

PARTICLE GROWTH AND AGGLOMERATION PROCESSES

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Contents

1. Introduction
 2. Product properties
 3. Rate processes
 - 3.1. Wetting and Nucleation
 - 3.2 Growth and consolidation
 - 3.3 Attrition and Breakage
 4. Regime maps
 - 4.1 Nucleation regime map
 - 4.2. Granulation regime map
 5. Population balance equations
 6. Granulation Equipment
 - 6.1. Drum granulation
 - 6.2. Pan granulation
 - 6.3. Fluidized bed granulation
 - 6.4. High Shear Mixer Granulators
 - 6.5. Dry Granulation
 - 6.5.1. Roll compaction
 - 6.5.2. Direct compression
 7. Equipment design
 8. Conclusion
- Glossary
Bibliography

Summary

Agglomeration is a size enlargement process used in the chemical process industries to impart better functionality to a product, thus avoiding common processing problems, and providing the consumer with a better product experience. Research into the controlling factors of this process is only in its infancy, requiring much further work to fully understand the methods and mechanisms of granulation. The article discusses the state of the art of granulation, from both a rate process and also equipment perspective.

1. Introduction

Agglomeration is an important process used in many industries to improve the characteristics of a material; giving benefits to the end user in the form of improved quality and function of the final product.

Many solid products are produced within the chemical industry and they are increasingly important owing to the shift of paradigm of production away from bulk base chemicals towards more highly specialized functional products, which add value through increased functionality and careful product design.

Unfortunately many of these chemicals are in solid form which proves a challenge for chemical engineers as it provides more problems over traditional liquid and gas based products due to fundamentally different governing physics. Many of problems stem from segregation of the final product where two solid components split from each other during transport leading to poorly distributed mixtures which can pose hazards to the quality of the produced product, and reduced performance in use. Problems also arise due to low bulk densities and difficult flow characteristics which make these powder products difficult to store and handle. These processing and material handling problems that occur within industry can be solved by agglomeration enabling, an increased bulk density product with a more free flowing nature and also less prone to segregation as individual constituents are held together in the formed agglomerate.

In addition to these problems agglomeration can act as a method to enhance a product, providing desired size distributions or product geometry for increased functionality, better consumer perception or protection of the end user from such hazards as dust and other possibly toxic or hazardous effects.

For these reasons many differing types of equipment have been developed to achieve these aims aiming to make, larger entities from powders, in which the individual primary particle are still visible, this process when carried out in a mixing device is usually described as granulation. Granulation and agglomeration processes tend to use a liquid or ductile solid (so called binder) to facilitate the build up of agglomerates.

Due to the wide range of industries and uses that granulation is used as a process for, ranging from fertilizer production through to fine chemical manufacture and pharmaceuticals and the differing scales of production ranging from kilograms/day to tones/hour there is a wide range of equipment designed for such uses, each with individual benefits and drawbacks. These devices are grouped together in Table 1.

| Method | Typical applications |
|--|--|
| Tumbling granulators - Drums - Discs | Fertilizers, iron ore, non-ferrous ore, agricultural chemicals |
| Mixer and Planetary granulators - Continuous high shear - Batch high shear | Chemicals, detergents, clays, carbon black pharmaceuticals, ceramics |
| Fluidized granulators - Fluidized beds - Spouted beds | Continuous: fertilizers, inorganic salts, detergents Batch: pharmaceuticals, agricultural chemicals, nuclear wastes |
| Centrifugal granulators | Pharmaceuticals, agricultural chemicals |

| | |
|---|--|
| Spray methods - Spray drying - Prilling | Instant foods, dyes, detergents, ceramics urea, ammonium nitrate |
| Compression agglomeration - Extrusion - Roll press - Tablet press - Moulding press - Pellet mill | Pharmaceuticals, catalysts, inorganic chemicals, organic chemicals, plastic preforms, metal parts, ceramics, clays, minerals, animal feeds |

Table 1: Agglomerator equipment designs and usual applications

Despite a large body of research on the subject there is still, as yet, no a-priori method of predicting granulation; there is as yet no method of knowing what will happen when a binder and powder are brought together within a specific mixer. Research continues around the world to develop this understanding in order to provide a predictive model of granulation.

As can be seen from Table 1 there is a significant number of granulation processes that involve ‘wet’ granulation (i.e. granulation with the use of a liquid binder) as opposed to more traditional ‘dry’ granulation techniques which use the ductability of a solid in order to produce agglomerates from the process.

2. Product properties

Agglomerated products are usually made to a specification that requires some knowledge of the use/ functionality of the material required. Many different “properties” can be used within industry to measure the quality of produced granules. Some examples of properties used are:

- **Size Distribution** – The size of the granules produced and the range that these sizes occur in. Generally it is expected that a narrow size distribution is best, as it likely leads to better batch homogeneity but can also be specified to be a certain width as post processing compression options are usually enhanced by a wider size distribution as a wider distribution is found to pack and compress better, leading to improved tablets.
- **Porosity** – The amount of free ‘air’ space within the granule. – The amount of air within the granule is significant as it provides for better dissolution ability, however usually at the trade off individual granule strength.
- **Attritional ability/ Dusting behavior** – The ability of the granule to loose material, producing constituent powder again. This is a problem as during packaging and transport significant attrition can occur, negating the use of agglomeration processes in the first place, leading to a product with a large amount of unintentional fines, and thus spoiling customer perception. The dusting behavior can also severely affect the safety of post-processing processes, leading to dust build up, provoking hazards of explosion and inhalation.
- **Dispersability** – Granules are usually designed to be dispersed into a medium as a method of use of the material, thus dispersability of the solid granules is of interest

and their time to break down is of interest within some industries.

- **Dissolution rate/time-** Dissolution rate and time are important factors, granules are usually produced to be dispersed in a medium, and thus are likely to be dissolved. These processes need to be carefully considered and the granules engineered for that particular case, as different final uses are likely to require significantly different dissolution times.
- **Strength-** As mentioned above individual granule strength is important for dusting and attritional behavior as well as for other post processing operations. It is generally assumed that there is a trade of between granule strength and dissolution rate.
- **Shape-** The shape of produced granules is of importance in some industries as it considerably influences bulk properties, such as flowability and processing behavior while also effecting customer perception.
- **Color-** the color of the produced product can influence customer perception
- **Homogeneity-** Homogeneity throughout the product, both between different batches and within the same batch is also of significance within the fine chemical and pharmaceutical industries, while spread of chemical throughout the individual granule is also of importance to many industries such as those of controlled release fertilizers.

Many of these factors are controlled by two primary factors, that of size and porosity. Controlling these is of fundamental importance in providing good quality product. The influence of granulator design on these properties is unknown, and is a subject of current research. It is known that each type of equipment, (Pan, Drum etc...) produces different properties of granule after set amount of time, careful selection of equipment to produce the desired "type" of granules is increasingly important, as differences between equipment and produced granule properties become more widely recognized, and quality of the produced agglomerates becomes more critical.

Measurement of these properties, usually results in a range of values between different granules tested. There is ongoing scientific debate about, both measuring the properties themselves, and then representing the produced results, to represent results are usually expressed in the form of a distribution, with the variable of interest on the abscissa and a measure of probability on the ordinate, with both cumulative and standard plots being acceptable.

3. Rate Processes

Wet granulation processes are of great interest to industrial manufactures of agglomerates owing to their ability to transform and mix dry powders for improved functionality; these methods have been described by combinations of many different idealized mechanisms, or so called rate processes that act to provide a basis for wet granulation process description.

- **Nucleation-** the binding together of particles to form a small granule. This mechanism reduces the number and mass of particles in the system while increasing the number and mass of granules.
- **Layering-** extra particles are bound to the surface of an existing granule. This action decreases the number and mass of individual primary particles in the system but

increases the mass of granules whilst leaving their number constant.

- Coalescence- where two granules successfully collide to form one granule. After collision, the two granules form a dumb-bell shaped intermediate. The collision is only successful if the net forces on the intermediate are sufficient to hold the newly formed granule together. Coalescence reduces the total number of granules but has no effect on their total mass.
- Abrasion transfer- occurs when material is transferred from the surface of one granule to another. This mechanism changes neither the total number nor the total mass of granules and very little work has been reported on abrasion transfer; its effects are often discounted.
- Crushing and layering- defined as the crushing of small granules by large granules into fragments with the subsequent adhesion of the fragments to the surface of the large granules. Decreasing the number of granules in the system, but not affecting their mass.

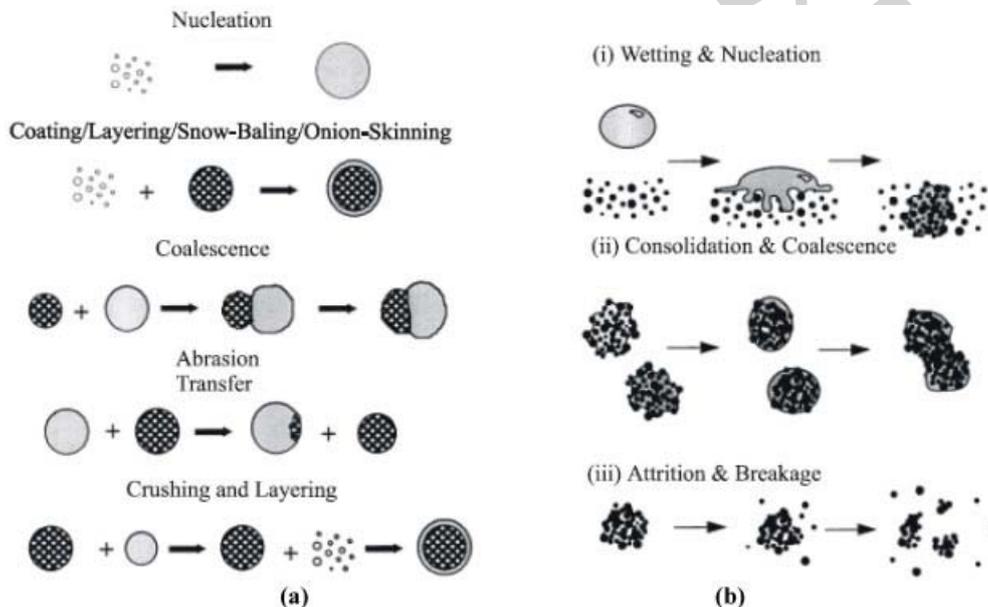


Figure 1: Schematic of granulation processes (a) traditional view Sastry and Fuerstenau (1973) *Mechanisms of agglomerate growth in green pelletization Powder Technology* 7 (2) 97, (b) modern approach Ennis (1996) *Agglomeration and size enlargement Session summary paper Powder Technology* 88 (3) 203-225

All the above mechanisms have been found to be present to some extent within a granulator, however, those which are more dominant and offer greater influence over final product properties, seem to be dependent on the type of granulator, and the input parameters both in terms of materials and operating conditions of the processes.

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