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Effects of punch geometry on powder movement during pharmaceutical tabletting processes

Brunel Eiliazadeh [a,*], Kendal Pitt [b], Brian Briscoe [a]

[a] *Department of Chemical Engineering and Chemical Technology, Imperial College, Prince Consort Road, London SW7 2AZ, UK*

[b] *Merck, Sharp & Dohme Ltd., Hoddesdon, Hertfordshire, UK*

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Abstract

This paper describes an investigation involving an experimental study of the movement of powder during the axially symmetrical uniaxial compaction of microcrystalline cellulose. The influences of simple flat faced cylindrical and concave curved faced cylindrical punch geometries are investigated which form tablets of non-homogeneous structure and possible poor mechanical integrity due to the development of uneven pressure distributions within the compacts.

Powder compaction occurs by the action of a compressive stress acting on a powder bed and the constrained powder moving according to the direction of the resulting forces generated by the initial application of the forming force. Changing the geometry of the punch and die set used will inherently change the direction of the resultant forces causing major changes in the direction of powder movement. The experimental study adopted a metal shot tracer method in conjunction with X-ray imaging, that has been designed to show how inherently changing the geometry of the punch, causes different amounts of powder movement to take place leading to the formation of a non-homogeneous structure within the tablet. Radial powder movements have also been investigated using the same metal shot tracer technique. The results presented show that the tablets were highly non-homogeneous in nature with high density regions present in the "top corners" and "middle bottom half", with respect to its compression axis of the flat faced tablets. High density regions were also noted in the "corners" of the convex curved faced tablets where powder was in contact with the die wall. Significant amounts of radial powder movement, with respect to the load axis were observed. This was particularly so for the convex curved faced variety, inducing large amounts of stored stress and strain, therefore, increasing the chances of mechanical failure.

Keywords: Powder compaction

* Corresponding author. Tel.: +44-7939-065742; fax: +44-1494-722731.
E-mail address: brunel.eiliazadeh@imperial.ac.uk (B. Eiliazadeh).

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1. Introduction

The study and application of the process of powder compaction has continued to develop over recent years and still continues to be a cheap and effective means of forming the desired object. The pharmaceutical industry routinely uses powder compaction to convert active drugs into metered solid dosage forms but fundamental problems such as "capping" and "lamination" still remain because of a generally poor understanding of the mechanisms involved with powder compaction. However, the solid dosage form is still the most common form of current drug delivery, as such tablets can be easily and cheaply manufactured on a large scale but more importantly, they can be administered to the patient in an established way. The necessity to be able to provide a unique identification of a tablet without resorting to the addition of colourants has led to the introduction of tablets of shapes that are more complex than one of a simple right circular cylinder (Newton et al., 2000); this is shown in Fig. 1.

The pharmaceutical industry currently favours manufacturing tablets of the convex curved face variety as such shapes are less susceptible to "edge chipping" and normally have a superior surface finish that is more durable. These attributes generally enhance the cosmetic appearance of the tablet. While the therapeutic function of the tablet is crucial, their mechanical properties are significant in terms of further processing and handling after the compaction operation. There have been previous methods in the past as described by Train (1957), Charlton and Newton (1985), Kandeil and De Malherbe (1977), Hersey and Train (1960), Ozkan and Briscoe (1996), Aydin et al. (1996), Marshall (1963), Sixsmith and McCluskey (1981) and many more to show the natural formation of density gradients in flat faced tablets but little or no definitive work has been carried out with regards to non-planar geometries. One such piece of work has been recently reported by Sinka et al. (2003).

In this investigation, we seek to show how inherently changing the geometry of the punch and die set used from a flat to convex curved geometry may enhance the formation of high density gradients in the tablets and cause eventual failure of the tablets by "capping" or "lamination".

2. Materials and methods

2.1. Materials

Microcrystalline cellulose (Avicel PH102, FMC, Philadelphia, PA, USA) has been used in this investigation as the compaction medium. Microcrystalline cellulose is a partially depolymerised derivative of

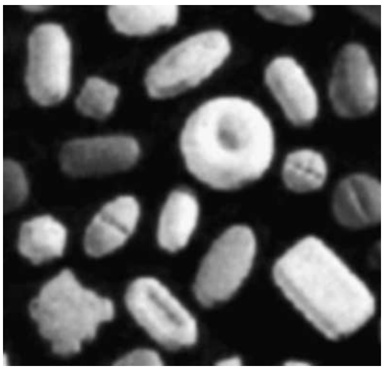

Fig. 1. Examples of the different shapes, sizes and colours of tablet currently available on the NHS.

Table 1
The mechanical properties of Avicel PH102

Compaction pressure	9.84 kN/cm^2
Tensile strength	0.8711 kN/cm^2
Permanent deformation pressure	15.3
Brittle fracture index	0.0821
Bonding index	0.0571
Reduced modulus of elasticity	1472

alpha cellulose. Microcrystalline cellulose is a white, odourless, tasteless, relatively free-flowing powder that is virtually free from organic and inorganic contaminants and is derived from purified speciality grades of wood pulp.

Microcrystalline cellulose is a very commonly used pharmaceutical compression excipient for drug delivery in solid dosage as in a tablet. It is a material which is known to be relatively ductile, hence the choice for its use for the easy compaction compared to other drug components such as Paracetamol which is known to be very brittle and difficult to compact. Avicel has bulk and full densities around 300 and 1520 $kg\,m^{-3}$, respectively. The particles are irregular with a size distribution of 20–200 μm. The mechanical properties of Avicel PH102 are shown in Table 1.

2.2. The metal shot tracer method

The method proposed and utilised by Aydin et al. (1996) has been further developed to produce not only relatively highly accurate density plots but also allows the radial powder movement to be measured by simply measuring the distance between the fine steel balls after compaction and comparing these distances with the distance between the steel balls prior to the implementation of the compaction process. Measuring the radial powder movement can be directly quantified using this method; other methods such as the conventional coloured layer technique assume that radial powder movement does not take place, hence in some cases can be highly inaccurate.

Both flat tablets and convex curved tablets were formed using this method. Convex face tablets were produced from a die diameter of 25 mm. The radius of face curvature of the tablets was 18.5 mm with the maximum punch face depth being 4.9 mm. Fig. 2 shows the geometrical dimensions of the non-planar tablets.

The die, supported upon a base, was filled with successive layers of the microcrystalline cellulose powder that incorporated the steel ball tracers of 2 mm diameter (Batch no. 1754, Atlas Ball & Bearing Co Ltd., Walsall, UK). Stainless steel balls were used in this investigation as the tracer material as they are easily found under X-ray imagery and are more readily available compared to other common tracer materials such as lead that is now particularly difficult to source with a consistent and accurate diameter. A template which was carefully machined having nine holes on its central line was inserted into the die following the addition of each successive layer and the steel balls were accurately inserted onto the powder layer through the template as shown in Fig. 3. After the die was filled, the layers of microcrystalline cellulose powder were compacted using a Lloyds Beal Compression Machine (Ammtek, USA). The locations of the fine steel balls before and after compaction were noted. In total 8 g of powder was used with an aspect ratio of 1.52. [1] In this investigation, the assumption is made that the steel balls do not significantly obstruct the regular powder flow; the steel balls themselves are measured between 10 and 100 times larger than the Avicel.

[1] The aspect ratio of a compact is defined as the height of the compact (38 mm) divided by the diameter of the compact (25 mm). The aspect ratios quoted in this investigation are calculated prior to compaction.

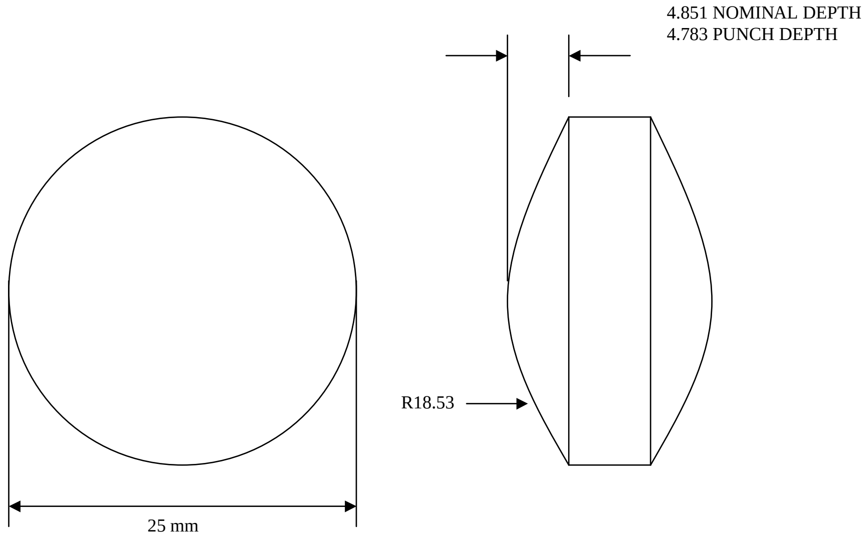

Fig. 2. Schematic showing the geometrical dimensions of the non-planar tablets.

Prior to compaction, and after the compaction process, in order to observe the positions of the fine steel balls in the microcrystalline cellulose compacts, the central planes of the compacts, including the fine steel balls were abstracted from the compacts by grinding and the remaining slice was photographed using a commercial X-ray instrument (43804 X-Ray System, Faxitron Series, Hewlett Packard). The identification of the precise location of the fine steel balls was performed by studying the X-ray films (Polapan 52, Polaroid, USA) in conjunction with a digital stereo microscope accessory. The density distributions were then calculated using the measured values of the initial locations (before compaction) and final (after compaction) distances between the fine steel balls (h_0 and h_1) and the original green density, ρ_0 by using Eq. (1), first used by Aydin et al. (1996).

$$\rho_b = \frac{\rho_0}{1 - [(h_0 - h_1)/h_0]} \tag{1}$$

where ρ_b is the density value at the location between the corresponding steel balls.

3. Results and discussion

In order to analyse the density distributions present within a tablet, the metal shot tracer method was used which clearly demonstrated how non-homogeneous a tablet mass becomes depending on the compaction pressure imposed. All of the tablets, whether they had a flat or convex curved faced geometry showed significant and uneven particle movement between the central and peripheral regions which ultimately produced the reported non-homogeneous structures. At the higher compaction pressures, higher densities were naturally observed for both curved and flat tablets but also, the variation in density distributions were also significantly increased.

3.1. Flat faced tablets

Fig. 4 shows the computed density distribution present in a flat faced tablet formed at a relatively high compaction pressure of 97.2 MPa. High density regions were found towards the “top corners” of the tablets

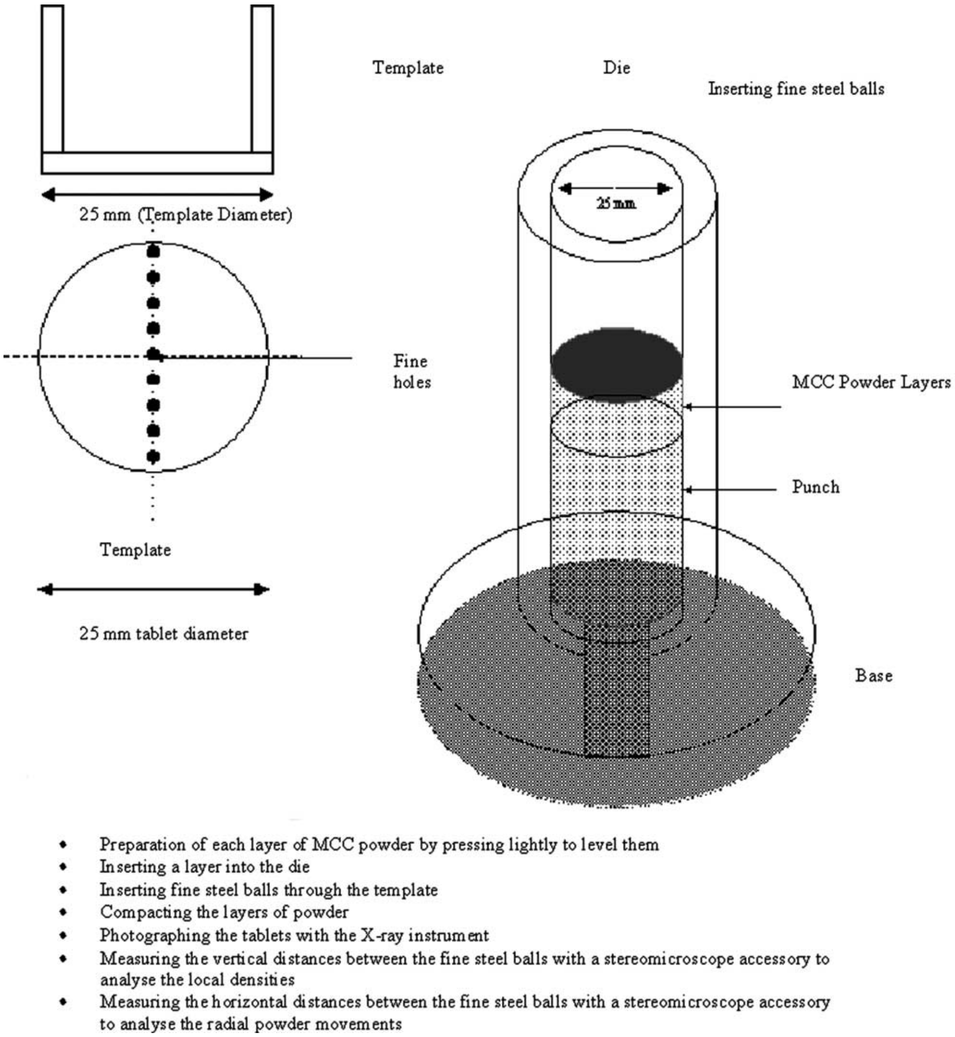

Fig. 3. Illustrations showing the experimental procedures involved with filling the die with the incorporation of steel balls.

and the "middle bottom half" with the high density in the middle bottom half usually being of slightly higher density than the top corners; recalling that this is a single ended compaction. The compressive forces that were acting on the powder particles, within the powder bed, were accommodated by an increase in local stress which may be resolved to show that they acted towards the bottom half of the tablets causing the formation of this region of high density together with stress relief within the part of the tablet adjacent to the moving top punch. Low density regions were also found to be present in the "bottom corners" of the tablets. The lack of perfect symmetry may reflect the intrinsic error of the method or a variation of initial powder density caused by imperfect die filling.

Fig. 5 shows the now classical Train Explanation for the development of the stress/density pattern within flat-faced tablets during single ended compaction which can be used to describe the general pattern of density gradients found within the tablets formed at various compaction pressures.

From Fig. 5, zone A and zone B are the resulting regions of high density in contact with the top punch. The x force component acts outward with respect to the loading axis, that is resisted by the die walls. The w

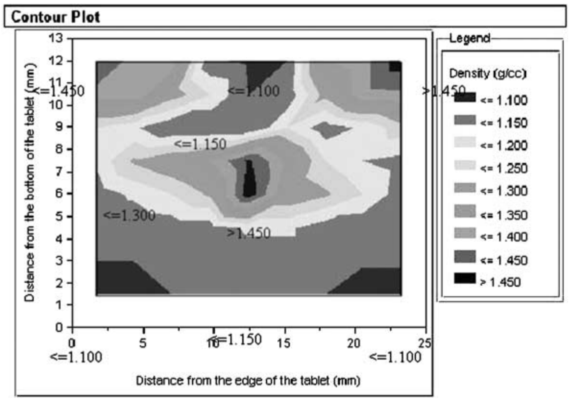

Fig. 4. Contour plot for the flat tablet formed at 97.2 MPa.

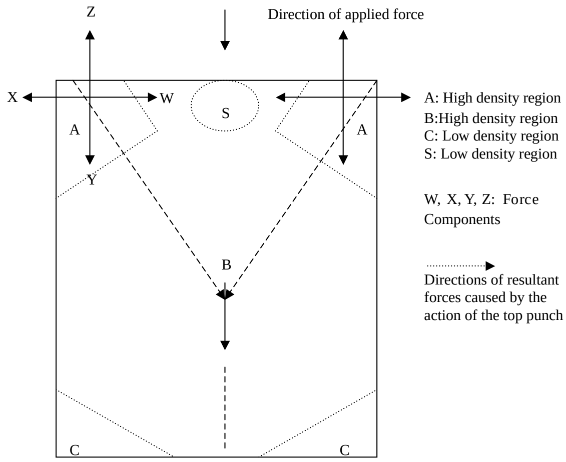

Fig. 5. Diagram showing the classical Train Explanation of the development of the pressure pattern within flat faced tablets (Train, 1957).

force component is also an outward stress that is resisted by the die walls. The vertical stress component, z, upwards is opposed by the descending top punch and the y component onto the particles below. Zone S and zone C all represent relatively low density regions.

Frictional forces at the die wall severely impede powder movement; this is seen as a force loss from the top to the bottom punch as was described by Ozkan and Briscoe (1996). As a result of these frictional effects, an uneven pressure distribution is setup which causes powder movement to be correspondingly uneven in nature. As a consequence, a heterogeneous density is produced as well as an associated internal stress field. The stress gradient will be directly related to the density gradient. Towards the bottom of the tablet, powder particles are experiencing less of a compressive force, hence the lower densities.

3.2. Convex curved faced tablets

A similar approach can be introduced into the analysis of the density fields developed for the convex curved surface tablets. Fig. 6 shows the density gradient plot for a convex curved tablet formed at 97.2

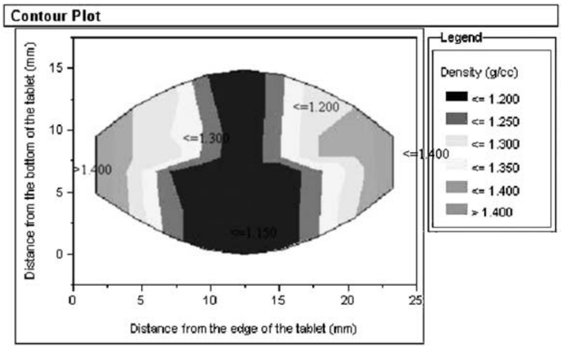

Fig. 6. Contour plot for the convex curved tablet formed at 97.2 MPa.

MPa. The results for the curved specimens show that the highest density regions tend to be closest to the die wall, at the edge of the central core for all compacts at all compaction pressures, with lower density regions occurring towards the centre of the tablets. In these regions of high density, the wall-friction was high, thus the powder movement was constrained in these areas compared to other areas of the tablet where powder movement was comparatively easier (Bal'shin, 1938; Kamm et al., 1947). Again note the imperfect symmetry in Fig. 6.

The resolution of the compression forces acting upon the powder particles using a concave curved faced punch shows that the resultant forces act inwards towards the centre of the tablet as is shown in Fig. 7. Fig. 7 represents the general pattern of density gradients for curved tablets formed at various compaction pressures. Fig. 7 actually resembles that shown in Fig. 5 but where the effective loading axis have been rotated by $\pi/2$.

Radial powder movement occurs to a larger extent in the case of convex curved tablets due to the inherent greater volume to fill in the centre of the tablet. As powder movement was restricted along the

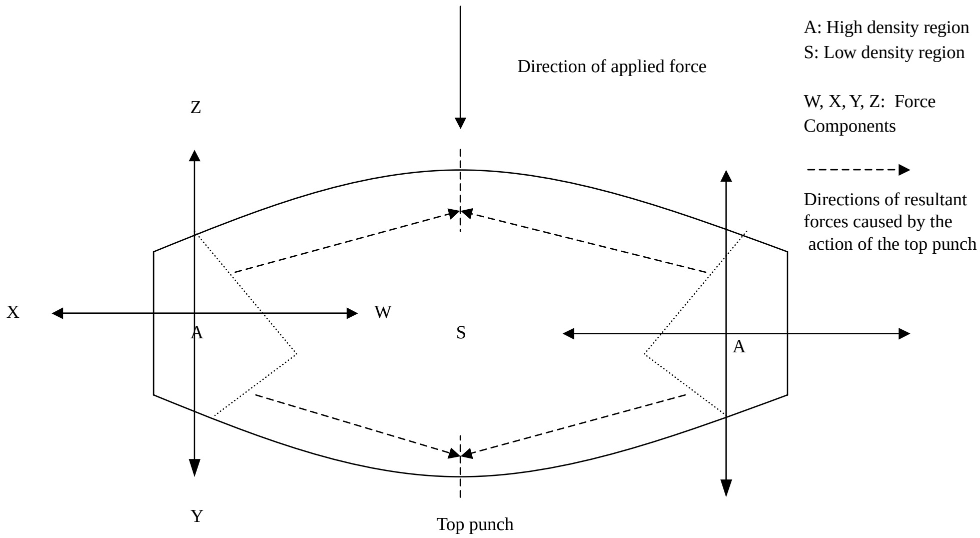

Fig. 7. Diagram showing an explanation of the development of the pressure pattern within convex curved faced tablets.

edges of the compact where powder was in direct contact with the die wall, density distributions were significantly higher compared to the flat faced tablets.

When a large curvature of the punch set was used (the curvatures of each face of a curved punch set used were always the same being concave), the net action of the resultant force was nearer to the moving punch face; this fact was also shown qualitatively by Sixsmith and McCluskey (1981). From Fig. 7, zone A is the region of high density and zone S represents the corresponding relatively low density regions. The x component is an outward stress that is resisted by the die walls. The w component is an outward stress that is resisted by the die walls. The vertical stress component, z, upwards is opposed by the descending top punch and the y component onto the particles below.

If the flat faced contour plots are compared with the convex curved contour plots, it is apparent that the density contour of the central region of the tablet can be found to have changed significantly. With the convex curved faced variety, die wall friction still impedes particle movement at the periphery but because of the nature of the punch shape, there is a much greater degree of radial movement in this region relative to the centre of the tablet than is found with the flat faced punch systems.

On examination, both the flat and curved tablets shows the high density or "harder" regions as well as the low density or "soft" regions are separated by well defined density, and thus stress boundaries especially where the difference in density between high and low density regions is high, for example at high compaction pressures. Sixsmith and McCluskey (1981) mentioned that lamination may occur along these stress gradient boundaries which are actually similar to those observed in the current investigation where the convex curved faced tablets were laminating consistently above 88 MPa. Delamination occurred along these internal stress/density boundaries where the tablet fractured into a series of distinct layers usually beginning in the centre of the tablet. With the convex curved faced variety, this corresponded to the areas of lower density in the centre of the tablets where crack propagation began and followed the relatively weak internal powder interfaces.

The variation of the density distributions within the convex curved faced variety, was seen to be much greater and also much more non-homogeneous than in the corresponding flat faced tablets. This indicates the presence of much more stored stress and strain and may be the reason why lamination was seen to be taking place.

3.3. Radial powder movements using the steel shot tracer method

Other methods to measure density distributions, such as the "coloured layer technique" devised by Train in 1957, have clearly shown that density gradients are present but due to limitations with the method, could not accurately account for lateral powder movement. In some cases, this inability to account for lateral powder movement has shown up as unrealistically high density values in excess of the "theoretical" density of microcrystalline cellulose itself at high compaction pressures. The metal shot method can be used to measure density distributions and radial powder movements accurately as we are simply measuring distances moved by the discrete location indicators. Fig. 8 shows the degree of radial powder movement taking place in flat faced tablet formed at 97.2 MPa.

At various compaction pressures, radial powder movement in the case of flat faced tablets generally follow the trends shown by Fig. 9. The length of the arrows shown reflects the relative extent of the powder movement and consequent local compaction.

Lateral powder movement in the case of the flat faced tablets, takes place primarily at the top corners of the tablet away from the die wall and at high compaction pressures in the centre of the tablet with minimal radial powder movement taking place in the bottom corners of the tablet. The effects of friction have been briefly described in Section 3.1; as force is externally dissipated from the top punch to the lower punch powder particles are under less of a compressive stress towards the bottom of the tablet. The result of this is the formation of low density regions but also the reduction of the radial powder movement. Generally

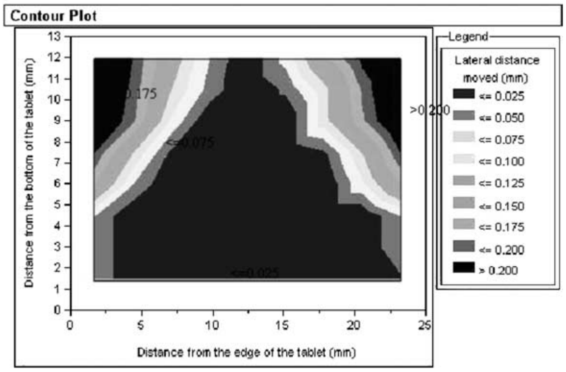

Fig. 8. Compacted lateral powder movement in a flat faced tablet formed at a compaction pressure of 97.2 MPa.

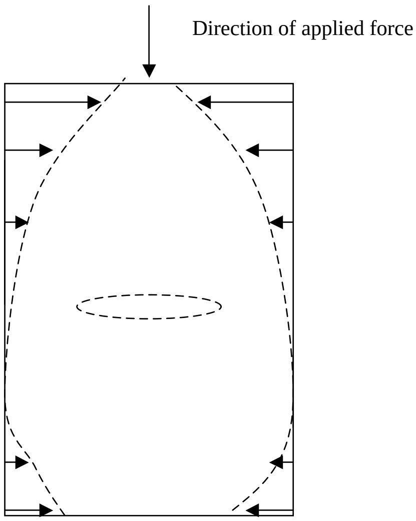

Fig. 9. Diagram showing the main areas of radial powder movement in a flat faced tablet.

speaking, the radial powder movement takes place according to the direction of the resultant forces as shown by Fig. 4. Powder may move in the vertical axis up to 10 mm at the higher compaction pressures, but it is also apparent from these results that for every 10 mm moved vertically, approximately 0.5 mm is also moved radially in the regions with high movement. This is only 5% of the vertical movement so in the case of flat faced tablets it can be considered to be fairly negligible at moderate compaction pressures.

Fig. 10 shows the corresponding degree of radial powder movement taking place in a convex curved tablet formed at 97.2 MPa of a similar aspect ratio. It is clear that the extents of the radial movement are at least a factor of three greater than for the flat face case given in Fig. 8.

At various compaction pressures, radial powder movement in the case of convex curved tablets follow the trends shown by Fig. 11.

The main radial powder movement, in the case of the convex curved tablets, takes place primarily at the edges of the tablet, adjacent to and away from the die wall, along the edges of the die punches even though frictional effects may be high in this region. Similar to the flat tablets, radial powder movement takes place

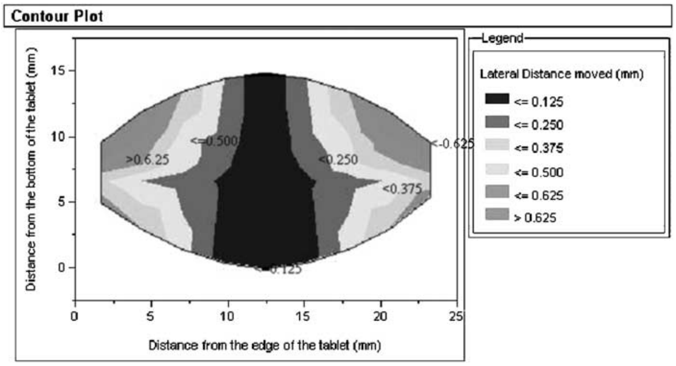

Fig. 10. Radial powder movement in a convex curved tablet formed at a compaction pressure of 97.2 MPa.

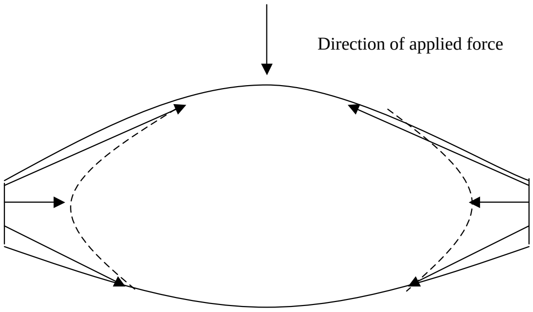

Fig. 11. Diagram showing the main areas of radial powder movement in a convex curved tablet.

according to the direction of the resultant forces as shown in Fig. 7. At the higher compaction pressures, more radial powder movement is seen to take place towards the bottom surface of the tablet as frictional effects seem to have more of an effect along the edges of the top and bottom punches rather than along the side edges of the die wall; this has been demonstrated by surface topography analysis in other work by the current investigators (Eiliazadeh et al., 2003). The powder may move vertically by up to 10 mm at high compaction pressures, but it is apparent from these results that for every 10 mm moved vertically, up to 1 mm is also moved radially in the regions with high movements. This is double the movement compared with the flat faced variety and can be explained due to the nature of the punch and die set used to form this shape of tablets where powder movement should occur towards the centre of the tablet, radially, due to the inherently larger volume present to fill.

Both radial and axial powder movement is severely restricted by frictional forces particularly where powder is in contact with the die wall. It is these frictional forces that cause an uneven pressure distribution and uneven powder movement throughout the powder compact. The result of this is a density gradient and large amounts of stored stress and strain within the tablet itself.

4. Conclusions

The further development of experimental methods, used in the past, has provided a better and more refined understanding of the movement of powder during the tabletting process. The metal tracer method has shown the formation of highly non-homogeneous structures particularly for the convex curved faced variety that was more susceptible to fracture failure due to increased amounts of stored stress and strain. Cracks were seen to be propagating along highly defined stress boundaries. Intrinsic inaccuracies present in the coloured layer technique used to measure density distributions were avoided by the use of the metal shot tracer method which also showed that large amounts of radial powder movement were taking place particularly with the curved tablets as there was inherently a greater volume to fill in the centre of the tablet due to the geometry of the punch and die set used. Radial powder movement was seen to be taking place away from the die wall for both flat and curved surfaces and inducing large amounts of stored stress and strain. In other related studies, surface topography investigations have shown the effect of this stored stress and strain; where they are high, shape distortions may take place due to stress relief. Surface topography has also shown the effects of friction during the compaction process.

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