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Effects of surface roughness and chrome plating of punch tips on the sticking tendencies of model ibuprofen formulations

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Abstract

The sticking of three model ibuprofen–lactose formulations with respect to compaction force and the surface quality of the upper punch were assessed. Compaction was performed at 10, 25 or 40 kN using an instrumented single-punch tablet press. Two sets of 12.5-mm flat-faced punches were used to evaluate the influence of surface quality. A third set of chrome-plated tooling was also used. Surface profiles (Taylor Hobson Talysurf 120) of the normal tooling upper punches indicated a large difference in quality. The punches were subsequently classified as old (Ra = 0.33 μm) or new (Ra = 0.04 μm) where Ra is the mean of all positive deviations from zero. Surface profiles of sample tablets were also obtained. Following compaction, ibuprofen attached to the face was quantified by spectroscopy. Punch surface roughness, compaction force and the blend composition were all significant factors contributing to sticking. Chrome plating of punch faces increased sticking at a low compaction force but decreased sticking at higher forces. Surface roughness of the tablets did not correlate with the corresponding data for sticking, indicating that this is not a suitable method of quantifying sticking.

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Introduction

A common problem encountered during tablet compaction is picking or sticking, which occurs when particles of the tablet formulation adhere to the punch face. The magnitude of this problem can vary from minor punch filming, where a thin layer of powder appears on the punch face, to major picking of the tablet surface.

Numerous methods have been employed to quantify sticking. Waimer et al (1999) studied the adhesion force as the upper punch is detached from the tablet face and claimed that compression force has a specific influence on the adhesion between the tablet and the upper punch, which is dependent on the compression behaviour of the excipients. Toyoshima et al (1988) stated that as sticking is the scratching of the tablet face, evaluation of tablet surface roughness is the best way to quantify the problem.

Pedersen (1999) claimed that sticking could sometimes be overcome by using methods such as chrome-plating the punch tip. However, Schumann & Searle (1992) evaluated the use of chromium electroplating and chromium nitride ion-bombardment as methods of eliminating sticking problems and they reported that chromium-electroplated tooling showed no improvement over untreated tooling, but the chromium nitride ion bombardment method overcame the problems encountered. In two earlier reports the effectiveness of boron-alloy coating of tablet tools was studied. While Tsiftsoglou & Mendes (1982) found that the treated tooling required lower ejection forces, produced tablets with better surface properties, reduced capping tendencies and punch-tip adhesion, Shah et al (1982) reported that the boron-alloy coating increased both ejection and adhesion forces.

The analgesic drug, ibuprofen, displays poor compaction behaviour and has a high tendency for sticking (Rasenack & Muller 2002). Aoki & Danjo (1998) stated that the degree of sticking of an ibuprofen formulation increased with increasing compression speed and decreased with increasing compression force.

The aim of this study was to quantify the sticking of ibuprofen and examine its dependency on punch-surface quality, compaction force and the blend composition of ibuprofen–direct compression lactose formulations. The effect of chrome-plated punches on sticking and the correlation between surface roughness of tablets and sticking was also investigated.

Materials and Methods

Materials

Ibuprofen crystals (BP) 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 0.5% Aerosil 200 and 0.5% magnesium stearate. Three formulations were used containing ibuprofen–lactose 29.5:69.5%, 49.5:49.5% or 69.5:29.5% (formulations I, II and III, respectively).

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). Flat-faced tooling (12.5 mm) was used and 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 per minute.

Surface characterisation

Two sets of 12.5-mm flat-faced tooling with different upper punch surface quality were used to assess sticking. Surface profiles of the upper punches were obtained from a Taylor Hobson Form Talysurf 120 (Taylor Hobson, UK) using inductive pickup (Resolution = 16 nm). The punches were subsequently classified as old or new based on the surface quality. Scans (5×5 mm) of the punch faces were measured and quoted as mean values ($\pm$ s.d.). A profile of the chrome-plated upper punch was obtained by the same method. 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, mean value of the five highest peaks on the surface profile. For the assessment of tablet-surface roughness, measurements of sample tablets were obtained by the same method as described above and mean Ra ($\pm$ s.d.) values quoted.

Scanning electron microscopy (SEM)
Upper punch faces were examined using a JSM-840 Scanning Electron Microscope (Jeol, Japan) with an accelerating voltage of 15 kV.

Sticking quantification
Following each 1-min compaction run, the upper punch was removed and the punch barrel was cleaned of any powder. Immediately, the punch face was immersed in 5 mL of 96% ethanol and gently agitated to allow the surface powder to dissolve. The solution was analysed by spectroscopy at 264 nm using a diode array spectrophotometer (845 2A; Hewlett Packard, Germany) and the amount of ibuprofen attached to the punch face determined (μg).

Results and Discussion

Figure 1 shows a typical surface profile obtained from the Taylor Hobson Talysurf 120 and is a representation of the profiles obtained for the old upper punch face. The surface roughness results (Table 1) and the SEM photographs (Figure 2) obtained show a large difference between the qualities of the two uncoated upper punch faces. Results indicate that the old punch face has far greater deviations and imperfections in its surface than the new punch.

Quantification of sticking (Figure 3) indicated that with an increase in the quantity of ibuprofen in the formulation, the amount of sticking to the upper punch also increased. This would be expected since there are more ibuprofen particles present in the die before compaction, so it follows that more particles will be in contact with, and therefore adhere to, the punch face during compaction. With formulation I there was little difference between the amount of sticking in relation to compaction force or punch surface quality. However, as the ibuprofen in the formulation increased to 49.5% (formulation II), and further to 69.5% (formulation III), the variation in sticking became more apparent. When using the old punch, sticking varied little with respect to compaction force, whereas differences between the results at 10, 25 and 40 kN were more pronounced with the new punch.

Statistical analyses, using a 3-way analysis of variance, showed that surface roughness, compaction force and blend composition were all individually significant

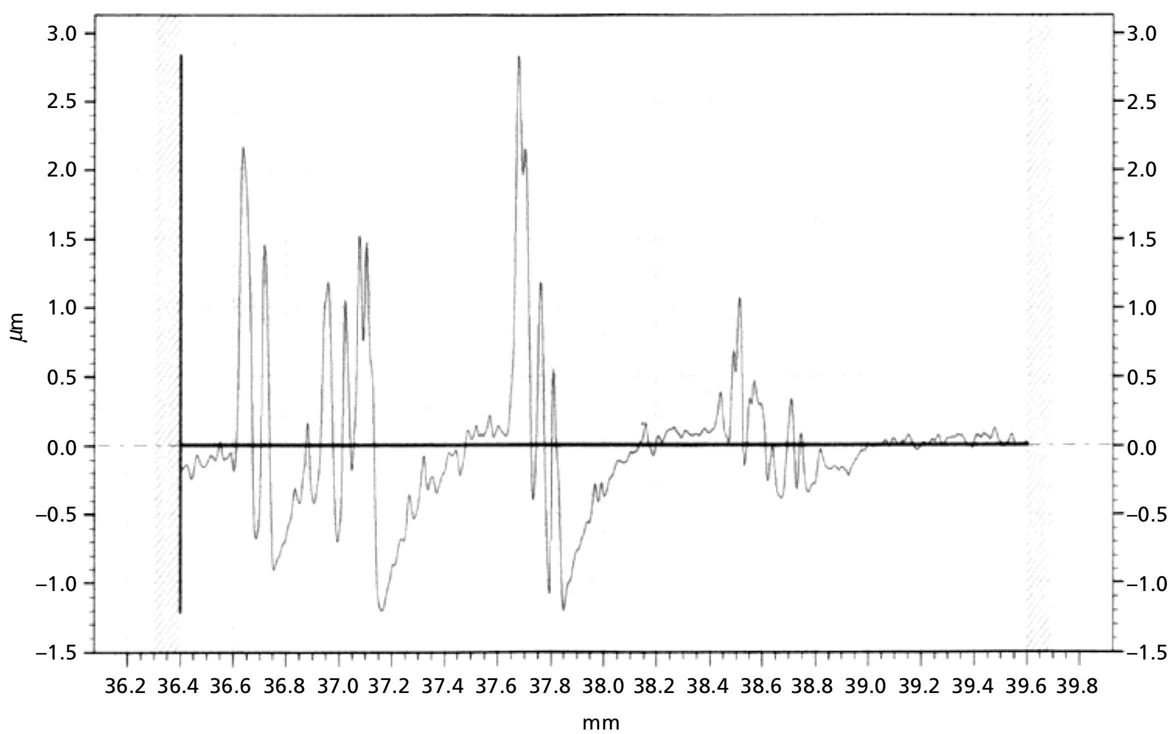

Figure 1 Example surface profile (Taylor Hobson, Talysurf 120), representative of old uncoated upper punch face.

Table 1 Surface roughness (Taylor Hobson, Talysurf 120) of 12.5-mm flat faced old uncoated, new uncoated and chrome-plated upper punch faces.

Surface profile parameter	Old punch	New punch	Chrome punch
Ra (mean of positive deviations (μm))	0.33 ± 0.05	0.04 ± 0.02	0.05 ± 0.01
Rt (maximum range (μm))	3.78 ± 0.86	0.44 ± 0.12	0.19 ± 0.03
Rz (mean of five highest peaks (μm))	2.20 ± 0.21	0.20 ± 0.06	0.10 ± 0.01

Data are means ± s.d., n = 5 for each data set.

($P < 0.05$) factors in tablet sticking. However, they also indicated that the interaction of all three factors was significant ($P < 0.05$) and, therefore, sticking was interdependent on punch-surface roughness, compaction force and blend composition. When using the uncoated upper punches, the highest level of sticking occurred with the new punch at a high compaction force and a high level of ibuprofen in the formulation. More sticking might be anticipated with the punch with the poorer surface quality. Tsiftsoglou & Mendes (1982) reported that boron-alloy coated tooling reduced punch-tip adherence and this was attributed to the separation of the punch tip and tablet face being a function of the adhesion force. This, the authors claimed, was related to the smoothness of the punch tip and therefore an increase in the number of imperfections on the punch tip would result in increased adhesion. However, an explanation for the apparent anomaly in our study may be that the imperfections in the old punch surface reduced the force of attraction between punch and tablet surfaces. The lack of these imperfections in the new punch face did not allow the punch to detach from the tablet surface as readily, thus increasing the level of sticking. When studying the effects of chromium nitride-ion bombardment and chromium electroplating on sticking, Schumann & Searle (1992) found that a punch with a more pitted surface provided less sticking problems than a smoother punch face and attributed this to the imperfections in the rougher punch face acting as loci that break the suction force between tablet and punch.

The surface quality of the chrome-plated upper punch (Table 1, Figure 2) was comparable with that of the new punch. The surface roughness data indicated a good quality smooth surface with few imperfections. As with the old and new punches, sticking with the chrome-plated punches was related to the ibuprofen content of the formulation. However, in relation to compaction force, the

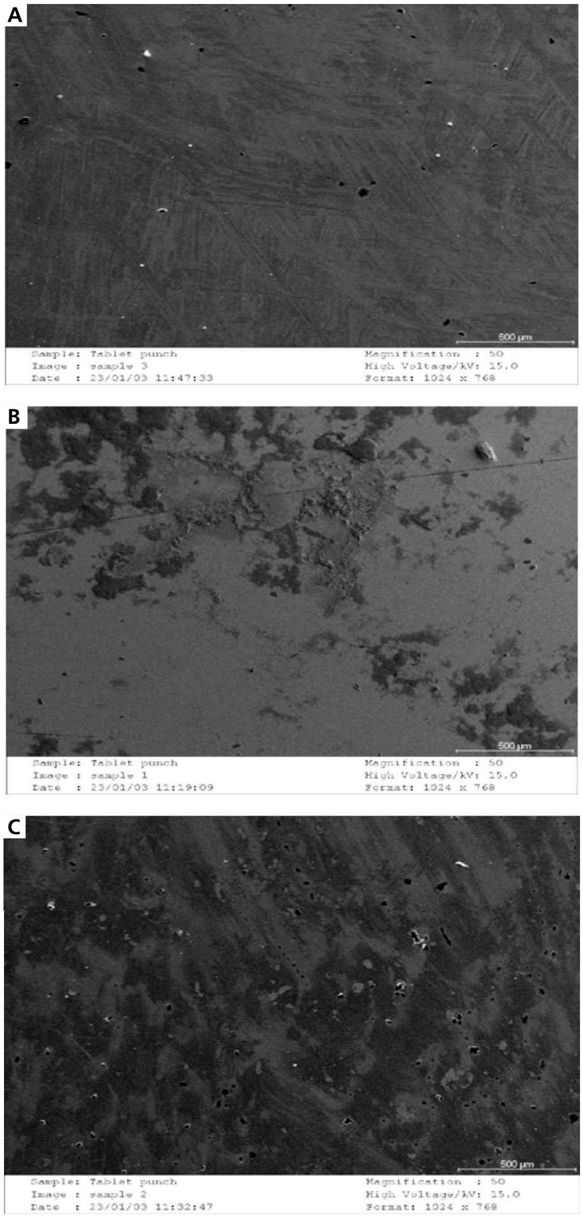

Figure 2 Scanning electron microscope photographs of new uncoated upper punch (A), old uncoated upper punch (B) and chrome-plated upper punch surfaces (C).

sticking results (Figure 3) when using the chrome-plated punch were markedly different and the greatest degree of sticking occurred at the lowest compaction force of 10 kN. The amount of sticking observed when using the chrome punch at 10 kN was greater than the highest levels reached when using the new punch at 40 kN for all three formulations. If the powder formulations in this study were electrostatically charged during the mixing or the die-filling processes (or both) an electrostatic interaction between the chrome-coated punch face and powder particles, which did not occur with normal un-coated tooling, may have caused an increase in adhesion levels. Triboelectrification of powder particles during processing operations may lead to adhesion effects (Eilbeck et al 1999) and as the surface material with which the powder is in contact is influential in the accumulation of electrostatic charge, it is feasible that adhesion due to electrostatic charging is also influenced by the material of the contact surface. Sticking at 25 kN and 40 kN when using the chrome punch was considerably lower than at the 10-kN compaction force (Figure 3) and was comparable with, or better than, the corresponding results when using the uncoated tooling for all formulations. The decreased sticking compared to the level at 10 kN may have been due to the higher compaction forces overcoming the electrostatic interaction between the chrome-plated punch face and the powder particles.

The surface roughness (Ra values) of sample tablets compacted using the new, old and chrome-plated punches are shown in Figure 4. Tablets of formulation III compacted at 40 kN using both old and new uncoated upper punches exhibited capping upon ejection from the die. Therefore, no surface roughness data for these tablets could be obtained. The surfaces of the tablets compacted at 10 kN were generally rougher than those compacted at 25 or 40 kN. In the case of the chrome-plated punches, this increased roughness at a low compaction force corresponded with the results of the sticking quantification, where more sticking of ibuprofen was observed at a low compaction force. However, this correlation did not apply to the results for uncoated punches, as less sticking was observed at 10 kN when using these punches. This indicates that the surface roughness of tablets is not a suitable method for evaluation of sticking. Furthermore, the roughness of the tablets does not appear to be a direct representation of the surface roughness of the punch faces. Comparison of the Ra values shows that those corresponding to the roughness of the tablet surfaces are generally higher than those of the punch surfaces. Podczeck et al (1999) reported that the composition of a powder formulation not only influenced the tablet properties but also the surface properties of the final compact. Therefore, surface roughness of tablets was not merely an imprint of the punch surfaces but was dependent on the formulation and the compaction forces used, and the current results concur with this finding. Although there was a general trend that tablets compacted at 10 kN had rougher surfaces than those compacted at higher forces, there were differences relating to formulations when using different punches. Formulation II produced smooth tablets at 10 kN when using the new punch whereas formulation III produced smooth tablets at 10 kN when using the old punch. An increase in the quantity of a fragmenting material has been reported to increase the surface roughness of a resulting tablet (Podczeck et al 1999). However, lactose is known to consolidate by some degree of fragmentation (Bateman 1988). In this study, an increase in the content of lactose did not result in increased surface roughness of the tablets produced. There appears to be a more complex relationship between tablet surface roughness, composition of the formulation and the punches used.

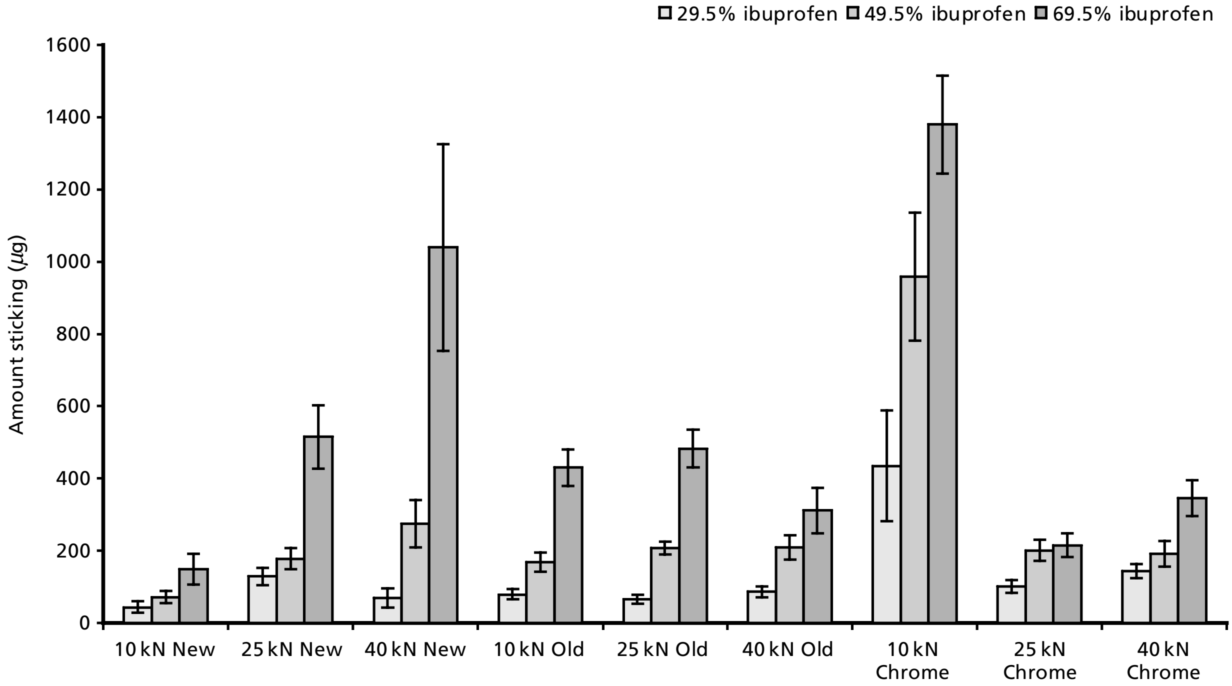

Figure 3 Sticking of ibuprofen with respect to compaction force, blend composition and punch surface (mean ± s.d., n = 25 for each data set).

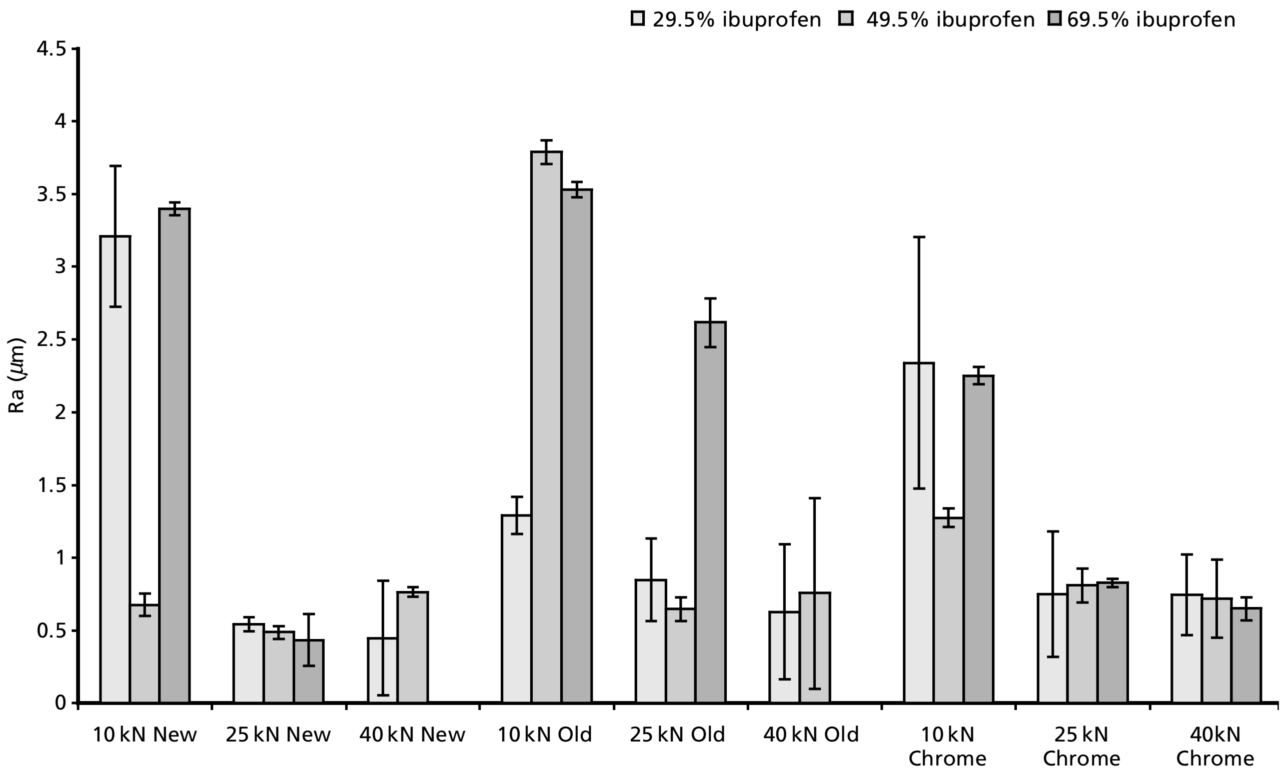

Figure 4 Surface roughness (Ra values) of ibuprofen–lactose DC tablets with respect to compaction force, blend composition and punch surface (mean Ra values ± s.d. n = 5 for each data set).

Conclusions

The relatively simple method developed during this research is a useful way of quantifying the adherence of ibuprofen to the upper punch following tablet compaction. The surface quality of the upper punch is an important factor and a smoother punch face does not necessarily correspond to a reduction in sticking. Chrome-plating of the punch face was ineffective at reducing ibuprofen adherence. The cause of sticking appears to be inter-relationship of the formulation, compression force, punch quality and punch surface material.

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