

Wall friction in the compaction of pharmaceutical powders: measurement and effect on the density distribution

A. Michrafy*, J.A. Dodds, M.S. Kadiri

Chemical Engineering Laboratory for Particulate Solids, UMR 2392, Ecole des Mines d'Albi, Campus Jarlard, 81000 Albi, France

Abstract

In this paper, the axial density profile of tablets of microcrystalline cellulose (MCC) powder compacted in nonlubricated die is investigated by finite element modelling (FEM). The Drucker–Prager/Cap model was adopted for the compaction behavior of powder. The material parameters of the model, including the die wall friction coefficient, were estimated from experimental data of die compaction where the initial density of powder is taken uniform. Changes of Young's modulus with density was measured with a four-point beam bending test. The results of the simulation of the compression and the decompression steps were used to calculate the axial density distribution. Comparison with the measured data presented in [A. Michrafy, M.S. Kadiri, J.A.D. Dodds, Wall friction and its effects on the density distribution in the compaction of pharmaceutical excipients, *Chem. Eng. Research and Design*, Vol. 81, Part A, September (2003)] is discussed.

Keywords: Compaction; Wall friction; Density distribution; Finite element modelling

1. Introduction

The density distribution resulting from a powder compaction process is often nonhomogeneous. This is mainly due to (1) interparticle friction and (2) die wall friction [2]. At the beginning of compaction, when the rearrangement of particles governs the process, mechanism (1) is dominant. When the contact pressure increases between particles and movement (rotations and slip) between particles become limited, die wall friction becomes the important parameter influencing the density distribution. Experimental investigations of the density distribution in tablets use complex techniques such as NMR radioscopy [3] or autoradiography [4]. In this paper, we investigate the possibility given by finite element analysis (FEA) applied to the compaction of microcrystalline cellulose (MCC) powder to compute the density distribution in the tablet. Results of this analysis are compared to measurements presented in Ref. [1].

2. Die wall friction

In single action pressing of powder in a cylindrical rigid die, the applied force F_u on the upper punch is transmitted to the powder and to the die wall with a radial force F_r . The force F_l transmitted to the lower punch is less than F_u due to friction between the powder and the die wall F_f and between the particles of the powder. To evaluate the friction coefficient under conditions similar to the compaction process, the Janssen–Walker analysis [5] is adopted. Subject to certain assumptions, the die wall friction coefficient μ is related to the mean axial stress σ_h at the depth h down from the top of the tablet, the aspect ratio h/D and the transfer ratio $\alpha = \sigma_r / \sigma_h$ by:

$$\mu = \ln(\sigma_h / \sigma_u) / (-4\alpha h / D) \quad (1)$$

where $\sigma_u = F_u / (\pi D^2 / 4)$ the axial stress on the upper punch, D is the diameter of the die, and σ_r is the radial stress.

In general, α depends on the axial stress and is not uniform over the height of tablet. However, in the work presented in Ref. [6], different powders were studied, and results show a constant value of α when the relative density

* Corresponding author. Tel.: +33 563 49 3162; fax: +33 563 493025.
E-mail address: michrafy@enstimac.fr (A. Michrafy).

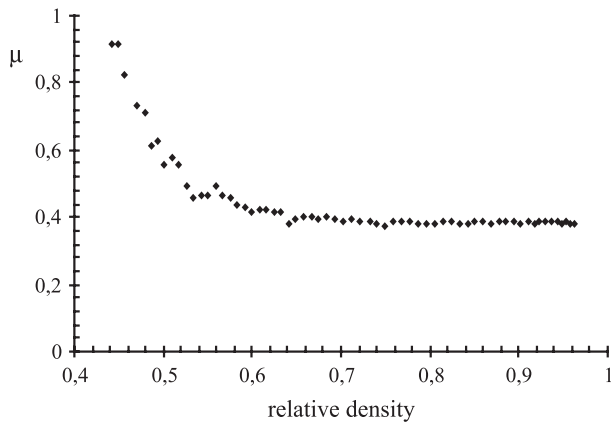


Fig. 1. Die wall friction coefficient vs. relative density ($\alpha=0.45$). Compaction of 0.31 g of MCC in nonlubricated die.

is lower than a certain value (≤ 0.8). Furthermore, in Ref. [7], measurements of α for different pharmaceutical powders show a constant value for low strain rate compaction. When modelling the compaction of ceramic powder [8], a constant value of the transfer ratio was used. In this study, and in the absence of measurement of the radial pressure, we calculate die wall friction from Eq. (1) by assuming the transfer ratio α to be constant. The value of α was fitted from the slope of the curve of radial pressure vs. axial pressure (see Fig. 5 in Ref. [7], low strain rate) and is typically found to be 0.45. The ratio σ_h/σ_u and h/D were measured during the compaction of 0.31 g of MCC. Putting these measured values in Eq. (1), the evolution of the friction coefficient (in case of $\alpha=0.45$) with the relative density can be plotted as in Fig. 1.

Fig. 1 shows that the friction coefficient decreases with increase in density and tends towards a constant value of 0.4, whilst the relative density reaches 50%. A similar variation of friction was obtained for the compaction of ceramic powder [9], where the friction mechanism was explained with the adhesion model of the contact between powder and die.

In the simulation of the compaction presented below, a mass of 2.75 g of MCC powder is compacted up to 60 MPa, and the die wall friction coefficient is calculated from Eq. (1). The evaluation is done at the end of the compaction, where the h/D was 2.01, and the transmission ratio was 0.65. In the absence of measurement of radial stress, the same value of α as in the compaction of 0.31 g was used. From Eq. (1), the resulting friction coefficient was 0.12.

3. Continuum modeling of compaction

In this model, the powder is considered to be a macroscopic porous continuum. This medium is characterized by material parameters such as the cohesion d , the internal friction angle β and by elastic properties such Young's Modulus E and Poisson ratio ν . The behavior of

powders depends on shear stress q and hydrostatic pressure p . The relation between p and q is defined by a yield function. Different models exist for the yield function, one of which is the Drucker–Prager/Cap model implemented in ABAQUS Software [10]. The Cap has an ellipsoidal shape, and its eccentricity R is a characteristic of the powder. The evolution of the Cap in the space (p, q) is assumed with an increasing function P_b of the volume change. This function is fitted from experimental data.

3.1. Powder parameters

The procedure for the determination of material parameters (d, β, R and P_b) is normally based on the triaxial compression test. However, for compaction of pharmaceutical powders, where the tablet shape is often cylindrical, a procedure for fitting material parameters can be based on data obtained from die compaction (axial stress and strain) and the transfer ratio α [8,11]. If α is constant, the stress path for the loading and unloading steps is a set of straight lines.

A mass of 2.75 g of MCC powder was compacted up to 60 MPa in nonlubricated rigid die at a velocity of 5 mm/min. Experimental data of axial strain and axial stress and the assumption that the transfer ratio is 0.45 give the resulting cohesion as 0.46 MPa and the internal friction angle as 41° . The Cap evolution is also fitted and plotted in Fig. 2. In these calculations, the eccentricity of the Cap was arbitrary taken to be 0.56. Sensitivity of results to the eccentricity value has not been systematically verified, but preliminary investigations seem to suggest it to be negligible. This is perhaps due to the simplicity of the loading path for compaction in cylindrical rigid die.

3.2. Elastic properties

The Young's modulus of MCC was characterized by a four-point beam bending test at different densities, and results were fitted as E (Gpa)=0.09 Exp(4.49 ρ_r). The determination coefficient was $r^2=0.98$. These results are agreement with those of the literature. However, inves-

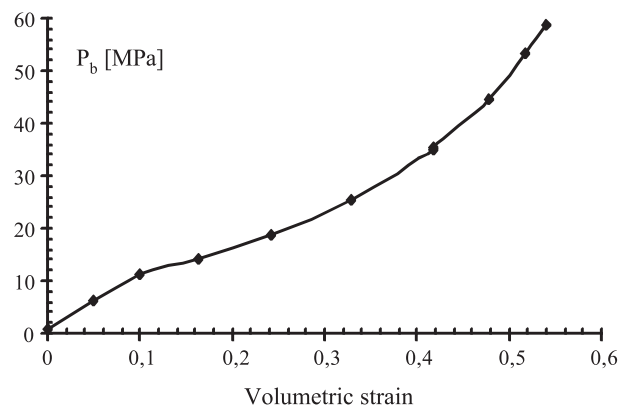


Fig. 2. Cap evolution vs. volumetric strain.

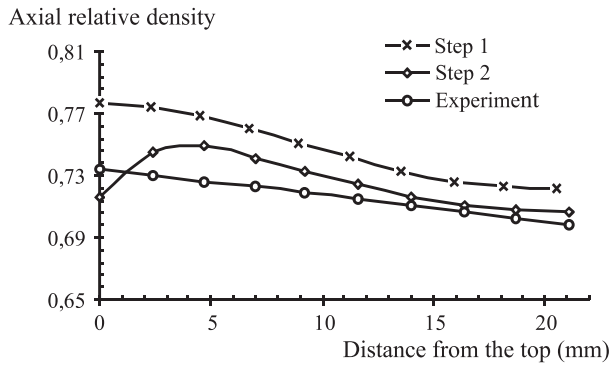


Fig. 3. Axial relative density profile over the height of the tablet. Compaction of 2.75 g of MCC up to 60 Mpa.

tigations of Poisson ratio of MCC give a dispersion of results. The value of 0.18 was used in the simulation.

3.3. Finite element analysis (FEA)

FEA is a numerical method used to solve the boundary problem of the compaction. The volume occupied by the powder bed of 2.75 g of MCC was meshed with a set of four-node axisymmetric elements. The initial density of the powder bed is assumed to be homogeneous and equal to 0.46. Material parameters and elastic properties defined below were used as input data. Punches and die were assumed to be rigid bodies. The die wall friction was fixed as 0.12. In the first step, the upper punch is moved 13.4 mm to compress the powder. In the second step, the upper punch is removed, and the tablet is free to decompress.

Because of the nonlinearity of the problem, successive incremental problems are solved. Stress and strain are calculated on the meshes, and relative density is deduced from the strain as

$$\rho_r = (\rho_0/\rho_i)\exp(-\varepsilon_{vol}) \quad (2)$$

where $\varepsilon_{vol}=\varepsilon_1+\varepsilon_2+\varepsilon_3$ with ε_i ($i=1,2,3$) as the principal strains, and $\rho_0=0.46$ as the bulk density before starting compaction. Fig. 3 shows the calculated relative densities along the height of the tablet at the end of step 1 and step 2. Measurements of the axial relative density of 2.75 g of MCC presented in Ref. [1] are also plotted for comparison.

Fig. 3 shows the calculated values decreasing from the top to the bottom of the tablet. In step 2, the maximum density is not obtained at the top of the tablet as in step 1. Experimental data are well approximated by the results of step 2, where the decompression phase is simulated.

4. Conclusion

Investigations using finite element modelling (FEM) to compute the density distribution of compacted MCC

powder indicate that it is possible to correlate powder parameters, wall friction and a simple loading path to give the density distribution of the compact. However, parameters such as α must be measured, and the sensitivity of results to R and ν must be reconsidered.

5. List of symbols

F_u	Upper force
F_l	Lower force
F_r	Radial force
h	Axial coordinate from the top of tablet
D	Internal diameter of die
μ	Wall friction
σ_h	Axial stress at height h (from the top of tablet)
σ_r	Radial stress
q	Modulus of the stress deviator
p	Hydrostatic pressure
ρ_0	Initial relative density
ρ_r	Relative density
$\varepsilon_i, i=1, 3$	Principal strains
$\varepsilon_{vol}=\varepsilon_1+\varepsilon_2+\varepsilon_3$	Volumetric strain
α	Radial to axial stress ratio
E	Young's modulus
ν	Poisson ratio
d	Powder cohesion
β	Friction angle of powder
R	Eccentricity of the Cap
P_b	Function of the evolution of the Cap

References

- [1] A. Michrafy, M.S. Kadiri, J.A.D. Dodds, Wall friction and its effects on the density distribution in the compaction of pharmaceutical excipients, *Chem. Eng. Res. Des.* 81 (Part A) (2003 September) 946–951.
- [2] D.J. Train, *Pharm. Pharmacol.* 8 (1956) 747.
- [3] G. Nebgen, *J. Pharm. Sci.* 84 (1995) 283–291.
- [4] H.H.M. Macleod, K. Marshall, *Powder Technol.* 16 (1977) 107–122.
- [5] R.M. Nedderman, *Statics and Kinematics of Granular Materials*, Cambridge University Press, 1992.
- [6] W.M. Long, Radial pressures in powder compaction, *Powder Metall.* 6 (1960) 73.
- [7] M.H. Es-Saheb, Uniaxial strain rate effects in pharmaceutical powders during cold compaction, *J. Mater. Sci.* 27 (1992) 4151–4159.
- [8] I. Aydin, B.J. Briscoe, K.Y. Sanliturk, The internal form of compacted ceramic components: a comparison of a finite element modelling with experiment, *Powder Technol.* 89 (1996) 239–254.
- [9] B.J. Briscoe, S.L. Rough, The effects of wall friction in powder compaction, *Colloids Surf., A Physicochem. Eng. Asp.* 137 (1998) 103–116.
- [10] Hibbit, Karlsson and Sorensen, *ABAQUS Theory Manual Version 5.7*, p. 4.4.4-1.
- [11] A. Michrafy, D. Ringenbacher, P. Tchoreloff, Modelling the compaction behaviour of powders: application to pharmaceutical powders, *Powder Technol.* 127 (2002) 257–266.