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The 7th World Congress on Particle Technology (WCPT7)

Auto-granulation of fine cohesive powder by mechanical vibration

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Abstract

As the size of individual particles is reduced below several microns, the interparticle cohesive forces begin to play a major role in the bulk powder behavior. Fine powders generally exhibit poor flowability as well as an affinity to agglomerate and form clusters due to this cohesion. This clustering behavior of dry, binderless particles is known as auto-granulation and can often cause difficulties in processing and handling of powders. In this study, a titania powder is vibrated under controlled conditions to induce clustering and promote agglomerate growth. The amplitude and frequency of the mechanical vibration is varied to view the effect of the input energy on the equilibrium agglomerate size. Furthermore, the densities of the formed agglomerates are measured to investigate the role of consolidation as a mechanism of auto-granulation. Given that the size of the agglomerates formed by this auto-granulation process is affected by the balance between the cohesive energy of the particles and the disruptive energy of vibration, this work provides insight into the mechanism controlling the growth of these agglomerates to an equilibrium size.

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1. Introduction

A wide range of products are produced by ceramic powder processing, including electronics, structural materials, chemical processing components, and refractories [1]. Whenever dealing with a dry powder process, difficulties may occur when the individual particles within the powder have an affinity to clump or cluster together due to interparticle, attractive forces. The scale of this clustering is dependent on the particle properties and can range from the creation of relatively small agglomerates up to large ones [2]. If during the process, the dispersion of the powder into individual particles is necessary, enough force must be applied to deform and break the particle clusters. This deformation is again dependent on particle properties as the attractive force between particles must be overcome for separation to occur [3].

When dealing with the cohesion between two particles, the contributing factors, in addition to attractive surface forces, are particle size and shape, and the degree of compression. With the larger particle sizes, the interactions are dominated by gravitational forces, but with the smaller particles adhesion has a much larger role [4]. This is the reason fine particles tend to aggregate with one another, especially when the size of individual particles becomes smaller than several microns. At this scale, the attractive forces between particles become comparable to the gravitational forces pulling those particles apart [5].

This paper investigates the auto-granulation behavior of a powder, i.e. the formation and growth of clusters in a dry, fine powder bed under vibration. Due to the powder bed being dry and void of any binder fluid, the primary mechanism of the auto-granulation behavior is the bulk cohesion of the particles. The effect of the mechanical vibration on the size and density of the formed granules is presented.

Nomenclature

A	amplitude of sinusoidal motion
a	acceleration due to vibration
E	energy of vibration
f	frequency of sinusoidal motion
k	wave number of sinusoidal motion
m	mass of sample under sinusoidal motion

2. Powder granulation

Fine cohesive particles with relatively high interparticle attractive forces have a tendency to clump together or self-agglomerate to form granules. This behavior has been studied in an effort to model the size enlargement, or granulation, process of cohesive particles. Ennis *et al.* [2] approached the modeling work by viewing the causes of granulation in particles at a micro-level. In the studied system, the particles were coated with a viscous binder and the sticking of two colliding particles depended on two competing factors: the energy dissipation of the binder layer and the rebounding kinetic energy of the collision [2]. As the size of the colliding particle directly affects the energy of collision, this theory explains the snowball-effect of granulation. If a small particle collides with a stationary large granule, the kinetic energy will be low. This increases the likelihood of the binder dissipating the impact energy of the particle onto the granule. Conversely, if a large agglomerate or a granule collides with another granule, the kinetic energy will be relatively large, more likely resulting in rebound.

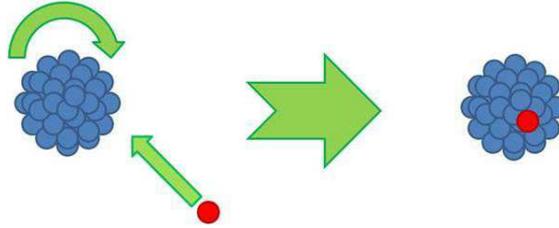


Fig. 1. Schematic of a fine particle attaching to a larger granule.

While the work by Ennis *et al.* [2] was primarily on granulation with a binder present in the system, the same premise can be applied to the binderless auto-granulation process, as the same two competing mechanisms of particle clustering and granule deformation occur in auto-granulation. Horio [5] has researched binderless granulation using the method of pressure swing granulation, where powder is both compacted and fluidized within a fluidized bed column by cyclically changing the direction and pressure of the airflow. This alternating compacting downward flow and upward fluidizing flow created granules with an inherent strength [6]. Analysis of the microstructure showed a core-rim structure, where the surface of the granule had a denser particle packing than the core. This led Horio [5] to consider that the motion of the granules around the chamber during the fluidization step leads to the surface deformation of the granules, increasing the particle packing. This compaction was a key step in the granules formation.

Agglomerate size prediction of a vibrated powder bed has been attempted using a force balance [7] and an energy balance approach [8]. Both have been shown to provide an accurate model for predicting agglomerate size in a vibro-fluidized bed. By balancing the cohesive forces/energies to the disruptive forces/energies of the vibration, previous models have shown an inverse relationship of vibration intensity with granule size [7,8]. The models presented are not applicable to mechanically vibrated powders though, with the absence of fluidizing air flow. Under only mechanical vibration, the mechanism of auto-granulation causes compaction of the particles within the granule, increasing the density and the strength of the granules. This compaction is negligible in the previous studies, but key for auto-granulation, and results in a very different behavior.

3. Experimental setup

3.1. Material system

For this study, the Cristal Global AT1 titania powder was used. This is a sulfate-processed titania with a d_{50} particle size of roughly 100 nm. Particle density was measured on a Micromeritics AccuPyc II 1340 and found to be 3952.6 kg/m^3 . Bulk powder flow properties were measured on a Schulze annular ring shear tester (RST-XS) and for major principal stresses ranging from 0.500 to 40 kPa, the average flow function for the powder was found to be 1.38. The cohesion of the powder over the range of major principal stresses is shown in Figure 2, with the powder increasing in cohesion with consolidation stress. As a precondition before mechanical vibration, the titania powder is sieved through a 1.4 mm sieve. This breaks up any large clusters in the powder that may be present due to powder storage and creates a uniform, fine powder bed for the start of the auto-granulation test. The mass of the powder bed was kept constant for all conditions at 15 g.

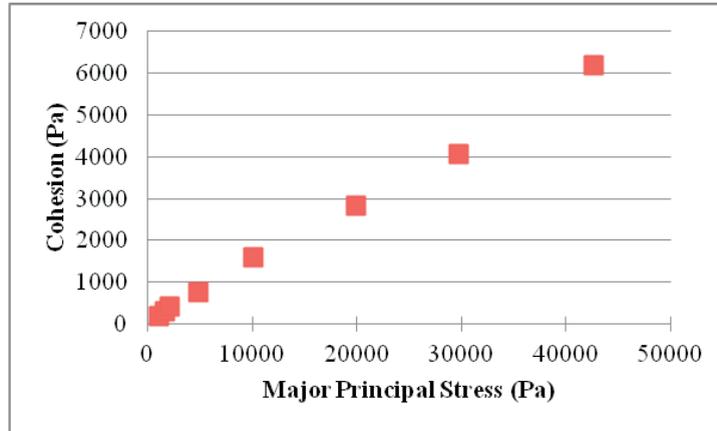


Fig. 2. Cohesion of titania powder measured by a Schulze annular shear cell.

3.2. Mechanical vibration

Mechanical vibration of the powder sample is achieved using an electrodynamic shaker (The Modal Shop Inc. K2007E01). An acrylic box with a side dimension of 60 mm to contain the powder sample is attached on top of the shaker. The shaker applies a vertical, sinusoidal motion to the powder bed with the amplitude and frequency of vibration being controlled for the test. Using simple harmonic motion, the energy of vibration, E , is calculated by Equation 1, where k is the wave number, A is the amplitude, m is the mass of the sample, and f is the frequency. The acceleration due to vibration, a , is calculated by Equation 2.

$$E = \frac{1}{2} k A^2 = 2m(\pi f A)^2 \quad (1)$$

(2)

$$a = A(2\pi f)^2$$

3.3. Granule characterization

The granules formed by mechanical vibration were tested to measure granule size and density. Size measurements were conducted by gently scattering the granules over a flat surface and allowing the granules to orient themselves on their plane of maximum stability. A high-resolution image was then taken of the granule population, with a pixel size of 0.058 mm. The pixel area of each granule was then measured and converted to a radius of a sphere of equivalent projected area. The entire population of collected granules at each vibration condition was imaged for sizing. Granule envelop density was measured using a Micromeritics GeoPyc 1360 to obtain the envelope density of the granules.

4. Results and discussion

4.1. Powder vibration

The various vibration conditions imparted on the powder to induce auto-granulation are shown in Table 1. For the first four test conditions, the amplitude was held constant with the frequency being altered. The last two test conditions had the frequency held constant at 50 Hz and the amplitude altered. This produced two vibration conditions with the vibration energy kept constant at 0.0073 J and two conditions with equal acceleration of 63.17 m/s².

Table 1. Vibration conditions of auto-granulation tests.

Test condition	Frequency(Hz)	Amplitude (mm)	Energy (J)	Acceleration (m/s ²)
1	35	1.00	0.0056	48.36
2	40	1.00	0.0073	63.17
3	45	1.00	0.0092	79.94
4	50	1.00	0.0114	98.70
5	50	0.80	0.0073	78.96
6	50	0.64	0.0046	63.17

Auto-granulation behavior becomes quickly evident in the fine powder bed, as shown in Figure 3. Within one minute, clusters form and grow within the fine powder bed to an easily observable size. For reference, the length of the container wall in Figure 3 is 60 mm. As expected with a snow-balling growth behavior, the granules continued to grow over time by consuming the finer particles within the powder bed at 5 minutes of vibration. The granules continue to grow until reaching an equilibrium maximum size limited by the vibration conditions. At the 50 Hz and 1.00 mm amplitude condition, granules were created and collected with vibration times of 5, 8, 11, 14, 17, and 20 minutes. These granules were then sized, with the d_{90} of the population shown in Figure 4. The data point at 20 minutes shows the average of 3 different runs, with the error bars giving the range of the measurements. The data show the granules size increases to an equilibrium value of about 1.5 mm after 14 minutes of vibration. As a result, for each vibration condition shown in Table 1, the time for vibration was kept constant at 20 minutes to ensure the granules have grown to their equilibrium size.

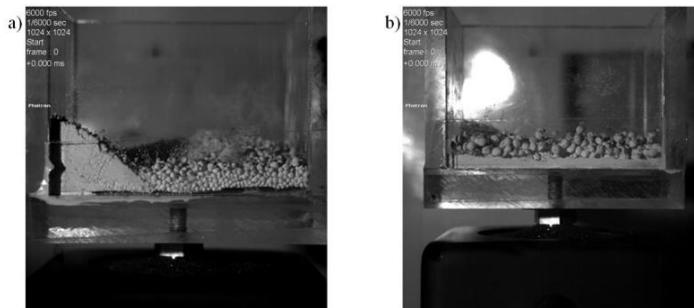


Fig. 3. High-speed imaging of powder under 50 Hz and 1.00 mm amplitude mechanical vibration after (a) 1 minute and (b) 5 minutes.

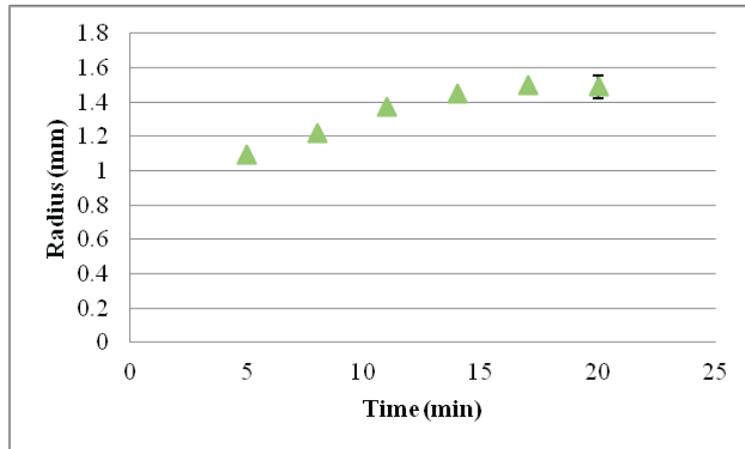


Fig. 4. The d_{90} of granule radius at 50 Hz and 1.00 mm as a function of vibration time.

4.2. Granule sizing

The entire population of granules produced due to auto-granulation behavior was imaged and sized. For each vibration condition, three repeats were made. The measured radius of the granules as a function of the vibration energy is shown in Figure 5, with the distribution of the radii represented by the d_{10} , d_{50} , and d_{90} . Each data point represents the average of the three measurements, with the error bars showing the range. The results show the d_{10} of the distribution remains relatively constant, but both the d_{50} and d_{90} increase with increasing vibration energy. This is in direct contrast to the behavior observed by Barletta *et al.* [7] and Zhou *et al.* [8], who modeled the behavior of a fluidized powder bed under vibration.

In Figure 6, the same granule radius data are shown as a function of vibrational acceleration. The critical values to note are the two data sets at vibration energies of 0.0073 J in Figure 5 and the two data sets at vibrational accelerations of 63.17 m/s^2 in Figure 6. These are run at separate vibration parameters, but the energy calculation given by Equation 1 and acceleration calculation given by Equation 2 are the same. The granule radii for the equal conditions are shown in Table 2. For vibrational energy, the equal data sets nearly overlap, with the difference in values being 0.01, 0.05, and 0.02 for the d_{10} , d_{50} , and d_{90} values, respectively. This confirms the controlling vibration parameter in relation to granule size is the vibrational energy input into the powder bed.

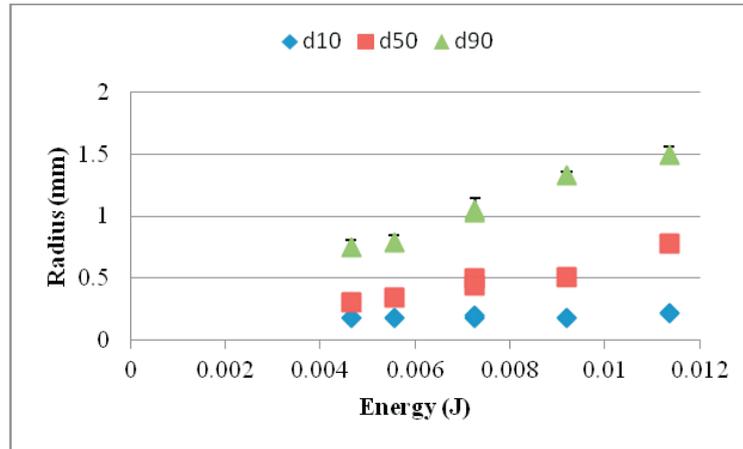


Fig. 5. Effect of vibrational energy on radius of granules produced by auto-granulation.

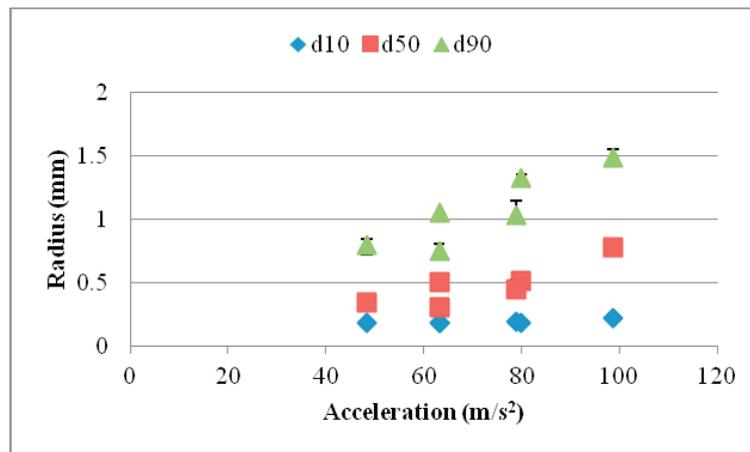


Fig. 6. Effect of vibrational acceleration on radius of granules produced by auto-granulation.

Table 2. Granule radius for vibration conditions with equal energy and equal acceleration.

Frequency(Hz)	Amplitude (mm)	Energy (J)	Acceleration (m/s ²)	Radius – d ₁₀ (mm)	Radius – d ₅₀ (mm)	Radius – d ₉₀ (mm)
40	1.00	0.0073	63.17	0.18	0.50	1.05
50	0.80	0.0073	78.96	0.19	0.45	1.03
50	0.64	0.0046	63.17	0.18	0.31	0.75

4.3. Granule density

For granule density measurements, a sample size of 30 granules from each test condition was used for measurement. The results, shown in Figure 7, have error bars representing the standard deviation of the data. The values show an increasing trend with vibration energy, correlating well with the granule sizing data. An increasing

density implies an increasing consolidation stress imparted on the powder. As shown Figure 2, the cohesion of the titania powder increases with increasing consolidation stress. Therefore, the powder within the granules created under higher vibration energies can be assumed to have a higher cohesion than the powder within the granules processed at lower energies.

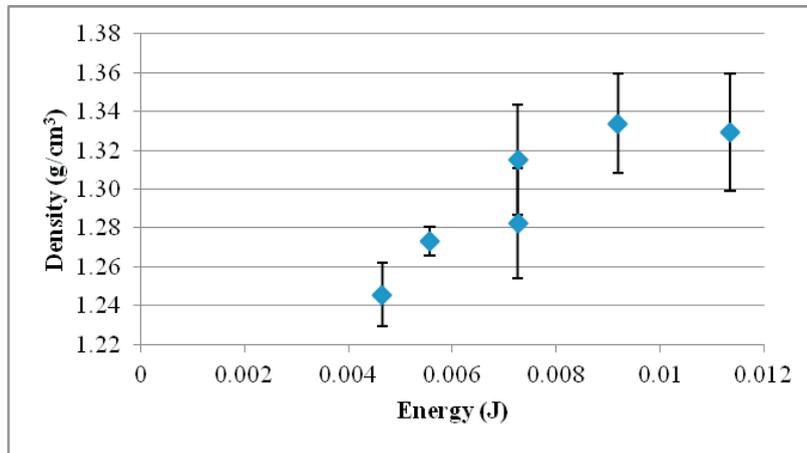


Fig. 7. Effect of vibrational energy on radius of granules produced by auto-granulation.

5. Conclusions

The work conducted showed that the Cristal Global AT1 titania powder exhibited a characteristic behavior known as auto-granulation. Auto-granulation is the snow-balling growth of particle clusters within a dry, fine powder bed due to the powder cohesion. These granules grow to an equilibrium maximum size over time dependent on the vibration conditions imparted on the powder bed. Both the granule size and density were found to increase with increasing vibrational energy. The increased granule density implies greater powder cohesion within the granule, which can explain the larger equilibrium granule size. This shows that the vibrational energy is not purely a disruptive force limiting the granule size, but consolidates the granules, increasing their internal cohesion and allows them to grow to a larger equilibrium size.

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