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The effect of some formulation and process variables on the surface roughness of film-coated tablets

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The effect of some formulation and process variables on the surface appearance of film-coated tablets has been examined by measuring the arithmetic mean roughness, Ra, values across the faces of tablets before and after they were coated with hydroxypropyl methylcellulose. For all tablet cores except those that were very porous, film coating resulted in an increasing surface roughness; for very porous cores a decrease was found. Tablets with rough surfaces were produced by coating with low molecular weight grades of the polymer; increaseing the polymer molecular weight resulted in a smoother finish with a minimum roughness at intermediate molecular weight grades. Increasing the polymer concentration above 2% w/v caused an increase in roughness as did increasing film thickness to 140 μm. There was a minimum in roughness at film thicknesses of 20 μm. The addition of pigment in low concentrations (0–25% v/v) caused a marginal increase in surface roughness but at concentrations above the critical pigment volume concentration, the surfaces were very rough. The results illustrate the potential of the method in the optimization of film formulations and process conditions during product development.

The physical appearance and surface texture of film-coated tablets are difficult to quantify and, although the use of photomicrography has helped in highlighting differences, comments on these variables have, in most cases, been limited to subjective assessments. In their study of the sugar coating process, Anderson & Sakr (1966) overcame this difficulty by measuring changes in the mean deviation from roundness around the circumference of the tablet. They were thus able to highlight small changes in the coating and relate these to changes in the processing conditions. A similar technique, but measuring changes in the surface roughness on the face of a tablet, has been used in this work to study the effect of some formulation and process variables on the surface appearance of film-coated tablets.

MATERIALS AND METHODS

All the tablets used were from batches used in previous work (Fisher & Rowe, 1976; Rowe, 1976; 1977; 1978). The tablets were prepared by compressing a standard placebo granule, consisting of lactose, starch and magnesium stearate, using an instrumented single punch tablet machine (Type F.3, Manesty Machines Ltd). The tablets were coated with a film formulation consisting of either a mixture of four parts hydroxypropyl methylcellulose (different molecular weight grades of Pharmacoat, Shinetsu Chemical Co. Ltd, Japan or Methocel 60 HG—Dow Chemical Co., U.S.A.) and one part ethyl cellulose (Grade N7 Hercules Powder Co. Ltd, U.S.A.) with 20% w/w glycerol as plasticizer, or hydroxypropyl methylcellulose alone. The formulations were all applied dissolved in a dichloromethane–methanol (70:30% v/v) solvent mixture using either a 6 inch diameter Wurster column or 24 inch 'Accelacota' (Manesty Machines Ltd). The pigments used—titanium dioxide (Grade 1700, D. F. Anstead Ltd, Essex) and F.D. and C. Yellow 5 Aluminium Lake (Colorcon Ltd, Kent) were both ball milled with part of the coating solution for 4 h before use. The film thickness was kept constant at 35 μm.

The mean Ra (arithmetic mean roughness) value (with standard deviation) was calculated from measurements on between six and twelve tablets using a Surfcom 30B fitted with a side skid pick up (Ferranti Ltd, Midlothian, Scotland) according to British Standard 1134 (1972). This system measures surface irregularities using a differential transformer type transducer fitted with a diamond-tipped stylus of radius 3 μm. The transducer is driven along the surface to be measured and the output is amplified and computed to record Ra values. For all tablets larger than 7·94 mm diameter a 4 mm traverse length with 0·8 mm cut off was used but for smaller tablets the traverse length was reduced to 2 mm. All measurements were taken across the diameter of the tablet.

RESULTS AND DISCUSSION

The results of increasing the molecular weight of the hydroxypropyl methylcellulose on the surface

roughness are shown in Table 1; tablets coated with the lowest molecular weight grade show the roughest surface. Increase in molecular weight causes a decrease in the surface roughness to a

Table 1. *Effect of molecular weight of hydroxypropyl methylcellulose on the surface roughness (Ra) of the resultant film coated tablet (tablet diameter 11·11 mm flat, Ra 1·09 ± 0·10 μm, solution concentration 2·5% w/v).*

Polymer	Approx. number average mol. wt	Ra μm
Pharmacoat 603	9 200	2·32 ± 0·27
Pharmacoat 606	11 300	1·73 ± 0·38
Pharmacoat 615	14 500	1·62 ± 0·27
Methocel 60HG v15	14 500	1·62 ± 0·27
Methocel 60HG v50	21 000	1·81 ± 0·32

minimum with intermediate molecular weight grades thus confirming the conclusions drawn from scanning electron photomicrographs of these surfaces (Rowe, 1976). Very rough surfaces were found when tablets were coated with solutions with high polymer concentrations (Table 2).

Table 2. *Effect of solution concentration on the surface roughness (Ra) of tablets coated with a film formulation containing Pharmacoat 606 (tablet diameter 11·11 mm, Ra 1·09 ± 0·10 μm).*

Solution concn % w/v	Ra μm
1	1·36 ± 0·18
2	1·51 ± 0·11
5	2·32 ± 0·26
8	4·83 ± 0·13
10	6·47 ± 0·52

These results are due to the effect of the polymer molecular weight and concentration on the viscosity of the coating solution and hence on the properties of the droplets of coating solution in contact with the tablet surface. During the coating process a solution of the polymer is atomized using a spray gun to produce droplets which, on contact with the tablet surface, spread and penetrate and ultimately coalesce to form a viscoelastic film. A guide to the change in droplet size with varying solution properties can be obtained using the equation (Fair, 1974):

$$\frac{\text{Dvm coating solution}}{\text{Dvm solvent}} = \left[\frac{\sigma \text{ solution}}{\sigma \text{ solvent}}\right]^{0.5} \left[\frac{\mu \text{ solution}}{\mu \text{ solvent}}\right]^{0.2} \left[\frac{\rho \text{ solvent}}{\rho \text{ solution}}\right]^{0.3}$$

where Dvm is the volume mean droplet diameter and σ, μ and ρ the surface tension, viscosity and density respectively.

Because of the large changes that can occur in the viscosity of hydroxypropyl methylcellulose solutions compared with the other variables, it can be seen that, of the three properties, viscosity will exert the greatest impact on droplet size. High viscosity solutions will result in large droplets with a relatively low surface area for evaporation while for low viscosity solutions the opposite will occur. Since the internal viscosity of the droplets will also influence the rate of evaporation of the solvent and the degree of penetration and spreading at the tablet surface, it can be seen that both extremes in viscosity will result in poor spreading and hence rougher surfaces—the low viscosity solutions because of high evaporation rates causing spray drying and the high viscosity solutions because of the inherent high internal viscosity of the droplet. Coating with solutions of medium viscosity should, therefore, produce the optimum film. Although increasing the molecular weight of the hydroxypropyl methylcellulose has an effect on the solution viscosity (Rowe, 1976) this is not so marked as the effect of increasing the polymer concentration, hence the marked increase in surface roughness of tablets coated with polymer concentrations in excess of 5% w/v (Table 2).

Imparting a secondary roughness by coating over the inherent roughness of the tablet core will result in either a smoother or rougher finish, depending on the relative magnitudes of the roughness of both core and film coating. This is shown by the results on the effect of tablet porosity (Table 3) in that film coating a core with a rough porous finish

Table 3. *Effect of tablet porosity on the surface roughness before (I) and after coating with film formulations containing (II) Pharmacoat 606 and (III) Methocel 60HG viscosity 50.* (Tablet diameter 10·0 mm, solution concentration 2·5% w/v).

Tablet porosity %	Surface roughness Ra μm		
	I	II	III
24·3	3·82 ± 1·22	2·99 ± 0·82	2·45 ± 0·20
20·2	2·41 ± 0·53	2·30 ± 0·29	2·25 ± 0·22
9·5	1·32 ± 0·23	1·89 ± 0·08	2·04 ± 0·18
6·2	0·88 ± 0·13	1·88 ± 0·22	2·14 ± 0·25

produces a coated tablet with a smoother finish. The results also show that a smoother finish can be produced by coating these cores with the higher molecular weight hydroxypropyl methylcellulose as opposed to using the standard 11 300 molecular weight grade. As the tablet porosity decreases these trends are reversed. The results on the effect of tablet size and shape (Table 4) show that even allowing for the slight porosity differences in the cores, the surfaces of the biconvex tablets are, in general, rougher than the flat tablets, which is consistent with the generally held view that the surface porosity, and hence surface roughness, at the crown of a biconvex tablet is marginally greater than at the edges due to force variation on compression. These differences are still apparent after coating.

The effect of film thickness (Fig. 1) is complex with a minimum roughness occurring at film thicknesses of 20 μm and then increasing with increasing film thickness up to 140 μm. The results have been confirmed by examination of the surfaces using scanning electron microscopy. The 9 μm thickness film had a very fine structure and appeared very smooth, while the 140 μm thickness film was very rough and had a distinctly 'flake-like' appearance as though the film had cracked and split on drying (Fig. 2). Coverage was complete at all thicknesses.

The addition of pigment in low concentrations to the film causes a marginal increase in surface roughness, but at high concentrations there is a marked increase (Fig. 3A). This point of inflection in the curve is known as the critical pigment volume

Table 4. *Effect of tablet size and shape on the surface roughness before and after coating with a film formulation containing Pharmacoat 606 (solution concentration 2·5% w/v).*

Tablet diam. mm	Shape	Porosity %	Surface roughness Ra μm before	after
6·25	flat	13·3	1·65 ± 0·24	2·15 ± 0·24
6·25	biconvex	13·3	2·07 ± 0·14	2·72 ± 0·49
8·0	flat	14·0	1·83 ± 0·34	2·59 ± 0·16
8·0	biconvex	14·7	2·40 ± 0·17	2·58 ± 0·35
10·0	flat	15·3	2·05 ± 0·58	2·39 ± 0·10

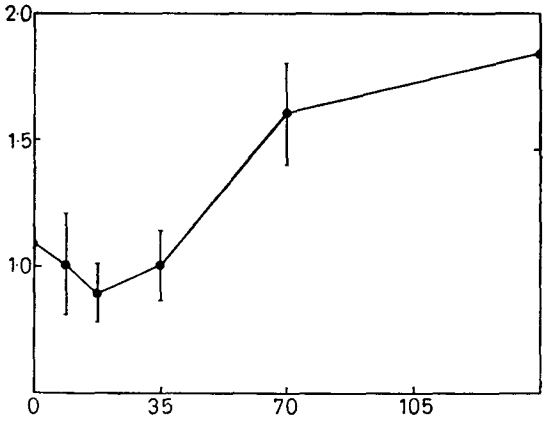

FIG. 1. The effect of film thickness (μm) (abscissa) on the surface roughness (μm) (ordinate) of tablets coated with a film formulation containing Pharmacoat 606. (Tablet diameter 11·11 mm, solution concentration 2·5% w/v).

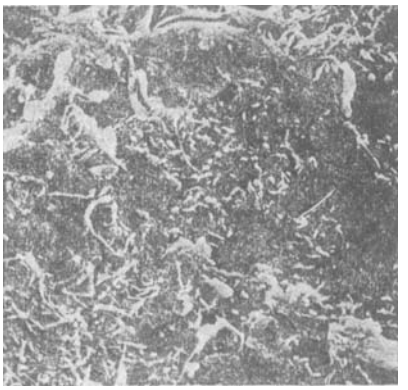

FIG. 2. An electron scanning photomicrograph of the surface of a tablet coated with a film formulation containing Pharmacoat 606—film thickness 140 μm (× 2400).

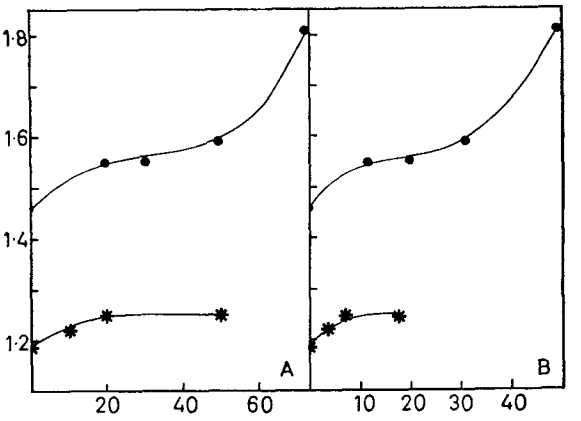

FIG. 3. The effect of pigment concentration (expressed as a % of the film former A as % w/w, B as % v/v) (abscissa) on the surface roughness (μm) (ordinate) of tablets coated with a film formulation containing Pharmacoat 606 ● F.D. & C. Yellow 5 Lake (ρ = 1·92 g ml^{-1}), * Titanium dioxide (ρ = 3·78 g ml^{-1}) (film thickness 35 μm, tablet diameter 11·11 mm, solution concentration 2·5% w/v).

concentration (usually expressed as a v/v concentration to correct for the densities of the pigments—Fig. 3B), and occurs when the ability of the polymer to completely cover the pigment particles is exceeded and the particles protrude through the film. This could be detected visually by a change from a high gloss finish at low pigment concentrations to more of a matt finish at the very high pigment concentrations.

The results show that differences in the surface finish of film-coated tablets can be highlighted by this method and illustrate its potential in the optimization of film formulations and process conditions during product development. The technique is non-destructive and can be used for both flat and biconvex tablets.

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