THE POSSIBILITIES OF FLUIDIZATION OF COHESIVE POWDERS IN CONES

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Selected hydrodynamic parameters are studied in the paper of a bed of a cohesive material, the diameter of a mean statistical particle being 37 μm. The bed of solids was aerated in a conical vessel and velocities were measured of incipient fluidization. It was established that a circulatory motion of the solid particles did not occur until at velocities exceeding that of incipient fluidization by a factor of three when the spouted bed regime was established. Another studied parameter was the mean velocity of descent of particles under the spouted bed regime. A theoretical relationship was derived for the latter quantity. A simple mathematical model was derived and experimentally tested for the distribution curve, *E*, characterizing the residence time distribution in the bed.

For gas–solid reactions with the reaction rate being proportional to the area of surface of the solids it is benefitial to work with powdered solids exhibiting relatively large interfacial area. However, efforts to maximize the interfacial area are limited by the fact that the flow properties of fine solids markedly deteriorate with decreasing particle size. In extremely fine powders cohesion plays an important role and aeration of such materials may be hampered by slugging or channeling while the bulk of the solids remains virtually immobile. This, of course, reduces the rate of renewal of the interface between the solids and the flowing gas. The channels by-pass the active parts of the bed with subsequent decline of the efficiency of the process. Because the above mechanism impairs also the conditions for heat transfer, exothermic reactions lead to hot spot formation with undesirable effects in the reactor. Methods how to suppress or eventually completely eliminate the appearance of immobile pockets in the bed already exist. The most common is the mixing of the batch in the space just above gas inlet. However, mechanical devices often cannot be mounted in the reactor either for construction reasons in case of high-pressure reactions, or owing to the presence of cooling coils in reactor necessitated by highly exothermic reactions. Another way of how to reduce the appearance of immobile pockets in reactors while favourably influencing the flow of the particulate solids is to shape the reactor vessel as a cone. This method is quite common in design of storage tanks and silos for granular solids.

We have tested the prospect of the application of cones also to fine-powdered materials. The idea is based on the fact that the regime within the cone well simulates that of a spouted bed ensuring intensive mixing of solids in the vicinity of gas inlet, that is a spot where the potentials of channel formation are high (the channels originate in the proximity of the distributing plate and spread upwards).

In this paper we have focused on the study of the motion of a compact, very fine granular material aerated in the apparatus shaped as a frustrum of a cone. However, situations can occur where the cone represents one element of the bottom of a large-scale multiple-cone reactor. Based on model experiments with one cone, the grid was designed to consist of conical elements. The function of the grid had been tested on a 400 mm experimental set-up.

THEORETICAL

The Velocity of the Solids in the Cone

When the gas is fed at the bottom of a conical vessel containing fine particulate solids one can distinguish two regions. In the neighborhood of the vertical axis the regime resembles that of the spouted bed. The solids here are entrained upwards by the gas. At the top the solids change their direction and move along the walls toward the distributing plate. Because volume expansion as well as radial dispersion of gas bubbles relatively deep in the cone is negligible one can take for the diameter of the spouted bed the diameter of the distributing plate. The required pressure drop for the transport at the mass flow rate G_s in spouted bed can be calculated using the following set of equations

$$\begin{aligned} \Delta p &= \Delta p_H + \Delta p_a + \Delta p_f , \\ \Delta p_H &= \varrho_g (G_s/G)(u_0/u_s) gH , \\ \Delta p_a &= u_s u_0 \varrho_G (G_s/G) , \\ \Delta p_f &= \Delta p_{f,g} + \Delta p_{f,s} , \\ \Delta p_{f,g} &= 2 f_g \varrho_g u_0^2 H/d_t , \quad \Delta p_{f,s} = 4 f_s H G_s u_s / 2d_t , \\ f_g &= 0.0791 \, \mathrm{Re}_t^{-0.25} \quad \text{for} \quad 3 \cdot 10^3 < (\mathrm{Re})_t < 10^5 \\ 4 f_s &= \frac{3 \varrho_g d_t C}{2 \varrho_s d_s} \left(\frac{u_g - u_s}{u_s} \right)^2 . \end{aligned} \tag{1}$$

C may be read off the graph as a function of Re[1]. The unknown variables in the set (*1*) are mutually constrained by the relations

$$G = u_0 \varrho_g = u_g \varrho_g e , \tag{2}$$

$$G_s = u_s \varrho_s (1 - e)$$

and by the assumption

$$u_s = u_g - u_t . \tag{3}$$

The mean velocity of the advancing particles in the space between the walls of the cone and the spouted bed area can be computed from

$$(G_s \, \pi d_t^2/4)/F(h) \, \varrho_s(1 - e') = v_s \,, \tag{4}$$

where e' ranges between the limits $e_{\text{bulk weight}}$ and $e_{\text{incipient fluidization}}$. $F(h)$ stands for the area of annulus at the height h which can be computed from

$$F(h) = \pi\{(d_t/2 + h \operatorname{tg} \alpha/2)^2 - d_t^2/4\} \,. \tag{5}$$

The maximum velocity that can be attained in the critical annulus (fictious surface through which the solids return into the spouted part of bed) equals the theoretical discharge velocity. Roughly, the flow of the material in the annulus under these conditions may be paralleled to the discharge of solids from a tank and the discharge velocity can thus be computed from familiar formular, *e.g.*[2]

$$F_s(\operatorname{tg} \theta_r)^{1/2}/C_w C_0 \, g^{1/2} \, \bar{\rho}_\Delta d_s^{2\cdot5} = 0\cdot161(d_{or}/d_s)^{2\cdot746} \,. \tag{6}$$

The coefficients C_w, C_0 can be found in standard books, *e.g.*[2]. The critical annulus may be put at the vertical distance from the distributing plate equalling about the diameter of the plate. Below this surface (that means at the tip of the cone) the character of the flow is rather atypical for the translatory type of motion and the continuous stream of solids disintegrates.

Flow Characteristics of Gas Passing through the Bed of Fine Powdered Solids

A simple mathematical model was formulated based on the idea that the gas on discharging from the distributing plate splits into two parallel sections containing dense and thin phase. This model seems to be an adequate approximation of the behaviour of the heterogeneous layer as confirmed by visual observations described in the preceding paragraph. To describe mixing in individual sections the model uses a series of ideally mixed cells (Fig. 1). Although the model disregards mutual mingling of gas from the two sections within the bed it appears quite satisfactory from the practical point of view.

For the distribution of the residence time one can derive for this model the following closed form expression

$$E(\theta) = f \frac{n^n}{(n-1)!} \beta^n \theta^{n-1} \exp(-n\beta\theta) + (1 - f) \frac{m^m}{(m-1)!} \left(\frac{\beta}{\alpha}\right)^m \theta^{m-1} \,.$$
$$. \exp(-m\beta\theta/\alpha) \tag{7}$$
$$f = Q_1/Q \,, \quad \theta = t/\bar{t} \,, \quad \alpha = \bar{t}_2/\bar{t}_1 \,, \quad \beta = \bar{t}/\bar{t}_1$$

Q is the volume flow rate through the equipment, Q_1 is the volume flow rate through section 1 containing the thin phase, n and m are respective numbers of the mixed cells in the first and the second section. $\bar{t}_1$, $\bar{t}_2$ are the mean residence times in the two sections and

$$V_1 = \bar{t}_1 Q_1 \,, \quad V_2 = \bar{t}_2 Q_2 \,, \quad \bar{t} = f\bar{t}_1 + (1 - f)\,\bar{t}_2 \,, \quad V = \bar{t}Q \,. \tag{8}$$

EXPERIMENTAL

The conical element of the set-up is represented by a frustum of a cone. The gas is fed into the cone through a perforated plate (grid) placed in the narrower bottom of the frustrum of a cone. Plates of wide ranging geometries were tested, that means plates differing both in the diameter of openings as well as the free area. It turned out though that for a detectable motion of the material within the cone and for a uniform aeration it was necessary to ensure high velocities within the openings of the discharge plate. This condition was met for our granular material only by the grids with the free area below 1% and the size of the openings below 0·9 mm. For the majority of experiments we used the plate exhibiting $\varphi = 0{\cdot}8\%$ and $d = 0{\cdot}9$ mm. The apex angle of the cone in spouted bed reactors working with coarse materials is usually around 30°. In view of the poor flow properties of fine granular solids we tested also geometries with apex angles below 30°.

Granulometric composition of the solids. A fine ground Si—Cu alloy (9 : 1) was used exhibiting the density of 2·364 kg/m^3. The mean bulk density was 1·083 kg/m^3. For comparison we used also the same material coarsely ground. Granulometric composition of both materials[3], *i.e.* the fine and the coarse one, is shown in Fig. 2. The diameter of a mean statistical particle of the fine and the coarse material was 37 μm and 650 μm respectively. The different composition of the material results from different regime of grinding applied on the basic raw material.

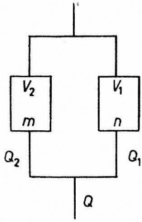

FIG. 1

Perfectly Mixed Cells Model

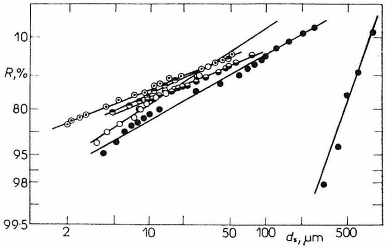

FIG. 2

Granulometric Composition of Fine and Coarse Material ○, ◒, ◓, ⊙, Samples of industrial Si—Cu alloys, ● Coarse-grain Si—Cu alloy and the same alloy ground to a finer grade.

Bottom with conical elements. In spite of undisputable advantages, in comparison with cylindrical geometry, the danger of arching in ordinary cones is always present. We have designed a new bottom of the reactor utilizing conical geometry but reducing the aptitude to arching in the apex of the cone (Fig. 3). The distributing grid consists of two horizontal, concentric, annular perforated plates separated by slanting walls. These walls viewed in the direction perpendicular to the grid have the shape of a triangle and the grid thus appears to consists of cones (Fig. 3). The angle of the cones and the geometry of the perforated plates (annuli) were designed utilizing the experience gathered during the experiments with one cone. In this arrangement the distribution of the residence time of the gas was studied which should reveal eventual bypasses or dead pockets. The experimental method was that of the response technique to a general input of the tracer (isotope Krypton 85). The response was picked up by the Geiger-Mueler tubes located just above the bed. The dynamic characteristics were determined by analyses of the time variations of the tracer both at the inlet and the outlet. The technique of the generation of the input and pick-up of the response curves has been described in detail elsewhere in the research report[4]. The characteristics of the bottom and the operating conditions are as follows: Diameter of the apparatus 400 mm, diameter of openings 0·8 mm, number of openings 539 + 229, the height of the freely dumped charge of solids (the bottom is attached to a cylindrical vessel) 680 mm, fine powdered, material, volume flow rate of gas 360—580 l/min.

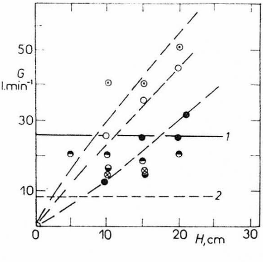

FIG. 3

Gas Flow Rate at Incipient Fluidization as a Function of Bed Depth

1 Calculated value[2] for $d_s = 0{\cdot}5$ mm, *2* experimental values for the coarse material in the cylinder of equivalent diameter. Velocities of incipient fluidization in the cone for the coarse material ◓ 10°, ◒ 15°, ⊗20°; minimum flow rate in the cone for the fine material ⊙ 10°, ○ 15°, ●20°.

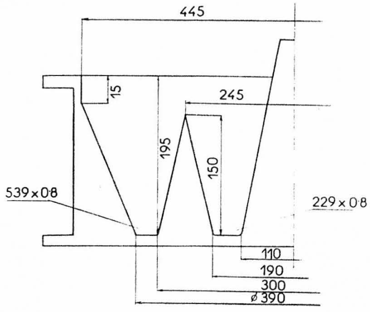

FIG. 4

Sketch of Axially Symmetric Grid with Conical Elements

Dimensions in mm.

RESULTS

The velocity of incipient fluidization in the elementary cone. Fig. 4 illustrates the effect of the angle on the minimum velocity u_b. The fine material requires higher flow rates of gas in comparison with the coarse one in order to bring about a detectable motion of the particles. The effect of the particle size distribution did not show for the fine material. The effect of the cone angle for the coarse material is not apparent. For the fine particles this influence can be explained by the arching which above 20° probably did not occur. For greater apex angles of the cone, however, the flow properties of the fine material again deteriorate. The coarse material, under the conditions as those in Fig. 4, at the minimum observable motion appears in the state of incipient fluidization and not the spouted bed. For a good mixing of both the fine and the coarse material the flow rates of gas must be increased about three times in comparison with those in Fig. 4. The minimum diameter of the perforated plate in the cone must be selected so as to prevent the particulate material from arching in the narrow part of the cone, an effect which would completely destroy the regime of spontaneous circulation. A first estimate of the diameter of the distributing plate can be based on the relation published by Stepanoff[5]

$$d_t > 4\tau_0(1 + \sin \varphi)/\varrho_s g \,. \tag{9}$$

The velocity of solid particles in the element cone. The velocity of solid particles in our experimental conditions was limited by the constraints at the discharge mentioned in connection with the preceding theoretical analysis. The empirically estimated values of the advancement (method of direct measurement of the velocity of tracer particles near the cone wall) range for the cone with the apex angle 20° and the gas flow rate 50–80 l/min between 1 and 2 cm/s. The agreement of the theoretically calculated values following from the previously given equations was within 30% rel. provided a steady regime had been reached. It should be noted that the results are valid for dry material. Although the majority of the experiments were carried out with the dried solids – the air was dried by a 0·8 m deep bed of silica gel and heated to 60°C – still the situation occurred when the motion in some experiments ceased for indefinite time (seconds to tens of seconds) to come back again later.

Further it should be noted that the process of circulation is not even for the "steady" mixing continuous. Instead, the mass of solids advances by "stick-slip" form of motion. The indicated data on the velocity of motion thus represent average values. The adverse effect of arching, which cannot be safely suppressed in simple cones, was disturbed by simultaneous function of a fixed disturbing jet located just above the distributing plate in the bottom of the cone. This jet, a 10 × 2 mm slot was located off center so as to obtain a stream of gas moving approximately horizontally in the tangential direction. The distance of the jet above the bottom was 2 cm and the distance from the walls about 2 cm. A satisfactory regime of mixing for the given geometry was achieved only with a simultaneous flow of gas both through the plate (30–40 l/min) and the jet (30 l/min); a reliable function of the cone can be ensured by two jets at opposite sides of the cone and with the opposite direction of the flow of the discharged gas.

The elementary cone, the cone without disturbing jets, does not apparently represent the optimum elementary unit from the view point of the arching to which contributes the whole surface of the wall of the cone. In large-scale experiments (isotope method of research of the residence time distribution) we therefore used the grid on the basis of screw cylinders. This though does not possess conical geometry but it is free of the adverse effect of the side walls of the cone on the formation of arches.

Mixing of gases in the model reactor with the bottom consisting of conical elements. The parameters of the experimental reactor given in the part Experimental were obtained from a series of numerical calculations consisting of fitting the experimental and calculated curves of the residence time distributions $E(\theta)$. The results are summarized in Table I. From the results it is apparent that the gas passing through the this phase moves practically at plug flow; in the dense phase along the walls of the reactor, with minor backmixing. In accord with the usual methods of relating the dispersion and mixed-cell models it is possible to estimate the coefficient of axial dispersion, and from the ratio of the volume of the thin and the dense phase to the total volume occupied by gas to estimate the average porosities in the two sections of the reactor and gas velocity. Additional measurements of the distribution of porosity (*e.g.* by independent γ-ray method) can further give precision to the data on the effective area of cross section and gas velocity to be used for modelling of gas–solid reactors. In the reactor with the bottom consisting of conical elements the content of the thin phase is about 1/10 (at stable conditions). Mixing of gas in the space

TABLE I

Experimental Data and Corresponding Model Parameters

Volume flow rate Q l/min	Mean residence time s	Value of residence time distribution			Parameters of model			
		θ_{max}	E/θ_{max}	$\bar{t}_2/\bar{t}_1$	Q_1/Q_2	V_1/V	n	m
390	14·42	0·523	0·887	2·0	0·3	0·18	5	3
425	12·60	0·535	0·847	2·0	0·15	0·08	5	3
515	9·20	0·595	0·897	1·9	0·20	0·12	5	3
590	9·44	0·526	0·854	1·8–2·2	0·2–0·3	0·143	5	3

between the upper level and the horizontal level reached by the walls of the cone is good and visual observation revealed also good motion of individual particles, a phenomenon advantageous for intensive heat transfer. However, as a disadvantage appears that for the lack of the sufficient quantities of the solid material the experiments could not cover greater depths of freely dumped material.

CONCLUSION

The engineering of the powdered solids–gas reactions appears both theoretically and experimentally little explored discipline although practical applications of such reactions are of industrial importance (*e.g.* the chemistry of organosilanes). Proper design of the reactor, owing to the rather unfavourable flow properties, is difficult. On purpose we have concentrated on the study of the motion of granular solids in vessels without mechanical mixing devices. A feasible alternative is the just described pseudo-fluidized bed. However, its stability poses a hydrodynamic problem. In this paper we have analyzed the use of the apparatus utilizing conical geometry which after a thorough analysis of the problem was thought to be capable of providing good flow properties for the solid phase. It turned out that a simple cone is not quite reliable for this purpose. A more suitable, although more complicated is the cone equipped with disturbing jets which also need not be always perfectly reliable at practical application to high-temperature reactions involving solids apt to sintering. In that case it is preferable to use the bottom with conical elements where the specific disadvantages of an individual cone are effectively suppressed. Moreover, the energy imparted to gas must be sufficient to set the solids into motion in order to ensure their reliable contact with gas. Practical application would mostly require the gas to be recycled. An economy consideration then would have to be used to compare this arrangement with a simple fluidized bed working with a coarse material. Such a material is generally less reactive but free of undue complications characteristic for fine powders.

LIST OF SYMBOLS

d	size of the opening
d_t	diameter of the spouted bed
d_s	solid particle diameter
d_{or}	equivalent diameter of disc with area equal to the discharge annulus
$E(\theta)$	residence time distribution function
f_g, f_s	friction factors
$F(h)$	area of cross section at distance h
F_S	mass rate of particles discharging from annulus into the spouted bed region
g	acceleration due to gravity
G	weight flow rate of gas
G_s	weight flow rate of particles, rate of entrainment
h	coordinate of height

H	height of bed
m	number of mixed cells
n	number of mixed cells
Δp	total pressure drop
Δp_a	partial pressure drop, kinetic term
Δp_f, Δp_g	partial pressure drop due to friction in phases
Δp_H	partial pressure drop, potential term
Q	volume flow rate of gas in reactor
Q_1, Q_2	volume flow rate of gas in sections with thin and dense phase
R	residue on the sieve with the mesh size d_s
Re_t	particle Reynolds number
$\bar{t}_1$, $\bar{t}_2$	mean residence time in sections with thin and dense phase
u_b	velocity of incipient fluidization
u_0	velocity of gas in spouted bed related to cross sectional area of vessel
u_g	real velocity of gas in spouted bed
u_s	velocity of particles in spouted bed
u_z	terminal velocity of particles
V	volume of aerated bed
V_1, V_2	volumes of sections with thin and dense phase
α	apex angle of cone
ϕ	angle of internal friction
ϱ	density
θ_{max}	dimensionless time corresponding to the maximum value of $E(\theta)$ distribution curve
θ_r	angle of slope (dumping angle)
τ_0	initial shear stress
φ	free area of plate

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