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A study of powder adhesion to metal surfaces during compression of effervescent pharmaceutical tablets

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Effervescent tablets were produced using four different formulations containing citric and/or tartaric acid and sodium bicarbonate with povidone and macrogol 6000. The same formulations were prepared with the addition of 1% sucrose ester powder. The adhesion of each formulation to the metal faces of tableting machine punch tips was determined using electron microscopy, surface roughness measurements and quantification of punch weight variations during tablet production. The basic formulations were inherently adhesive and produced tablets with a weak, porous structure which were qualitatively and quantitatively rougher than conventional, non-effervescent compressed tablets. Both formulations containing tartaric acid produced tablets with a lower surface roughness and with less tendency to stick to tablet punch faces than the two formulations containing citric acid alone. The addition of a water-soluble sucrose ester had a beneficial effect especially on the formulations with inherently high adhesive tendencies.

The commercial production of effervescent pharmaceutical tablets requires careful selection of formulation components and processes to obtain tablets of the required specification (Aumonier 1970; Moulin 1972; Aiache 1974, 1975; Faguet 1976; Faguet et al 1977; Boymond 1979; Lachman & Lieberman 1980). Formulations which provide optimal disintegration or dissolution times, or produce pleasant tasting solutions for oral administration, frequently have poor compression properties.

As most effervescent tablets are formulated to react chemically with water, it is necessary that the residual moisture content of the granules for compression is minimal (usually below 0·5% w/w). As a result the bonds formed between and within the granules are weak when compared with conventional pharmaceutical tablets.

Official compendia usually require effervescent tablets to disintegrate completely within 1 or 2 min. This is achieved by using a tablet with a relatively large diameter, resulting in an increased specific surface area compared with most pharmaceutical tablets, making a larger surface available to the reacting liquid. The combination of large diameter, thin compacts prepared from weakly bound granules of low moisture content frequently results in porous, brittle compacts that can only be formed at relatively high compression pressures. To achieve a clear solution from the formulation, all the components must either be soluble or react to produce soluble compounds. Such components may have low intrinsic anti-adherent properties when their moisture content is low but they are hygroscopic and may take up moisture to produce intrinsically adhesive granules. This situation can be exacerbated during tablet compression, with the result that material adheres to the compression tool faces and there is 'picking out' of large areas of the compact surface on completion of a compression cycle.

Specialized equipment and formulations can effect large changes in the appearance of effervescent tablets (Sendall et al 1983). To find the effectiveness of these changes requires some quantitative evaluation of tablet surface properties coupled with a qualitative assessment. In the present study, the effect of varying the formulation of an effervescent granulation, and the incorporation of a novel anti-adherent was examined by different techniques. Surface roughness profiles were obtained for 25 mm diameter flat-faced tablets and low-resolution contours were produced by digital processing of the profiles. Arithmetic mean roughness (R_a) values were also obtained and used to characterize surface quality (Rowe 1979).

The surfaces of tablets of all the formulations were examined by scanning electron micrography and quantitative measurement of adhesion to compression tool faces in a single-punch machine was monitored.

MATERIALS AND METHODS

Effervescent tablet formulations

Granulations were prepared from mixtures of organic acids and sodium bicarbonate with povidone

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(Table 1) in a small planetary mixer. After mixing, the materials were granulated with anhydrous industrial methylated spirits. The wet granules were then tray dried at 45 °C, milled through a 2·5 mm aperture screen using a small mill (Apex Equipment Ltd, UK) and then blended with Macrogol 6000 passed through a 0·5 mm screen in an 8 litre cube mixer (type KB20 Erweka, GmbH, FRG).

Table 1. Composition of effervescent formulations used in the study.

	Formulation (% w/w)			
Ingredient	1	2	3	4
Sodium bicarbonate	55·5	61·1	50·4	51·7
Citric acid	42·3	36·7	31·6	—
Tartaric acid	—	—	15·8	46·1
Povidone	0·5	0·5	0·5	0·5
Macrogol 6000	1·7	1·7	1·7	1·7

For the studies on punch tip adhesion, 1 part of food grade sucrose ester (TAL 160, Contract Chemicals, Liverpool, UK) was dry blended with 99 parts of tablet granulation in a cube mixer (Erweka GmbH, Frankfurt, FRG). The lubricated granulations were compressed using a Manesty type RD3 rotary press (Manesty Machines, Liverpool, UK) equipped with 25 mm diameter flat-punches with bevelled edges, or a single punch machine (Manesty type E2) equipped with 12·5 mm diameter flat faced punches.

Before compression, punch faces were cleaned using industrial methylated spirits, tablets were sampled after 100–300 compression cycles.

Electron microscopy

Samples of each formulation compressed into 25 mm diameter tablets were prepared for electron microscopy (Type 35C, JEOL, Japan).

Surface roughness studies

Surface rugosity was studied using a Surtronic 3 display/traverse unit (Rank Taylor Hobson Ltd, Leicester, UK) which interprets data obtained from a pick-up arm that traverses a stylus across the surface to be characterized, in terms of the arithmetic mean roughness (R_a). The unprocessed information can also be passed to a microcomputer. The apparatus is designed to characterize smooth (in powder technology terms) metal or ceramic surfaces and great care was required in setting it to obtain reproducible results with the rough surfaces encountered in the present study.

Preliminary experiments showed that large variations in R_a values could be expected because of the nature of the surface, the short traverse length and the large maximum and minimum values of the height of the surfaces being examined. The apparatus was first used in the R_a mode with a cut-off of 0·8 mm and the standard stylus of 5 μm radius giving an R_a determination over a distance of 4·5 mm.

Mean R_a values were calculated making 30 determinations on each formulation by traversing the pick-up across three dimensions of each of ten tablets. The tablets were mounted in a Perspex holder to eliminate movement. Then profiles of surface roughness were obtained with the Surtronic 3 in traverse mode. In this mode the pick-up traverses 25 mm at 0·25 mm s^{-1} the result being an output voltage proportional to the vertical displacement of the stylus (1 V representing a displacement of 100 μm) which was recorded by an XY chart recorder operating at a chart speed of 2 mm s^{-1}. The ratio of chart and traverse speeds provided a compromise between good resolution of the stylus heights with the necessity of obtaining a trace length of approximately 250 mm.

To obtain a complete picture of the tablet surface, the diametral traverse recording was followed by a 2 mm shift of the tablet under the stylus, perpendicular to the traverse direction. Thirteen parallel chords about 2 mm apart were therefore traversed and recorded for a 25 mm tablet of each formulation sampled after about 100 compression cycles.

The analogue traces obtained were then digitized by a microcomputer (BBC model B, Acorn Computers Cambridge, UK) connected to a PL Graphics system digitizer (B. S. Dollamore Ltd, Burton-on-Trent, UK). The 'peak and trough' positions of the trace were thus translated into digital values and stored, 250–300 points representing a typical diametral traverse. The resolution of the profile was such that ten changes in surface height could be identified per millimetre of tablet surface. A computer program was subsequently developed to classify the surface heights into bands of 20 μm enabling the percentage of points at any height to be determined (see Fig. 1).

Adhesion studies

Adhesion of material to the tips of punches was monitored by compression of each formulation, prepared with and without antiadherent at a force of about 15 kN on the single punch tablet machine equipped with 12·5 mm diameter flat-faced punches. The punches were cleaned as described earlier

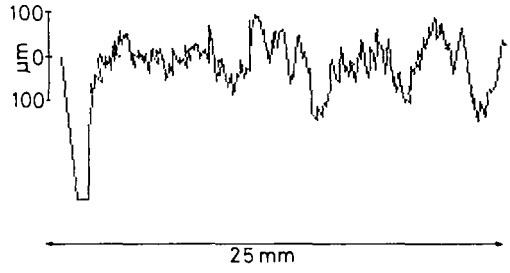

FIG. 1. Typical diametral scan of a 25 mm tablet after digitization and reconstruction by microcomputer. The data from the scans is summarized in Table 3. The height range within which most points occur is seen to be narrowest for those formulations which exhibit the lower R_a values.

and the upper punch weighed before loading the hopper. After completing a pre-selected number of compression cycles, the upper punch was removed and weighed to within 0·1 mg. The data obtained were tabulated and displayed graphically.

RESULTS

Average surface roughness (R_a)

Table 2 shows the mean of the average surface roughness (R_a) values determined. The R_a values for formulations without anti-adherent are much greater than those with anti-adherent, and those formulations containing tartaric acid (3, 4) have a lower R_a than those containing citric acid alone. The mean R_a values are all less than 10 μm while maximal peak values lie between ten and twenty times the R_a value (Table 3). It thus appears that R_a values reflect the overall surface quality, values of 3 μm or less representing the surfaces of conventional tablets, while values of 5 μm or greater indicate a surface of poor appearance and large variations in surface height over the tablet surface (see note, Table 2).

Table 2. Surface roughness (R_a) values for effervescent formulations with and without anti-adherent (mean ± s.d.).

	Formulation number			
	1	2	3	4
	R_a values (μm)			
Without anti-adherent	6·99 ± 1·63	5·35 ± 0·83	4·50 ± 1·39	4·04 ± 0·84
With 1% w/w anti-adherent	3·49 ± 0·89	2·99 ± 0·27	2·46 ± 0·39	2·69 ± 0·52

Non-effervescent tablet produced from starch–lactose granules under similar conditions: mean 3·13 ± 0·62.

Formulation 4 which contains tartaric acid and sodium bicarbonate, gave tablets with over 75% of the surface within a 40 μm height range, while at least 40% of the surface of other tablets prepared with formulations was outside a 60 μm height range (Table 3). The formulations containing citric acid (1, 2, 3) had similar surface height ranges indicating that the percentage composition does not affect surface roughness as much as the inclusion or exclusion of specific components.

Table 3. Percentage of tablet surface above and below a reference line for four 25 mm diameter effervescent tablets of different formulation.

Height of profile/μm	Formulations			
	1	2	3	4
<−100	13·383	2·753	11·973	5·272
−100	7·495	1·733	4·367	1·620
−80	11·242	4·333	7·907	2·763
−60	16·667	7·086	11·596	5·240
−40	16·488	11·547	13·743	28·072
−20	13·954	13·765	14·006	32·899
0	9·172	13·994	13·479	16·577
20	5·639	11·445	9·450	5·049
40	2·819	9·534	6·212	1·302
60	1·392	7·443	4·104	0·349
80	0·750	5·149	1·996	0·127
100	0·000	0·280	0·000	0·000
>100	0·999	10·936	1·167	0·730

For each formulation, irrespective of the degree of roughness, there were proportionally more surface cavities than asperities, both in overall terms and at individual profile contours (Table 3). This is consistent with a 'picking out' mechanism being responsible for major alterations in surface profiles such as are found in effervescent tablets.

Adhesion studies

Table 4 and Fig. 2 show that the addition of anti-adherent to the formulations tested after 100 com-

Table 4. Increase in weight in mg of 12·5 mm upper punch against number of compression cycles for four effervescent tablet formulations without and with (*) anti-adherent.

Formula	Number of cycles								
	0	1	5	10	20	40	60	80	100
1	0	0	0·7	1·6	2·8	7·0	9·1	9·7	7·0
1*	0	0	2·4	3·8	2·1	2·1	1·9	1·1	1·1
2	0	0·2	0·7	1·7	3·3	6·8	7·3	5·3	7·1
2*	0	0	0	0	0·1	0·3	0·7	1·4	1·9
3	0	0·4	0·8	1·2	1·9	3·6	4·4	5·3	8·1
3*	0	0·2	0·2	0·5	0·5	0·3	0·7	1·0	0·7
4	0	0·1	0·7	1·7	1·9	2·0	2·1	1·9	2·1
4*	0	0·3	0·3	0·5	0·3	0·1	0·2	0·7	1·1

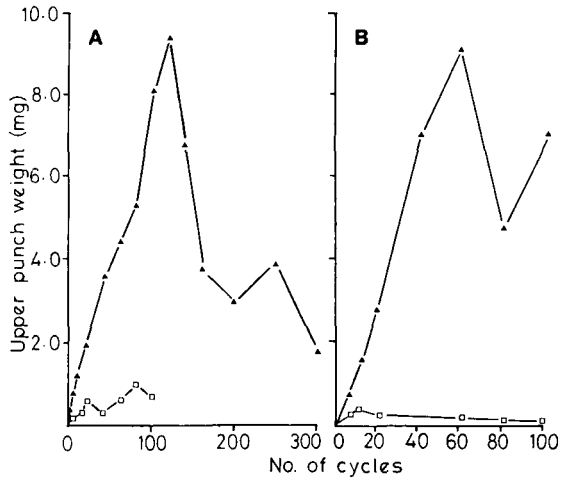

FIG. 2. (A) Plot showing change in punch weight against number of compression cycles for formulation 3 without (▲) and with (□) anti-adherent. (B) Plot showing change in punch weight over 100 compression cycles for formulation 1 without (▲) and with (□) anti-adherent.

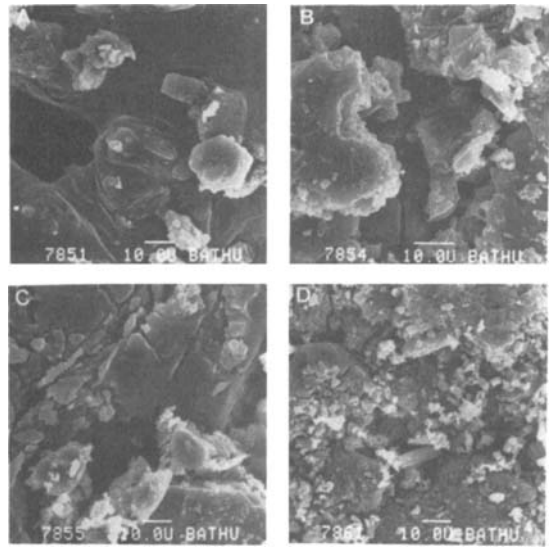

FIG. 3. Scanning electron photomicrographs showing surface detail of tablets compressed from (A) Formula 1; (B) Formula 2; (C) Formula 3; (D) Formula 4.

pression cycles reduced the adhesion of material to punch faces to less than 2 mg in all cases. The largest improvement was effected in formulation 1, while there was minimal improvement with formulation 4 which was the least adhesive initially. This formulation also had the lowest R_a value. Therefore it can be inferred that increased surface roughness is caused by the removal of material from the surface and that the adhesive forces between the punch face and the granulation are greater than the bonding forces within the compressed tablet. The rate at which material is added to the punch face is also formulation-dependent, and is most rapid with the two formulations containing citric acid alone.

The compression forces appear to cause material to leave the tool surface once a film of material has bonded to the metal. This caused the punch weight to vary in a cyclical fashion over periods of 20–60 compression cycles, the compression and adhesive forces acting in opposition as compression continued.

Scanning electron micrographs of tablet surfaces from formulations 1–4 showed highly porous, open structures with little intergranular bonding. At low magnification the surface of tablets with low R_a values (formulation 4) had a fine, uniform structure; at higher magnification (Fig. 3) the surfaces of tablets containing citric acid (formulations 1–3) were observed to have many more cavities and a more open structure than formulation 4. In general, the micrographs correlated well in observed surface quality with the observed R_a values and the roughness profiles obtained.

CONCLUSIONS

Effervescent pharmaceutical tablet formulations are inherently adhesive and the tablets produced have a weak, porous structure. Tablet surfaces have a much higher average roughness than conventional tablets and the surface height may change by over 100 μm within a 100 μm distance.

Formulations containing tartaric acid produced tablets having a lower surface roughness, and less adherence to tablet tools than those containing citric acid.

R_a values may be used to quantify tablet surface quality. Great care must be taken to establish test conditions, and correlate observed quality with numerical R_a determinations. Low values (<3μm) indicate surfaces approaching the quality of conventional (non-effervescent) pharmaceutical tablets.

The water soluble sucrose ester tested at 1% w/w significantly decreased adhesion to tablet punch faces of the more adhesive formulations. Decreasing the adhesive properties of the granulation should decrease significantly the surface roughness of the tablet, and consequently improve the quality of the finished product.

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