JPP
Journal of
Pharmacy and Pharmacology

JPP 2004, 56: 947–950

Received January 15, 2004
Accepted April 7, 2004
DOI 10.1211/0022357023736
ISSN 0022-3573

Effect of punch tip geometry and embossment on the punch tip adherence of a model ibuprofen formulation

Matthew Roberts, James L. Ford, Graeme S. MacLeod, John T. Fell, George W. Smith, Philip H. Rowe and A. Mark Dyas

Abstract

The sticking of a model ibuprofen–lactose formulation with respect to compaction force, punch tip geometry and punch tip embossment was assessed. Compaction was performed at 10, 25 or 40 kN using an instrumented single-punch tablet press. Three sets of 'normal' concave punches were used to evaluate the influence of punch curvature and diameter. The punches were 10, 11 and 12 mm in diameter, respectively. The 10-mm punch was embossed with a letter 'A' logo to assess the influence of an embossment on sticking. Flat-faced punches (12.5 mm) were used for comparison with the concave tooling. Surface profiles (Taylor Hobson Talysurf 120) of the upper punch faces were obtained to evaluate the surface quality of the tooling used. Following compaction, ibuprofen attached to the upper punch face was quantified by spectroscopy. Increasing punch curvature from flat-faced punches to concave decreased sticking. Altering punch diameter of the concave punches had no effect on sticking when expressed as μg mm^{-2}. The embossed letter 'A' logo increased sticking considerably owing to the probable concentration of shear stresses at the lateral faces of the embossed logo.

Introduction

Sticking of ibuprofen to the upper punch during direct compression tableting has previously been examined with respect to punch surface quality and surface material of the punch tip (Roberts et al 2003).

Punch geometry has been shown to be an important factor in the properties of compacted tablets (Aulton & Tebby 1975; Sixsmith 1980; Aulton 1981; Newton et al 2000) and the movement of powder during the compaction process (Sixsmith & McCluskey 1981). Increasing punch curvature was found to increase the hardness of the periphery relative to the centre of tablets, with 'normal' concave punches (diameter (D)/curvature radius (R) = 0.67), resulting in almost uniform distribution of hardness across the tablet surface (Aulton & Tebby 1975). Increasing punch curvature increases the amount of material enclosed within the punch tip during compression, which is relatively protected until the main body of the compact is compressed to low porosity and acts as a secondary punch face to compact the protected material (Sixsmith 1980).

Punch embossing in tablet manufacture is a method often used to produce recognisable tablets for the purposes of appearance and marketing. Waimer et al (1999) studied the influence of tablet engravings with respect to adhesion using an instrumented upper punch to measure the 'pull-off' force as the upper punch detached from the tablet face. The authors claimed that strong adhesion was caused by the concentration of shear stresses that occur at the lateral faces of the engraving during compression and that stress distribution within the tablet is a very important factor in adhesion. With two formulations known to stick, pull-off force increased with steeper angles of the engravings and with increasing compaction force.

The aim of the present study was to investigate the influence of changing from flat-faced punches to normal concave punches on the sticking of ibuprofen to the upper punch face. The influence of a typical punch embossment was also investigated using a normal concave punch with a letter 'A' logo.

School of Pharmacy and Chemistry, Liverpool John Moores University, Byrom Street, Liverpool, L3 3AF, UK

Matthew Roberts, James L. Ford, Philip H. Rowe, A. Mark Dyas

FMC BioPolymer, Avenue Mounier 83, 1200 Brussels, Belgium

Graeme S. MacLeod

School of Pharmacy and Pharmaceutical Sciences, University of Manchester, Oxford Road, Manchester, M13 9PL, UK

John T. Fell

Manesty, Kitling Road, Knowsley, Merseyside, L34 9JS, UK

George W. Smith

Correspondence: James L. Ford, School of Pharmacy and Chemistry, Liverpool John Moores University, Byrom Street, Liverpool, L3 3AF, UK. E-mail: j.l.ford@livjm.ac.uk

Materials and Methods

Materials

Ibuprofen crystals (B.P.) were supplied by M&A Pharmachem (UK). Direct compression lactose (Tablettose 80) was supplied by Meggle GmbH (Germany). Colloidal silica (Aerosil 200) was supplied by Degussa (UK). Magnesium stearate was supplied by BDH (UK) and 96% ethanol by Hayman (UK). All materials were used as supplied.

Formulations

Each formulation contained 69.5% w/w ibuprofen, 29.5% w/w lactose DC, 0.5% w/w Aerosil 200 and 0.5% w/w magnesium stearate.

Powder mixing

The ibuprofen, lactose and Aerosil were blended in a 500-mL glass jar using a tumbling powder mixer, consisting of a motor (Heidolph, Germany) and clamp, for 10 min at 40 rev min^{-1}. Following the initial blending stage, the powder was sieved using a 1-mm aperture sieve (Endecotts, UK) to remove any agglomerates. Magnesium stearate was added and the formulation blended for a further 5 min.

Compaction

Tablets were compacted at 10, 25 or 40 kN using an F3 single-punch tablet press (Manesty, UK) instrumented with strain gauges to measure upper punch compaction force. Strain gauges were connected, via a junction box (Bruel & Kjoer, Germany), to a chart recorder (SE120; ABB, UK). We used 11- and 12-mm normal concave, 10-mm embossed normal concave and 12.5-mm flat-faced tooling; target tablet weight was 400 mg. Each compaction run (i.e. running time of tablet press for each data set) was 1 min and production speed was 19 tablets min^{-1} (Roberts et al 2003). Although different diameter punches were used during the study, compaction forces were reported rather than compaction pressures for several reasons. First, although the forces applied were acting over different surface areas, it was impossible to determine whether all the particles at the punch–tablet interface (i.e. where sticking occurs) were subjected to the same pressures. Also, the levels of sticking were corrected for the differences in punch surface area and it was also considered to be more practically relevant to quote compaction forces rather than compaction pressures.

Surface characterization

Surface profiles of the upper punches were obtained from a Taylor Hobson Form Talysurf 120 (Taylor Hobson, UK) as previously described (Roberts et al 2003). For the 10-mm concave punch embossed with the letter 'A' logo, surface scans of the areas not embossed were obtained. Consequently, some of the areas scanned were less than 5 mm in length (e.g. the enclosed area within the letter A). The surface profile parameters obtained were: Ra, the mean value of all positive deviations of the surface profile from zero; Rt, the maximum range (highest peak to lowest trough on the surface profile); and Rz, the mean value of the five highest peaks on the surface profile.

Sticking quantification

Following each 1-min compaction run, sticking to the upper punch face was quantified as previously described by Roberts et al (2003). As punches of different curvature and diameter were used, the surface area over which the sticking was evaluated varied. The amount of sticking was therefore reported as $\mu g\, mm^{-2}$.

Punch face surface area

The surface area (A) of the flat punch face was calculated using the following equation:

$$A = \pi r^2 \qquad (1)$$

where r is the punch radius.

The surface area (A) of the concave punch faces were calculated using the following equation:

$$A = \pi (r^2 + h^2) \qquad (2)$$

where r is the punch radius, and h is the height of curvature.

The height of the punch curvature (h) was calculated from the following equation:

$$h = (t - W)/2 \qquad (3)$$

where t is the overall thickness of the tablet, and W is the thickness of the central cylinder.

Statistical analysis

Results of the sticking quantification using the various punch and compaction force combinations were analysed using the Minitab statistical package. A two-way analysis of variance was used to assess any significant ($P < 0.05$) differences in the levels of sticking observed. A one-way analysis of variance was used to assess any significant ($P < 0.05$) differences between the surface roughness measurements of the punches used.

Results and Discussion

Surface quality of the upper punch faces was previously found to be an important factor in the sticking of ibuprofen (Roberts et al 2003). The Taylor Hobson Talysurf profiles of the upper punches used in the present study are shown in Table 1. Ra values for all punches were comparable and although Rt and Rz values obtained differed slightly, results indicated good quality smooth surfaces with few imperfections. Statistical analysis revealed no significant ($P > 0.05$) differences between the surface roughness of the punches used.

Table 1 Taylor Hobson Talysurf 120 surface profiles of upper punch faces used

Surface profile parameter	11-mm concave punch	12-mm concave punch	10-mm embossed concave punch	12.5-mm flat punch
Ra (μm)	0.06 ($\pm$0.03)	0.04 ($\pm$0.001)	0.05 ($\pm$0.01)	0.04 ($\pm$0.02)
Rt (μm)	0.58 ($\pm$0.10)	0.54 ($\pm$0.12)	0.21 ($\pm$0.08)	0.44 ($\pm$0.12)
Rz (μm)	0.22 ($\pm$0.07)	0.13 ($\pm$0.02)	0.10 ($\pm$0.04)	0.20 ($\pm$0.06)

Ra, mean value of all positive deviations of the surface profile from zero; Rt, maximum range (highest peak to lowest trough on the surface profile); Rz, mean value of the five highest peaks on the surface profile.

The sticking quantification results were corrected for surface area of the upper punches using Equation 1 (flat-faced punches) or Equation 2 (concave punches). Examination of the sticking results (Figure 1) indicated that when using flat-faced tooling, sticking to the upper punch face increased with compaction force, resulting in a large amount of sticking at 40 kN. This observation has previously been reported by Roberts et al (2003). A similar relationship, although much less pronounced, between compaction force and sticking occurred when using the 'normal' concave tooling (11 and 12 mm diameter). However, changing from a flat-faced punch to a concave punch reduced sticking of ibuprofen to the upper punch face at higher compaction forces. Sticking was therefore dependent on punch curvature. When flat-faced punches are used the compression force causes local stresses within the powder bed and the resultant force acts towards the bottom half of the compact to form an area of high density. A protected region develops between this area and the upper punch, and as punch geometry changes so does the central region of the tablet (Sixsmith & McCluskey 1981). With an increase in punch curvature there is an increase in both axial and lateral movement of the powder bed and a greater tendency to form the area of high density, which increases the protective effect on the area between it and the punch face. Owing to the increase in curvature from a flat-faced punch to a concave punch, the protected region, which develops between the main body of the compact and the moving punch face, is only subjected to compressive stresses and not shear and compressive stresses (Sixsmith and McCluskey 1981). This effect appears to have reduced the adhesion of powder to the upper punch face during compaction. Sticking to the 11-mm concave punch face did not differ significantly ($P > 0.05$) from sticking to the 12-mm concave punch face and therefore punch diameter did not appear to influence sticking when expressed as μg mm^{-2}.

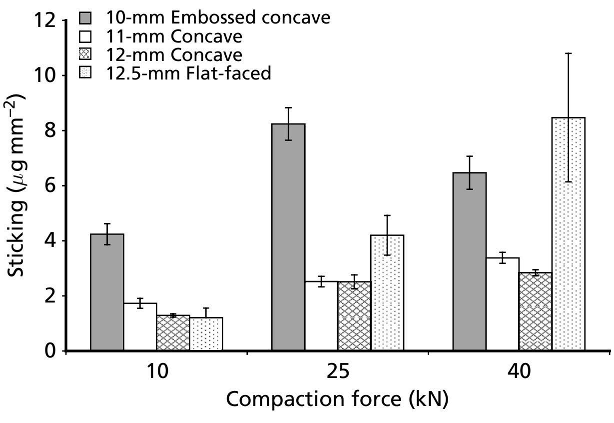

Figure 1 Effect of compaction force on the sticking of an ibuprofen–lactose DC formulation to the upper punch face using 10-mm embossed concave, 11- and 12-mm concave and 12.5-mm flat-faced punches. Mean values $\pm$ s.d., n = 25 for each data set.

The presence of the 'A' logo on the embossed concave punch increased sticking significantly ($P < 0.05$) compared with the non-embossed concave punches (Figure 1). The logo was chosen as the candidate engraving because of the predicted high levels of adhesion. When using punch engravings to apply tablet design, letters or symbols with enclosed areas or with sharp corners are known to be problematic (Young 1995). The increase in adhesion with the presence of an engraving has been ascribed to the concentration of shear stresses at the lateral faces of the engraving (Waimer et al 1999). This explanation appears to be true in the case of the present results. Sticking to the embossed punch appeared to be less dependent on compaction force than the flat-faced punch, with an increase in sticking observed from 10 kN to 25 kN, but little difference between sticking at 25 kN and 40 kN. Sticking at 40 kN when using the embossed concave punch was comparable with that of the flat-face punch at the same compaction force.

Conclusions

Ibuprofen adhesion to the upper punch face during compaction was reduced by increasing punch tip curvature but was independent of punch tip diameter. Embossing of a normal concave punch increased adhesion to levels greater than that observed with a flat-faced punch at 10 kN and 25 kN, and to comparable levels at 40 kN. These findings corroborate previous reports with regard to the problem of sticking and the importance of punch geometry and embossment during tableting. This research is a continuation of earlier studies into the problem of sticking during compaction, specifically with regard to ibuprofen.

References

Aulton, M. E. (1981) Effect of compaction pressure and punch curvature on the indentation hardness profiles of some compressed tablets. *Pharm. Acta Helv.* **56**: 332–336

Aulton, M. E., Tebby, H. G. (1975) Hardness distributions over tablet faces with different curvatures. *J. Pharm. Pharmacol.* **27** (Suppl.): 4P

Newton, J. M., Haririan, I., Podczeck, F. (2000) The influence of punch curvature on the mechanical properties of compacted powders. *Powder Technol.* **107**: 79–83

Roberts, M., Ford, J. L., MacLeod, G. S., Fell, J. T., Smith, G. W., Rowe, P. H. (2003) Effect of punch surface roughness and chrome plating of punch tips on the sticking tendencies of model ibuprofen formulations. *J. Pharm. Pharmacol.* **55**: 1223–1228

Sixsmith, D. (1980) Punch tip geometry effects on powder compression. *J. Pharm. Pharmacol.* **32**: 854–855

Sixsmith, D., McCluskey, D. (1981) The effect of punch tip geometry on powder movement during the tableting process. *J. Pharm. Pharmacol.* **33**: 79–81

Waimer, F., Krumme, M., Danz, P., Tenter, U., Schmidt, P. C. (1999) The influence of engravings on the sticking of tablets. Investigations with an instrumented upper punch. *Pharm. Dev. Technol.* **4**: 369–375

Young, L. (ed.) (1995) *Tableting specification manual*. 4th edn, American Pharmaceutical Association, Washington DC