Particuology 7 (2009) 408-413

Contents lists available at ScienceDirect

Particuology

journal homepage: www.elsevier.com/locate/partic

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### Variation of granule mass fraction with coordination number in wet granulation process

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#### A R T I C L E I N F O

Article history: Received 7 September 2008 Accepted 15 July 2009

*Keywords:* Granules Primary particle Coordination number

#### ABSTRACT

In granulation, fine particles combine to form a coarse granule in the form of a particle matrix partially or fully saturated with a binder liquid. The final product of granulation possesses a wide variety of granule size distributions with surface mean diameters which differ with operating conditions. The final granule size depends on the operating conditions, e.g. operating gas velocity, inlet air temperature, initial feed particle size, and viscosity of the binder. The objective of this paper is to find out the uniformity in the relation between the granule mass fraction in the final granule size distribution and the number of feed particles present in the granules. The total number of granules obtained depends on the experimental conditions but the granule mass fraction and the number of feed particles forming a single granule are independent of operating variables, feed material and method of granulation. The paper purports further to compare the uniform nature of mass fraction of the granules in final granule size distribution and the primary particles required to form that particular granule size irrespective of experimental conditions of granulation.

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#### 1. Introduction

Granulation is a process by which fine particles combine to form coarse particles. It is a key process in various industries like pharmaceutical, food processing, detergent, fertilizers, etc. Granulation process is carried out in several ways depending upon the product required like drum granulation, fluidized bed granulation, rotor granulation, etc. In respect to addition of liquid the granulation process is divided into two types, wet granulation and dry granulation. In dry granulation, no liquid is added, the dry powders are compressed and form granules/tablets by sintering. Dry granulation process is mostly applied in pharmaceutical preparations. In wet granulation process fines are stuck together by combining liquid bridges among all the primary particles. Wet granulation process is further divided into two types: (i) Layering and (ii) Agglomeration. Layering is that type of granule formation process where binder solution is deposited on the entire surface of the core particle and the solute part remains on the core particle as an onion layer after evaporation of solvent part. This type of single particle growth is called layered growth. Agglomeration is multi-particle growth that results from adhering of small particles by forming liquid or solid

bridges between them, leading to the formation of larger particles called "agglomerates". Wet granulation process occurs in three stages—mixing of dry particles, addition of binder liquid and mixing of fine particles with the binder solution and formation of wet granules, the last and final stage is to drive off the liquid by supplying hot air and formation of dry granules from wet granules. According to Iveson, Litster, Hapgood, and Ennis (2001) there are fundamentally three rate steps determining wet granulation mechanism:

- wetting and nucleation,
- consolidation and growth,
- breakage and attrition.

These are shown schematically in Fig. 1 and often take place simultaneously in the granulation equipment.

After the granulation is performed, a wide distribution of granule size is obtained from which an average size of granules, i.e. surface mean diameter of the granules is obtained. The granules obtained are of various sizes, formed by different possible combinations of primary particles. The size and number of granules formed are varied due to different experimental conditions. Adetayo, Litster, and Desai (1993), Iveson, Litster and Ennis (1996), Iveson and Litster (1998a, 1998b) have studied the mechanism and growth kinetics of granule formation although the process is not well understood and difficult to control. The significant analysis of



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<sup>1674-2001/\$ –</sup> see front matter © 2009 Chinese Society of Particuology and Institute of Process Engineering, Chinese Academy of Sciences. Published by Elsevier B.V. All rights reserved. doi:10.1016/j.partic.2009.07.001



Fig. 1. Wet agglomeration process. (a) Wetting and nucleation, (b) consolidation and growth, and (c) breakage and attrition.

final particle size distribution is also not carried out. In this paper, an attempt is being made to bring out the uniformity in the relation between the granule mass fraction in the final granule size distribution and number of feed particles which are forming these granules. To establish this uniformity the granulation experiments have been conducted at different operating conditions, which lead to formation of a wide variety of granule size (Hemati, Cherif, Saleh, & Pont, 2003; Pont, Saleh, Steinmetz, & Hémati, 2001; Smith & Nienow, 1983a, 1983b). The purpose of present work is to show that in spite of adopting different experimental conditions the variation of mass fraction of a particular granule size ( $N_{Gi}$ ) with number of feed particles forming that granule ( $N_i$ ) shows a uniform behaviour.

## 2. Concept of coordination number and its application in granulation

The granules formed during the agglomeration process can be viewed as the arrangement of particles in a structured packing matrix. Thus the number of particles involved in this formation is an important parameter deciding the size of the granules. The number of contact points on a particle is defined as coordination number (Roux, 2004; Suzuki & Oshima, 1983a, 1983b; Suzuki & Oshima, 1985a, 1985b). It is one of the important parameters used to specify the particle arrangement in a randomly packed collection of particles as exhibited by granules formed during granulation process.

The concept of coordination number is useful in ascertaining the intra-granular porosity and in determining the final granule size. The higher the coordination number the lesser the spacing between the particles forming the granules will be and hence the closer the particles will be packed. The initial particle size which is varied during different experiments also has an impact on coordination number and hence the final granule size.

#### 3. Experimental set-up and details

The schematic diagram of experimental unit is shown in Fig. 2. Urea particles have been taken in a fluidized bed of 2.54 cm inner diameter and 30 cm height with a sintered plate distributor. The granular urea (average particle size approximately equal to 2 mm) is crushed and then sieved to prepare cuts for granulation studies. Three cuts of particles, with average size of 327.5 µm (maximum size 355 µm and minimum size 300 µm), 390 µm (maximum size  $425 \,\mu\text{m}$  and minimum size  $355 \,\mu\text{m}$ ) and  $512.5 \,\mu\text{m}$  (maximum size  $600 \,\mu\text{m}$  and minimum size  $425 \,\mu\text{m}$ ), are used in the granulation studies. Sieve analysis is used to measure average granule size after the granulation is completed. Saturated solution of urea (prepared at room temperature) is used as binder solution. A predetermined quantity of binder solution is sprayed on to the top surface of fluidized bed in one shot over a short period varving from 2 to 6 s. using a 1 mm diameter spray nozzle. The spray nozzle was positioned at about 1-2 cm above the fluidized bed surface to avoid binder solution drifting to the wall. The average droplet size of binder is 80 µm analyzed by image analysis, which is smaller than the feed particle size and hence uniformly sticks on the surface of the feed particles.

Granulation experiments were performed at different operating conditions using combination of the following parameters:

- (1) Initial particle size, *D*<sub>pi</sub>: 327.5, 390, 512.5 μm.
- (2) Operating velocity, U<sub>og</sub>: 65.8, 98.8, 131.7 cm/s.
- (3) Inlet air temperature,  $T_{gi}$ : 30, 37, 50 °C.



**Fig. 2.** Schematic diagram of the experimental set-up. (1) Blower, (2) rotameter, (3) silica gel tube, (4) heater, (5) fluidized bed, (6) pressure probe, (7) manometer, (8) and (9) thermocouple, and (10) sprayer.

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