Advanced Powder Technology 25 (2014) 195–210

Contents lists available at SciVerse ScienceDirect

Advanced Powder Technology

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

Review paper

# Monitoring and control of coating and granulation processes in fluidized beds – A review

Carlos Alexandre M. da Silva [a], José Júnior Butzge [a], Marcello Nitz [b], Osvaldir P. Taranto [a,*]

[a] School of Chemical Engineering, University of Campinas – UNICAMP, Brazil
[b] Mauá Institute of Technology, Praça Mauá, 1, 09580-900 São Caetano do Sul, São Paulo, Brazil

ARTICLE INFO

Article history:
Received 21 August 2012
Received in revised form 30 March 2013
Accepted 11 April 2013
Available online 28 April 2013

Keywords:
Fluidization
Coating
Granulation
Monitoring
Fluidization regime
Particle size
Moisture content

ABSTRACT

This review presents a compilation of works of the main techniques for monitoring and control fluidization regimes, particle size and moisture content during coating and granulation processes in the fluidized bed. The development of monitoring and control systems for coating and granulation of particles is highly desirable, not only to allow the operation in a stable bubbling fluidization regime, which intensifies heat and mass transfer, but also to ensure strict quality specifications for products, such as, uniform particle size distribution, low moisture content and good flowability. This paper focuses on the discussion of methods used and results obtained in studies on monitoring and control of granulation and coating process in the fluidized bed reported in the literature in the last decades. Pressure fluctuation signal analysis is widely discussed as a tool of regime monitoring. To monitor particle size, techniques such as, Near Infrared spectroscopy (NIR), Focused Beam Reflectance Measurements (FBRMs), among others are presented in detail. As for moisture content tracking, the methods are reviewed like acoustic signals, capacitance, microwave resonance and spectroscopy. It is evident that although these processes are highly complex, the techniques presented here have evolved mainly due to the efforts of several research groups, showing great potential for applications in industry, emphasizing the importance of this research field.



**Contents**



* Corresponding author. Address: Av. Albert Einstein 500, 13083-852 Campinas, São Paulo, Brazil.
E-mail address: val@feq.unicamp.br (O.P. Taranto).

0921-8831/$ - see front matter 
http://dx.doi.org/10.1016/j.apt.2013.04.008

**Nomenclature**

| | |
|---|---|
| $A_{bed}$ | cross section area of the bed ($m^2$) |
| $D_{50}$ | mean size of particle (μm) |
| $D$ | diameter of column (m) |
| $d_p$ | particle diameter (m) |
| $F_p$ | force of the particles (N) |
| $f_0$ | frequency of the signal (Hz) |
| $g$ | distance between two photodetectors (m) |
| $H$ | height of bed particles (m) |
| $K_C$ | proportional gain of the controller (V/%) |
| $Q$ | squared distance between two distributions of the two attractors |
| $S$ | statistical parameter (–) |
| $V_C(Q)$ | variance of $Q$ |
| $X_{50}$ | average diameter (m) |
| $\theta_r$ | angle of repose (°) |
| $\sigma_p$ | standard deviation of pressure fluctuation (Pa) |
| $\tau_I$ | integral time constant (s) |
| $\phi$ | particle sphericity (–) |

## 1. Introduction

Particle granulation and coating processes have been receiving a lot of attention from various manufacturing segments, and mainly from the pharmaceutical industry, as they significantly improve the quality of final products by increasing the mechanical resistance of particles, providing controlled release of active principles, protecting from microorganisms and external physical agents such as excessive heat, moisture and light exposure. Besides, they provide better processing conditions by increasing size and density of the particles.

The coating process is the coverage of the solid particles by a solution or suspension that is sprayed onto the bed and heated air is used for drying. The process remains for a certain residence time for the occurrence of mixing and dispersion of the particles liquid on them. Granulation is a common unit operation in chemical, pharmaceutical and food industry. Most of the granulation systems use wet agglomeration in fluid bed equipment. The granulation process transforms powders into aggregates, thus increasing the size of a solid mixture or one solid material, by a controlled agglomeration. The physical mechanisms which occur in the granulation operation (wetting, drying and growth) are similar to the coating process. However, the expected increase in particle size in this process is considerably higher compared to the coating process. In the coating process, the formation of agglomerates is undesirable. The challenge of the film coating process is to apply the spray droplets uniformly and to dry them at the proper rate, so that both agglomeration and elutriation are avoided. Both processes are very complex and affected by several variables. Therefore, they are difficult to be controlled.

The stability of the fluidization regime during coating and granulation operations performed in a fluidized bed is very important. When defluidization occurs, regions with no activity appear, the agglomeration of particles takes place, heat and mass transfer coefficients are reduced and that may cause process interruption within minutes. In extreme situations, this can result in total collapse of the bed. The moisture content and the diameter of the particles are critical parameters as they impact the stability of fluidization; therefore they must be both monitored and controlled [1,2] .

As it is very important to maintain stable fluidization during operations that coat and granulate solids, the implementation of real time monitoring and control strategies have become a requirement at international level. This is mainly so in the pharmaceutical industry, aiming at ensuring strict specifications for product quality control, validate the development of new drugs and to adapt production processes to the PAT (Process Analytical Technologies) specifications. The pharmaceutical segment has been investing heavily in the automation of their plants for the coating and granulation of solid particles.

It is extremely desirable that a real time monitoring and control system is used in the coating and granulation of particles in order to enable a bubbling stable fluidization regime, which will lead to a homogeneous distribution of the characteristics of the solid material being processed to what concerns density, moisture content, size distribution, flowability, etc. It is very important to use monitoring and control systems in fluidization operations in order to ensure high reproducibility in the quality of the product, enhancements to what concerns security aspects of the process, reduction of the number of failures in the batches and reduction in the costs of energy and human resources. Nevertheless, variables that are commonly monitored at coating and granulation plants (moisture and temperature of the outlet air, bed temperature, pressure drop and temperature variation throughout the bed, moisture content of particles), neither provide information needed to identify the incipient of defluidization nor generate information on the particle diameter. Therefore, such variables have limitations when used as control variables in a closed-loop control.

The main contribution of this work was to analyze the most relevant research, focused on developing techniques and methodologies for monitoring and control coating and granulation processes in fluidized beds. This article will cover some of the techniques applied to monitor the fluidization regime and to identify fluid-dynamic instabilities in particle coating and granulation processes, highlighting time series analysis of pressure fluctuation. Researches on the main on-line monitoring techniques of moisture content and particle size will also be discussed and compared to off-line measurement techniques. This article also proposes to examine some difficulties and future prospects on the development of strategies to monitor and control coating and granulation processes.

## 2. Monitoring strategies of variables that impact the stability of the system

### 2.1. Fluidization regime

The stable bubbling fluidization regime is essential to ensure high performance of the processes involving coating and granulation of solid particles as in this regime heat and mass transfer between the solid and fluid phase is intensified, which enables the controlled growth of particle size, that is, the process does not reach unstable situations with extreme agglomeration. According to Maronga [3], depending on the conditions within the bed, as the wet particles collide and generate liquid bridges, and if there is excessive moisture, many particles can agglomerate and cause the bed defluidization, phenomenon that is known in literature as *wet quenching*. In general, inappropriate combinations of operating conditions (mass of solids, spraying liquid flow rate, fluidization air flow rate, bed temperature) can intensify adhesion forces between the particles at non-desired levels, making the instability of the fluidization system inevitable.