrent mode, but the correlations are also said to be valid for horizontal

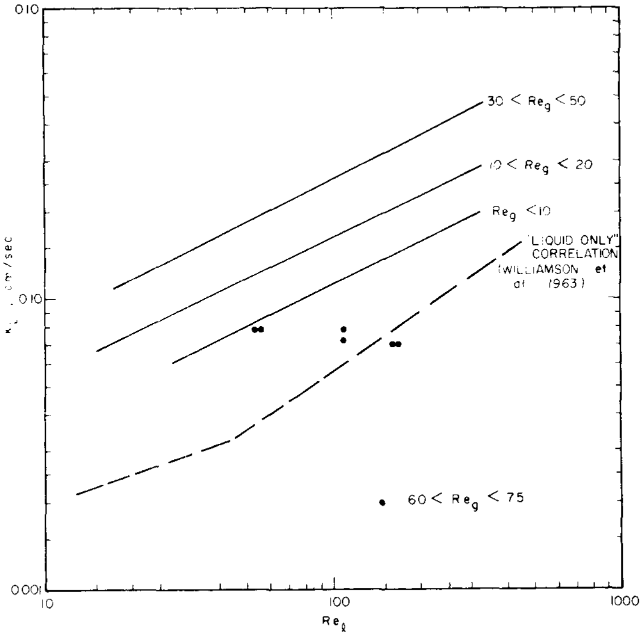

Fig. 9. Comparison of mass transfer coefficients at various gas rates.

and upflow orientations. For the points in Figure 9, values of liquid saturation were estimated at 0.25 for the lower liquid rates, and increased to 0.38 for the higher rates. An estimate of an average film thickness of 0.09 cm (at 25% saturation) was made by dividing the liquid holdup by the surface area of the catalyst particles. The data points in Figure 9 do not yield the lowest liquid saturations among the experimental runs. Several runs at the low end of the liquid rate range, with gas flow Reynolds numbers in the 40 to 50 range, had liquid saturations of 0.16 to 0.17. Thus, the estimates of liquid saturation do not appear to account for the low mass transfer coefficients.

The existence of a pulsing regime in two-phase concurrent flow in packed beds, as observed by Larkins et al. (1961), and Weekman and Myers (1964), may be the explanation of the low mass transfer coefficients. In pulsing flow, flat plugs of high density material (that is, mostly liquid) traverse the entire column. Conversion of the liquid in these plugs might be expected to be low in comparison with the liquid flowing as a thin film over the packing, as it does in the gas-phase-continuous regime. For the air-water system, Weekman and Myers found that the transition from gas-continuous to pulsing flow occurred in the superficial liquid velocity range of 0.65 to 1.3 cm/s for superficial air velocities below about 30 cm/s. However, pulsing flow for the air-water system did not occur at air rates below about 11 cm/s. These regime boundaries may not be applicable to the present system because it is a hydrogen-hydrocarbon system, and it is upflow rather than downflow. The runs in question in the present mode were at hydrogen velocities of 4 to 5 cm/s, and liquid rates were in the range of 0.7 to 2 cm/s. It seems fair to conclude that pulsing flow is a good possible explanation for the low coefficients, but proof of this must await more extensive flow studies.

The work of Mashelkar and Sharma (1970) on packed bubble columns is related but not directly comparable to the present work. Their measurements of k_La were made with a noncatalyzed gas-liquid reaction with flow over an inert packing. That is, in their experiments the chemical reaction took place near the gas-liquid interface and in the present work the reaction took place at the liquid-solid interface. Their measurements of k_La over ¾ in. glass spheres showed practically no liquid rate dependence over their range of superficial liquid velocity of 0.1 to 0.5 cm/s, but k_La increased from about 0.03 to 0.05 s^{-1} as the superficial gas rate increased from 5 to 18 cm/s. It is interesting that their values of the mass transfer coefficient did not fall off in this range of gas rates. Perhaps the onset of pulsing flow takes place at higher gas velocities for larger packings. Weekman and Myers (1964) observed a variation in pulse frequency with packing size in their work with 0.149-in. to 0.255-in. spherical packing.

ACKNOWLEDGMENT

Research was sponsored by the U.S. Atomic Energy Commission under contract with the Union Carbide Corporation.

NOTATION

a = superficial catalyst surface area
A, B, C = empirical constants in Equation (2)
C^* = equilibrium hydrogen concentration in liquid phase
C_s = hydrogen concentration in liquid phase at catalyst surface
D_p = catalyst particle diameter
k_L = mass transfer coefficient
R = rate of reaction
Re_g = Reynolds number, gas phase $D_pV_g\rho_g/\mu_g\epsilon$
Re_l = Reynolds number, liquid phase $D_pV_l\rho_l/\mu_l\epsilon$
V_g = superficial gas velocity
V_l = superficial liquid velocity
X = concentration of α-methylstyrene

Greek Letters

ϵ = nonsolid fraction of the bed
ρ_g = gas density
ρ_l = liquid density
μ_g = gas viscosity
μ_l = liquid viscosity

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Manuscript received June 10, 1974; revision received August 9 and accepted September 6, 1974.