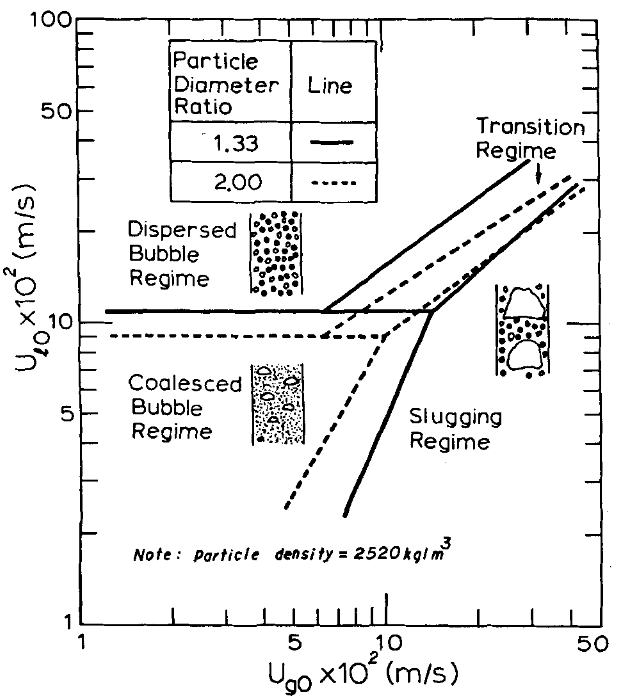

Figure 4. Flow regime map for binary mixtures in fluidized states with particle diameter ratios of 1.33 and 2.0.

bubble regime for the particle diameter ratio of 1.33 is higher than that for the particle diameter ratio of 2. Likewise, for a given liquid velocity, the gas velocity required to reach the boundary between the coalesced bubble regime and slugging regime for the particle diameter ratio of 1.33 is higher than that for the particle diameter ratio of 2.

Comparing Figure 4 with Figure 3, it is important to note that there is a strong correlation existing between the flow regime map and the solids mixing map for the binary mixtures considered. Evidently, the complete mixing state occurs largely in the coalesced bubble regime and the slugging regime and slightly in the transition regime. The partial intermixing state occurs largely in the dispersed bubble regime and the transition regime and slightly in the coalesced bubble regime. The complete segregation states occur solely in the dispersed bubble regime.

ACKNOWLEDGMENT

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NOTATION

W_1 = weight of particle 1, kg
W_2 = weight of particle 2, kg
U_{lo} = superficial liquid velocity, m/s
U_{go} = superficial gas velocity, m/s

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Effect of Distributor to Bed Resistance Ratio on Uniformity of Fluidization

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The effect of the distributor-to-bed resistance ratio on the uniformity of fluidization is analyzed. A new model for the cause of channeling through a fluidized bed is proposed, and a criterion for uniform fluidization, giving rise to the condition of full fluidization, is established.

The performance of a fluidized bed depends largely on the satisfactory design of its distributor. Apart from supporting the weight of the bed during downtime and preventing the back flow of particles into the plenum section, the major function of the distributor is to distribute the fluidizing medium across the base of the bed so that the fluidized condition is maintained over the entire cross section. It has been known that to maintain a stable operation of the bed, the pressure drop of a certain magnitude needs to be established through the distributor.

Numerous papers have been published on the determination of the required distributor pressure drop or on its relation to the bed pressure drop (Hiby, 1964; Siegel, 1976; Mori and Moriyama, 1978; Sathiyamoorthy and Rao, 1979; Qureshi and Creasy, 1979). Among approaches presented in these papers, that based on Siegel's model is considered to be the simplest for deriving a stability criterion for a fluidized bed (Qureshi and Creasy, 1979); it has been used to calculate the relative pressure drop through a porous or perforated distributor for stable fluidization (Qureshi and Creasy, 1979; Sirotkin, 1979). The objectives of this work are to propose a new or improved model for the cause of channeling in a fluidized bed through critical analysis of Siegel's model and to establish a new criterion for uniform fluidization, giving rise to the condition of full fluidization.

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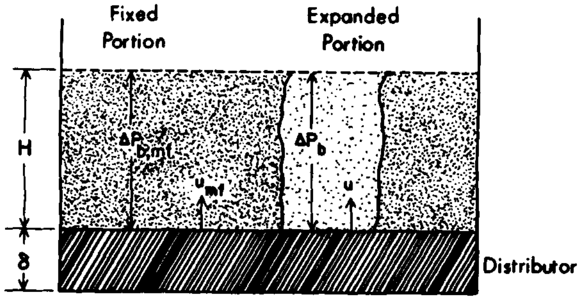

Figure 1. Local expansion of the bed on the porous distributor.

CHANNELING THROUGH A FLUIDIZED BED

When a perturbation causes a local increase in the velocity of fluidizing medium through an originally uniform bed of nonsticky particles at minimum fluidization, this velocity will exceed the minimum fluidization velocity locally, Figure 1. This, in turn, will cause the bed to expand locally, thereby altering the pressure drop through the portion of the bed; this will also change the local pressure drop through the distributor plate. Siegel (1976) has considered that channeling tends to occur if the overall local pressure drop decreases with an increase in the velocity of the fluidizing medium. Mathematically, it can be expressed as:

$$\frac{d(\Delta P_d + \Delta P_b)}{du} < 0 \tag{1}$$

Siegel (1976) has used this model in connection with a porous distributor to show that channeling will occur through a gas-fluidized bed of small particles ($Re_t < 0.2$) provided that $R < 0.14$; here, R is the ratio of the distributor pressure drop to the bed pressure drop at minimum fluidization. This value of R, 0.14, is regarded by Siegel to be a stability criterion or fluidization. Qureshi and Creasy (1979) have used Siegel's model for a perforated distributor to show that channeling will take place if $R < 0.072$. However, by means of a global analysis, it will be shown that this is not the case.

The pressure drop through a porous distributor can be calculated as

$$\Delta P_d = u r_d \tag{2}$$

where r_d is the resistance of the distributor. At minimum fluidization, it yields

$$\Delta P_{d,mf} = u_{mf} r_d = R\ \Delta P_{b,mf}$$

and, therefore, the resistance of distributor, r_d, is defined as

$$r_d = R\left(\frac{\Delta P_{b,mf}}{u_{mf}}\right) \tag{3}$$

From Eqs. 2 and 3, we obtain

$$\frac{\Delta P_d}{\Delta P_{b,mf}} = R\left(\frac{u}{u_{mf}}\right) \tag{4}$$

The pressure drop through an expanded or fully fluidized bed is defined as

$$\Delta P_b = H\rho_s(1-\epsilon) \tag{5}$$

The local bed expansion would not be expected to affect appreciably the overall bed height; hence, the bed height is assumed to stay constant. At minimum fluidization,

$$\Delta P_{b,mf} = H\rho_s(1-\epsilon_{mf}) \tag{6}$$

From Eqs. 5 and 6, we obtain

$$\frac{\Delta P_b}{\Delta P_{b,mf}} = \frac{1-\epsilon}{1-\epsilon_{mf}} \tag{7}$$

The bed expansion can be obtained from an available correlation as (Richardson and Zaki, 1954)

$$\frac{u}{u_t} = \epsilon^n \tag{8}$$

For small particles ($Re_t < 0.2$), we have

$$\frac{u_t}{u_{mf}} \simeq 78 \text{ and } n \simeq 4.65$$

From Eqs. 4 and 7, the relative overall local pressure drop is obtained as

$$\frac{\Delta P_t}{\Delta P_{b,mf}} = \frac{\Delta P_d}{\Delta P_{b,mf}} + \frac{\Delta P_b}{\Delta P_{b,mf}} = R\frac{u}{u_{mf}} + \frac{1-\epsilon}{1-\epsilon_{mf}} \tag{9}$$

By assuming that $R = 0.1 < 0.14$ for illustration, the relative pressure drop through the distributor, that through the bed and the relative overall local pressure drop have been calculated from Eqs. 4, 7 and 9, respectively. The results are shown in Table 1 and Figures 2 and 3. For a constant $\Delta P_{b,mf}$, it can be seen that at first the overall local pressure drop, ΔP_t, decreases with an increase in the velocity as shown in Figure 3, because the decrease in the pressure drop through the bed, ΔP_b, due to its expansion is greater than the increase in the pressure drop through the distributor, ΔP_d, due to the velocity increase, Table 1 and Figure 2. Furthermore, the overall local pressure drop, ΔP_t, will be smaller than $\Delta P_{t,mf}$, and thus, the local velocity u, will increase. The decrease in ΔP_t will eventually cease at a certain velocity u_c, and ΔP_t will attain its minimum, Figure 3. Beyond this velocity, the decrease in the pressure drop through the bed, ΔP_b, will become smaller than the increase in the pressure drop through the distributor, ΔP_d, as illustrated in Figure 2. Thus the overall local pressure drop, ΔP_t, will increase with an increase in the velocity, and therefore (Figure 3)

$$\frac{d(\Delta P_t)}{du} > 0 \text{ for } u > u_c$$

However, the overall local pressure drop, ΔP_t, will still be smaller than $\Delta P_{t,mf}$ until the velocity reaches u_f, as indicated in Figure 3; therefore, the local velocity, u, will continue to increase. This is contrary to Siegel's model, because it stipulates that only under the condition of

TABLE 1. RELATIVE DISTRIBUTOR, BED AND OVERALL LOCAL PRESSURE DROPS FOR THE BED WITH A POROUS DISTRIBUTOR

$\frac{u}{u_{mf}}$	1.0	1.1	1.2	1.3	1.4	1.5	1.6	1.7	1.8	1.9	2.0	2.1	2.2	2.3	2.4	2.5
$\frac{\Delta P_d}{\Delta P_{b,mf}}$	0.10	0.11	0.12	0.13	0.14	0.15	0.16	0.17	0.18	0.19	0.20	0.21	0.22	0.23	0.24	0.25
$\frac{\Delta P_b}{\Delta P_{b,mf}}$	1.0	0.9867	0.9742	0.9626	0.9517	0.9413	0.9315	0.9221	0.9132	0.9046	0.8964	0.8885	0.8809	0.8736	0.8665	0.8597
$\frac{\Delta P_t}{\Delta P_{b,mf}}$	1.1	1.0967	1.0942	1.0926	1.0917	1.0913	1.0915	1.0921	1.0932	1.0946	1.0964	1.0985	1.1009	1.1036	1.1065	1.1097

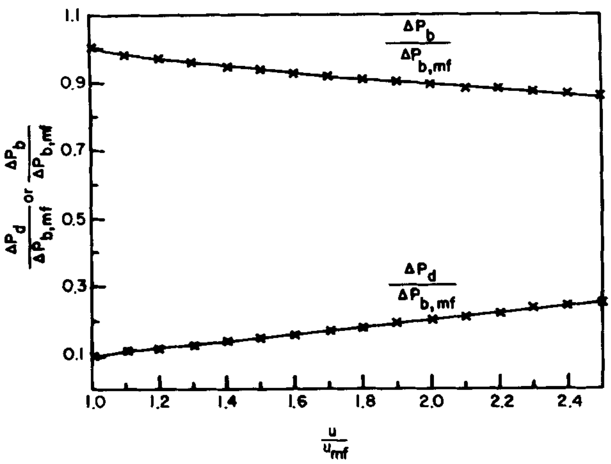

Figure 2. Relative pressure drops through the bed and the porous distributor.

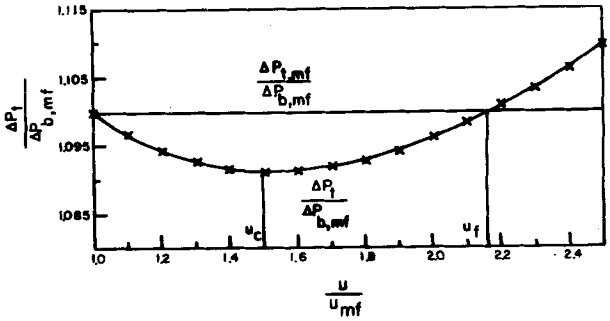

Figure 3. Relative overall pressure drop, including those through the distributor and the bed.

$$\frac{d(\Delta P_t)}{du} < 0$$

will the local velocity increase.

At the velocity, u_f, the overall local pressure drop, ΔP_t, will be equal to $\Delta P_{t,mf}$; thereafter, it will exceed $\Delta P_{t,mf}$, thus preventing the local velocity to increase further, and consequently, the channeling will not occur. This is also contrary to Siegel's model, because it stipulates that channeling will occur in the bed at $R = 0.1 < 0.14$. In practice, we also see that, in spite of Siegel's model, some beds can operate successfully with the values of R as low as or lower than 0.1 without channeling. The velocity, u_f, corresponds to the full fluidization velocity, because after this velocity no portion of the bed can remain in the fixed state.

The analysis given in the preceding paragraphs indicates that even when inequality (Eq. 1) is valid at minimum fluidization, the local velocity of the fluidizing medium cannot continue to increase without bound to form a channel. The condition for channeling through a fluidized bed of nonsticky particles should be defined in the range of velocity $u_{mf} < u \leq u_t$; within this range, the following condition should be satisfied for a channel to form in the bed of nonsticky particles.

$$ur_d + H(1-\epsilon)\rho_s < u_{mf}r_{d,mf} + H(1-\epsilon_{mf})\rho_s \tag{10}$$

In this case, if the local velocity, u, through a uniform bed at minimum fluidization is perturbed upward, it will continue to increase until u_t is reached. Before the local velocity reaches u_t overall local pressure drop, ΔP_t, is smaller than $\Delta P_{t,mf}$ estimated by Eq. 10, and when it is equal to or exceeds u_t, a channel will be established.

According to the present model as expressed by inequality (Eq. 10), the criterion for the formation of a channel in the bed with a porous distributor, for which r_d is essentially constant in the laminar flow region and consequently $r_{d,mf} = r_d$, is

$$r_{d,mf} < \frac{H\rho_s(\epsilon - \epsilon_{mf})}{u - u_{mf}} \tag{11}$$

Because

$$H\rho_s(1-\epsilon_{mf}) = u_{mf}r_b,$$

inequality (Eq. 11) can be rewritten as

$$\frac{r_{d,mf}}{r_b} < \frac{u_{mf}}{u - u_{mf}}\frac{\epsilon - \epsilon_{mf}}{1 - \epsilon_{mf}} \tag{12}$$

Substituting Eq. 8 into Eq. 12 and rearranging, we obtain

$$\frac{r_{d,mf}}{r_b} < \frac{\left(\dfrac{u}{u_{mf}}\right)^{1/n} - 1}{\left(\dfrac{u}{u_{mf}}\right) - 1}\cdot\frac{1}{\left(\dfrac{u_t}{u_{mf}}\right)^{1/n} - 1} \tag{13}$$

Obviously,

$$\frac{\left(\dfrac{u}{u_{mf}}\right)^{1/n} - 1}{\left(\dfrac{u}{u_{mf}}\right) - 1}$$

decreases with an increase in u, and, therefore, the minimum value of $r_{d,mf}/r_b$ in the range $u_{mf} < u \leq u_t$ is at u_t. Thus, if

$$\frac{r_{d,mf}}{r_b} < \frac{1}{\left(\dfrac{u_t}{u_{mf}}\right) - 1} \tag{13a}$$

a channel may be formed in the bed. For small particles ($Re_t < 0.2$),

$$\frac{u_t}{u_{mf}} \simeq 78,$$

as mentioned earlier and therefore,

$$\frac{r_{d,mf}}{r_b} < 0.013$$

This value is one-tenth of that given by Siegel's approach.

For the perforated distributor, r_d, cannot be considered to be independent of u; it is well known that (Bird et al., 1960)

$$\Delta P_d = ur_d = \zeta\frac{u^2}{2g}\rho_f \tag{14}$$

or

$$r_d = \zeta\frac{u}{2g}\rho_f \tag{15}$$

We can resort to this velocity dependence of the resistance through the perforated distributor to develop a criterion for channeling for the bed with a perforated distributor, which is slightly different in form from inequality (Eq. 13a) derived for the bed with a porous distributor. From Eq. 15, the resistance through the perforated distributor at the minimum fluidization velocity, $r_{d,mf}$, is

$$r_{d,mf} = \zeta\frac{u_{mf}}{2g}\rho_f \tag{16}$$

Then, the resistance at any velocity u, r_d, can be expressed as

$$r_d = r_{d,mf}\frac{u}{u_{mf}} \tag{17}$$

From inequality (Eq. 10) and Eqs. 17 and 8, we obtain for the perforated distributor

$$ur_{d,mf}\frac{u}{u_{mf}} + H(1-\epsilon)\rho_s < u_{mf}r_{d,mf} + H(1-\epsilon_{mf})\rho_s$$

or

$$\frac{r_{d,mf}}{r_b} < \frac{\left(\dfrac{u}{u_{mf}}\right)^{1/n} - 1}{\left(\dfrac{u}{u_{mf}}\right)^2 - 1}\cdot\frac{1}{\left(\dfrac{u_t}{u_{mf}}\right)^{1/n} - 1} \tag{18}$$

TABLE 2. DISTRIBUTOR-TO-BED RESISTANCE AND PRESSURE DROP RATIO FOR UNIFORM FLUIDIZATION

Porous Distributor										
$\frac{u}{u_{mf}}$	1.5	2	3	4	5	6	7	8	9	10
$\frac{r_{d,mf}}{r_b}$	0.1174	0.1036	0.0859	0.0746	0.0666	0.0606	0.0558	0.0519	0.0486	0.0459
$\frac{\Delta P_d}{\Delta P_{b,mf}}$	0.1761	0.2071	0.2576	0.2984	0.3330	0.3634	0.3906	0.4152	0.4378	0.4587
Perforated Distributor										
$\frac{u}{u_{mf}}$	1.5	2	3	4	5	6	7	8	9	10
$\frac{r_{d,mf}}{r_b}$	0.0470	0.0345	0.0215	0.0149	0.0111	0.0087	0.0070	0.0058	0.0049	0.0042
$\frac{\Delta P_d}{\Delta P_{b,mf}}$	0.1057	0.1381	0.1932	0.2387	0.2776	0.3116	0.3418	0.3691	0.3941	0.4171

The ratio

$$\frac{\left(\frac{u}{u_{mf}}\right)^{1/n} - 1}{\left(\frac{u}{u_{mf}}\right)^2 - 1}$$

also decreases with an increase in u, and the minimum value of

$$\frac{r_{d,mf}}{r_b}$$

in the range of $u_{mf} < u \le u_t$ is at u_t. Therefore, channeling will occur in the bed of small particles ($Re_t < 0.2$, $u_t/u_{mf} \simeq 78$ and $n \simeq 4.65$) with a perforated distributor, if

$$\frac{r_{d,mf}}{r_b} < 1.644 \times 10^{-4}$$

This value is much smaller than that obtained by the approach based on Siegel's model which is 0.072 (Quershi and Creasy, 1979). Note that the required pressure drop through the distributor is very small for preventing channeling in the bed.

CRITERION FOR UNIFORM FLUIDIZATION

From the analysis given in the preceding section, it is clear that the distributor-to-bed pressure drop ratio for preventing channeling in the bed cannot be used as a criterion for designing a distributor. For example, when $R = 0.1 > 0.013$, channeling will not occur in the bed with a porous plate, but if the operating velocity is smaller than $2.16u_{mf}$, we see from Table 1 and Figure 3 that the overall local pressure drop of the fluidized part is smaller than $\Delta P_{t,mf}$, and, thus, partial, but not full, fluidization may occur in the bed. Only after the full fluidization velocity, u_f, is attained, will the overall local pressure drop of a fluidized portion of the bed be greater than $\Delta P_{t,mf}$, causing the whole bed to fluidize. Therefore, to guarantee the full fluidization of the bed at a given operating velocity of u, it is necessary that

$$ur_d + H(1-\epsilon)\rho_s \ge u_{mf}r_{d,mf} + H(1-\epsilon_{mf})\rho_s \qquad (19)$$

This expression gives rise to the following criterion for uniform fluidization of the bed with a porous distributor.

$$\frac{r_{d,mf}}{r_b} = \frac{\left(\frac{u}{u_{mf}}\right)^{1/n} - 1}{\left(\frac{u}{u_{mf}}\right) - 1} \cdot \frac{1}{\left(\frac{u_t}{u_{mf}}\right)^{1/n} - 1} \qquad (20)$$

Similarly, for the bed with a perforated distributor, we have the following criterion for uniform fluidization.

$$\frac{r_{d,mf}}{r_b} = \frac{\left(\frac{u}{u_{mf}}\right)^{1/n} - 1}{\left(\frac{u}{u_{mf}}\right)^2 - 1} \cdot \frac{1}{\left(\frac{u_t}{u_{mf}}\right)^{1/n} - 1} \qquad (21)$$

The results calculated from Eqs. 20 and 21 are given in Table 2. The distributor-to-bed pressure drop ratios for the porous and perforated distributors at various operating velocities are also shown in Table 2, which have been calculated respectively from the following expressions;

$$\frac{\Delta P_d}{\Delta P_{b,mf}} = \left(\frac{u}{u_{mf}}\right)\left(\frac{r_{d,mf}}{r_b}\right) \qquad (22)$$

for the porous distributor, and

$$\frac{\Delta P_d}{\Delta P_{b,mf}} = \left(\frac{u}{u_{mf}}\right)^2\left(\frac{r_{d,mf}}{r_b}\right) \qquad (23)$$

for the perforated distributor. Since these results are within the range used in practice (Qureshi and Creasy, 1979), the present approach or model for determining the criterion for uniform fluidization may be reasonable.

From Eq. 20 the criteria of channeling, partial fluidization and full fluidization can be determined for the fluidized bed with a porous distributor. For example, when $u = u_t$, we obtain

$$\frac{r_{d,mf}}{r_d} = \frac{\left(\frac{u_t}{u_{mf}}\right)^{1/n} - 1}{\left(\frac{u_t}{u_{mf}}\right) - 1} \cdot \frac{1}{\left(\frac{u_t}{u_{mf}}\right)^{1/n} - 1} = \frac{1}{\left(\frac{u_t}{u_{mf}}\right) - 1}$$

For small particles ($Re_t < 0.2$), for which $u_t/u_{mf} \simeq 78$ and $n = 4.65$, we have

$$\frac{r_{d,mf}}{r_b} \simeq \frac{1}{78-1} = 0.013$$

At this value channeling will occur in the bed because the overall local pressure drop through the fluidized portion of the bed, ΔP_t, before the local velocity reaches u_t is smaller than $\Delta P_{t,mf}$, thus causing the local velocity to continue to increase; when it reaches u_t, a channel will be established. When $u = u_{mf}$, we cannot directly calculate $(r_{d,mf}/r_b)$ from Eq. 20; however, by defining

$$\frac{u}{u_{mf}} - 1 = x,$$

we can write

$$\frac{\left(\frac{u}{u_{mf}}\right)^{1/n} - 1}{\left(\frac{u}{u_{mf}}\right) - 1} = \frac{(1+x)^{1/n} - 1}{x}$$

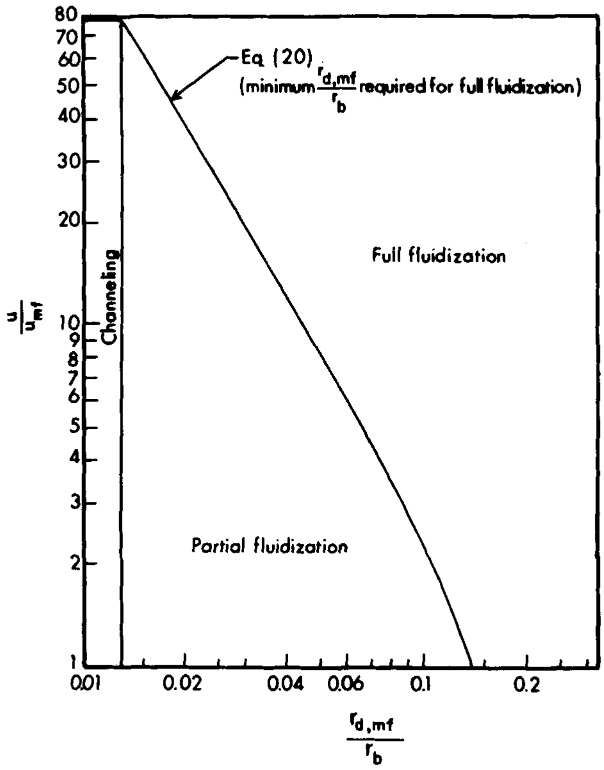

Figure 4. Regions of the channeling, partial fluidization and full fluidization for the bed with a porous distributor.

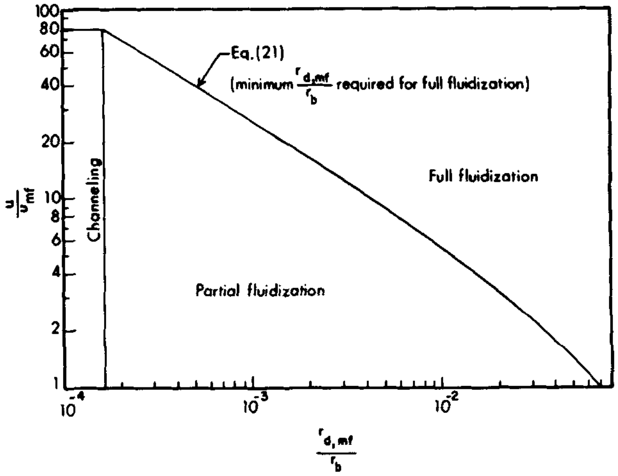

Figure 5. Regions of the channeling, partial fluidization and full fluidization for the bed with a perforated distributor.

Furthermore, we see that

$$x \rightarrow 0$$

when

$$u \rightarrow u_{mf}$$

Since

$$\lim_{x \to 0} \frac{(1+x)^{1/n} - 1}{x} = \frac{1}{n},$$

we obtain

$$\frac{r_{d,mf}}{r_b} = \frac{1}{n} \frac{1}{\left(\frac{u_t}{u_{mf}}\right)^{1/n} - 1} = \frac{1}{4.65}\left[\frac{1}{(78)^{1/4.65} - 1}\right] \simeq 0.14$$

When the value of $(r_{d,mf}/r_b)$ is 0.14 or greater, the overall local pressure drop through the fluidized portion of the bed, ΔP_t, will be greater than $\Delta P_{t,mf}$, and thus the local velocity can not increase beyond the minimum fluidizing velocity, u_{mf}; this will not lead to the partial fluidization and the full fluidization will persist in the bed. The value of $(r_{d,mf}/r_b)$ in the range from 0.013 to 0.14 will cause partial fluidization, if the value of (u/u_{mf}) is smaller than that calculated by Eq. 20. Similarly, for the perforated distributor, we obtain $r_{d,mf}/r_b = 1.46 \times 10^{-4}$ when $u = u_t$, and $r_{d,mf}/r_b = 0.07$ when $u = u_{mf}$.

Figure 4 plots Eq. 20 indicating the regions of the channeling partial fluidization and full fluidization for the porous distributor. Similarly, Figure 5 plots Eq. 21 indicating the regions of the channeling, partial fluidization and full fluidization for the perforated distributor.

CONCLUSION

1. The results of the present global analysis appear to indicate that Siegel's model for the onset of fluidized bed channeling is not valid.

2. The condition under which channeling will occur in the bed has been established. It is:

$$\frac{r_{d,mf}}{r_b} < \frac{1}{\frac{u_t}{u_{mf}} - 1}$$

for the bed with a porous distributor, and

$$\frac{r_{d,mf}}{r_b} < \frac{1}{\frac{u_t}{u_{mf}}^2 - 1}$$

for the bed with a perforated distributor.

The value of the distributor-to-bed resistance ratio for the occurrence of channeling based on the present approach, as given above, is much smaller than that based on Siegel's model.

3. The nonoccurrence of channeling cannot assure the uniform fluidization. The criterion for it should be based on the condition of full fluidization in the bed. Based on the present approach, the criterion is

$$\frac{r_{d,mf}}{r_b} = \frac{\left(\frac{u}{u_{mf}}\right)^{1/n} - 1}{\left(\frac{u}{u_{mf}}\right) - 1} \cdot \frac{1}{\left(\frac{u_t}{u_{mf}}\right)^{1/n} - 1}$$

for the porous distributor, and

$$\frac{r_{d,mf}}{r_b} = \frac{\left(\frac{u}{u_{mf}}\right)^{1/n} - 1}{\left(\frac{u}{u_{mf}}\right)^2 - 1} \cdot \frac{1}{\left(\frac{u_t}{u_{mf}}\right)^{1/n} - 1}$$

for the perforated distributor.

The calculated values of the distributor-to-bed pressure drop ratio, $(\Delta P_d/\Delta P_{d,mf})$, based on the present approach are reasonably consistent with the available data.

ACKNOWLEDGMENT

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NOTATION

H = height of bed
ΔP_b = pressure drop through the bed
$\Delta P_{b,mf}$ = pressure drop through the bed at the minimum fluidization velocity
ΔP_d = pressure drop through the distributor
$\Delta P_{d,mf}$ = pressure drop through the distributor at the minimum fluidization velocity
ΔP_t = overall pressure drop
R = distributor-to-bed pressure drop ratio
Re_t = Reynolds' number at the particle terminal velocity
r_b = static bed resistance
r_d = distributor resistance
$r_{d,mf}$ = distributor resistance at the minimum fluidization velocity
u = superficial fluid velocity
u_{mf} = minimum fluidization velocity
u_t = particle terminal velocity
ϵ = void fraction of the bed
ϵ_{mf} = void fraction of the bed at the minimum fluidization velocity
ρ_f = density of fluid
ρ_s = density of particles
ζ = orifice coefficient

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Formation of Inclusions in Terephthalic Acid Crystals

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INTRODUCTION

In recent years improved processes for the manufacture of terephthalic acid (TPA) have led to the development of processes for the direct esterification of TPA with ethylene glycol. This route to the formation of polyethylene terephthalate (PET) has significant economic advantages (Hizikata (1977)) when compared with the route employing dimethyl terephthalate (DMT). A requirement of the direct esterification route is a pure grade of TPA virtually free of impurities which cause unwanted coloring of the product PET. The impurities in TPA manufactured by the oxidation of paraxylene are typically intermediate and byproducts of the oxidation such as 4-carboxybenzaldehyde (4-CBA), o- and m-phthalic acid, p-toluic acid and p-acetylbenzoic acid. 4-CBA is probably the most difficult of these impurities to remove and its concentration in TPA serves as a practical criterion of TPA purity.

Purification of TPA has been the subject of intensive research of the years and a multitude of techniques have been proposed. A summary of these techniques appears as Table 1. A large number of the TPA purification techniques employ crystallization (from the vapor or from the liquid) as a purification step (Maclean, 1960; Kurtz, 1965; Olsen, 1970). It has been reported (Fujita et al., 1968) that recrystallization alone does not normally reduce 4-CBA concentrations in TPA crystals to the desired level. In a recent paper Myerson and Gaines (1982) reported that TPA crystals underwent an irreversible change of habit (known as crystal aging) when immersed in their own saturated solution. The rate of this change of shape was reported to be a strong function of temperature and resulted in an increase in crystal purity. It is the purpose of this study to present several observations which may explain the limitations of crystallization or aging as a purification method.

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TPA RECRYSTALLIZATION AND AGING

Recrystallization and aging experiments were conducted in a batch suspension crystallizer shown in Figure 1. It consists of a

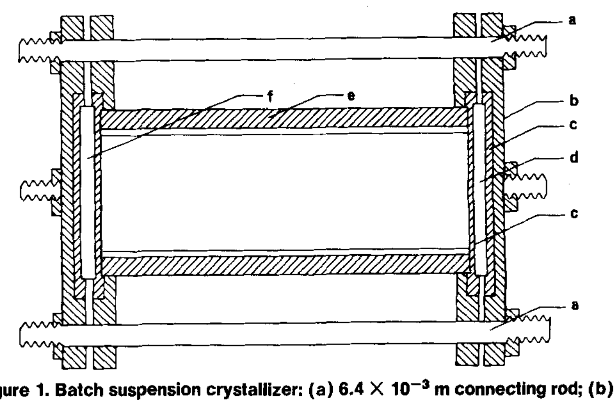

Figure 1. Batch suspension crystallizer: (a) 6.4×10^{-3} m connecting rod; (b) 1.016×10^{-1} m diameter by 6.4×10^{-3} m thick steel frame plate; (c) teflon gasket; (d) 5.08×10^{-2} m diameter by 6.4×10^{-3} m thick tempered sight glass; (e) 3.81×10^{-2} m nominal diameter schedule 80 stainless steel pipe; (f) 3.81×10^{-2} m OD tempered sight glass.