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The effect of punch's shape on die compaction of pharmaceutical powders

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ABSTRACT

This paper investigates the compaction of pharmaceutical powders using different shapes of punches. We introduce a model of mechanical behaviour Drucker–Prager Cap (DPC), using the approach of compressible continuous media. The model parameters that are depending on the material density, were identified from experimental data and a calibration process was applied on Microcrystalline Cellulose (MCC) powder. In addition, the mathematical formulation of the boundary problem of compaction in rigid tools brings back to an optimization problem with constraint, which is solved by finite element method. The Drucker–Prager Cap model, which is implemented in Abaqus/Standard software, was employed using a user subroutine, USDFLD. Three kinds of typical pharmaceutical tablets are considered: flat-face tablet and concave face tablet with two different depths. Results of simulations of die compaction cycle as compression, decompression and ejection, reproduce the powder compaction process for the studied shaped punches. The effects of the punch's shape on the compaction process were observed on the distribution and the maximum of stress and density in the compact. Examination of the density gradient according to the shape, suggests a capping tendency, which increases with the punch depth. This study illustrates the potentiality of the FEM method, which could be used as an efficient tool to predict the density and the stress distributions into shaped compacts and to provide a diagnostic of the capping problems.

1. Introduction

The pharmaceutical industry has invested vast amounts of time and money in the study of the powder compaction. This expenditure is quite reasonable because the tablet can be self-administered by the patient; it can be manufactured with several forms and different colours and introduces a constant dose of active ingredient. The tablet is obviously more profitable to manufacture than parenteral dosage forms that must be administered, in most cases by trained personnel. So, more than 80% of the drugs in USA are formulated to produce oral dosage forms. Compared to oral dosage forms, tablets are the manufacturer's dosage form of choice because of their relatively low cost of manufacture, package and increased stability [1]. Contrary to the metallic powders, the compression of pharmaceutical powders has no difficulties linked to the complicacy of the tablet shape. We find simple forms in general (flat, concave…). As a result, the difficulty of the flow of material for complex forms does not meet (or little) in the pharmaceutical tablets. However, several phenomena may lead to non-conformity of tablets. The friction between powder and die during the compaction process leads to heterogeneity of the density distribution into the tablet. This heterogeneity continues during the decompression and ejection phases and leads to a dispersion of mechanical characteristics and sometimes to heterogeneity of the active ingredient for the scored tablets. Because of the powder properties and the parameters of compaction process, the tablet expansion during decompression and ejection phases is well known in the pharmaceutical industry as a redoubtable phenomenon for production. Thus, the interest to control the compaction process parameters and to analyse the fundamental properties of powders is very important.

The aim of the pharmaceutical powder compaction is to produce a good tablet without capping, with a sufficient mechanical strength, with uniform weight and with other properties. However, considering the powder (or powder mix) properties, which are very sensitive to handling, to provenance, or to manipulation, the success of the compaction process and the manufacturing of a good tablet need an understanding of the fundamental properties. These properties, which can be physicochemical and/or mechanical, allow explaining how a formulation could act during compaction.

Moreover, unsuccessful tablets are not always due to formulation. The process parameters as speed of compaction, punch shape, lubrication [2], temperature [3] and humidity changes, and state of maintenance of punches and die are often responsible of disturbance during production.

Considering the fact that about 70% of pharmaceutical tablets are biconvex, we are interested in this work by the effect of the punch shape on the powder behaviour during compaction.

The computational modelling of powder compaction has typically been carried out by two different approaches: the discrete method

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and the continuum method. In this work, the continuum model method was used. The compaction behaviour of pharmaceutical powders can be studied using the principles of continuum mechanics at macroscopic level, i.e. phenomenological models. A variety of continuum models from the soil mechanics literature have been developed from experiments on different geo-materials as described by Drucker et al. [4], Schofield and Wroth [5], Di Maggio and Sandler [6], Gurson [7] and Green [8]. Most of these models are governed primarily by elliptical caps that determine the densification yield loci during the compaction process. However, elliptical caps fail to capture the shearing phenomenon in powders, which is extremely important during the decompression and the ejection phases of powder compaction. Only the Drucker Prager/Cap (DPC) model is able to capture these phenomena because of the presence of a shear yield surface in addition to an elliptical cap. Hence, the DPC model has gained wide acceptance as a good constitutive model for modelling powder compaction. DPC models have been used for the analysis of compaction of pharmaceutical powders. These models can represent the densification and hardening of the powder, as well as the interparticle friction. DPC model have been used for pharmaceutical powders by A. Michrafy et al. [2], S. Kadiri et al. [9,10], J.C. Cunningham et al. [11], C.-Y. Wu et al. [12], G. Frenning [13], and recently by L.H. Han et al. [14] and T. Sinha et al. [15,16].

This work investigates the pharmaceutical powder compaction in cylindrical flat and curved punches. The approach is based on the detailed calibration of the powder behaviour in flat punch using DPC model and the analysis of punch curvature effects on the density distribution during the compaction cycle. The material parameters of the model were identified by an experimental procedure. Numerical simulation of the compaction process using finite element method, gives us access to displacement, strain and stress fields in the tablet. The unloading and ejection phases are often neglected or the resolution method is not specified. Because the structure must release strain energy to remain in equilibrium, it is important to use adequate methods to avoid unstable response. In this study, the unloading step was simulated successfully using the Riks method, implemented in Abaqus. Results of the distribution of density gradients according to the punch deep curvature and tendency to the capping of tablets were discussed.

This paper is organized as follows: Section 2 presents the materials and methods; results are introduced in Section 3, where we present the experimental results of parameter identification and the numerical simulations. Finally, Section 4 summarizes the conclusions.

2. Materials and methods

2.1. Materials

The microcrystalline cellulose Vivapur® 102 (MCC 102) is often used as a pharmaceutical excipient. Characteristics of the powder MCC 102 provided by JRS (J. Rettenmaier and Sohne) is shown in Table 1. This powder having good flowability, compressibility and compactability, was used to identify the model parameters. A scanning electron microscopy image of the powder MCC 102 is presented in Fig. 1.

The bulk density which is defined as the ratio of the mass over the volume of powder, is determined by measuring the volume of a known mass of powder sample, that may have been passed through a sieve into a graduated cylinder, or a measuring vessel, or by measuring the mass of a known volume of powder that has been passed through a volumeter into a cup. The bulk properties of powders depend on preparation, treatment and storage of the sample, i.e. how it was handled. In this work, various masses were filled in a graduated cylinder with a known diameter. The obtained bulk density of MCC 102 is approximately 0.31 ± 0.01 g/cm^3.

Table 1
Properties of MCC Vivapur® from J. Rettenmaier and Sohn.

Powder	Mean particle size	True density	Bulk density
MCC Vivapur 102	90 mm	1.59 ± 0.002 g/cm^3	0.31 ± 0.02 g/cm^3

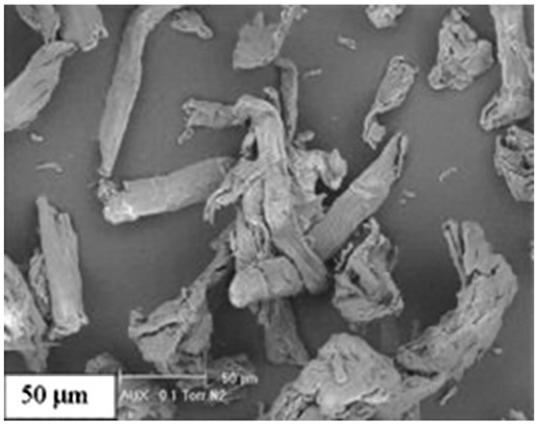

Fig. 1. Scanning electron microscopy image of powder MCC (Vivapur® 102).

In addition, the relative density is expressed as:

$$\textit{Relative density} = \textit{Bulk density}/\textit{True density}. \quad (1)$$

Thus, the initial relative density ρ_0 is equal to 0.195.

2.2. Drucker–Prager Cap model

A short presentation of the DPC model is made here. For more details, this model was described in several papers [9,12,14–16]. The Drucker–Prager Cap model is implemented in the Abaqus Software. The yield function is defined with three surfaces represented in Fig. 2: the shear failure surface F_s defining the correlation between the cohesion d and the internal friction angle β, the elliptical surface (or cap surface) F_c which can expand or contract according to the volumetric strain and the transition surface F_t between F_s and F_c. The evolution of the cap surface is described with the hardening function p_b which is the position of the cap on hydrostatic pressure axis for each density state.

$p = 1/3(\sigma_x + \sigma_y + \sigma_z)$ hydrostatic (compressive) stress; $q = \{(1/2)[(\sigma_x - \sigma_y)^2 + (\sigma_y - \sigma_z)^2 + (\sigma_x - \sigma_z)^2]\}^{1/2}$ Mises equivalent shear stress; where σ_x, σ_y, and σ_z represent the principal directions of stress.

Six parameters are required to define the yield surface of the modified DPC model: β, d, p_a, R, p_b and α and two elastic parameters, Young's modulus E and Poisson's ratio ν, are required for describing the elastic behaviour of powders. In order to identify these parameters, we use experimental tests with instrumented die, shear cell and diametrical crushing.

The powder is characterized by mechanical properties (d, β, E) which evolve with the relative density of the powder, a constant

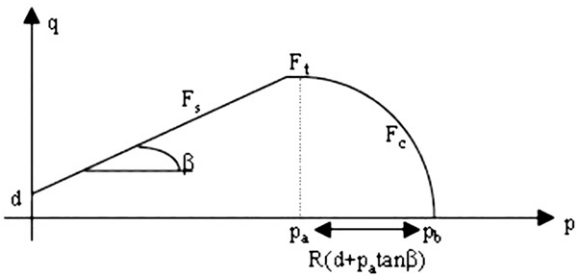

Fig. 2. Drucker–Prager/Cap model presented in the (p,q) plane.