

Particle size control by torque measurements during drying and granulation from solutions

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Abstract

The fluidized bed process presented in this paper is suitable to recover solids from solution in form of granules. Solution is sprayed into the bed and, as the 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 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. In this paper, the results of torque measurements and their correlation with particle size are presented.

Key words: granular flows, torque, Cuette-device, particle size

1. Introduction

A special fluidized bed process developed by Hungarian researchers [1] serves to recover solids in form of granules, directly from solutions or suspensions. An initial charge of particles chemically similar to the material being 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 kept constant 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 the City College of the City University of New York (CCNY) and the Research Institute of Chemical and Process Engineering, Hungary. The work was supported by the US – Hungarian Science & Technology Joint Fund and by the Hungarian National Foundation for Fundamental Researches. 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 depend on particle size. The group at CCNY has carried out experiments by a Cuette-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 Cuette-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 optical

microscope. Angle of repose and flowability values were measured according to ASTM methods. Bulk densities were determined by using a graduated cylinder.

The experimental device consisted of an inner, rotating cylinder and an outer stationary tube. Both were covered by coarse sandpaper 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 $v_p=0,16-0,65$ m/s) by an electronically controlled DC motor. The torque on the shaft was continuously measured and recorded by an electronic data acquisition system and a PC. The maximum measurable torque was 0,44 N-m. 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 torque signals, which can be evaluated most easily, 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 particles motion in this case was set at a velocity of $v_s=0,0001$ 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 mm , while the rotating cylinder was only 80 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 particle bed in the modified Cuette-device was lower, and both 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 $12,7 \text{ mm}$). For this reason, the shear region never penetrated to the standing cylinder across the whole shearing gap, also observable by eye.

3. Results and discussion

The curves shown in *Fig.3.* represent the results of torque measurements obtained for five different mean particle sizes ($d=0.3, 0.52, 0.72, 0.9, 1.43 \text{ mm}$ respectively) of granulated sucrose particles with narrow size distribution. The sixth lowest 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 force, which was necessary to adjust the basis 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).

Using different rotation speeds ($n=80 \text{ and } 120 \text{ rpm}$), the torque was observed to increase at increasing particle size as seen in *Fig.4* for three different preset break values ($M_o=0.043, 0.057 \text{ and } 0.092 \text{ N-m}$). 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, 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 $d=0.8-0.9 \text{ mm}$ granule diameter. Above that size, the dependence seems to be 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 $v_s=0.0001\text{m/s}$, obtained by a mass flow of particles of $2.0\text{-}2.2\text{ kg/h}$, was chosen to be negligibly small as compared to the peripheral velocity of rotation, (this latter being $v_p= 0.22\text{ m/s}$). 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 torque 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 $M= 0.297\text{ N-m}$ in the static and $M=0.299\text{ N-m}$ in the sliding bed. This is the 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 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 particle systems with narrow size distribution shown in *Fig.5*. The mean particle sizes were $d=0,45\text{ mm}$ and $1,1\text{ mm}$, 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.*, $d=0,49\text{ mm}$ and $0,92\text{ mm}$ 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 $d=0.4\text{-}0.6\text{ mm}$ size distribution were initially filled into the fluidized bed. The modified Cuette-type device (see *Fig.2*) was connected to the granulator, where larger particles with $d=0.8\text{-}1.0\text{ mm}$ size distribution were continuously introduced. A screw feeder as shown in *Fig.2* ensured the flow of particle through the Cuette-device with $v_s=0.0001\text{ m/s}$ axial velocity. Torque was continuously measured and recorded. Mean particle sizes were calculated from these measured values and plotted in *Fig. 7*.

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_q :

$$M_q = \pi \rho_B g L^2 R^2 \sin \phi \left[1 - \frac{U}{U_{mf}} \right] \quad (1)$$

This Equation shows quadratic dependence in function of the height of the sheared layer L and on the radius of the inner cylinder R .

Linear dependence was found on bulk density ρ_B , on the inner friction coefficient of the particle bed, $\sin \Phi$, and on gas velocity U , relative to the minimum fluidization velocity U_{mf} . No direct relation was shown by this equation with the particle diameter.

This Equation was obtained for shearing particles at and below the 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, independently 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 shear zone, observable from above 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 therefore most likely that the effective radius of the shearing zone measured from the center of rotation R_{eff} is increasing with increasing particle size. This behavior may influence the torque even at constant internal friction or shear rate in the particle bed and change the radius of rotation. Therefore, it looks to be reasonable to apply a term R_{eff} instead of the radius R of the rotating cylinder. The change of the radius of the rotation could be mathematically described as $R_{eff} = [f(d)]^{1/2} R$, where $f(d)$ is a function depending on particle size. This may be

an explanation to the results obtained in the present study. Function $f(d)$ can be a complex relationship, which is not cleared yet. Thus, the torque depends also on particle size in addition to the parameters of Equation 1. However, to clear up this dependence, more detailed experimental study, as well as further theoretical consideration are needed to get a firm basis of these observed phenomena.

4. Conclusions

A modified Cuette-type shearing device with a relatively small rotating inner cylinder and broader gap for particles was constructed. During this work, it was tested 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 mm mean diameters, it was shown that the measured torque values increased significantly as a function of mean particle size. This relation was also observed for sliding particle beds (moving axially at low velocity) and for particle systems with considerably broader particle size distribution. Experiments under transient conditions, i.e. in a bed where smaller particles were gradually replaced by larger ones, also confirmed the applicability of this torque measurement method to estimate the particle size in the bed.

From these results, it was concluded that this type of Cuette-device could be advantageously 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, *mm*
- M – torque value (with particles), *Nm*
- M₀ – preset torque value (without particles), *Nm*
- n – rotational speed, *rpm*

- t – time, *sec*
v_p – peripheral velocity of rotated cylinder, *m/s*
v_s – sliding velocity, *m/s*

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Table 1. Characteristics of the particles

	Sucrose size range mm	Sucrose mean particle size mm	Form of particles	Angle of repose	Flowability cm ³ /s	Bulk density kg/m ³
1	0,2-0,4	0.3	prismatic*	39.0	13.3	740
2	0,4-0,63	0.5	prismatic*	39.1	12.8	760
3	0,63-0,8	0.7	rounded prism	39.8	11.9	770
4	0,8-1,0	0.9	near spherical	40.9	11	760
5	1,25-1,6	1.4	near spherical	40.9	8.9	790

* without sharp tips and edges

Figures

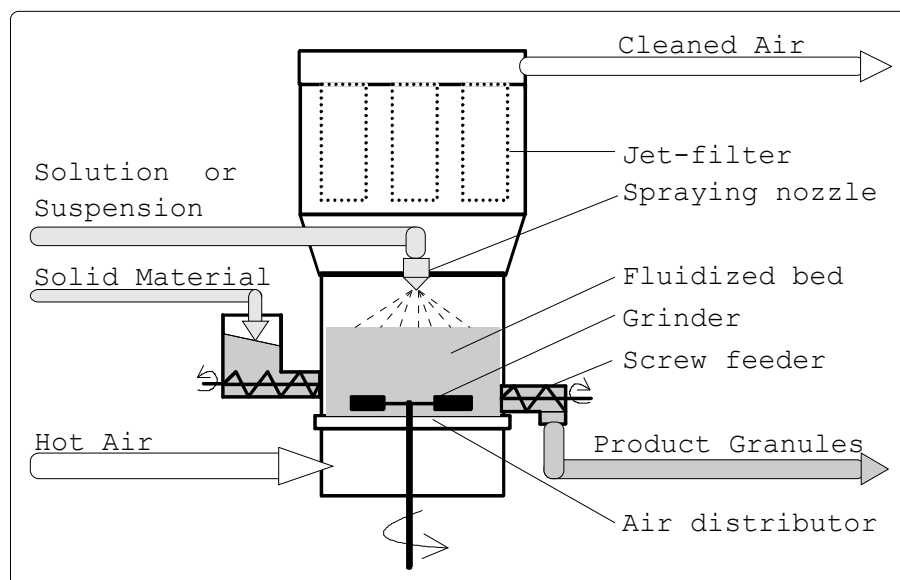


Figure 1. Fluidized bed drier and granulator

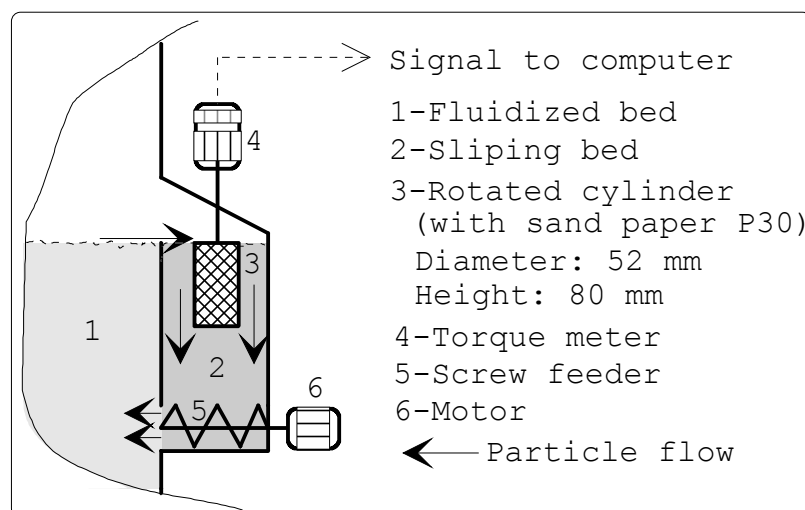


Fig. 2. Modified Couette-device connected to the fluidized bed granulator

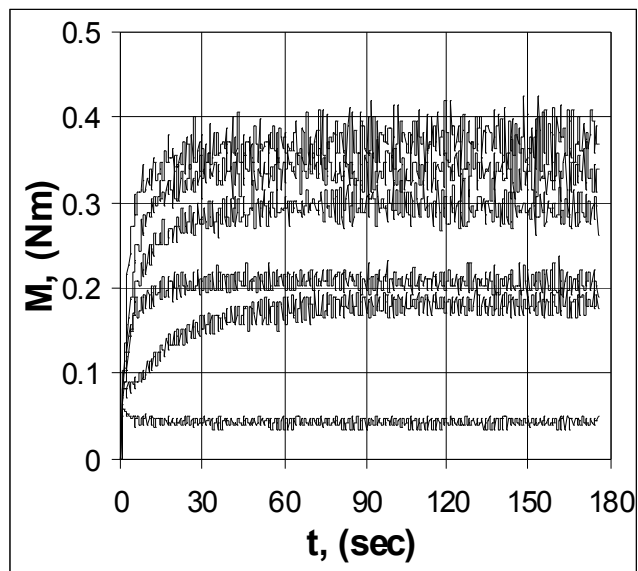


Figure 3. Torque vs. time

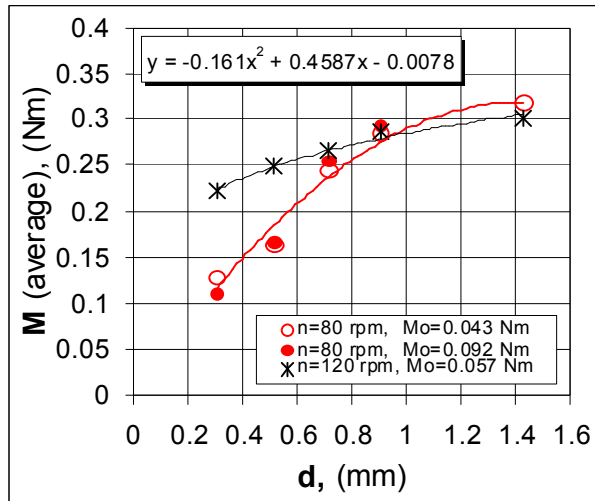


Figure 4. Mean particle size vs. average torque

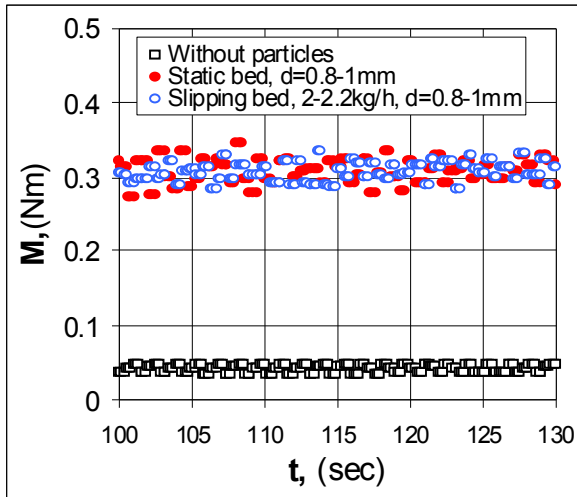


Figure 5. Torque vs. time in static and slipping beds

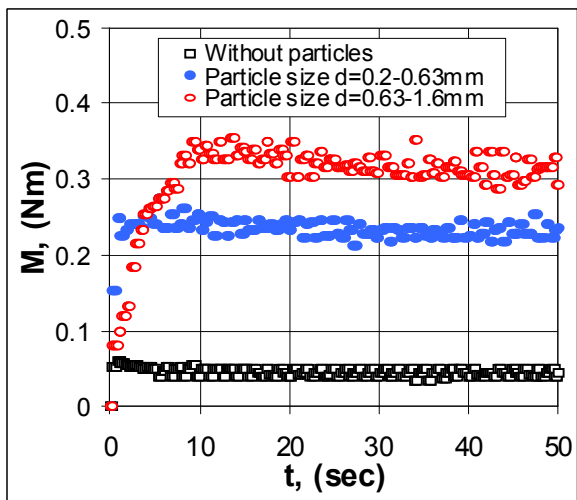


Figure 6. Torque vs. time at different particle size distributions

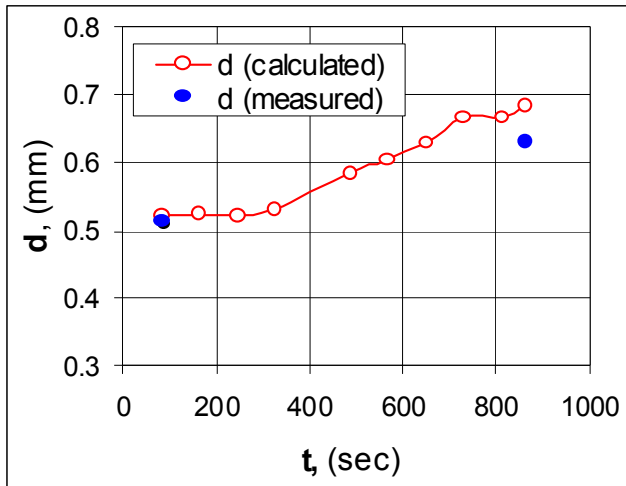


Figure 7. Average particle size calculated from torque values and measured by sieve analysis