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Scale-up of detergent granules in a high shear mixer

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ABSTRACT

Wet granulation is a process where primary powder particles are made to adhere to form multi-particle entities called granules and this is achieved by using a binder. A simple scale-up of a granulation unit is rather difficult. This becomes even more difficult in a practical situation as the manufacturers make compromises in their scale-up rules on geometries due to cost reasons. Earlier work reported in literature on scale-up emphasized geometrical similarity and proposed various dimensionless numbers for scale-up. Recent work in this area describes granulation process as a multi-scale operation. The final product quality is determined by both macro-scale operation (like rotor speed in a high shear granulator) and micro-scale operation (like particle formation and interaction). Few researchers also talk about an intermediate approach between the macro and micro scales. In this approach, the key transformations such as binder distribution, nucleation, growth, consolidation and breakage were selected based on their relevance to the desired product attributes. The desired product attributes reported in an intermediate approach are chemical homogeneity, granule size, size distribution and granule density etc. There is little published literature on the scale-up of batch granulators in spite of their wide and varied use in the chemical process industries. The aim of this paper is to study the scale-up of detergent powders using a reactive binder processed via wet granulation in a high shear mixer. Three widely used scale-up rules viz. constant Swept Volume, constant Tip Speed and constant Froude Number were tested to identify the right parameters to be scaled for improvements at industrial scale. Bulk properties of the granulation product and mechanical properties of single granules from a desired size range were monitored to decide on the parameters to be scaled. From the bulk properties evaluated, Froude Number emerged as the right approach for scale-up for the system under investigation. However, mechanical properties of single granules did not fit the scaling rules. The strength and yield pressure of individual granules processed at large scale were higher compared to that at a small scale process. Known models of Heckel, and Kawakita and Ludde were used to fit the bulk compression data to estimate single granule properties; which show similar trends as observed in a single granule compression. The Heckel parameter ($1/K$), and Kawakita and Ludde parameter ($1/b$) which represents mechanical strength were estimated to be higher for large scale process compared to that for small scale process. In other words, achieving similar particle size distribution does not guarantee similar mechanical properties of individual granules. Hence similarity in single granule properties as well as bulk properties may be a right approach for the scale-up of wet granulation process.

1. Introduction

According to Cameron et al. [1], granulation process, like other solid handling operations, is among the least understood of processes. Therefore, granulation has been more of an art than science until a decade ago (Litster [2]). A simple scale-up of a granulation unit is rather difficult. This becomes even more difficult in a practical situation as the manufacturers make compromises in their scale-up rules on geometries due to cost reasons. There are different schools of thought about what dimensionless number to choose for scaling up these processes. York et al. [3] explained how the difference in the scale of operation could influence the product properties. Scale of operation does not just mean small and big machines as it sounds; but it should be beyond that looking into aspects like geometric similarity, fill volume etc. Taking an agglomerator unit as an example, at small scale the heat transfer of the agglomerator mass is high compared to that of the product mass. Therefore, the energy input by the motor has little impact on the temperature of the product. However, this balance changes when one scales up to bigger scale equipment, where longer continuous running can change the operating temperatures by several degrees. Changes in temperature can lead to different agglomeration profiles, as most of the binders used for agglomeration are liquid and temperature has an influence on its viscosity. Thus, changes in temperature can radically change the potential for absorbance of the binder as well as the mechanical properties at impact. Humidity in air also works as a good binder; therefore high humidity in air can increase the degree of agglomeration in the same setup.

The commercial agglomerated detergent products have certain specifications on particle size distribution. Due to this reason, typically the undersize and oversize parts of the product are screened out. In some

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cases, the oversize is fed back directly whereas in other cases, it is ground into smaller particles and then fed back to the agglomeration unit. Any scale-up work on agglomeration process must take into account the impact of these recycled streams as part of the raw material that enters the agglomeration process. Mangwandi et al. [4] showed how to obtain a maximum product yield with a minimum level of recycle ratio for a particular binder to solid ratio at a specific granulation time.

The granulation process is a multi-scale operation, where final product quality is determined by both macro-scale operation (like rotor speed in a high shear granulator) and micro-scale operation (like particle formation and interaction). A successful scale-up must take into account both the scales. The basic aim behind the scale-up is to achieve identical product attributes on both micro-scale and macro-scale when one goes from a small scale to a large scale process [5].

A macro-scale approach is based on different parameters like Froude Number, Tip Speed, Swept Volume, Stokes Number, Reynolds Number and Power Number [6–9]. These parameters have been used as a basis to determine operating conditions for scale-up studies in the past. As an example, power drawn (a measurable parameter) in a vertical granulator used for pharmaceutical granulation is used to determine the residence time of granules (e.g., endpoint in a batch mixer or fill level in a continuous mixer). In this work, we are investigating three most widely used parameters viz. Froude Number, Tip Speed and Swept Volume for scale-up of detergent granules. The objective of this work is to indicate improvements at industrial scale of wet granulation.

The constant Froude Number is defined as:

$$Fr = \frac{(2\pi N)^2 r}{g} = \text{constant} \tag{1}$$

where

N	mixer rotational speed, rps
r	mixer blade radius, m
g	gravitational acceleration, m/s^2

Qualitatively, the Froude Number is the ratio of centrifugal force to gravitational force and describes the state of fluidization in the mixer [10].

The constant Tip Speed is defined as:

$$V = 2\pi r N = \text{constant} \tag{2}$$

where

N	mixer rotational speed, rps
r	radius of the plough, m

Constant Tip Speed, which can also be referred as peripheral speed, maintains equal material motion in both the smaller and larger scales of operation. This eliminates any possibility of generation of stagnant area in the moving material [11].

Constant Swept Volume to Batch Size or Volume Ratio, Q_s/Q_b is defined as:

$$\frac{Q_s}{Q_b} = \frac{\pi r^2 h n N}{Q_b} = \text{constant} \tag{3}$$

where

r	mixer plough radius, m
h	blade height, m
n	number of blades
N	mixer rotational speed
Q_b	batch volume, m^3

Relative Swept Volume can be used to compare different mixing equipment designs and size scales [12]. It considers the volume of product swept away by the impeller of mixing blade in a given period of time, combining the affects of product fill level, impeller speed and impeller design.

A micro-scale approach focusses on the key transformations during the process and on defining mechanistic correlations between these transformations and desired product attributes at the scale of individual granules.

Mort el al. [13] took an intermediate approach between macro and micro scales. They described the mechanisms in terms of key transformations such as binder distribution, nucleation, growth, consolidation and breakage. These transformations were selected based on their relevance to the desired product attributes such as chemical homogeneity, granular size, size distribution and granule density. They concluded that the challenge lies in maintaining the similarity of each transformation during scale-up.

There is little published literature on scale-up of batch granulators in spite of their wide and varied use in the chemical process industries. The early published work of Wang and Fan [14], and Schofield [15] were concerned about scale-up of simple powder mixing and blending operations in equipment such as Tumbler, Ribbon, and Plough mixers. They emphasized the need for preservation of geometric similarity and propose general rules which are widely considered for scale-up via wet granulation based on macro-scale approach.

Granulator flow studies carried out by Litster et al. [16] showed that there are two powder flow regimes viz. bumping and roping. The powder flow goes through a transition from bumping to roping as impeller speed is increased. The roping regime gives good bed turn over and stable flow patterns. This regime is recommended for good liquid distribution and nucleation. This study recommended that, assuming geometrically similar granulators, impeller speed should be set to maintain constant Froude Number during scale-up rather than constant Tip Speed to ensure operation in the roping regime.

The aim of this paper is to study the scale-up of detergent powders processed via wet granulation in a high shear mixer. The effect of using a reactive binder for granulation on scale-up of the process is evaluated to identify the right parameters to be scaled. Three widely used scale-up rules for wet granulation were studied and they are based on

a. Constant Froude Number, Fr
b. Constant Tip Speed, V
c. Constant Swept Volume to Batch Size or Volume Ratio, Q_s/Q_b.

The granulation product was analyzed for both single granule properties and bulk properties to arrive at the right parameter for the scale-up of detergent powders.

2. Materials and methods

2.1. Processing of granules

A mixture of sodium carbonate ($d_{4,3} = 131$ μm) and sodium chloride ($d_{4,3} = 466$ μm) was used as the base powder. The particle size distributions of sodium carbonate and sodium chloride were determined by Laser Diffraction using Malvern Mastersizer Scirocco 2000 (model: ADA 2000) and the results are shown in Fig. 1. In this paper, the nomenclature 'particle' is used to define the primary materials in the range of 100 μm to 500 μm. These are agglomerated to get large and multi-particle entities called 'granules'.

The reactive binder used for granulation was Linear Alkyl Benzene Sulphonic Acid (96% active). The granulation at smaller scale was