

Process Intensification in a Trickle-Bed Reactor: Experimental Studies

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Process intensification has been the focus of increasing attention in recent years. A volume reduction of distillation and absorption units by a factor of 100 or more using rotating beds has been claimed. Industrial trickle-bed reactors are bulky because the liquid flow and interphase transport of the reacting species are governed by the earth's gravity, as in the case of distillation columns. To explore the possible process intensification in these reactors, we have measured the reaction rates of hydrogenation of α -methyl styrene with palladium as catalyst in the rotating beds of spherical particles and metal foam, which acted as catalyst support. To quantify the intensification achieved, the reaction rates were compared with those of conventional trickle beds. The enhancement in the reaction rates was in the range of 30–40 times in a centrifugal force field of about 450 times the gravitational force field. An industrial reactor of 60 m³ could be replaced with a rotating bed < 1.5 m³ in volume. It appears that there is a possibility of further process intensification. However, the volume reduction is possible only for mass-transfer limited reactions. © 2005 American Institute of Chemical Engineers AIChE J, 51: 3186–3192, 2005

Keywords: process intensification, Higee (high gravity), trickle-bed reactor, rotating packed bed, rotating trickle-bed reactor

Introduction

Process intensification in distillation and absorption equipment has been the focus of increasing attention in recent years. In conventional columns, the achievable vapor and liquid throughputs and interphase mass transport are inherently governed by the gravitational field. These can be enhanced in a rotating annular packed bed [also known as Higee (high gravity)] because the liquid flows under the influence of a centrifugal force field of 350–1000 times the gravitational force field. It has been claimed that such process intensification can lead to a volume reduction of the equipment by a factor of 100 to 1000. This in turn leads to lower capital costs and inherently safer design because the liquid holdup is smaller than that in conventional equipment. Ramshaw,¹ at Imperial Chemical In-

dustries (ICI), UK and recently Trent et al.,² at Dow Chemicals, reported the commercial use of rotating packed beds. An appraisal of the process intensification in the rotating packed beds has been presented by Rao et al.³ The centrifugal field permits the use of a packing with high specific area in the range of 1000–4000 m²/m³, which is about five to ten times larger than the conventional packing. The liquid flows as thin films because of the centrifugal force and this leads to a reduction in the liquid-side resistance to mass transfer. Sivalingam et al.⁴ carried out studies on mass transfer from gas to solid through the liquid film flowing over a rotating string of spheres. They found that the enhancement in the transfer rate is five to seven times on the rotating string of spheres compared to that with the gravity flow. Therefore, the enhancement in the volumetric liquid-side transfer coefficient could be as high as 25–70 in rotating packed beds of spheres. Therefore, for the reaction systems in which the rate-limiting step is the liquid-side mass transfer, a similar volume reduction of the reactor could be achieved. Rao et al.⁵ proposed a novel rotating packed bed in

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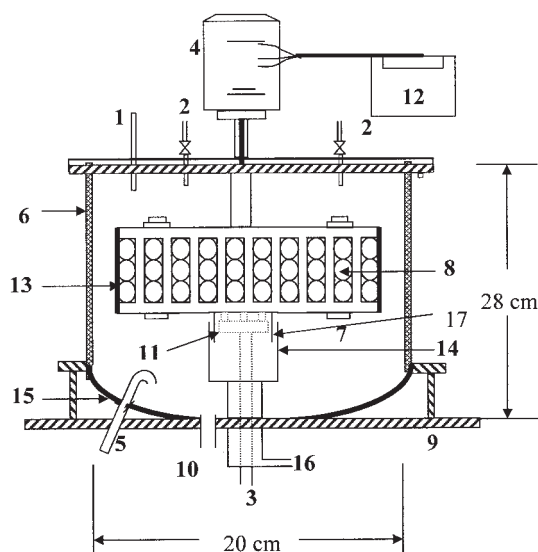


Figure 1. Model trickle-bed reactor.

1: Manometer tap; 2: gas outlet; 3: liquid inlet; 4: motor; 5: gas inlet; 6: glass wall; 7: annular packed bed; 8: alumina catalyst beads; 9: stand; 10: liquid outlet; 11: distributor; 12: AC inverter drive; 13: cylindrical side plate with rectangular slots; 14: cup for collecting splash liquid; 15: elliptical dish; 16: splashed liquid outlet; 17: cylindrical baffle.

which the slip velocity between gas and liquid is as high as 10–30 m/s in contrast to 1–3 m/s in the conventional columns. A similar volume reduction can be achieved if the rate-limiting step is the gas-side mass transfer.

Trickle-bed reactors are used for hydrodesulfurization, hydrodemetallization, hydrodenitrification, hydrogenation, and oxidation. These units are bulky because the flow of the liquid in these reactors and interphase transport of reactants are inherently governed by the gravitational field, as in the case of distillation and absorption columns. The volumes of some of these reactors are in the range of 150–200 m³.⁶ Van Hasselt et al.⁷ showed that the volume of a trickle-bed reactor would be in the range of 430 to 600 m³ for a throughput of 20,000 barrels per day for “deep” hydrodesulfurization of vacuum gas oil required to meet future emission standards. Further, they suggest the use of special packing and countercurrent flow of the gas and liquid to reduce the volume. The rotating packed beds permit countercurrent flow, or even crosscurrent flow, which may be superior to the former in that the reverse reaction can be reduced because the H₂S is removed as it is formed. They may find application for the Fisher–Tropsch synthesis of wax, liquid-phase hydrodesulfurization of kerosene and naphtha, and hydrogenation of aromatics and olefins in solvent manufacture. The rotating beds are not new; the rotating-basket reactors have been used for the determination of intrinsic kinetics.⁸

The objective of the present study is to explore the possible process intensification in rotating trickle-bed reactors. Toward this end, we have chosen the hydrogenation of α -methyl styrene to cumene using palladium as catalyst, which is known to be a mass-transfer-limited reaction above 40°C. We have carried out the experimental studies on a rotating trickle-bed reactor using a bed of spherical alumina particles impregnated with palladium as well as with metal foams plated with palla-

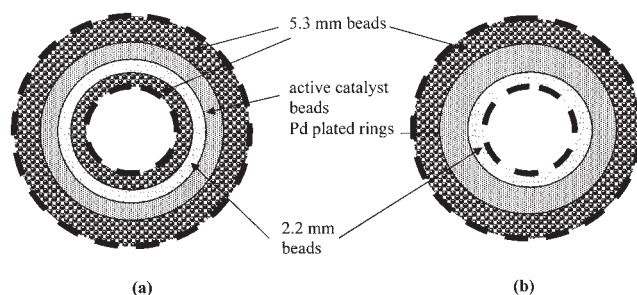


Figure 2. Arrangement of inert catalyst layers and metal-foam (top view).

Radial width of layers (cm): (a) outer 2.5, active 1.5, inner 1.0, innermost 1.0; (b) outer 3.1, metal-foam 2.2, inner 0.7.

dium. To quantify the process intensification, we have compared the rates in rotating trickle-bed reactors with those in conventional trickle-bed reactors reported in the literature.

Experimental

Rotating trickle bed

Figure 1 shows the experimental rotating trickle-bed reactor. It consisted of an outer casing, housing for the bed, a liquid distributor, and a cup for collecting the liquid that was splashed at the inner periphery of the bed housing. The side of the casing was a cylindrical glass tube sandwiched between two metal plates. To facilitate the liquid withdrawal, a stainless steel elliptical dish was attached to the lower end of the glass wall as shown in the figure. The housing was attached to the shaft of the motor mounted on the top plate. The rotational speed of this induction motor was varied continuously between 500 and 2500 rpm using an inverter drive.

The housing of the bed was made of two annular disks separated by two inner and outer slotted cylindrical shells and were bolted to provide a rigid housing for the bed. The dimensions of the housing were: height, 4.5 cm; inner diameter, 9 cm; and outer diameter, 21 cm. The free area for the flow of gas and liquid on the inner and outer plates was 50% of the total.

To ensure uniform flow of liquid over the active catalyst, the bed of catalyst particles was sandwiched between the layers of inert particles as shown in Figure 2a. Similarly, the metal-foam rings were sandwiched between the layers of inert alumina particles as shown in Figure 2b.

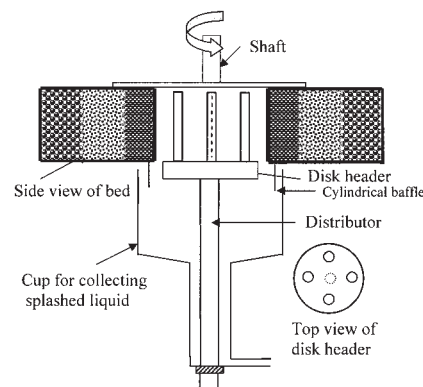


Figure 3. Liquid distributor and bed assembly.

Table 1. Physical Properties of γ -Alumina Particles

Mean diameter, mm	2.2, 1.5
Packing density	797.6 kg/m ³
External porosity	38.18%
Internal porosity	62.80%
Particle density	1.17 g/cm ³
Pore volume	0.5366 cm ³ /g
External surface area	1690, 2489 m ² /m ³

The liquid distributor and the bed assembly are shown in Figure 3. The liquid distributor was made of a disk-shaped header. The liquid inlet tube was attached to the header at the center. Four tubes were attached near the outer periphery as shown in Figure 3. These were provided with eight equispaced circular holes of 0.3 mm diameter. The spacing of holes on the four tubes was staggered such that the liquid issued as 32 jets directed toward the inner periphery of the housing. A clearance of 0.5 cm between the tubes and the inner periphery was kept to ensure that the jets reached the latter almost horizontally. At very low liquid flow rates, some of the alternate holes were blocked by winding a Teflon[®] tape over the holes to achieve such a condition.

If the bottom plate of the housing was a circular disk, a part of the liquid reaching the inner periphery gets splashed and falls onto the bottom disk. It flows on the disk as a thick layer without proper contact with the gas phase.⁹ To prevent the bypassing of liquid, an annular disk and a cup arrangement were provided to draw the splashed liquid separately and to measure its flow rate. A cylindrical baffle was fitted to the bottom disk to prevent the flow of splashed liquid into the casing. The clearance between the baffle and the cup was large enough to allow free flow of hydrogen from the casing to the inner periphery of the bed.

Catalyst preparation

The alumina catalyst particles were prepared by impregnating them with palladium, as suggested by Herskowitz et al.¹⁰ The visual observations indicated that the palladium was uniformly distributed throughout the particles. We have used

particles of 2.2 and 1.5 mm in diameter. The physical characteristics of particles supplied by the vendor (Norton Co., Philadelphia, PA) are given in Table 1.

The metal-foam rings were supplied by Recemat[®] International B.V. (The Netherlands). Their structure and characteristics as supplied by the vendor are given in Table 2. The metal-foam bed was made by stacking five annular rings: four of 1 cm height and one of 0.5 cm height. To deposit palladium on these rings, we followed the electroless-deposition method suggested by Lowenheim¹¹ and Henry.¹² First, the metal-foam rings were given an alkaline soak for 30 min in a boiling solution with a composition of 15 g/L NaOH and 15 g/L Na₂CO₃. After soaking, the rings were thoroughly rinsed with distilled water. These rings were then immersed in a plating bath containing a solution of 2 g/L PdCl₂, 160 mL/L NH₄OH (27%), 26 g/L NH₄Cl, and 10 g/L NaH₂PO₄·H₂O at 50°C. The solution was stirred with a magnetic stirrer to eliminate gas bubbles and to maintain uniform composition in the holes and crevices. Upon completion of the electroless deposition, the solution, which was dark in color, became clear (colorless). After the deposition, the foam rings were rinsed thoroughly with distilled water and dried in a stream of hot air. The difference in the weight of metal rings before and after the plating gave the exact amount of palladium deposited. The visual observation of the metal foam indicated that it was black in color and the palladium was uniformly deposited. The catalyst particles were activated by heating to 370°C in a stream of hydrogen for 24 h. However, the metal foams were used as such after washing with distilled water.

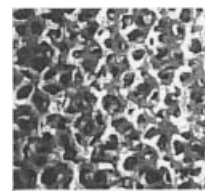
Setup

The experimental setup is shown in Figure 4. Keeping the bed rotating, first nitrogen and then hydrogen were passed through the casing for about 1 h each to ensure the casing was filled with hydrogen before the start of the run. Then, the liquid was circulated through a water bath and the bed using a peristaltic pump (Cole-Parmer Instrument Co., Vernon Hills, IL). A surge tank was used to dampen the fluctuations in the flow. The rotational speed and the liquid flow issuing out of the

Table 2. Properties of RECEMAT[®] Metal-Foam

Property	RECEMAT [®] (2733)	RECEMAT [®] (1723)
Estimated avg. pore diameter, mm	0.6	0.9
Specific surface area, m ² /m ³	2500	1700
Material	Nickel-chromium	Nickel-chromium
Dimensions of the rings		
ID, mm	102	102
OD, mm	149	149
Density, g/cm ³	0.60	0.65
Porosity	0.9	0.9

Metal foams



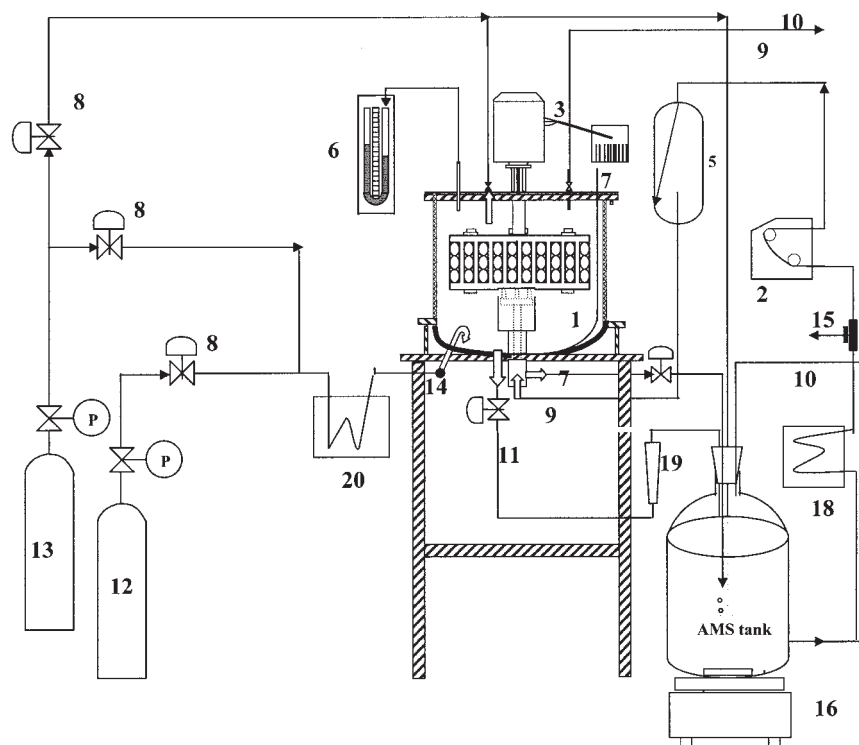


Figure 4. Experimental setup.

1: Reactor; 2: peristaltic pump; 3: A/C motor; 4: feed tank-cum-presaturator; 5: surge tank; 6: manometer tapping; 7: digital thermometer; 8: gas flow control valves; 9: liquid inlet; 10: gas outlets; 11: liquid outlet; 12: H₂ gas cylinder; 13: N₂ gas cylinder; 14: gas inlet; 15: sampling port; 16: magnetic stirrer; 17: cylindrical baffle; 18: water bath; 19: rotameter; 20: water bath.

bed were monitored using a digital stroboscope (Nova-Strobe DB plus, Cole-Parmer) to observe the nature of the flow. We carried out studies at 2200 and 1900 rpm and have reported the centrifugal acceleration, computed at the inner periphery of the catalyst layer. The liquid came out of the bed as fine droplets and they appeared to be uniformly distributed throughout the outer periphery. No special provision was made to circulate the hydrogen through the bed. Because of the centrifugal force field the gas is drawn in through the inner periphery and forced out through the outer periphery.

Liquid samples were drawn through the sampling port at regular intervals and analyzed for the cumene concentration. A gas chromatograph (Model 5765; Nucon Engineers, Delhi, India) with a 2 m long bentone-34 column and a dual-flame ionization detector was used for the analysis. A stream of hydrogen was passed through the coil immersed in a water bath before it entered the casing and the excess hydrogen was vented from the reactor. In all the runs, the α -methyl styrene was presaturated with hydrogen. The liquid was circulated through the bed with a peristaltic pump (Model 78022-10, Cole-Parmer). The recirculation rate and the splashed liquid flow rate were monitored. The rotational speed was measured with the digital stroboscope and cross-checked with the inverter drive readings. All the experiments were carried out at a temperature of 40°C and 1 atm pressure.

Results and Discussion

The reaction rate was determined from the rate of change of the concentration of cumene in the charge with time using the equation

$$r = \frac{V}{V_b} \frac{dC}{dt}$$

where V is volume of the liquid charge to the setup (cm³), V_b is volume of the active catalyst bed (cm³), C is the concentration of cumene (mol/cm³), and t is time (s).

Figure 5 shows the reaction rates with the alumina particles in the rotating beds. The liquid superficial velocity was computed based on the inner radius of the active layer. The rate is higher for the smaller size particles. Under the conditions used,

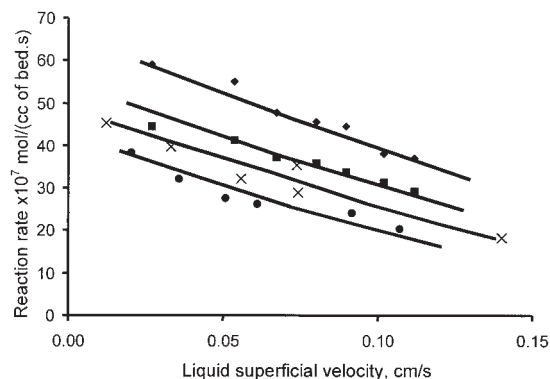


Figure 5. Reaction rates with γ -alumina particles.

◆, 1.5-mm beads: 485 g (2200 rpm), 1.0 mass % Pd; ■, 1.5-mm beads: 374 g (1900 rpm), 1.0 mass % Pd; ×, 2.2-mm beads: 483 g, 1.0 mass % Pd; ●, 2.2-mm beads: 374 g, 1.0 mass % Pd.

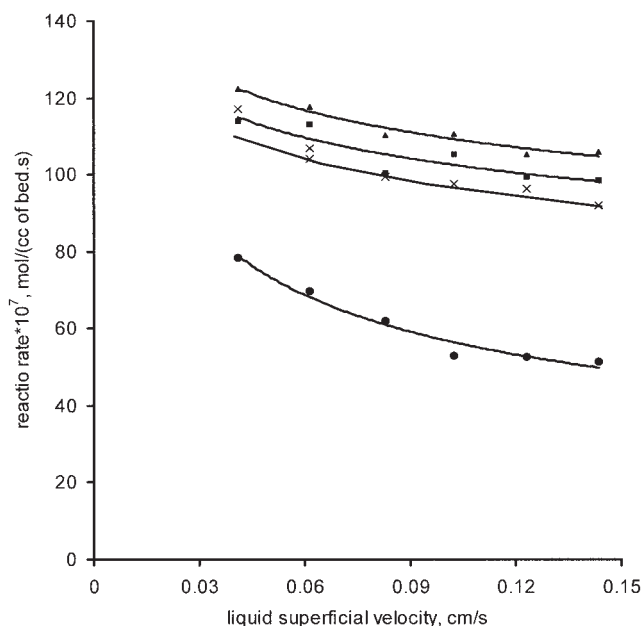


Figure 6. Reaction rates with metal foams at 360 g (1900 rpm).

●, RECEMAT® (1723): 0.5 mass % Pd; ■, RECEMAT® (1723): 1.0 mass % Pd; ▲, RECEMAT® (1723): 1.5 mass % Pd; ×, RECEMAT® (2733): 2.5 mass % Pd.

the kinetics of hydrogenation of α -methyl styrene to cumene on Pd is known to be irreversible, first order with respect to dissolved hydrogen and zero order with respect to α -methyl styrene. Further, it was in a mass-transfer-controlled regime. Therefore, the hydrogen transfer to the surface was the limiting step. Because the smaller particles have larger specific area, the rate was higher for smaller size. The rate increased with increasing centrifugal force and decreased with increasing liquid velocity as the liquid film became thinner.

Figure 6 shows the reaction rates with the foam metal. Note that the reaction can occur only on the surface of the catalyst because it is nonporous, unlike the alumina particles. Even with alumina particles, the reaction occurs in a thin outer shell of the particles. Therefore, the trend in the reaction rate was similar to that with the alumina particles as the reaction took place. The rates increased as the liquid film became thinner with increasing centrifugal force field. However, the rate was greater for the smaller specific area for the metal foams. It is unlikely that there is a liquid maldistribution. Even if it was present, the extent of liquid maldistribution may not differ significantly between the two foam packings. Burns and Ramshaw¹³ observed the changing nature of the liquid flow with rotational speed over a reticulated wire-mesh packing. At low rotational speeds, the liquid flow covered the "pore" openings of the network of the filaments as films, which they termed *pore flow*. With increasing rotational speed, the pore flow turned into a droplet flow in which the tiny droplets jumped from one filament to the other. If the film flow was laminar, there would be concentration gradients within the film, as is the case with the pore flow. However, the periodic mixing of liquid within the film leads to higher mass-transfer rates. Such a mixing is possible with the drop flow. The nature of flow may depend on the size of the pore openings of the foam metal. There are

indications that higher specific area does not always lead to higher mass-transfer rates.³ The understanding of the nature of liquid flow and of possible liquid maldistribution could help to enhance the process intensification.

Further, the rates with the alumina particles were substantially lower than those of the metal foams, even though their specific surface areas were comparable. In the case of the former, the area covered by the liquid pendular rings, held between the adjacent particles, would be very small, unlike that for the gravity flow. However, it cannot account for the rate being half that of the foam. Munjal et al.¹⁴ reported the gas-liquid interfacial area in a rotating bed of spherical particles. Their data indicate that the gas-liquid interfacial area could be less than half of the specific packing area. There could be a partial wetting of the bed, which may depend on the type of packing. There is a need for deeper understanding of the nature of liquid flow and its texture in the rotating beds.

To quantify the process intensification, that is, the volume reduction that could be obtained with the rotating beds, we require the reaction rate data for the conventional trickle-bed reactor. Herskowitz et al.¹⁰ and McManus et al.¹⁵ reported the reaction rates in trickle beds under similar conditions based on the mass of catalyst. We have converted their rate data to volume basis using their particle density and bed porosity; their maximum reaction rates were 4.9×10^{-7} and 1.44×10^{-7} mol/(cm³ of bed s⁻¹). The former used catalyst of 0.75 mass % Pd and 1.34 diameter, whereas the latter used 1.5 mass % Pd and 3.3 mm diameter. The rates depend on the mass % Pd and the size of the particle. The experimental rate data for this reaction system under similar conditions have been reported in trickle beds, on strings of beads, and single pellets with different percentages of Pd loading and particle diameters by several groups of investigators. Therefore, these data had to be reduced to a common basis for the use as a reference scale for quantifying the process intensification. It is reasonable to use the surface area rather than the volume of bed or the mass of the particles as the basis for the reaction rates, given that the reaction occurs within a thin outer shell of the particles. The

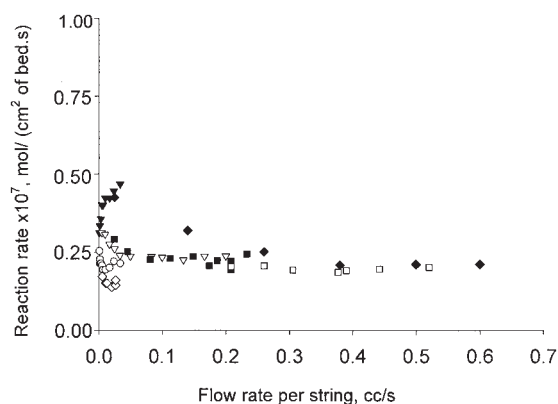


Figure 7. Comparison of reaction rates of different investigators on specific area basis.

○, Herskowitz et al.¹⁰: bed, 0.75 mass % Pd; ▼, Herskowitz et al.¹⁰: bed, 2.5 mass % Pd; ▽, Subramaniam¹⁸: string, 2.5 mass % Pd; ■, Sivalingam⁴: string, 2.5 mass % Pd; □, Satterfield et al.¹⁷: string, 1.0 mass % Pd; ◆, Funk et al.¹⁹: single pellet, 1.0 mass % Pd; ◇, McManus et al.¹⁵: bed, 1.25 mass % Pd.

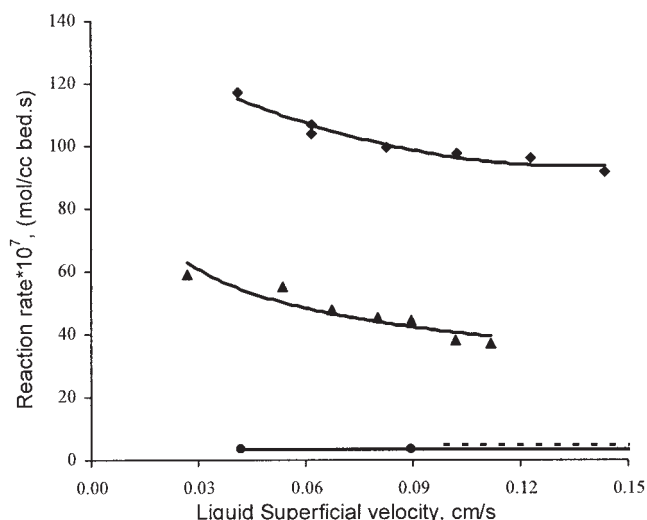


Figure 8. Comparison of reaction rates for different substrates.

◆, RECEMAT® (1733): 1.5 mass % Pd, 360 g; ▲, 1.5-mm alumina beads, 1.0 mass % Pd, 485 g; ●, conventional trickle bed: 1.0 mass % Pd; ---, Herskowitz et al.¹⁰

data reported by Morita and Smith¹⁶ and Satterfield et al.¹⁷ indicate that the intrinsic kinetic constant varies linearly with mass percentage of palladium up to 5%. Therefore, one would expect that the reaction rate data based on the surface area and palladium mass % of different investigators should be in agreement. Sivalingam et al.⁴ computed the reaction rate data reported by several workers in terms of $\text{mol}/(\text{cm}^2 \text{ mass \% Pd}^{-1})$. The liquid rate per string was computed by considering that the bed is composed of several strings of spheres. The number of strings was set as the number of spheres that can be packed in a circular cross section of the bed perpendicular to the direction of flow in a triangular pitch with spheres touching each other. Figure 7 shows the comparison of the rates reported by different groups on a common basis. There was a considerable deviation in the rates reported by different investigators at very low flow rate, but the rate was in good agreement for most cases and it was nearly constant beyond the liquid flow rate of $0.05 \text{ cm}^3/\text{s}$. Therefore, we may consider the reaction rate to be $0.22 \times 10^{-7} \text{ mol}/(\text{cm}^2 \text{ mass \% Pd}^{-1} \text{ s}^{-1})$.

Because the process intensification requires the quantification in terms of volume reduction, we have converted the reaction rate from unit area basis to unit volume basis for the bed for 2.2-mm particles. The rate is $3.14 \times 10^{-7} \text{ mol}/(\text{cm}^3 \text{ s}^{-1})$ for 1.5 mass % Pd.

Figure 8 shows the comparison of the reaction rates in rotating beds with alumina beads and metal foam and in conventional trickle beds computed as mentioned above. To show direct comparison, we have plotted the data of Herskowitz et al.¹⁰ and have presented only the highest rates for the alumina particles and the metal foam. It can be seen that the rate with the RECEMAT® (1723) metal foam was the highest. The rate in the conventional trickle-bed reactor was in the range of 3.57×10^{-7} to $2.85 \times 10^{-7} \text{ mol}/(\text{cm}^3 \text{ mass \% Pd}^{-1} \text{ s}^{-1})$. The highest rate with metal foam was in the range of 120×10^{-7} to $110 \times 10^{-7} \text{ mol}/(\text{cm}^3 \text{ mass \% Pd}^{-1} \text{ s}^{-1})$. The enhancement in the rate with rotating beds was 33 to 39 times higher than

that of the conventional trickle bed reactors. Because the reaction rate would be greater with larger beds and higher rpm, the volume reduction of similar magnitude or more could be achieved. The intrinsic kinetic rate for this reaction is $596 \times 10^{-7} \text{ mol}/(\text{cm}^3 \text{ s}^{-1})$ at 50°C with 1.0 mass % Pd and 1 atm hydrogen pressure. This rate is about five times the highest rate observed. At these conditions the intrinsic kinetic regime is being approached, and thus further intensification may not be possible.

In the present study, the reaction rate was controlled by the liquid-side mass transfer. For the case in which the reaction rate is controlled by the gas-side transport, the novel rotor design proposed by Rao et al.⁵ can be used to achieve process intensification. If the rate is controlled by the reaction kinetics, it is unlikely that the process intensification can be achieved with a rotating trickle-bed reactor.

Conclusions

The hydrogenation rates of α -methyl styrene were measured in a rotating trickle bed using alumina particles and metal foam as catalyst supports. The rates with metal foam were nearly twice as great as those with spherical particles of the same specific area. Further, the metal foam with smaller specific area gave higher rates. These trends in the reaction rates were attributed to the nature of liquid flow over the packing. It was shown that a volume reduction by a factor of 30 to 40 is attainable with moderate centrifugal acceleration of 500 g (4900 m/s^2), if the liquid-side mass transfer is the controlling step.

Acknowledgments

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