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Wet granule breakage in a breakage only high-hear mixer: Effect of formulation properties on breakage behaviour

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ABSTRACT

Wet granule breakage can occur in the granulation process, particularly in granulators with high agitation forces, such as high-shear mixers. In this paper, the granule breakage is studied in a breakage only high-shear mixer. Granule pellets made from different formulations with precisely controlled porosity and binder saturation were placed in a high-shear mixer in which the bulk medium is a non-granulating cohesive sand mixture. After subjecting the pellets to different mixing time in the granulator, the numbers of whole pellets without breakage are counted and taken as a measure of granule breakage. The experimental results showed that binder saturation, binder viscosity and surface tension as well as the primary powder size have significant influence on granule breakage behaviour. It is postulated that granule breakage is closely related to the granule yield strength, which can be calculated from a simple equation which includes both the capillary and viscous force of the liquid bridges in the granule. The Stokes deformation number calculated from the impact velocity and the granule dynamic strength gives a good prediction of whether the granule of certain formulation will break or not. The model is completely based on the physical properties of the formulations such as binder viscosity, surface tension, binder saturation, granule porosity and particle size as well as particle shape.

1. Introduction

In granulation process, it is generally accepted that three rate processes exist, namely wetting and nucleation, consolidation and growth, and attrition and breakage [1]. Although the process of granulation is still not completely understood, significant progress has been made on the understanding of granulation growth mechanism in the last decade or so. However, wet granule breakage in granulators is less well understood than either nucleation or growth. Understanding the breakage mechanism is very important as breakage of wet granules will influence and may control the final granule size distribution, especially in high-shear granulators. In some circumstances, breakage can be used to limit the maximum granule size or to help distribute a viscous binder. The study of wet granule breakage is also important from process and product quality perspective. Few investigators have described or studied wet granule breakage in granulation processes. Some preferential growth mechanisms in tumbling granulation may involve attrition or breakage of weak granules (crushing and layering, abrasion transfer) [2]. However, breakage is much more likely in higher intensity mixer and hybrid granulators. The limited work on wet granule breakage focuses on these processes.

Wet granule breakage has been identified clearly in high shear mixer experiments by other means. Among them colored tracer granules or liquid are the mostly used methods for identifying wet granule breakage [3,4]. Van den Dries [5] studied the influence of process and formulation variables on the granule breakage phenomena in a higher shear mixer and found that increasing binder viscosity and decreasing particle size increases granule strength. An extensive review on breakage in granulation is given by Reynolds et al. [6,7]. However, all the experimental work on granule breakage were carried out in granulators where both coalescence and breakage occur at the same time. This makes it difficult to separate the breakage mechanism from coalescence as both of them occur at the same time.

The objective of this work is to study the effect of formulation properties in a breakage only granulator so as to better understand the breakage mechanism. The design and methodology for the breakage only granulator is described and the effect of binder saturation, binder viscosity and primary particles size on wet granule breakage is reported.

2. Experimental

2.1. Materials

Glass ballotini with two different size ranges, −63+45 μm and −20 μm as well as broad size distribution lactose were used as the primary particles. The size distributions of the −20 μm glass ballotini

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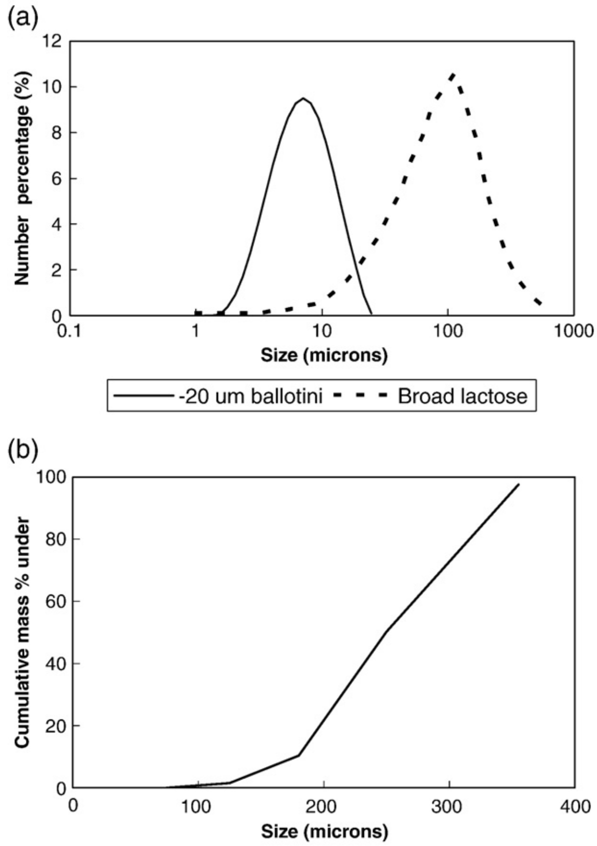

Fig. 1. (a) Size distribution of −20 μm glass ballotini and broad lactose (Malvern Master sizer). (b) Size distribution of the inert sand (sieve analysis).

and the lactose are shown in Fig. 1a. The glass ballotini were from Mo-Sci Specialty Products, L.L.C. MoO, USA and were spherical in shape and easy to characterise. The broad lactose was from Wyndale Lactose Pty Ltd in New Zealand and was chosen as a non-spherical powder which is widely used in pharmaceutical industries. The aspect ratios of the powders from image analysis and other properties of the powders are listed in Table 1. The liquid binders used were water and silicone oils with different viscosities ranging from 0.1 Pas to 30 Pas (Sigma Aldrich). The characteristics of binders are listed in Table 2. The formulations used for the breakage experiments were listed in Table 3.

2.2. Granule pellet preparation

Cylindrical pellets of 5 mm in diameter and 5 mm in height were made in a stainless-steel press. Firstly a known amount of solid and binder were kneaded together in a plastic bag until the binder was evenly distributed throughout the powder. The amount of material necessary to give pellets of 40% porosity at each binder saturation value was calculated (0.1 to 0.14 g for broad lactose and 0.15 to 0.19 g for glass ballotini) and was loaded into the chamber of the press. The ram was placed in position and pushed down until it was tightly up against a 5 mm spacer. Then the load was released and the pellet gently pushed out. The liquid contents of some water bound pellets were measured and was found to be very close to the liquid contents of the bulk powders, indicating that the mixing of powder and binder was uniform. Fig. 2a and b shows a typical example of the pellets made from the press.

Although the pellets were very small, they were not necessarily of uniform porosity. It is expected that particles near the moving ram are packed more tightly than those at the stationary end which are partially protected by the friction of the powder against the press wall. Thus the calculated pellet porosity is an average value only. The pellets formed are however, rather uniform in appearance and no cracks were observed on the pellet surfaces in most cases. Twenty pellets were made for each experiment.

2.3. Breakage experiments

An industrial food processor (Hobart FP62) was used as the laboratory scale high-shear mixer. The Hobart is a vertical axis granulator with a stainless steel bowl of 300 mm in diameter and 140 mm in height, and a nominal 6 L volume. The impeller in this work consisted of two 11° bevelled blades (see Fig. 3). The blade geometry was chosen to be similar to impellers found in typical Hobart high-shear mixer granulators. All the experiments were conducted with an impeller speed of 500 rpm. At this speed a fully steady state toroidal or roping behaviour was always observed.

To study the breakage mechanism alone, a bed of 2 kg of an inert mixture of sand and 48.4 g of 100 mPas silicone oil was added to act as a carrier bed for the twenty granules. The dry sand size distribution is shown in Fig. 1b. The flow properties of the bed was measured using a Shultz shear cell and are given in Table 4 with dry sand properties shown for comparison. This sticky sand bed was chosen as a model for the cohesive wet mass in a real granulator. The sticky sand more realistically represents the transfer force from the impeller blades and transmits stress through the powder bed for a cohesive granulating wet mass. However, the sticky sand bed does not agglomerate during mixing. Therefore the properties of the bed do not change with time as happens during granulation. Each bed of sand was used for three consecutive experiments.

At the start of each experiment, 20 pellets of the desired formulation were added to the sticky sand bed in the mixer. At 15 second intervals, the mixer was stopped and the contents of the bowl was hand sieved through a 3.38 mm sieve to remove granule survivors. Survivors were defined as granules which stayed above the 3.38 mm sieve. During the experiment, only whole granules were counted as survivors. Any half-sized or smaller granules were

Table 1
Properties of primary powders

Materials	Density (kg/m^3)	$D_{3,2}$ (μm)	$d_{4,3}$ (μm)	Aspect ratio
−63+45 μm Glass Ballotini	2524.9	54.9	58.4	0.98
−20 μm Glass Ballotini	2572.3	5.9	7.8	0.98
Broad Lactose — 100 mesh	1562.3	42.2	103.3	0.62

Table 2
Liquid properties

Binder	Density (g/ml)	Viscosity (PaS)	Surface tension (N/m)
Water	0.997	0.0009	0.072
0.1 PaS silicone oil	0.968	0.10	0.0212
1 PaS Silicone oil	1.09	1.0	0.0215
30 PaS silicone oil	1.1	30.0	0.0215

The silicone oil properties are from Sigma-Aldrich and the water properties are from Perry's chemical engineering handbook [8].

Table 3
Formulations used in the experiments

Powder	Binder type	Binder saturation
−63+45 μm glass ballotini	water	0.3, 0.5, 0.6, 0.8, 1.0
	1 PaS silicone oil	0.3, 0.5, 0.6, 0.8, 1.0
Broad lactose	1 PaS silicon oil	0.3, 0.5, 0.6, 0.8, 1.0
−20 μm glass ballotini	Water	0.3, 0.5
	0.1 PaS silicone oil	0.3
	1 PaS silicone oil	0.3, 0.5, 0.6
	30 PaS silicone oil	0.3, 0.5, 0.6*

*For −20 mm ballotini at saturation values higher than 0.6, the pellet becomes deformed when pushed out due to sticking on the press wall.