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### Breakage in granulation: A review

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#### Abstract

The study of breakage in granulation is important from a process and from a product quality perspective. Breakage is considered an important rate process in granulation, and plays roles in granule homogeneity and strength. Understanding this rate process has important implications in the design and control of the granulation process. From a product perspective, the study of breakage has important implications for the subsequent processing, transport, handling and final use of granular products. Breakage behaviour of granules can be a strong signature of the consistency of properties between nominally identical granular products. This paper reviews the study of breakage from the process scale down to the single granule and sub-granule scale, discussing largely experimental results complemented with some modelling results.

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#### 1. Introduction

The process of granulation is used in a wide range of industries, including mineral processing, agricultural products, detergents, pharmaceuticals, foodstuffs and speciality chemicals. Typically, fine powders are agglomerated together to form larger particles, or granules. In wet granulation, for example, liquid is used to stick the constituent particles together. Granules generally have a variety of advantages over fine powders in that they flow well, pose less environmental hazards, and dissolve or disperse better.

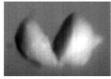
The process of granulation still remains relatively poorly understood. However, it is generally accepted that granulation is a combination of three rate processes, namely wetting and nucleation, consolidation and growth, and attrition and breakage (Iveson et al., 2001). In addition to the obvious growth retardation, attrition and breakage help to improve granule homogeneity (van den Dries et al., 2003) and granule strength by promoting consolidation. The importance of the study of granule breakage is in two principal areas.

Firstly, understanding breakage as a rate process and part of the granulation process allows improved process design and specification. Specifically, due to the importance of breakage in homogenising a batch of granules, improved knowledge of this process can lead to more controlled product quality. This leads onto the second role of improved understanding of granule breakage. Study of breakage of granules as products of the process can provide information of the behaviour of granules under further processing, handling and transporting conditions. In addition, deformation and breakage of granules can be used as a product quality tool to assess the granule properties. For example, it has been shown by Fu et al. (2004a) that the coefficient of restitution is very sensitive to variability in granule composition and structure.

This review paper covers the subject of breakage in granulation from a number of different length scale perspectives. At the process scale, breakage is important in enhancing the material distribution and eventual strength of the product granules. Knowledge of how operating parameters and equipment design influence the breakage process can help to improve the properties of the granular products. Knowledge of the true rates of breakage can improve modelling and prediction of granulation behaviour. At the single granule scale, extensive studies have been made to characterise

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(a) Binderless granule

ranule (b) Solid granule

(c) Wet granul

Fig. 1. Example of the typical impact fracture of the three generic types of granules under moderate impact conditions. These granules are between 4 and 5 mm in diameter: (a) binderless granule; (b) solid granule; (c) wet granule.

granule strength and behaviour under static and dynamic conditions. Understanding of how different variables effect the strength of granular materials again will assist in improving the properties of granular products. Also knowledge of breakage behaviour at the single particle scale can improve our understanding of breakage at the process scale. Sub-granule scale experimental studies can provide an understanding of how different variables and components contribute to the apparent granule strength, giving a physical basis for how to improve granule properties. In reviewing the modelling of granule breakage, a similar scale approach is adopted. Population balances are a powerful tool for modelling the influence of various rate processes on the properties of large groups of granules. Micro-mechanical modelling of granules allows further insight into the breakage behaviour of granules.

Granulation in itself is a broad topic. In this case a granule, or an agglomerate, refers to a body that consists of constituent particles held together. Here, we define three generic types of granules that will be discussed. Firstly, a binderless granule is described, whereby the constituent particles are held together by micro-scale forces, typically van der Waals forces. Secondly, a solid granule refers to a granule where the constituent particles are held together by solid bonds. Thirdly, a wet granule is described as a granule, which contains interstitial liquid. Although these three generic cases could all be described as granules, it is expected that they will exhibit different breakage behaviour due to the different nature of the constituent particle bonding forces. Fig. 1 illustrates the typical fragmentation of the three types of granules under moderate impact conditions.

In addition to different granule classifications, there are a wide range of practical processes to create granules, and a wide range of techniques to characterise the breakage of granules. It is not the aim of this review paper to be completely exhaustive in reporting all of these. At the process scale, principally, studies using high shear mixer granulators are reviewed and to a lesser extent fluidised beds. This is due to the increased perceived importance of the breakage process in these granulators and the focus of the literature on investigating breakage in this type of equipment, rather than any attempt at excluding other processes.

#### 2. Breakage at the process scale

Some of the early studies of attrition and breakage in the granulation process were carried out by Capes and Danckwerts (1965) and Sastry et al. (1977). They proposed that mechanisms by which granules grow in tumbling drum granulators involved *crushing and layering*. This mechanism is now generally considered as *attrition and breakage* (Ennis and Litster, 1997), and describes the breakage of wet or dried granules due to impact, wear or compaction in the granulator or during subsequent product handling.

In reviewing experimental studies of breakage during granulation processes, two broad groupings of research can be found. These are, firstly, studies of the process where breakage is inferred from observation of some ensemble property such as the temporal granule mean size, and secondly, studies where breakage is identified directly, often through addition of coloured dyes to create *tracer* granules, providing data from which breakage kinetics can be extracted.

#### 2.1. Observations of the breakage process

Knight et al. (2000) examined size enlargement of melt granules with time and impeller speed in a vertical axis high shear mixer. They found great variation in agglomeration behaviour with impeller speed. In particular, it was found that an increase in impeller speed exhibited an increase in the extent of granule growth. However, this pattern did not continue indefinitely, and at high impeller speeds, there was a noticeable reduction in the extent of granule growth (see Fig. 2). They found that granule size distributions were bimodal throughout the granulation process. It was argued that the bimodal distribution persisted for long times due to the breakage of large granules into small fragments. In addition, it was found that there was a considerable reduction in the fraction of relatively large granules above 1 mm at high impeller speeds. They deduced that these observations were evidence of a breakage process. They also observed a reduction in the size of the granules when increasing the mixer speed from 800 to 1500 rpm for 1 minute at the end of an 800 rpm batch (shown in Fig. 2). However, Iveson et al. (2001) argue that changes to the granule size distribution, on their own, are insufficient evidence for wet granule breakage. For example, an increase in impeller speed could contribute to an increase in rebound of colliding granules due to the increased impact velocities. This would lead to a reduction in the coalescence probability, although an increase in collision rates would also be likely. Knight et al. (2000) support their deduction through further analysis of the granule morphology. They found that low impeller speed granules exhibited high sphericity, whereas those from high impeller speed experiments have a more irregular shape, again consistent with a breakage process.

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