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## Dry granulation of organic powders—dependence of pressure 2D-distribution on different process parameters

Thibaut Lecompte<sup>a,\*</sup>, Pierre Doremus<sup>a</sup>, Gérard Thomas<sup>b</sup>, Laurent Perier-Camby<sup>b</sup>, Jean-Claude Le Thiesse<sup>c</sup>, Jean-Claude Masteau<sup>c</sup>, Laurent Debove<sup>a</sup>

<sup>a</sup>Laboratoire 3S, INPG, 1025 rue de la piscine, F 38041 St Martin d'Hères, France

<sup>b</sup>Laboratoire Spin, LPMG UMR-CNRS 5148, Ecole Nationale Supérieure des Mines de, St Etienne, 158 Cours Fauriel, F42023 St Etienne, France <sup>c</sup>Rhodia, Centre de recherche de Lyon, 55 av. des frères Perret, 69190 St Fons, France

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#### Abstract

Nowadays dry granulation of powders has become a very important research topic because it is the most economic way of granulation, making the particle handling easier, and avoiding the loss of material during particle processing, or particle transfers. This kind of process has been deeply studied but a better knowledge appears necessary to control the great number of parameters of the process. This is particularly important in cases where the nature of the powder may lead to very complex phenomena during compaction.

In order to try and optimise dry granulation process for organic compounds, a roll press has been designed with a series of instruments enabling to control the compaction process. The apparatus consists of three parts: a vertical container with rotating steel blades avoiding arches into which the powder is poured, a feeder transferring the powder towards the rolls; the feeder is equipped with an horizontal helical screw in a cylindrical draft tube (10 mm in internal diameter, 500 mm long) and in the end of the feeder, a junction allows the change from the cylindrical symmetry of the feeder to the prismatic symmetry existing in the roll gap. The roll press (0–500 kN, load per unit length  $0-10^4$  kN m<sup>-1</sup>) has been developed to record different major classical parameters: the roll speed, the roll gap, the press strength, the rotation angle, and the feeding rate (between 0 and  $20 \text{ g s}^{-1}$ ). In comparison with different kinds of roll press described in the literature, in this work an original instrumentation system has been developed to catch specific data. The 3D-pressure distribution profiles at the interface between powder and the roll wall and the drive torque applied to the rolls were measured. A large-sized smooth steel-made roll (240 mm diameter, 50 mm width) has been chosen to compare the results to the industrial scale.

The results obtained with an organic compound exhibited the dependence—sometimes unexpected—of the rotation angle, the feeding and the rotation speed on the pressure distribution, the roll width, and the drive torque. © 2005 Elsevier Ltd. All rights reserved.

Keywords: Roll compaction; Powder; Organic materials; Pressure distribution; Driving torque; Screw feeder

#### 1. Introduction

The rolling compaction has been used since many decades in several industrial processes. Even though roll compaction has been extensively studied, a lack of knowledge does remain because of a great number of parameters, especially when using organic powders. This study aims at reaching a better understanding of the phenomena occurring during the roll compaction to optimise cohesion and dissolution of

\* Corresponding author.

E-mail address: lecompte@hmg.inpg.fr (T. Lecompte).

the final granules. An instrumented roll press has been developed to record pressure profiles on the powder/rolls wall interface, moment applied on the rolls to drive them and the gap between the rolls.

During powder granulation three steps can be distinguished: (i) feeding of powder to the roll gap, (ii) compaction of powder into dense tapes, (iii) breaking of tapes into granules. The powder can be fed by two ways: by gravity or by force feeding using single or multiple screw feeder (Pietsch, 1991). In our work, a forced feeding has been chosen to obtain a more constant powder flow, as to estimate the influence of each variable on the characteristics

Table 1 Meaning former compaction experiments

Reference	Roll diameter (mm)	Roll width (mm)	Max.wall speed $(mm s^{-1})$
Simon (2000)	130	50	100
Michel (1994)	100	46	100
Dec and Komarek (1992)	304.8	50.8	48
Jérôme et al. (1991)	250	40	353
Shima and Yamada (1984)	80	20.2	100
Tundermann and Singer (1969)	85.2	50.8	669
Katashinskii and Vinogradov (1965)	175	38-79	28
Lecompte et al.	240	50	377

of the final tapes. During the compression step, the powder is compacted by passing between two rolls to produce tapes with controlled strength. The third step—breaking tapes—will not be discussed in this paper.

Johanson (1965a,b) and Katashinskii and Shtern (1983a,b) have shown in their theories of rolling compaction that the bigger the roll diameter the more satisfying the cohesion properties of the compacts produced. Serris et al. (2002), studying uniaxial compression of several organic powders, have observed that the longer the underpressure and up-to-pressure duration, the more dense and cohesive the compacts. Johanson (1965a,b) introduced the nip angle, where the powder starts to be driven by the roll walls. This nip angle value is less than 10° and does not depend on the roll diameter. Then increasing the roll diameter brings about an enlargement of the compaction area and increases the time during which powder grains are submitted to the pressure. As a consequence the tapes will acquire a better cohesion. Table 1 shows the list of different roll diameters used during earlier experiments. In most cases, quite small diameters are chosen. These diameters correspond to roll granulators available in the market. The originality of the present study lies in the fact that the instrumented roll press has totally been developed in our laboratory, with the objective of simulating industrial conditions: rolls with quite a big diameter and large enough to avoid edge effects, under a huge maximum force. Such an experimental assembly extends the scope of the study with results allowing more reliable extrapolation to larger industrial units.

Torque measurements during compaction have been neglected in earlier experiments. However, the torque monitoring appears really interesting to reach a better understanding of this process and would be helpful for modelling. Therefore a torque sensor has been set on a driving axle of our roll press.

Simon and Guigon (2003) (Guigon and Simon, 2003) have observed that the density was varying along the tapes produced. This has been connected to the geometry of the feeding screw and rotational motion introducing heterogeneous feeding. To investigate on this observation, our roll has been equipped with three pressure sensors, located at differ-

ent points around the diameter and along the roll's width, to measure the radial and axial pressure distribution.

This original set of equipment has been assembled and used to study the behaviour of an organic powder, and find out the best conditions to produce satisfactory tapes in the best way.

#### 2. Press technology

A roll press has been designed with a series of instruments enabling to control the compaction process. Fig. 1 shows a roll press set up with two main parts: the feeding unit and the roll press assembly.

#### 2.1. Container, feeding box and screw (Fig. 1, Mark 1)

First, the powder is poured into a vertical container with rotating steel blades avoiding arches. Then with a screw feeder the powder is fed between the rolls. Simon and Guigon (2003) (Guigon and Simon, 2003) have observed that feeding obtained by only one endless screw were neither perfectly stable nor homogeneous and had some effects on the tape density distribution. In this work, a single horizontal screw—closer to industrial conditions—has been used. At the end of the feeder, a junction allows the change from the cylindrical symmetry of the feeder to the prismatic symmetry existing in the roll gap. The motor coupled with the screw was chosen to reach feeding flow rates just over  $22 \text{ g s}^{-1}$ .

### 2.2. Press and rolls

The roll press (Fig. 1, Mark 2) can be used either in speed controlled mode or in force controlled mode. According to the previous comments, the rolls (Fig. 1, Mark 3)—240 mm diameter—are very large for a lab testing. The choice of the



Fig. 1. Overview of the roll press set up: (1) feeding system; (2) hydraulic press; (3) rollers; (4) actuator.

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