j = $(R'/1 + R')\ (1 - \Sigma g x_1) - 1.0$
k = $(1 + gR')/(1 + R')$
K = y/x = distribution coefficient
L = heavy or extract phase rate, moles, weight, or volume per unit time
M = number of theoretical stages below the feed stage $M + 1$
n = subscript referring to any stage which may take on any value from 1.0 to N
N = total number of theoretical stages including partial reboiler if any but excluding partial condenser if any
R = external reflux ratio at top or raffinate end of column, amount of reflux/amount of product
R' = external reflux ratio at bottom or extract end of column, amount of reflux/amount of product
S = fresh solvent rate, moles, weight, or volume per unit time
S_E = solvent rich material recovered in the extract solvent recovery equipment, moles, weight, or volume per unit time
S_n = $K_n V_n / L_{n+1}$ = component stripping factor where the subscript n can take on any stage number from 1.0 to N
V = light or raffinate phase rate, moles, weight, or volume per unit time
x = concentration in heavy or extract phase, units consistent with units on rates
y = concentration in light or raffinate phase, units consistent with units on rates

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Manuscript received January 2, 1959; revision received December 17, 1959; paper accepted December 28, 1959.

Effect of Uniformity of Fluidization On Catalytic Cracking of Cumene

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The effect of uniformity of fluidization on the catalytic cracking of cumene, with a silica-alumina catalyst, was studied in a 3-in.–diameter reactor operated at 800°F. Superficial gas velocities were varied from 0.06 to 0.24 ft./sec. for bed heights of 1.5, 3, 5, and 8 in. The percentage conversion in a fluidized bed was found to be lower than in a fixed bed and to be dependent on the type of catalyst support used. A porous plate support was up to 30% more efficient than a screen support, which is attributed to its better gas-distribution characteristics and to its ability to introduce the discontinuous phase as smaller gas bubbles. Indications are that the first few inches of bed are very effective in bringing in contact the continuous and discontinuous phase, and therefore most of the conversion due to the interaction of the two phases takes place here. This can be explained in terms of the rapid growth of gas bubbles as they proceed through the bed.

The nature of the fluid and solid flow in a fluidized bed has been investigated in many studies (*2a, 4, 5, 6, 25, 27*) and the fluidization process has been described (*8* to *13* and *18*). The general solid pattern has been established as being up the center of the column and down the sides (*4, 5, 16*) with the downward-flowing solid particles creating some back mixing of the gas along the sides (*4, 5*). The importance that the homogeneity of dispersion of fluid and particles can have on the properties of a fluidized bed has been realized, and ingenious methods have been proposed to measure bed uniformity (*1, 3, 17, 24*), but unfortunately studies have been mainly of a qualitative nature. Gilliland and coworkers (*6*) in kinetic studies of a gas-phase homogeneous reaction concluded that plug flow is a good assumption for kinetic studies and that the lowering of conversion in a fluidized bed was due mainly to gas bypassing through the gas-solid phase of the bed.

Shen and Johnstone (*23*) studied the catalytic decomposition of nitrous oxide, and Mathis and Watson (*15*) studied catalytic cumene dealkylation in both fixed and fluidized beds under isothermal bed conditions. The conversion obtained under fluidized-bed conditions was always less than for the fixed bed and was attributed to ineffective contact caused by bypassing gas. Both Shen and Johnstone (*23*) and Mathis and Watson (*15*) have proposed mathematical models to aid in the understanding of the behavior of the bed. To make mathematics manageable, quite a few assumptions have to be made and a high degree of error can be expected from such models, but as yet these two studies have not yielded enough data to make any conclusions of the general applicability of the models.

The aim of the present study was to investigate the effect of fluidization on the catalytic cracking of cumene. The cumene-cracking reaction was chosen because of its desirable cracking characteristics and because the reaction mechanism has been studied extensively (*2, 20, 21, 26*).

Special emphasis in this investigation was placed on the effect of uniformity on the cracking reaction, since it was expected that this variable would have a significant effect on the level of conversion attained in a fluidized bed.

MATERIALS AND APPARATUS

The cumene used in the kinetic study was of pure-grade quality that was purified by passing through silica gel to remove cumene hydroperoxide and other polar impurities. After purification it analyzed

Cumene	98.68%
Ethylbenzene	1.30%
Cumene hydroperoxide	0.02%

Albert Gomezplata is at present with the University of Maryland, College Park, Maryland.

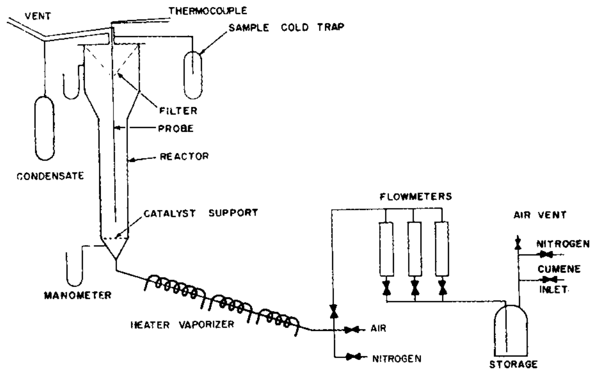

Fig. 1. Diagram of equipment.

The silica-alumina catalyst, which analyzed 13% aluminum oxide, had a surface area by B.E.T. analysis of 490 m. sq./g. The 100- to 200-mesh fraction was used after fines had been removed by fluidizing the catalyst for a period of 8 hr. at a moderate gas rate.

A flow diagram of the equipment is presented in Figure 1. Briefly, it consisted of a cumene storage tank, flow meters to measure the cumene feed, a 6-ft. heater to vaporize and preheat the cumene, and a 3-in.–diameter reactor to contact the cumene and catalyst. The reactor had a catalyst disengaging section along with a vent system designed to keep the reactor at atmospheric pressure and had provision for either a porous plate or a screen as a catalyst support. Compressed nitrogen was used to pump cumene through the system. Stainless steel was used throughout in the heater, reactor, and top assembly.

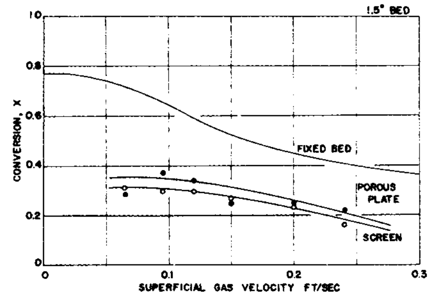

Fig. 2. Conversion vs. superficial gas velocity.

METHODS OF PROCEDURE

Before a run was started, the desired amount of catalyst was placed in the reactor and the equipment heated while air was passed through the reactor. When the apparatus reached operating temperatures, which took several hours, a 10-min. nitrogen purge was started, after which the cumene feed was applied. With practice the bed temperature could be adjusted to 800°F. within 20 min. and kept to ±5° of that temperature. A small flow of nitrogen continually flushed the sample line until a sample was taken. The normal sampling time was 10 min. After the sample had been taken, the cumene feed was stopped and the residual cumene in the boiler was allowed to evaporate. A 15-min. nitrogen purge followed, and then air was put through the reactor which caused the catalyst temperature to increase. When the temperature reached 1,000°F., the variac controlling the bed temperature was turned off until this temperature began to fall, indicating the end of the carbon burning cycle. The sample collected was transferred to a small vial and analyzed. Gas chromatography was found to be a quick and accurate method of analysis for both impurities and the fraction converted.

RESULTS

Preliminary Study

When the catalyst was fluidized with air in a plastic column, a marked difference in the uniformity of a fluidized bed was observed depending on whether a porous plate or a screen was used as a catalyst support. With a porous plate as a catalyst support, a very definite bed expansion, up to 20%, was observed even at low gas velocities. At these low gas velocities channeling does occur but usually at several places in the bed. At higher gas velocities gas passes through the dense phase in small gas bubbles that seem to be evenly distributed throughout the bed. The small bubbles at the walls combine rapidly and grow larger as they progress through the bed, and it can be inferred that a similar action takes place within the bed. From visual observations it is questionable whether the solid circulation pattern is up the center and down the sides when a porous plate is used.

With a fine-mesh screen used as the catalyst support a 60-deg. cone distributor or a 3-in. Berl saddle calming section was found necessary to obtain a symmetrical bed with gas flow through the center. Even then there was a marked difference in fluidization with a screen. At low gas velocities, channeling can be observed at only one point in the bed, with little or no bed expansion, and even at higher gas velocities, only a small percentage of bed expansion is obtained. The greater part of the gas flow was through the center of the bed, and a larger bubble size compared with that observed with a porous plate was noticed. A greater solid turbulence was evident by the considerable ejection of particles that made observation of the upper boundary of the bed impossible after a few seconds of gas flow.

The difference that supports can make was also noticed by Grohse (*17*) while determining instantaneous density profiles of fluidized beds with X-ray absorption techniques. His results indicate that a porous-plate support produces a bed that is longitudinally uniform in density and that a bed with a screen support has a greater density at the bottom, levels off in the middle, and has a lower density at the top.

It is evident that these differences in the contact of the disperse and dense

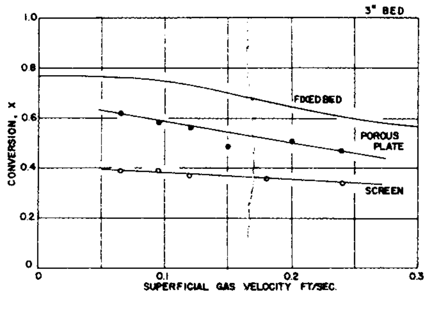

Fig. 3. Conversion vs. superficial gas velocity.

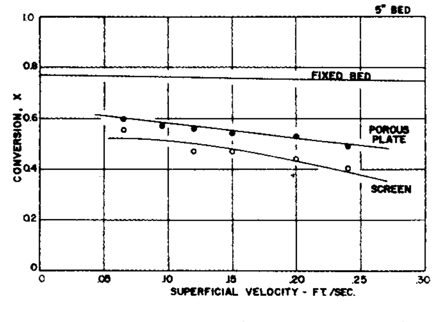

Fig. 4. Superficial gas velocity vs. conversion.

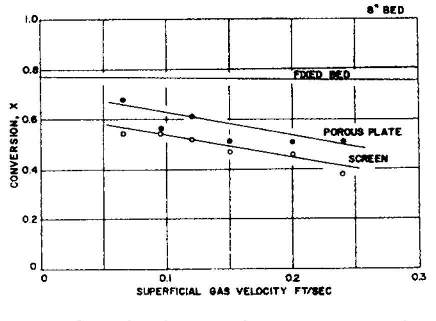

Fig. 5. Superficial gas velocity vs. conversion.

phases may lead to different reaction rates when the solid is used as a catalyst for a reaction.

Fixed-Bed Study

It was attempted to determine fixed-bed data with the same equipment as used in the fluidized-bed study; however at the low gas velocities required to reproduce the same *W/F* ratios used in the fluidized study, high thermal conversion was obtained (as high as 12%) and side reaction overshadowed the cracking of cumene. But the cracking of cumene by silica alumina catalyst has been studied extensively (*2, 14, 19, 20, 26*), enabling the conversion expected from a fixed bed to be calculated.

Kinetic studies have shown that the cracking of cumene is controlled by a single-site surface reaction (*2, 20*) and that the steady state reaction per unit of catalyst surface can be expressed by

$$\frac{dn}{dt} = k_3 B_o \frac{Ps - 1/K_E P_m P_n}{Ps + GK_m P_m + G\Sigma K_{Ii} P_{Ii} + G} \qquad (1)$$

The adsorption-equilibrium constants for cumene products and common inhibitors have been tabulated (*14*). Of special importance is cumene hydroperoxide, which is a very strong inhibitor that accumulates on storage.

The rate equation can be integrated to give an expression for the total conversion to be expected from an integral reactor (fixed-bed reactor).

$$2\frac{W}{F} k_3 B_o M^3 = (M^2E + C - MJ)\ln(1-x')$$
$$+ (M^2E + C + MJ)\ln(1-x') - 2Cx' \qquad (2)$$

where

$$E = 1 + GK_I P_I + G/P$$
$$J = 2G + GK_m + GK_I P_I$$
$$C = G + GK_m - 1$$
$$M^2 = (K_E + 1)/K_E$$

Equation (2) can be used to predict the conversion expected in a fixed bed when quantitative data of the impurities of the cumene feed are available, provided that diffusion transport effects are not present and that the activity of the catalyst is assumed proportional to the B.E.T. surface area. The possibility of diffusion-transport effects was eliminated by the very fine-mesh catalyst used. The predicted conversion seems to agree with the results of the fluidized-bed study. Conversions close to equilibrium were predicted for all bed heights except the 1½-in. bed. It was at this bed height that a noticeable decrease in conversion took place in the fluidized-bed data.

The cumene cracking reaction results in a volume change and consequently an increase in gas superficial velocity with bed height. In this paper results are based on the inlet superficial velocity for simplicity.

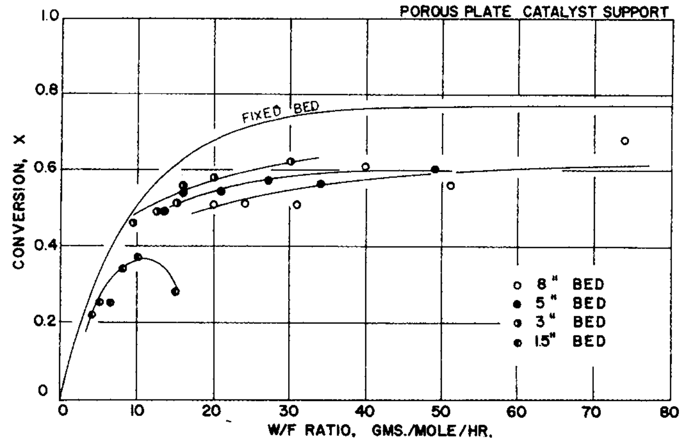

Fig. 6. Conversion vs. *W/F* ratio.

Fluidized-Bed Study

Thermal cracking was found to be negligible at the space velocities studied, with only 0.6% observed at low gas rates. The purified cumene cracked to benzene and propylene with very few side reaction products (compared with a 1.3% ethylbenzene impurity in the feed). Catalyst fouling was negligible over several runs made for 80 min. Reproducibility of the data was generally within 2% conversion, and frequent check runs were made. The catalyst was changed after each series of runs, although no indication was found of catalyst deactivation with time.

The results of runs made in the reactor with both a screen and porous plate, in which bed height and superficial velocity were varied, are shown in Figures 2 to 5, together with the expected conversion in a fixed bed.

The experimental results show that for the cracking of cumene over the range of conditions studied a lower conversion is obtained in a fluidized bed than in a fixed bed of similar size. This is in agreement with the results of Shen and Johnstone (*23*) and Mathis and Watson (*15*), who also obtained lower conversion in their fluidized beds. The lower conversion can be attributed to the gas that bypasses the bed in the form of bubbles or channels. The bypassing gas does contribute to the conversion in the bed, since the over-all conversion is always greater than it is for the solid-gas phase alone. It is reasonable to assume that this extra conversion is obtained from both the transfer of reactants and products between the continuous and the discontinuous phase and any reaction taking place in the discontinuous phase from catalyst entrained in that phase. It then follows that the intimacy of contact between the two phases and the distribution of the gas phase will have an effect on the conversion obtained, and the experimental results verify this conclusion. Over the range of conditions studied, up to 30% difference in conversion was noticed between a porous plate and a screen support, which have been observed to give a marked difference in bed uniformity in the plastic reactor. With a porous plate, which gave a smoother fluidiza-

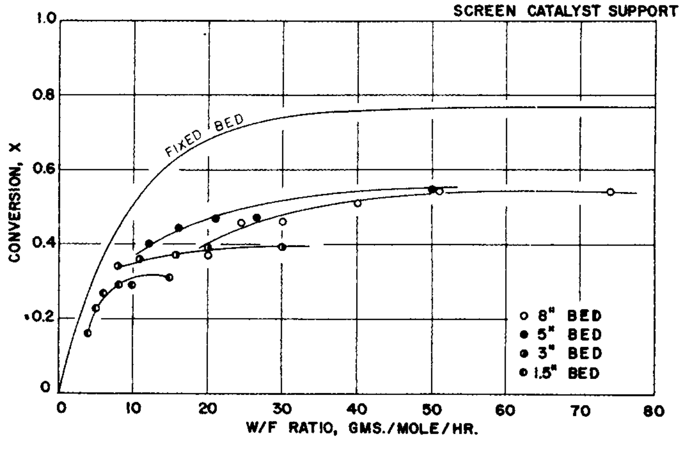

Fig. 7. Conversion vs. *W/F* ratio.

tion than a screen, larger conversions and greater bed efficiencies were obtained. These can be explained in terms of the smaller gas bubbles of the discontinuous phase, formed at the bottom of the bed, that expose a large surface for interaction with the continuous phase and which are distributed evenly over the total cross section of the bed. On the other hand, with a screen support larger bubbles are present in the bed, with most of the bubbling action taking place in the center; a lower conversion can thus be expected with a screen. In both cases the bubbles are observed to combine rapidly as they pass up the bed, and the subsequent influence that they have on conversion emphasizes the need for either standardizing equipment for experimental studies or finding a method to characterize the uniformity of a bed.

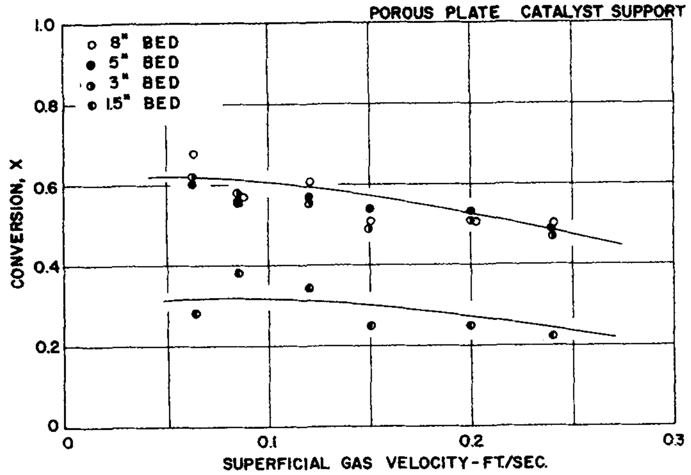

Fig. 8. Conversion vs. superficial gas velocity.

Mathis and Watson (*15*) proposed to characterize bed uniformity by the height-to-diameter ratio (L/D) of the bed; however this conclusion is not applicable to all beds, as is evident from Figures 6 and 7, where the same (L/D) ratio does not fix the level of conversion but is dependent on whether a screen or a porous plate is used as a catalyst support. However in view of the apparent independence of gas distribution on the inlet conditions when a porous plate is used, it may well be that in this case the (L/D) ratio would characterize the uniformity of the bed. An attempt was made to use the minute pressure fluctuations of the bed to predict bed uniformity, but the attempt was unsuccessful owing to the difficulty of isolating the pressure fluctuations in the bed from those introduced by other parts of the equipment.

The data of Mathis and Watson were obtained at a higher cracking temperature than those of the present study and cover a wider range of superficial velocities, although with relatively few points. It is difficult to compare results, even though the same reaction is involved, because of the dependence of reaction rate on the purity of the cumene used. A similar decrease in conversion with increased superficial gas velocity in the fluidized bed can be noticed, although an optimum velocity at which the fluidized bed conversion approaches the conversion in a fixed bed was not noticed. Shen and Johnstone (*25*) in their kinetic studies of catalytic decomposition of nitrous oxide did not notice an optimum velocity effect either. This velocity pinch effect is dependent on the curvature of the fixed-bed data, and in view of the extreme difficulty found in taking experimental fixed-bed data and the fact that an unusual high depression of conversion is evident in Mathis and Watson's fixed-bed results, it seems likely that their data are in error.

When all the runs made with a porous plate are grouped together as shown in Figure 8, very interesting results are observed. For beds of 3, 5, and 8 in., approximately the same conversion is obtained at similar superficial gas velocities, and the conversion vs. velocity curves follow similar trends. A higher conversion is obtained at low gas velocities with a leveling off at higher velocities. Similar trends are observed with a screen as shown on Figure 9, with the exception that only the 5- and 8-in. beds are similar. It should be noted that the conversion values approached in the case of a porous plate are higher than those approached in the bed with a screen and in both cases less than those predicted for a fixed bed. In this study conversion in the continuous phase closely approaches the thermodynamic equilibrium of 0.77 (*22*) conversion for all bed heights above 3 in. With this in mind, one notes that in the case of the porous plate the results indicate that essentially all the interaction between phases takes place below the first 3 in. of the bed. This is understandable in terms of the small bubbles present at the bottom and their rapid growth as they proceed up the column. With a screen, on the other hand, gas bubbles are also present at the bottom of the bed, but they are bigger and not evenly distributed, since most of the gas flow takes place up the center of the bed. A very high degree of solid turbulence results. In a bed with a screen, because of the inherently larger bubble size, lower conversions are obtained than with a porous plate. An upper limit in conversion is obtained, but a higher bed is needed to reach the maximum interaction conditions. This must be attributed to the larger percentage of catalyst entrained in the gas phase by the violent solid turbulence of the bed. Other investigators have not been able to notice these effects because their studies were not conducted under conditions where the

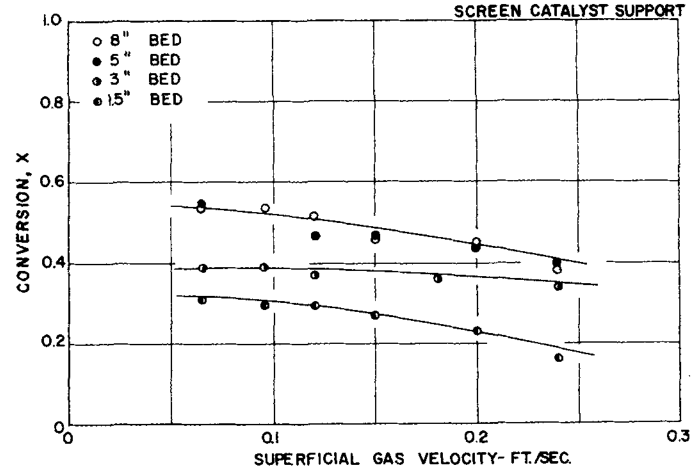

Fig. 9. Conversion vs. superficial gas velocity.

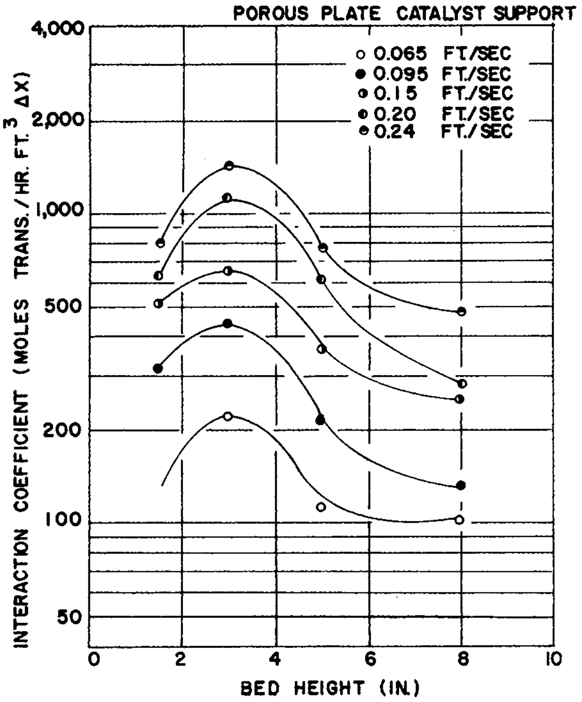

Fig. 10. Interaction coefficient vs. bed height.

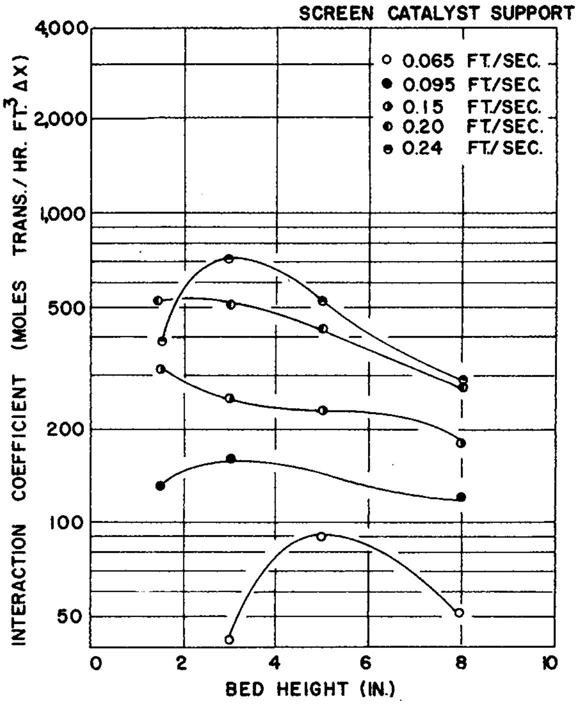

Fig. 11. Interaction coefficient vs. bed height.

conversion expected of a similar fixed bed became essentially independent of the height of the bed.

The mathematical models of fluidized beds that have so far been proposed are all based on Toomey and Johnstone's (*25*) theory of a discrete fluidized gas-solid mixture. The phases are assumed to constitute parallel reactors, and a mass transfer coefficient is used to account for the interaction between the phases, calculated from the difference in over-all conversion from that expected for just the gas-solid phase. The coefficient is evaluated per unit volume of bed and is assumed constant for the entire bed. Shen and Johnstone proposed two models. In one, progressive flow was assumed for both phases; in the other, progressive flow was assumed for the gas phase with complete mixing of the gas-solid phase. Mathis and Watson considered just the case of progressive flow in both phases but assumed that there was catalyst entrainment in the gas phase and introduced the back reaction in a pseudo first-order equation used to describe the cracking rate. In the present study a new simpler model, using the same approach as Mathis and Watson (*15*), was derived. This model except for the method of development and basic units is similar to the one proposed by Shen and Johnstone (*23*) for progressive flow in both phases. The refinements introduced by Mathis and Watson were not considered because of the difficulty of evaluating the extent of catalyst entrainment in the gas phase and having to account for the back reaction scheme at the high level of conversions attained with cumene.

Fluidized Reactor Model

The bed is assumed to be composed of two parallel reactors, one containing the continuous phase and the other the discontinuous phase. The discontinuous phase consists of all the bypassing gas; the continuous phase consists of the solid-gas mixture under conditions similar to those present at incipient fluidization. A pseudo mass transfer coefficient per unit bed volume is introduced to account for over-all conversion above that predicted for the dense-phase reactor. Both reactors are assumed to be at the same temperature and pressure conditions, and it is further assumed that progressive gas flow exists in both phases and that the reaction will follow a first-order kinetic scheme.

A material balance around the continuous-phase reaction element dZ results in

$$\frac{dx_A}{dZ} + \left[\frac{(K_{AB}A) + \left(\frac{kW}{L}\right)}{F_A}\right] x_A - \frac{(K_{AB}A)}{F_A} x_B = \frac{kW}{LF_A} \qquad (3)$$

A similar material balance around the discontinuous phase yields

$$\frac{dx_B}{dZ} = \frac{(K_{AB}A)}{F_B} (x_A - x_B) \qquad (4)$$

Equations (3) and (4) are simple, ordinary differential equations which can be solved by assuming progressive gas flow, constant temperature and pressure, even distribution of catalyst in phase A, and a constant interaction coefficient. The boundary conditions are $x_A = x_B = 0$ at $Z = 0$. The solutions are

$$x_A = \left[\frac{F_B g_1 + 1}{(K_{AB}A)}\right] R_1 e^{g_1 Z} + \left[\frac{F_B g_2 + 1}{(K_{AB}A)}\right] R_2 e^{g_2 Z} + 1 \qquad (5)$$

$$x_B = R_1 e^{g_1 Z} + R_2 e^{g_2 Z} + 1 \qquad (6)$$

where

$$g_1 = \frac{1}{2F_A F_B} [-b + \sqrt{b^2 - 4F_A F_B d}]$$

$$g_2 = \frac{1}{2F_A F_B} [-b - \sqrt{b^2 - 4F_A F_B d}]$$

$$b = (K_{AB}A)(F_A + F_B) + F_B \frac{kW}{L}$$

$$d = (K_{AB}A) \frac{kW}{L}$$

$$R_1 = \frac{g_1}{g_2 - g_1}$$

$$R_2 = \frac{g_2}{g_1 + g_2}$$

The over-all conversion is represented by

$$x = \frac{F_A x_A + F_B x_B}{F_A + F_B} \qquad (7)$$

Values for x_A and x_B can be substituted in the above equation and then simplified to

$$x = Q_1 e^{g_1 z} + Q_2 e^{g_1 z} + 1 \qquad (8)$$

where

$$Q_1 = R_1 \left[\frac{F_A F_B g_1 + (K_{AB}A)(F_A + F_B)}{(K_{AB}A)(F_A + F_B)} \right]$$

$$Q_2 = R_2 \left[\frac{F_A F_B g_2 + (K_{AB}A)(F_A + F_B)}{(K_{AB}A)(F_A + F_B)} \right]$$

To evaluate the interaction coefficient from experimental results, a trial-and-error solution is necessary. A value of $K_{AB}A$ is assumed, and the value of over-all conversion calculated from Equations (5) to (8). If it agrees with the experimental value, then the correct value of K_{AB} has been assumed. The interaction coefficients were calculated by a trial-and-error solution with an IBM 650 digital computer. The results are shown in Figures 10 and 11 and are plotted vs. bed height with superficial gas velocity as a parameter. In the case of a bed with a porous plate the interaction coefficient increases rapidly with bed height, goes through a maximum at approximately 3 in., and then decreases almost proportionally to bed height. With the screen the results are erratic in that some curves have an inflection at 3 in., while others just decrease with bed height. However two facts are evident: with a screen the decrease of interaction coefficient with bed height is more gradual than with a porous plate, and the coefficients are lower for the former.

The plots of interaction coefficient vs. bed height support the concept that practically all interaction between phases takes place in the first few inches of bed. The coefficients obtained with a porous plate increase quickly to a maximum and then decreases with bed height. With a screen the decrease is more gradual, and at some gas velocities no initial increase is evident. Since the coefficient is evaluated per unit of reactor volume, a coefficient decrease proportional to the bed height signifies that no more effective transfer is taking place between the phases; such a trend is noticed after the first few inches of bed height. Shen and Johnstone also found that their coefficients varied proportionally to bed height. Transfer coefficients can be transformed dimensionally to interaction coefficients by dividing the absolute pressure by the product of the gas constant, absolute temperature, and transfer coefficient. When one converts their data, interaction coefficients of 280 to 2,900 are obtained which are in the same range as those of the present study even though both systems are different. The coefficients found by Mathis and Watson on the other hand are much higher than those in the present study, and this again can be attributed to the high depression of conversion obtained in their fixed-bed data.

The greater interaction between phases at low bed heights indicates that the interaction coefficient used in the parallel reactor model is not constant over the height of the bed but is definitely higher in the lower section. These differences should be known, and studies should be started to evaluate these interaction coefficients as a function of bed height, since the parallel reactor model seems to be a practical approach to the design of a fluidized reactor from fixed-bed data.

ACKNOWLEDGMENT

The authors are indebted to Phillips Petroleum Company for providing the fellowship that made this study possible. Appreciation is extended to the Hercules Powder Company for supplying the cumene and to the American Cyanamid Company for the catalyst.

NOTATION

A = cross-sectional area, sq. ft., catalyst site
B_o = concentration of active sites per unit area, moles/m. square
D = reactor diameter, ft.
F = feed rate, moles/hr.
G = constant related to chemisorption of cumene
J = inhibitor molecule
K_E = equilibrium constant for reaction cumene → benzene + propylene
K_{AB} = interaction coefficient, moles /cu. ft./unit conversion difference
k = reaction-rate constant, g. moles/hr./g. catalyst
L = total bed height, ft.
P = pressure, atm.
r = reaction rate, g. moles reacted/hr./g. catalyst
x = conversion as mole fraction

Subscripts

A = continuous phase
B = discontinuous phase
I_i = inhibitors
m = benzene
n = propylene
s = cumene

Superscripts

= ratio of fraction converted to fraction converted at thermodynamic equilibrium
— = over-all value

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Manuscript received May 27, 1959; revision received December 4, 1959; paper accepted December 9, 1959.