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Effect of mechanical vibration on the size and microstructure of titania granules produced by auto-granulation

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ABSTRACT

Auto-granulation is the growth of particle clusters of fine, cohesive powders due to agitation of the bed, such as mechanical vibration. This clustering occurs without the addition of any binder to the system and the granules reach an equilibrium size due to the balance between disruptive and adhesive forces experienced by the clusters during process operations. For this reason, it is important in powder processing to be able to characterize this behavior. In this study, a sub-micron titania powder is mechanically vibrated under controlled conditions to induce clustering and promote auto-granulation. The amplitude and frequency of the vibration are varied to view their effect on the equilibrium granule size. Furthermore, imaging of cross-sections of the granules is conducted to provide insight into the internal microstructure and measure the packing fraction of the constituent particles. It is found that under all vibrational conditions investigated the particles exhibit a core–rim microstructure.

1. Introduction

When dealing with fine powders, as the particle size is reduced, the attractive interaction between particles increases in significance, dominating over the effect of gravitational forces. This leads to an increase in powder cohesion with finer sizes, and promotes a behavior where the small particles cluster into larger agglomerates [1]. Powders that have a propensity to agglomerate can create problems in powder processing [2], such as causing heterogeneities in the powder structure which is undesirable, such as in extrusion [3]. This behavior of size enlargement is common in many powder processes, such as granulation [4,5]. For this reason, there is an interest to be able to understand how size enlargement of powders occurs under given conditions.

Auto-granulation is a granulation process that occurs within a fine powder without any additives. The powder clusters simply become loosely bound due to the highly cohesive nature of the inter-particle contacts. Ku et al. [6] found that auto-granulation can be induced in a powder bed by agitation using mechanical vibration. For a given vibration amplitude and frequency, the particles cluster to a maximum equilibrium size with an inherent strength. This equilibrium size is dependent on the mechanical vibration intensity and interparticle adhesion.

The process of how the powder forms clusters during auto-granulation is not completely understood. In traditional wet granulation, the theory at the micro-level involves two competing factors: the energy dissipation of the binder layer and the rebounding kinetic energy of the collision between two colliding particulate entities. The mechanism of growth occurs due to the coalescence of like-sized particulate units, such as particles-to-particles, agglomerates-to-agglomerates, or granules-to-granules [7]. This would result in a hierarchal microstructure, where the internal structure exhibits evidence of the multi-scale growth process. Such granules would be visibly comprised of clusters, with those clusters formed from smaller clusters. This results in a pattern that continually reduces in scale until small clusters comprised of individual particles are observed.

Without the presence of a binder layer, there is only inter-particle adhesion to dissipate the collision energy between two like-sized particulate units. Unless the adhesive properties of the particles are relatively large, the collision would most likely result in rebound [4]. The mechanism of growth during auto-granulation was proposed by Ku et al. [6] to be a snow-balling process, where fine particles are taken up by larger granules by sticking onto their surface. This particle-to-granule clustering occurs due to the mismatch in size and consequently the mismatch in mass, where the kinetic energy of the collision due to the fine particles would be insignificant as compared to the adhesive energy of the much larger granule. As a greater number of fine particles stick to the surface of the granule, the granule grows larger [6]. This snow-balling process would produce a homogeneous microstructure, as the granules would be comprised of a singular sized building block: the fine particles of the powder.

Similarly to auto-granulation, pressure swing granulation is also a binderless granulation process that produces granules with an inherent strength [8]. In pressure swing granulation, the powder is placed in a

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fluidized bed column and the air flow within the column is cyclically changed between a compacting, downward flow and fluidizing, upward flow through the bed [8]. The granules produced by this method exhibit a microstructure with a core–rim structure, where the surface of the granules has a denser particle packing than the core [9,10]. Horio [10] suggested that the motion of the granules around the chamber during the fluidization step leads to surface deformation of the granules, creating the denser outer shell. This denser outer shell also explains the inherent strength of the granules, as the increased packing fraction at the surface creates an increased mechanical strength [10].

Golchert et al. [11] investigated the effect of granule microstructure on the compressive strength of the granule. This study suggested that the breakage behavior is heavily dependent on the structure of the granule, with both the mechanism and extent of breakage experienced by the granule changing between samples. The propagation of cracks was shown to be dependent on the network of contacts between the particles within a granule [11]. Therefore, the ability to characterize the internal microstructure of a granule is of extreme importance to understand the granule strength.

Models to predict granule size in vibro-fluidized beds have been shown using both force balance [12] and energy balance [13] approaches. In a vibro-fluidized bed, the bulk powder is subjected to a fluidized air flow along with the mechanical vibration [12]. This creates a different environment to a simply mechanically vibrated powder bed, with the absence of fluidizing air flow. While studies have been made into the behavior of powder beds under mechanical vibration [14,15], none have observed or characterized auto-granulation behavior of the powder.

The main aim of this study is to determine the effect the mechanical vibration has on the auto-granulation behavior. This is achieved by evaluating the effect of the significant parameters of the mechanical vibration on the equilibrium granule size. Furthermore, the internal microstructure of the formed granules is analyzed to provide insight into the mechanism of granule growth.

2. Experimental setup and methodology

2.1. Material system

The powder sample used for this study was AT1 titanium dioxide powder, supplied by Cristal Global (Jeddah, Saudi Arabia). This powder is made of smooth, rounded primary particles with a size of roughly 100 nm, as shown in Fig. 1. The appearance of the powder is white and cohesive, with loose clusters easily forming during powder handling and storage. For each test, the mass of the powder used was kept constant at 15 g. To test the powder at a reproducible state, a precondition step was used, where the powder was sieved through a 1.4 mm (14 mesh) sieve to break up any large clusters before testing.

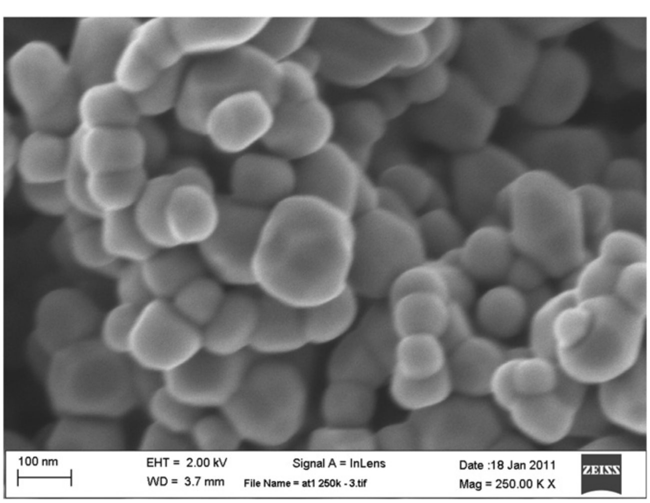

Fig. 1. SEM image showing particles of Cristal Global AT1 titania powder.

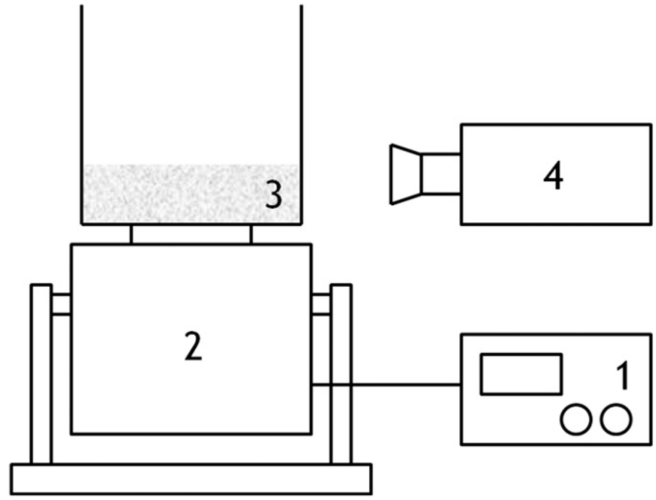

Fig. 2. Experimental setup showing the (1) signal generator, (2) electrodynamic shaker, (3) powder bed in acrylic container, and (4) high speed camera.

2.2. Mechanical vibration

An electrodynamic shaker (Model K2007E01, The Modal Shop Inc., Ohio, USA) was used to apply the mechanical vibration to the powder bed. A schematic diagram of the experimental setup is shown in Fig. 2. A signal generator (Model TG315, Thurlby Thandar Instruments Ltd., Huntingdon, UK) creates a digital, sinusoidal wave, which is converted to a mechanical vibration by the electrodynamic shaker. The powder is placed in an acrylic box with side dimensions of 60 mm, which is vibrated by the electrodynamic shaker in a vertical motion. The motion is monitored using a FASTCAM SA5 high speed camera (Photron, California, USA) to independently check the applied frequency and amplitude and to observe the auto-granulation process.

For this study, a range of vibration frequencies and amplitudes were used, as shown in Table 1. The amplitude and frequency of each test condition were converted to a vibration energy associated with one period, E, using simple harmonic motion (Eq. (1)).

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

where k is the wave number, A is the amplitude, m is the mass of the sample, and f is the frequency. The acceleration, a, and power, P, are given by Eqs. (2) and (3), respectively.

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

$$P = Ef = 2\mathrm{m}f^3(\pi\mathrm{A})^2 \quad (3)$$

Table 1
Test conditions for mechanical vibration of the powder.

Test condition	Frequency (Hz)	Amplitude (mm)	Energy (mJ)	Acceleration (m/s^2)	Power (W)
1	35	1.00	5.6	48.36	0.20
2	40	1.00	7.3	63.17	0.29
3	45	1.00	9.2	79.94	0.41
4	50	1.00	11.4	98.70	0.57
5	50	0.80	7.3	78.96	0.36
6	50	0.64	4.6	63.17	0.23