Analysis of Failure Mechanisms during Powder Compaction

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Abstract

Capping mechanisms during the compaction of pharmaceutical powders were explored. Both experimental and numerical investigations were performed. For the experimental study, an X-ray Computed Microtomography system has also used to examine the internal failure patterns of the tablets produced using a compaction simulator. Finite element (FE) methods have also been used to analyse the powder compaction. The experimental and numerical studies have shown that the shear bands developed at the early stage of unloading appear to be responsible for the occurrence of capping. It has also been found that the capping patterns depend on the compact shape.

Keywords : Powder Compaction, Failure, Capping, Finite Element Methods, X-ray Computed Tomography

1. Introduction

Powder compaction is a general process to transform a powder into a coherent specimen (i.e, green body, or tablets) in a number of industries. The process can be divided into three distinct stages: (i) die filling, during which the blend of powders is deposited into the die cavity under gravity by a feed shoe running over the die opening [1-6]; (ii) compaction, the powder is compressed inside a die by punches, the die and punches can be of different shapes so that various shaped products can be produced; and (iii) ejection, after the compaction, the green body/tablet is ejected from the die. The properties of tablets are influenced by the powder behaviour during all the above three stages [7]. Therefore, understanding the powder behaviour during each stage is very important and has attracted significant attention over the past several decades.

The produced green bodiess/tablets need to possess certain mechanical strength so that they can maintain their integrity during the process and be strong enough to sustain any possible load experienced during the post-compaction processes, such as handling. Hence, any defects are not tolerable during the process. Such defects, however, often occur during the process. Therefore, understanding the failure mechanisms has attracted considerable attention. In this study, we will focus on capping --- one of the most common failure phenomena encountered in compaction of pharmaceutical powders. Due to the diversity of pharmaceutical blends, increase of newly discovered drug substances and the complexity of process conditions, no universal theory for capping has been widely accepted and the capping mechanisms are not fully understood yet. Thus the capping mechanisms are further examined in this study through combined numerical analysis using advanced finite element methods (FEM) and physical visualisation using x-ray computed tomography (XRCT).

2. Methods and Results

Lactose powder, which is widely used in the pharmaceutical industry as a diluent, was considered in this study. Compaction experiments were conducted on the powders using a compaction simulator. The tablets produced were examined using X-ray computed microtomography (XRCT). In this study, a desktop high-resolution micro-CT system (Skyscan 1072, SkyScan, Aartselaar, Belgium) with a spatial resolution of 5 μm was used to visualise the failure patterns of pharmaceutical tablets.

Finite element methods (FEM) were used to model the powder compaction. The material properties and die-wall friction, calibrated from experiments with the instrumented die described above, were input into a commercial package, ABAQUS, in which the Drucker-Prager-Cap model was implemented. In this study, we first analyse the same cases as the calibration experiments with the instrumented die, in order to validate the simulations. Further simulations were carried out to examine the influence of tablet geometry by choosing punches with different surface curvatures.

Four different shaped lactose tablets were considered. All tablets are of same diameter of 8mm, but were compressed with standard toolings used in pharmaceutical industries so the top and bottom surfaces for these four different shaped tablets are of different surface curvatures. It has been observed from the experiemtns using compaction simulator that capping occurs for all four cases considered in this study. Fig.1 shows the photograpies of capped tablets of different shapes. It is clear that a cone-shaped capping pattern is induced for flat-faced tablets (Fig.1a) [8], while the inclination angle of failure surfaces apparently decreases as the surface curvature increases so that a planar failure surface is observed for deep convex tablet (Fig.1d). In the same experiments, it has also been noticed that some tablets produced were visually intact or were not completely capped as shown in Figs. 1a, 1c, 1d, but developed a chipping-like failure pattern (Fig.1b). Those tablets have been further examined used XRCT. XRCT images of four different shaped tablets are shown in Fig.2, which shows the vertical cross-section of the tablets. It is clear that even for those intact tablets (Figs.2a and 2c) and chipped tablets (Fig.2b) internal micro-cracks are clearly detected. It has also shown that for the flat-faced tablets (Fig.2a) the cracks were developed from top edge towards the mid-centre of the tablets [9], which is consistent with the cone-shaped capping pattern shown in Fig. 1a. For convex tablets, the micro-cracks were developed along relatively planar surfaces (Figs.2b and 2c) when compared to those in flat-faced tablets (Fig.2a). Again the patterns of these micro-cracks are similar to the capping patterns observed visually (Fig.1). It is believed that these micro-cracks will lead to capping during the post-compaction processes.

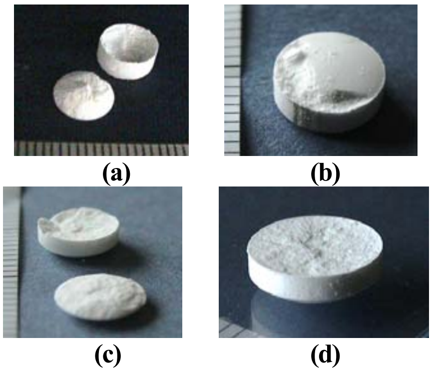

Fig. 1. Photographies of capped (a) Flat-faced tablet; (b) Shallow convex tablet; (c) Standard convex tablet and (d) deep (double radius) convex tablet.

Finite element alanysis were also performed to investigate the capping mechanisms for making those four different shaped tablets. Previous studies [8,9] reveals that the capping patterns were associated with intensive shear band developed during decompression stage of the powder compaction. Thus only shear stress distributions during decompression are presented for four different cases considered (see, Fig.3). It can be seen from Fig.3 that there are intersive shear zones developed around the top edge of the tablets. The patterns of these shear zone depends upon the shape of the tablets. Comparing Fig.3 with Fig.2, it is clear that the patterns of the shear zone are consistent with those of the micro-cracks developed in the powder compactions. This suggests that capping could be related to shear bands developed during decompression stage. However, the lacking of correlation of stress developed with the strength of materials considered in the present FE models make it difficult to predict the crack formation and development during the powder compaction which are relevant to capping, which deserve further investigation.

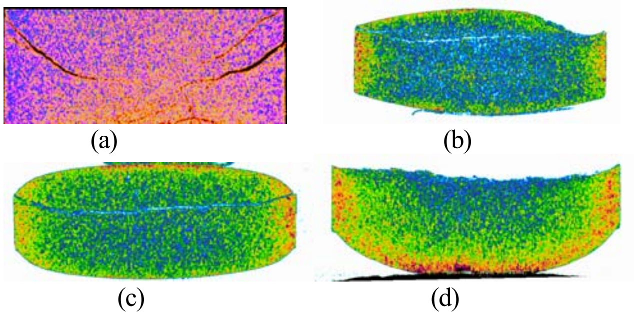

Fig. 2. X-ray computed micro-tomography images of (a) Flat-faced tablet; (b) Shallow convex tablet; (c) Standard convex tablet and (d) deep (double radius) convex tablet.

3. Summary

Experimental and numerical investigatiosn were performed to investigate the capping phenomena during powder compaction. Tablets of four different shapes were considered. It has been shown that capping patterns are depenedent upon the shape of powder compacts and are consistent with the patterns for the shear bands developed during decompression stage of the compaction.

4. Acknowledgements

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