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

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Reaction rates of selective hydrogenation of phenylacetylene (PA) and styrene were studied in a packed-bed reactor with concurrent gas and liquid upflow. The surface reaction rates were expressed by the reaction between adsorbed PA or styrene and dissociated hydrogen atoms competing for the same active sites. The contributions of mass transfer rate were determined, and better selectivity conditions were discussed.

SCOPE

Since concurrent operation is not limited by flooding, gas-liquid reactions requiring a solid catalyst are generally carried out in a gas-liquid upward concurrent flow in industrial applications. During development work on a reactor to convert PA to styrene in a mixture of styrene and xylene with hydrogen in the presence of a supported

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palladium catalyst at minimum loss of styrene to ethylbenzene (EB), it was essential to evaluate the relations between the mass transfer rates and the true surface reaction rates.

Average concentration of PA was less than 4000 ppm in the mixture, and PA is more reactive than styrene. Thus the mass transfer rate of the reactants (PA and hydrogen) was particularly important in discussing the overall reaction rates and in determining an appropriate design of a reactor. The kinetics of hydrogenation of PA were studied by White and Litt (1975) on a rotating catalyst disk and by Matsumoto et al. (1972) over platinum metals. No study has been reported, however, on the reaction kinetics of this hydrogenation in a catalyst bed with gas-liquid concurrent upflow. Also, there has been no published information on the mass transfer rate at the solid and liquid interface in the system, except for Snider and Perona (1974), the authors (1974), and Goto et al. (1975); yet these studies did not examine the mass transfer rate in the system other than taking measurements.

In the present study, which pays particular attention to the mass transfer rate from liquid to a catalyst surface, kinetics of the selective hydrogenation was analyzed.

CONCLUSIONS AND SIGNIFICANCE

For the experimental conditions used (PA = 500 to 7 000 ppm, styrene = 0.2 to 0.6 mole fraction, hydrogen pressure = 1.22 to 4.43 atm, $Re_L = 5$ to 9 and $Re_G = 3$ to 5), the hydrogenation reactions were under mixed diffusion-surface reaction control. The contributions of the mass transfer rate to the overall reaction rate were well determined by applying the authors' results.

Hydrogenation rate of PA or styrene was expressed by the reaction between adsorbed PA or styrene molecules and dissociated hydrogen atoms competing for the same active sites. Individual chemical reaction rate constants were determined, and the relations between the selectivity and the operating conditions were studied, the results showing close agreement between observed data and calculated values.

CHOICE OF FLOW DIRECTION

To decide the flow direction of gas-liquid concurrent flow, preliminary selective hydrogenation experiments were done in both directions in a catalyst bed, and concurrent upflow resulted in far better selectivities than downflow. Similar results were observed by Otsuka et al. (1968) in hydrodesulfurization process: upflow was about 30% superior to downflow in desulfurization efficiency. Thus the upflow reactor system was chosen.

EXPERIMENTS

The reactor used was made from 3.2 cm ID by 30 cm length pipe. The volume of the reactor that was not utilized for the catalyst loading was filled with inert glass beads of similar size to the pellets in the palladium catalyst beds. The catalyst and glass beads were retained between support grids. The catalyst consisted of 0.35 cm diameter by 0.32 cm alumina cylinder pellets coated with palladium. The calculated superficial surface area per unit weight of catalyst was 10.4 cm^2/g. The catalyst bed was 0.91 cm deep, and the weight of catalyst was 7.15 g.

Commercial hydrogen was used for the studies. The flow of hydrogen was measured by a wet test meter. A back pressure regulator on the effluent hydrogen controlled the reactor pressure. Analyses for PA and EB were made by gas chromatography.

The reactor was operated as a differential reactor, with small changes in the liquid phase composition during passage through the reactor. The average composition of the PA mixture ranged from 500 to 7 000 ppm in the styrene and *o*-xylene mixture, and the composition of the styrene and xylene mixture ranged from 0.2 to 0.6 mole fraction of styrene. The reaction temperature and the pressure were varied from 50° to 80°C and 1.22 to 4.43 atm, respectively.

CORRELATION OF EXPERIMENTAL DATA

The following conclusions were drawn from the experimentally determined reaction rates, plotted as functions of concentrations of PA and styrene, and from the hydrogen pressure in Figures 1, 2, and 3.

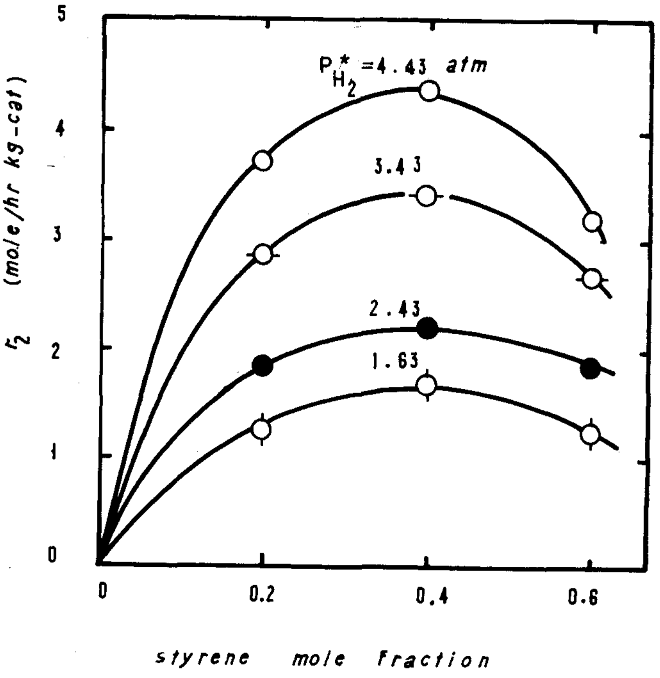

Fig. 1. The variation of hydrogenation rates of styrene r_2 with the styrene concentration.

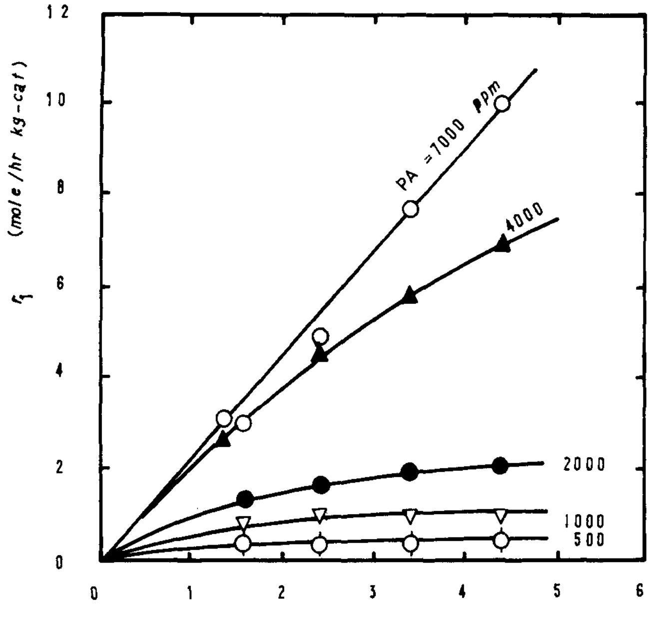

Fig. 2. The variation of the rates of PA hydrogenation r_1 with the pressure of hydrogen.

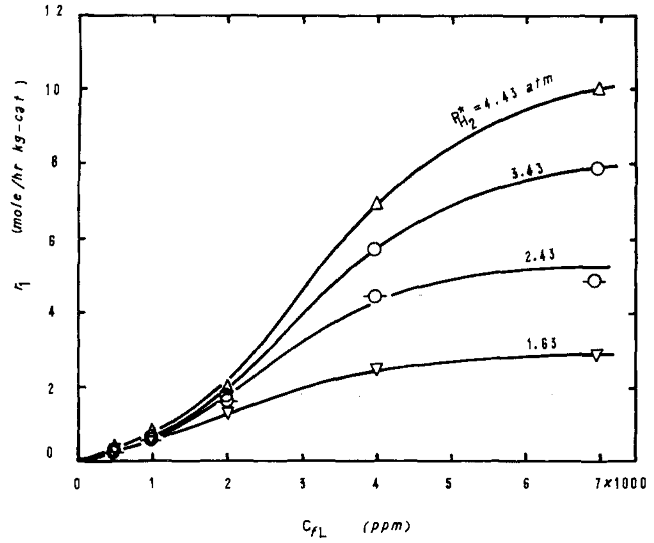

Fig. 3. The variation of r_1 with the concentration of PA in the liquid C_{fL}.

1. The gas phase, being essentially pure hydrogen, offered negligible resistance to the transport of hydrogen.

2. Mass transfer resistance inside the catalyst pellet is small, as the reaction is restricted to the exterior surface where the catalyst resides.

3. A maximum is observed in the hydrogenation rate of styrene plotted as a function of concentration of styrene (Figure 1), an impossible condition if mass transfer of styrene were rate controlling.

4. The reaction rate is proportional to the hydrogen pressure with higher concentration of PA. Thus, in that range the mass transfer of hydrogen must be rate controlling (Figure 2).

5. If adsorption of PA were rate controlling, the reaction rate would be proportional to the concentration of PA (Figure 3). The diffusion rate, however, must be accounted for in some extent, as the concentration of PA is very low and hydrogenation rate is much faster than that of styrene.

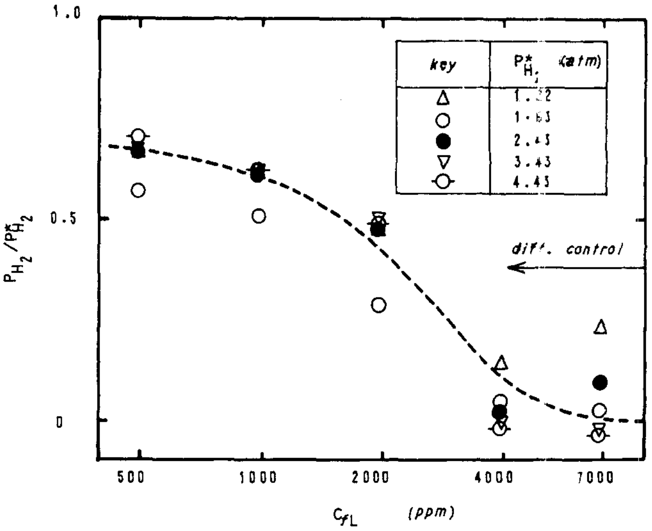

Fig. 4. Effective hydrogen concentration at the catalyst surface $P_{H_2}/P^*_{H_2}$ vs. C_{fL}.

6. If desorption of products were rate controlling, the reaction rate would be nearly independent of the concentration of PA and of hydrogen pressure.

It was concluded, therefore, that the reactions were under mixed diffusion-surface reaction control.

CATALYST SURFACE CONCENTRATION OF HYDROGEN AND PA

Mass transport onto a catalyst surface is the subject of interest in this study. The reactor was operated as a differential reactor with hydrogen saturated feed; therefore, gas-liquid interface resistance was neglected.

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

$$R_H = k_{DH}a(C_{H_2}^* - C_{H_2}) \tag{1}$$

The liquid phase at the gas-liquid interface was assumed to be saturated for hydrogen gas, and the saturated value $C_{H_2}^*$ was obtained from the reactor pressure and the estimated Henry's law constant H (Katayama et al., 1967). The mass transfer rate for PA is

$$R_f = k_{Df}a(C_{fL} - C_f) \tag{2}$$

For a differential reactor, the arithmetic mean value calculated from the inlet and terminal conditions of the bed was used as C_{fL}.

Liquid-solid mass transfer coefficient k_{DH} and k_{Df} were calculated from the correlation of the authors (1974). Then, the PA and hydrogen concentrations at the catalyst surface were calculated with observed PA and hydrogen consumption rates and the estimated mass transfer rate which are given by the following: For Re_L less than 10

$$Sh = 48Re_G^{1/4} \tag{3}$$

For Re_L greater than 10

$$Sh/Sh_o = 1 + 4Re_G^{0.55}/Re_L^{0.7} \tag{4}$$

where

$$Sh_o = 0.75Re_L^{1/2}Sc^{1/3} \tag{5}$$

with $Re_G = 0$. Some problems, however, are present. First, these data were obtained by using diffusion current methods in the system of an active particle in an inert bed, the concentration field around the single particle probably being different that it would be if all particles were active. There was some question about the applicability of the results to usual packed beds (Satterfield, 1975). Second, those correlations were measured at a fixed Schmidt number ($Sc = 1\,170$ for water solution). Thus the results should be applied very carefully to the systems with different Schmidt numbers.

The reasonable concentration profiles on a catalyst surface as shown in Figures 4 and 5 are probably an answer to the first problem. For the second problem, it was found that any correction of Sc on Equation (3) gave only unsatisfactory results (smaller transfer rates than the observed rates). It is probably true that the liquid-solid mass transfer in catalytic bed with gas-liquid concurrent upflow is only dominated by turbulence caused by gas stream and is little affected by physical properties of the liquid. This may be supported by the fact that once gas is introduced into the liquid-solid system, the Sherwood number increases dramatically [twenty to forty times greater than the original level as shown in Figure 1 in the previous work (1974)].

DEVELOPMENT OF KINETIC EQUATIONS

The most satisfactory interpretation of the experimental results postulates that the reactions are expressed by surface reactions between adsorbed PA or styrene and that two adsorbed hydrogen atoms compete for the same active sites. The rate equations corresponding to this mechanism, with Langmuir type of adsorption isotherm assumed, are for PA

$$r_1 = -\frac{k_1K_fK_HC_fP_{H_2}}{(1+K_fC_f+K_sC_s+\sqrt{K_HP_{H_2}})^3} \quad (6)$$

and for styrene

$$r_2 = -\frac{k_2K_sK_HC_sP_{H_2}}{(1+K_fC_f+K_sC_s+\sqrt{K_HP_{H_2}})^3} \quad (7)$$

These equations were rearranged into [for example, Equation (6)]

$$(C_fP_{H_2}/r_1)^{1/3} = (1/k_1K_fK_H)^{1/3}(1+K_fC_f + K_sC_s+\sqrt{K_HP_{H_2}}) \quad (8)$$

for convenience in evaluating the constants.

The four constants were calculated by the following procedure.

1. A plot of $(C_fP_{H_2}/r_1)^{1/3}$ vs. $\sqrt{P_{H_2}}$ gives a straight line, and the intercept gives $(1 + K_fC_f + K_sC_s)/(k_1K_fK_H)^{1/3}$. The slope gives $\sqrt{K_H}/(k_1K_fK_H)^{1/3}$, as shown in Figure 6.

2. Similarly, from the kinetic data of hydrogenation of styrene at different concentrations without the presence of PA, a plot of $(C_sP_{H_2}/r_2)^{1/3}$ vs. $\sqrt{P_{H_2}}$ as shown in Figure 7 gives the ratios of the intercepts as

$$[\text{intercept}_1/\text{intercept}_2] = (1+K_sC_{s_1})/(1+K_sC_{s_2}) \quad (9)$$

which was used for determining K_s. The slopes were not used for calculation owing to lack of accuracy.

3. From the ratio of the slope to the intercept in Figure 6, K_H was determined by

$$[\text{slope/intercept}] = \sqrt{K_H}/(1+K_fC_f+K_sC_s)^{1/3} \quad (10)$$

where, as shown in Figure 6, the intercepts made no difference at different levels of PA concentration in the range of 500 to 2 000 ppm. This implies that

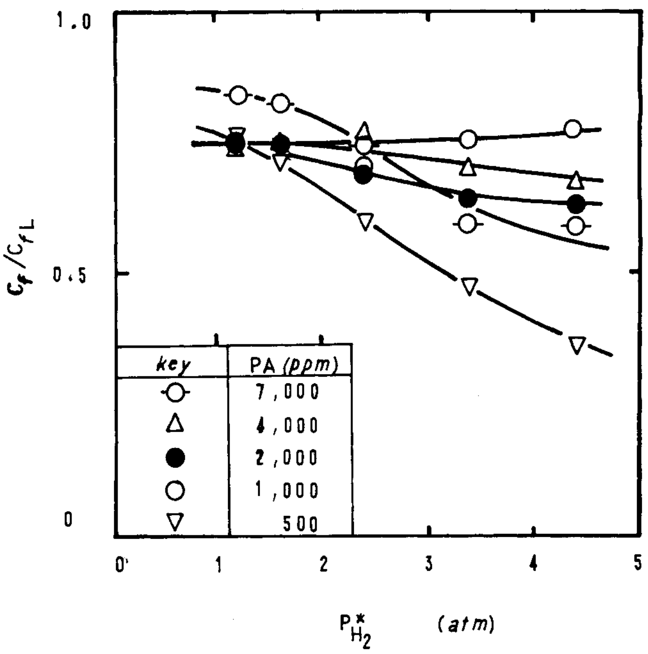

Fig. 5. Effective PA concentration at the catalyst surface C_f/C_{fL} vs. P_{H_2}.

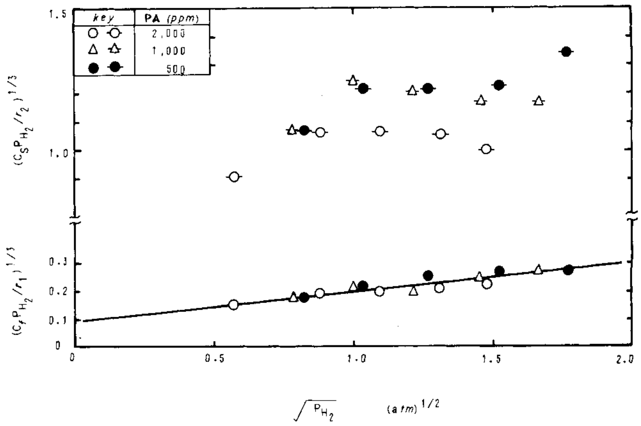

Fig. 6. $(C_fP_{H_2}/r_1)^{1/3}$ or $(C_sP_{H_2}/r_2)^{1/3}$ vs. $\sqrt{P_{H_2}}$.

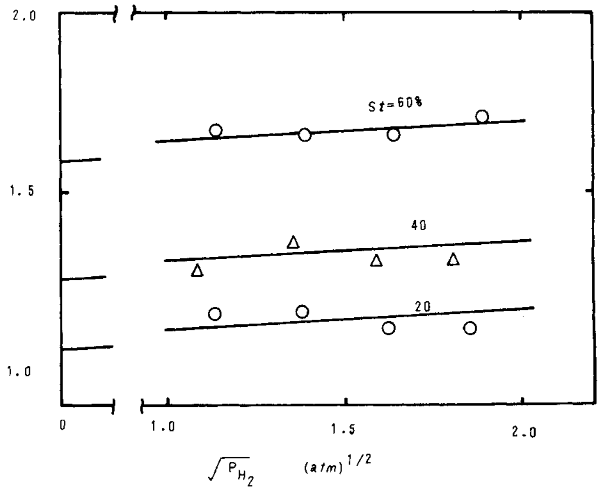

Fig. 7. $(C_sP_{H_2}/r_2)^{1/3}$ vs. $\sqrt{P_{H_2}}$.

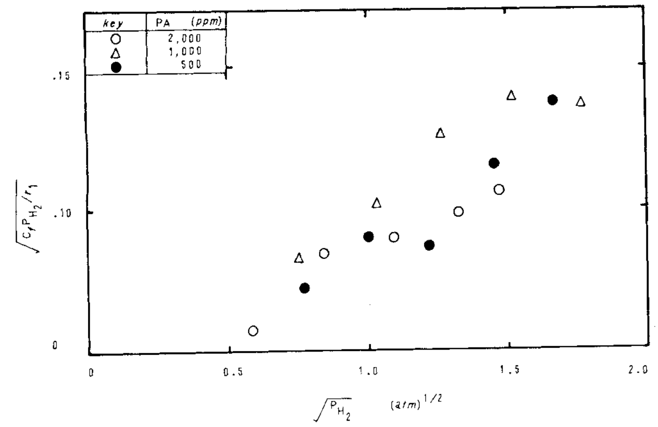

Fig. 8. $(C_fP_{H_2}/r_1)^{1/2}$ vs. P_{H_2}.

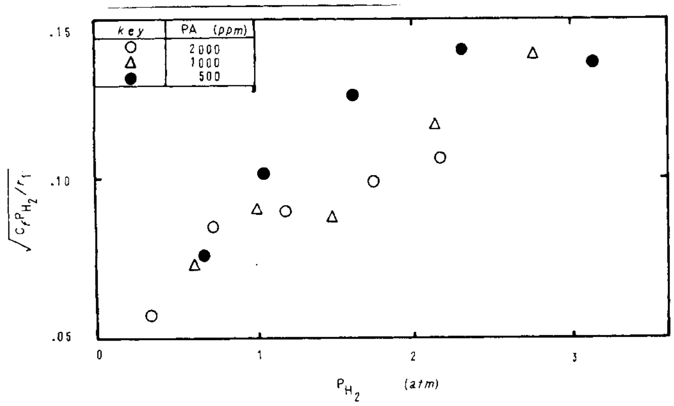

Fig. 9. $(C_fP_{H_2}/r_1)^{1/2}$ vs. $\sqrt{P_{H_2}}$.

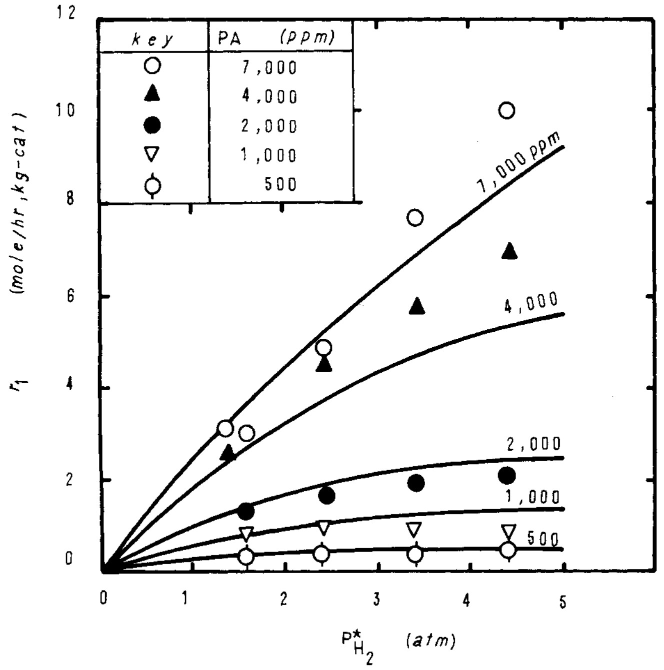

Fig. 10. PA hydrogenation rates r_1. Calculated values (solid lines) are compared with the observed values.

$$1 + K_fC_f + K_sC_s \cong 1 + K_sC_s \qquad (11)$$

From Equations (10) and (11), K_H was determined.

4. The ratio of set of hydrogenation of styrene with and without the presence of PA gives

$$[(C_sP_{H_2}/r_2)_a^{1/3}/(C_sP_{H_2}/r_2)_b^{1/3}] = \frac{1 + K_fC_f + K_sC_s + \sqrt{K_HP_{H_2}}}{1 + K_sC_s + \sqrt{K_HP_{H_2}}} \qquad (12)$$

where suffix a is with PA and b is without PA. Since K_s and K_H had been determined, K_f was calculated from Equation (12). To examine the magnitude of K_fC_f for the experiments in Figure 6, the contributions of K_fC_f to $1 + K_fC_f + K_sC_s$ were found to be 1 to 5% in these experiments. Thus, the assumption of Equation (11) was approved.

5. Once K_f, K_s, K_H were calculated, k_1 was determined from either the intercept or the slope of the line in Figure 6.

6. Reaction rate constant k_2 was determined by the reaction rate ratio of PA and styrene.

7. Estimated kinetic constants at 50°C were $K_f = 7.3$, $K_s = 0.29$, $K_H = 3.6$, $k_1 = 274$ and, $k_2 = 56$ in moles per liter, hour units.

In Figure 6, the hydrogenation rate of styrene resulted in poor linearity in the plot. The reason may be that the effective hydrogen concentration on the catalyst surface close to the adsorbed styrene molecule would be a little different from that to PA.

The other mechanisms, such as adsorbed PA or styrene molecules reacting with adsorbed dissociated hydrogen atoms on different active sites or adsorbed hydrogen molecule on the same or different active sites, were rejected because of poor linearity in an Equation (8) type of plot or a minus value of the estimated constant (Figures 8 and 9).

Although White and Litt (1975) and Matsumoto et al. (1972) have studied the hydrogenation of PA in the presence of palladium and platinum catalysts, respectively, the reaction mechanism had been left uncleared.

As references, the hydrogenation of α-methylstyrene (AMS) with the presence of palladium catalysts was studied by Babcok et al. (1957). For hydrogen pressure above 3 atm, the surface reaction takes place between adsorbed AMS and dissociated hydrogen atoms adsorbed on different active sites of the catalyst, and with pressure below 3 atm, adsorbed AMS reacts with dissociated hydrogen to compete for the same active sites.

SELECTIVITY AND OPERATING CONDITIONS

Since the kinetic mechanisms had been determined, the effects of operating conditions on the selectivity were studied.

The rate equation of the reactions are Equations (6) and (7), and correlations between PA and hydrogen concentration in the bulk liquid and that on the catalyst surface at steady state are

$$C_f = \frac{C_{fL}}{1 - r_1/k_{Df}aC_f} \qquad (13)$$

For a differential reactor with hydrogen saturated feed

$$P_{H_2} = \frac{P_{H_2}^*}{1 - (r_1 + r_2)/k_{DH}aHP_{H_2}} \qquad (14)$$

To find the initial values of C_f and P_{H_2}, Equations (13) and (14) were solved by the Newton-Raphson method with initial values of C_{fL} and $P_{H_2}^*$. Then, Equations (6),

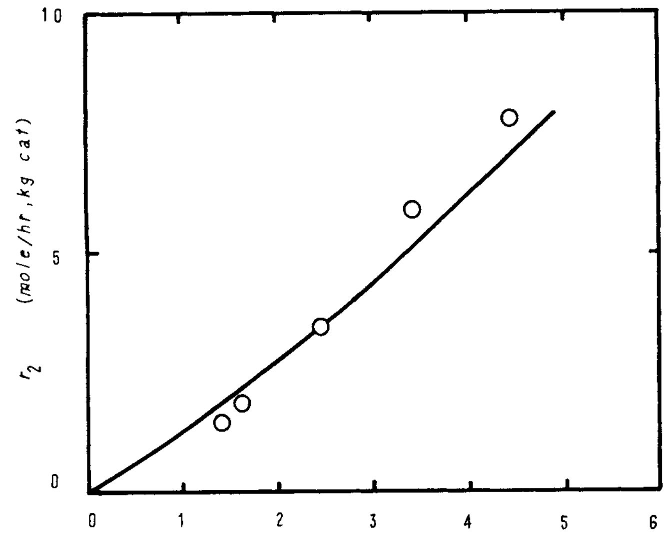

Fig. 11. Hydrogenation rates of styrene r_2. Comparison of calculated values with the observed ones.

(7), (13) and (14) can be solved simultaneously for instantaneous C_f and C_E (C_s was kept constant).

Thus, instantaneous PA concentration in the liquid phase is given by

$$C_{fL} = C_f(1 - r_1/k_{Df}aC_f) \tag{15}$$

and PA conversion is given by

$$Y = 1 - C_{fL}/C_{fLo} \tag{16}$$

The calculated values were compared with the observed data in Figures 10 and 11. Thus, it may be concluded that the agreements are fairly good.

Finally, the effects of various operating factors on the selectivity will be discussed. Let us define the instantaneous selectivity as

$$S = \frac{r_1}{r_2} = \frac{k_1 K_f C_f}{k_2 K_s C_s} = \frac{k_1 K_f C_{fL}}{k_2 K_s C_s (1 - r_1/k_{Df}aC_f)} \tag{17}$$

From Equation (17), we see that the selectivity apparently increases with smaller C_s and larger $k_{Df}a$ or C_f, it has a minimum value to P_{H_2}. The selectivity reaches a minimum value at $P_{H_2} \cong 4$ atm at 50°C, which may correspond to $P_{H_2}{}^* = 7$ to 10 atm according to the operating conditions.

Some calculation results are tabulated in Table 1 to show the effects of some factors on the selectivity.

Significant effects of temperature on the selectivity were not observed. Similar results were observed on the selectivity in hydrogenation of PA, styrene, and AMS by White and Litt (1975).

TABLE 1. EFFECTS OF HYDROGEN PRESSURE AND MASS TRANSFER COEFFICIENT ON THE SELECTIVITY. INITIAL PA IS 2 000 PPM AND STYRENE IS 0.4 MOLE FRACTION IN THE REACTION MIXTURE

$P_{H_2}{}^*$ (atm)	P_{H_2} (atm)	k_{Df} (cm/hr)	Selectivity†
1.63	0.44	34	0.41
7.00	3.92	34	0.28
10.00	7.00	34	0.29
1.63	0.20	61	0.43

† r_1/r_2 at 10% PA conversion.

There are very few comparable studies on selective reactions proceeding in this kind of multiphase system. Thus we hope that the interest of this study might extend beyond that of the reactions actually studied.

NOTATION

a = surface area of catalyst per unit weight
C_H, C_f, C_s, C_E = concentration of hydrogen, PA, styrene, and EB at catalyst surface
d_p = particle diameter
d_h = $\epsilon d_p/1.5(1 - \epsilon)$
D = molecular diffusivity
H = Henry's constant
k_D = mass transfer constant
K = kinetic constant in a Hougen-Watson type of equation
Re_L, Re_G = $\rho u d_h/\epsilon\mu$ Reynolds number, L for liquid and in main gas stream, respectively, atm
r_1, r_2 = reaction rate of PA and styrene, respectively
R_H, R_f = mass transfer rate of hydrogen and PA, respectively
Re_L,Re_G = $\rho u d_h/\epsilon\mu$ Reynolds number, L for liquid and G for gas
Sc = $\mu/\rho D$, Schmidt number
Sh = $k_D d_h/D$, Sherwood number
u = mean fluid velocity
Y = conversion
ϵ = void
ρ = fluid density
μ = viscosity

Subscripts

f = PA
H = hydrogen
s = styrene
L = liquid phase
o = initial condition

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