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A novel nucleation apparatus for regime separated granulation

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Abstract

A novel nucleation apparatus is presented for the production of narrow sized nuclei from various powder and binder liquid combinations. Mono-sized binder liquid droplets are produced by a specially designed mono-disperse droplet generator. The droplet generator is positioned above a conveyor belt, transporting a powder bed through the spray zone of the droplet generator. By nucleating powder on a conveyer belt, the nucleation mechanism is completely separated from all other granulation mechanisms due to the lack of relative motion between primary particles and/or formed nuclei. Nucleation tests were performed using chalcopyrite and limestone powders with water as the binder liquid. At all operating conditions, the formed nuclei were found to originate from multiplicities of drops that merged on the powder bed surface. Investigation of the dynamics of nuclei formation showed that powder-binder liquid combinations with fast penetration dynamics result in less variation in the number of droplets from which nuclei originate. Smaller and more narrowly distributed nuclei were also achieved by increasing powder speed through the spray zone.

Keywords: Mono-sized drops; Regime separated granulation; Nucleation; Granulation; Mono-sized granules

1. Introduction

Granulation and control of granulation processes have become increasingly significant in a range of formulation industries, including pharmaceuticals, agricultural chemicals, mineral processing, food technology and detergents. Granulation of powders is generally desirable to assist with dust minimization, handling and storage and more importantly, the delivery of active ingredients in pharmaceuticals and detergents. Granulation is defined as agglomeration by agitation in the presence of a binder liquid. Thus for granulation to occur there is generally some form of agitation of a powder, during and after the addition of a liquid binder. Current well-known granulation processes include the use of fluidized beds, flat bed, pan or disc, drum and mixer granulators, all of which use agitation to encourage granulation. The desired results of granulation processes are to produce granules with the required physical properties and with a narrow size range. Current granulators produce granules with a wide size distribution resulting in segregation and large recycle streams of granules outside the required size range. Further, due to the complex internal mechanisms, current granulators are difficult to design and to scale up rationally [1]. Ennis and Litster [2] separated the internal granulation mechanisms in three rate processes, these being wetting and nucleation, consolidation and coalescence and breakage and attrition.

Extending this idea, we propose the concept of a regime separated granulator in which key regimes are physically separated so that they can be controlled to a much greater extent than is now possible. Fig. 1 illustrates this concept. Most combinations of desired product qualities could be achieved in two-stage granulation where the first stage gives controlled nuclei formation and the second stage gives controlled growth and consolidation. A combination drop controlled nucleation and layered growth gives potential for very narrow granule size distributions.

Hapgood and co-workers [3,4] demonstrated the critical importance of the nucleation and liquid binder distribution process in determining the size and spread of the final granule size distribution. They described several nucleation regimes, including the drop controlled regime in which there is a one to one correspondence between the drop size distribution and the granule size distribution. A key parameter in determining the nucleation regime was the dimensionless spray flux. Their work helped quantify and explain earlier experimental studies on the

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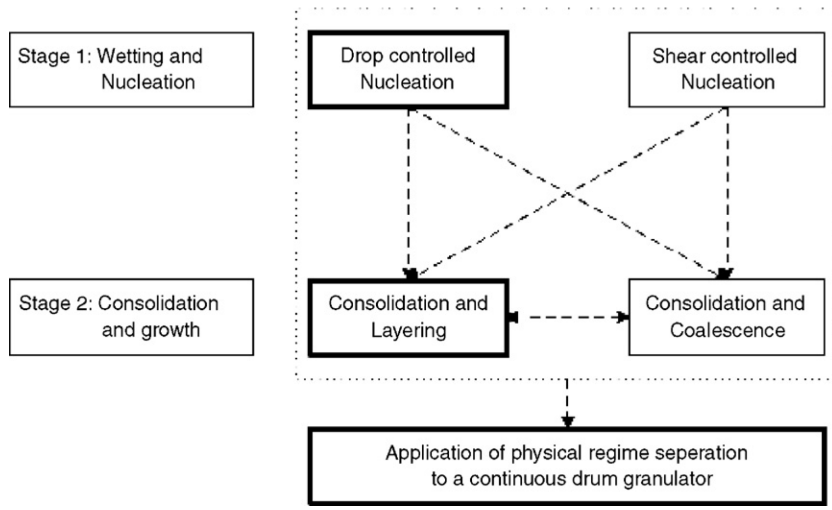

Fig. 1. Concept of regime separated granulation.

effect of powder flux and spray nozzle parameters on granule size distributions [5–7].

Monosized drops can be produced from a showerhead arrangement using an acoustically vibrated jet [8]. Schaafsma et al. [9] used a similar approach with a single vibrated nozzle in fluidized bed granulation. In this paper, we completely separate the nucleation zone of a granulator by passing a thin powder layer on a conveyor belt, an approach described qualitatively by Rumpf [10] but which has received little attention since. By using a monosized drop generator as the spray nozzle and independent control of the powder speed on the belt, it should be possible to guarantee an extremely narrow nuclei granule size distribution.

2. Theory

A liquid flowing through a circular orifice may drip or form a laminar, wavy or turbulent jet, depending on the nozzle flow [11,12]. The disintegration of a laminar liquid jet is sensitive to natural as well as imposed disturbances. Natural disturbances may exist out of all sorts of background noise such as velocity relaxation, whereas imposed disturbances may consist of mechanical vibration or electrical forces [13]. Investigations by Sakai et al. [13] show that for low viscosity liquids, the surface waves imposed by disturbances grow following the Rayleigh equation [14,15]. Weber [16] generalized Rayleigh's linear stability analysis to the case of viscous fluids. He also considered the effects of air drag on the moving jet at high jet velocities. Drop formation using high viscosity liquids has been extensively studied by Goedde and Yuen [17].

For any disturbance to result in breakup of a laminar cylindrical jet of low viscosity into droplets, Plateau [18] found that the disturbance wavelength λ has to be larger than the circumference of the liquid jet.

$$\lambda \geq \pi d_j \quad (1)$$

This restriction can be expressed in terms of the dimensionless wave number:

$$ka \leq 1 \quad (2)$$

where a is the jet radius and $k=2\pi/\lambda$. Therefore:

$$ka = \frac{\pi d_j f}{u_j} \quad (3)$$

in which d_j, f and u_j are the jet diameter, disturbance frequency and jet velocity respectively. In the literature, the jet radius, diameter and velocity in Eq. (3) are often substituted by the internal diameter d of the nozzle and the average liquid velocity u in the nozzle. The same simplifications are made here.

The stability of the production of mono-sized drops by imposed disturbances is further affected by the operating conditions and physical properties of the liquid. Sakai and Hoshina [19] provide an in-depth experimental study of the effects of these conditions and properties on the stability of mono-sized drop formation. Four empirical relations were determined marking the boundaries of an operational envelope

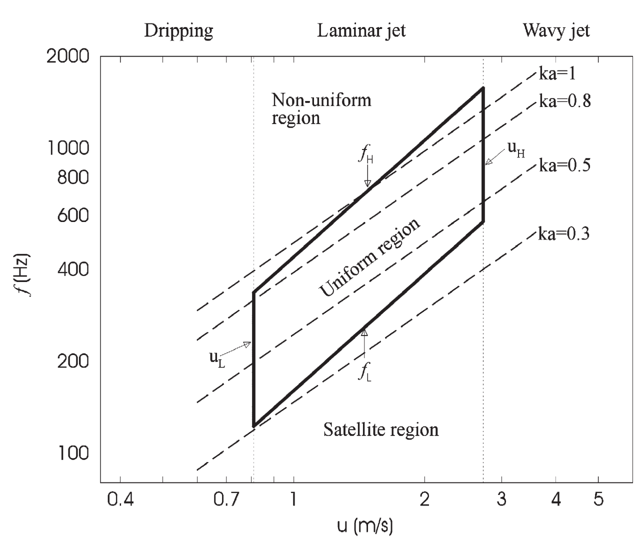

Fig. 2. Stability region for mono-sized droplet formation according to Sakai and Hoshino [19].