Method for Particle Size Control During the Drying and Granulation in Fluidized Bed

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Abstract
The fluidized bed process presented in this paper is suitable for recovery of solids from solutions in the form of granules. The solution is sprayed into the bed and as solvent evaporates, solid material is deposited on the surface of fluidizing particles. During this process, particle growth takes place by surface layering and/or agglomeration. A special grinder is used in the fluidized bed to control particle growth by causing selective disintegration of large particles. For steady state operation, the most important task is to determine the existing particle size distribution in the fluidized bed. A special method was developed to measure torque and stress fluctuations* in the bed of particles. Using a correlation between torque and stress fluctuations and particle size, this method and device can be used for direct control of the rotation speed of the grinder to produce granules of given size in the fluidized bed. Results of torque and stress measurements and their correlation to particle size are presented.

Key words: Granular Flows, Torque measurements, Couette device, Measurements of particle size.

1. Introduction
A special fluidized bed process, developed by Hungarian researchers [1], serves to recover solids in the form of granules directly from solutions or suspensions. An initial charge of particles, chemically similar to the material in solution, is fluidized by hot air. Solution is sprayed into the fluidized bed where the solvent evaporates and solid material is deposited onto the surface of the fluidized particles. During this process, particles are growing either by surface layering or by agglomeration. To ensure steady state operation, the mean particle size has to be stabilized by feeding (or recycling) fine particles into the equipment and/or by employing special grinder-rollers rotating within the fluidized bed. Mean particle size and particle size distribution can be adjusted by the feeding rate of solids and/or by the geometrical parameters and rotational speed of the grinder-rollers.
The granulated product is taken off continuously from the bed. The process is shown schematically on Fig.1. To control the steady state operation, the most important task is to determine the existing particle size in the bed. To this end, development work was carried out during joint research between a group at the City College of CUNY and another at the Research Institute of Chemical and Process Engineering, Hungary. The work was supported by the US – Hungarian Science & Technology Joint Fund. The new concept and experimental set up used in Hungary were based on theoretical principles established at CCNY and on earlier laboratory experiences performed in New York. The current measurements were carried-out in Hungary, while evaluation of the results was done during a combined effort of the two groups.

The starting idea was that the characteristics of the torque signal captured during rotational shearing of a bed of particulate solids may depend on particle size. The group at CCNY has carried out experiments on a Couette-type device [2] to determine the dependence of the dimensionless torque on relative gas velocity and dimensionless shear rate. Particles of different materials and sizes were used with gas velocities at and below minimum fluidization including the case of zero gas velocity, i.e., packed bed conditions. Similar devices were also used for continuous shearing of fine fluidized powders by other workers [3-4]. These latter measurements were intended to determine the „viscosity” and other characteristics of particulate beds fluidized by gas as a function of gas velocity.

2. Experimental

For the present work, a modified Couette-device somewhat similar to that employed by Tardos et al. [2], was used (see Fig.2) to study the effect of particle size on the measured torque during continuous shearing of dry, granulated sucrose particles. Characteristics of the granular material with five different mean particle sizes between \(d=0.3\) and \(1.4\ mm\) are given in Table 1. Particle sizes were determined by sieve analysis. Granule shapes
were observed by light microscope and the angles of repose and flowability values were measured according to ASTM methods. Bulk densities were determined using a graduated cylinder.

![Diagram of experimental device]

**Fig. 2. Modified Couette-device connected to the fluidized bed granulator**

The experimental device consisted of an inner, rotating cylinder and an outer stationary tube. Both were covered by coarse sand paper with a wall friction coefficient higher than the internal friction coefficient of the particle bed. The inner cylinder could be rotated between \( n = 60 \) and 240 rpm (corresponding to a peripheral speed of \( v_p = 0.16-0.65 \, \text{m/s} \)) by an electronically controlled DC motor, while the torque on the shaft was continuously measured and recorded by an electronic data acquisition system and a PC. The maximum measurable torque was \( M_{\text{max}} = 0.44 \, \text{Nm} \). During preliminary experiments it was found that a relatively low rotational speed was more advantageous for the purpose of this work. Therefore, for the runs reported here, the inner cylinder was rotated at a rotational speed of \( n = 80 \) and 120 rpm, that was about one third of the maximal speed applied earlier by Tardos et al. [2]. Similarly to their experiments, the device was open at the top, i.e. the particle bed was unconstrained from above, with a constant level of particles. The bed could hence dilate to the critical state to easily accommodate the imposed shearing. In this way, a geometrically simple, regular flow could be formed in the gap between the rotating and stationary cylinders.

Preliminary tests carried out with gas flows at or above minimum fluidization showed that there was no significant difference in torque generated at different particle sizes. These tests also gave evidence that the easiest evaluable torque signals were obtained when no gas flow was used. Therefore the majority of tests were made under this condition. In addition to using stationary particle beds, experiments were also carried out with particles continuously moving downwards along the gap between the cylinders. This operation simulates the operation of a measuring cell used for particle size control for a continuously working granulator where the particles must steadily be exchanged in the measuring cell. The downward particle motion in this case was set at a velocity of \( v_s = 0.0001 \, \text{m/s} \), corresponding to a 13 minute mean residence time in the cell: this was considered suitable for process control purposes. This motion had no visible influence on the torque values measured, as shown below.

The diameter of the outer standing cylinder and that of the inner rotating cylinder were \( D = 106 \, \text{mm} \) and \( D_c = 52 \, \text{mm} \), respectively. Thus, the width of the gap between the two
cylinders was \( l_g=26\,\text{mm} \). The height of particle bed was \( 100\,\text{mm} \), while the rotating cylinder was only \( 80\,\text{mm} \) high.

The equipment and experimental conditions used for the present studies were somewhat different from those employed by Tardos et al. [2]: the height of the particle bed in the modified Cuette-device was lower, while the height and diameter of the rotating cylinder was significantly smaller. The gap between the cylinders was more than two times wider (26 mm instead of 12.7 mm). For this reason, the shear region never penetrated to the standing cylinder across the whole shearing gap (this observation was made by eye).

Table 1. Characteristics of the particles

<table>
<thead>
<tr>
<th></th>
<th>Sucrose size range mm</th>
<th>Sucrose mean particle size mm</th>
<th>Form of particles</th>
<th>Angle of repose</th>
<th>Flowability cm³/s</th>
<th>Bulk density kg/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.2-0.4</td>
<td>0.3</td>
<td>prismatic*</td>
<td>39.0</td>
<td>13.3</td>
<td>740</td>
</tr>
<tr>
<td>2</td>
<td>0.4-0.63</td>
<td>0.52</td>
<td>prismatic*</td>
<td>39.1</td>
<td>12.8</td>
<td>760</td>
</tr>
<tr>
<td>3</td>
<td>0.63-0.8</td>
<td>0.72</td>
<td>rounded prism</td>
<td>39.8</td>
<td>11.9</td>
<td>770</td>
</tr>
<tr>
<td>4</td>
<td>0.8-1.0</td>
<td>0.9</td>
<td>near spherical</td>
<td>40.9</td>
<td>11</td>
<td>760</td>
</tr>
<tr>
<td>5</td>
<td>1.25-1.6</td>
<td>1.4</td>
<td>near spherical</td>
<td>40.9</td>
<td>8.9</td>
<td>790</td>
</tr>
</tbody>
</table>

• without sharp tips and edges

3. Results and discussion

The diagrams shown in Fig.3. represent the results of torque measurements obtained for five different mean particle sizes \((d=0.3, 0.52, 0.72, 0.9, 1.43\,\text{mm})\) of granulated sucrose particles with narrow size distribution. The sixth curve refers to the torque measured in the empty device, i.e. with no particles. This represents only the friction of bearings and a small preset brake that was necessary to adjust the base-level of the torque. During measurements, this value was subtracted from all measured values. After an initial transient period, the measured torque remained essentially constant for each granule fraction (apart from stochastic fluctuation around a mean value caused mainly by irregular interactions between the rotating cylinder and the particle bed).

Fig. 3. Torque vs. time

\[
y = -0.1526x^2 + 0.4425x - 0.0013
\]

Fig. 4. Mean particle size vs. average torque
Using different rotation speed (n=80 and 120 rpm) the torque was observed to increase at increasing particle size as seen in Fig.4 for three different preset break values (\(M_o=0.043, 0.057\) and \(0.092\) Nm). This value had no influence, nor had the slow downwards motion of the particles as was evidenced by comparing these data with those obtained with no sliding particle motion as seen in Fig.5. As Fig.4 shows, the average values of the measured torque signal were in close relation with particle size, especially in the region below \(d=0.8-0.9\) mm granule diameter. Above that size, the dependence seemed less pronounced, at least for this geometry and dimensions of the device.

To check how a continuous measuring cell based on this principle could work, measurements were performed with particles moving downwards within the gap along the rotating cylinder. A sliding velocity of \(v_s=0.0001\) m/s, obtained by a mass flow of particles of 2.0-2.2 kg/h, was chosen to be negligibly small as compared to the peripheral velocity of rotation, (this latter being \(v_p=0.22\) m/s). Two experiments were performed under similar condition, with the difference that a static bed was used in the first case and a sliding bed in the other. The torques measured for particles having 0.9 mm mean particle size vs. time are plotted on the same diagram in Fig.5. From this, it is obvious that there is no significant difference between the two series of data: the mean torque value is \(M=0.297\) Nm in the static and \(M=0.299\) Nm in the sliding bed. This is evidence that continuous torque measurements can be done to check the granule size at a given moment within the granulator.

Experiments were also performed using polydisperse particle systems having much broader particle size distributions instead of the almost uniform granule fractions given in Table 1. Torque values obtained for these broad fractions do not show broader scattering of data than obtained for narrow particle size distribution as shown in Fig.5. The mean particle sizes were \(d=0.45\) mm and \(1.1\) mm, respectively. Averaging the torque values plotted in Fig.6, and calculating the mean particle sizes from these averages with the function given on Fig. 4., \(d=0.49\) mm and \(0.92\) mm were obtained, respectively; this can be considered as acceptable accuracy for such a preliminary experiment.

To simulate experimentally the situation of measuring particle sizes in a continuously working granulator, a continuous transition from one size to the other was simulated. Sucrose particles with \(d=0.4-0.6\) mm size distribution were initially filled into the...
fluidized bed. The modified Couette-type device (see Fig.2) was connected to the granulator where larger particles, with \( d=0.8-1.0 \) mm size distribution, were continuously introduced. A screw feeder as shown in Fig.2 ensured the flow of particle through the Couette-device; this was found to be \( v_s=0.0001 \) m/s (axial velocity).

Torque was continuously measured and recorded. Mean particle sizes were calculated from these measured values and plotted in Fig.7.

![Fig. 7. Average particle size from torque and direct measurement](image)

From this figure, it is clearly seen that the transition from the smaller to the larger particle size could be well traced by this method. At the beginning, and towards the end of this run, samples were taken for sieve analysis to determine the real mean particle size. These were found to be in good correlation with those obtained from the measured torque.

To correlate these findings with the theory and experimental results of Tardos et al. [2], some discussion is needed. To evaluate theoretically the experimental data, the following general expression was proposed [2] for the torque \( M \):

\[
M = \pi \rho_B g L^2 R^2 \sin \phi \left[ 1 - \frac{U}{U_{mf}} \right]
\]

This shows quadratic dependence on the height of the sheared layer, \( L \), on the radius of the inner cylinder, \( R \), and linear dependence on bulk density, \( \rho_B \), on the inner coefficient of friction of the particle bed, \( \sin \phi \), and on gas velocity, \( U \), relative to the minimum fluidization velocity \( U_{mf} \). No direct relation is shown by this equation with the particle diameter.

This equation was obtained for shearing particles at and below minimum fluidization velocity, within a relatively narrow gap along the surface of the rotating cylinder having relatively large diameter (i.e. rather small curvature) and length. During rotation, the shearing region always extended over the whole width of particle bed within the gap, independent of particle size. The main difference in the device used for the present experiments relative to the earlier one is that we used a much wider gap between the two cylinders, where the shearing region did not extended to the stationary wall. According to our observations, the width of the shearing zone (at least at the observable free top surface of the particle bed and for zero gas velocity) was in direct relation with the particle size: it was narrower for small particles and broader for larger ones. It is likely
therefore, that the effective radius of the shearing zone from the centre of rotation, \( R_{eff} \), is increasing with increasing particle size. This behaviour may directly influences the torque even at constant internal friction or shear rate in the particle bed and, change the radius of the rotating cylinder, \( R \), in the equation above. The change in the size of the radius could be mathematically depicted as, \( R_{eff} = [f(d)]^{1/2} R \), where \( f(d) \) is a particle size dependent function having the overall form shown in Fig. 4. This may give some explanation for the results obtained in the present study and would suggest a more complex relationship of the torque that in this case would depend also on particle size in addition to the parameters of equation (1). A more detailed experimental study and new theoretical consideration are needed to get a firm basis of these observed phenomena.

4. Conclusions

A modified Couette-type shearing device having a relatively small rotating inner cylinder and a broad gap for particles was constructed and tested during this work to study the effect of particle size on the torque generated on the shaft of the DC driving motor. Using five different particle sizes of granulated sucrose particles from \( d = 0.3 \) to \( 1.4 \text{ mm} \) mean diameters, it was sown that torque values increased significantly as a function of mean particle size. This was also observed for sliding (axially slowly moving) particle beds and for polydisperse particle systems. Measurements under transient conditions i.e., with a bed of larger particles continuously replacing a bed of smaller particles, also confirmed the above results.

From these results, it can be concluded that this type of Couette-device can advantageously be used to measure the mean granule size in a continuously working granulator. Further technical questions have to be solved for the final construction of such a device. To elucidate the basic theoretical aspects referring to the distribution of particle velocities and shear forces along the shear region, as well as their relation to particle size, further experimental and theoretical studies have to be carried out.

5. Nomenclature

d – particle size, \( \text{mm} \)
M – torque value (with particles), \( \text{Nm} \)
\( M_0 \) – preset torque value (without particles), \( \text{Nm} \)
n – rotational speed, \( \text{rpm} \)
t – time, \( \text{sec} \)
\( v_p \) – peripheral velocity of rotated cylinder, \( \text{m/s} \)
\( v_s \) – sliding velocity, \( \text{m/s} \)

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