J. Pharm. Pharmacol. 1981, 33: 1–4
Received July 4, 1980

0022–3573/81/010001–04 $02.50/0

The effect of the particle size of an inert additive on the surface roughness of a film-coated tablet

R. C. ROWE

ICI Pharmaceuticals Division, Alderley Park, Macclesfield, Cheshire, U.K. SK10 2TG

The effect of the particle size of a model inert additive—a dolomite, commercially available in a wide range of grades with varying particle sizes—on the surface roughness of a coated tablet has been studied using a stylus surface roughness measuring instrument. With all grades with a maximum particle size below 10–15 μm there was only a marginal increase in roughness on the addition of the material at low concentrations with a marked increase at concentrations in excess of 20–30% v/v. With the largest particle size material (mean size 18 μm) there was a marked increase in surface roughness at low concentrations with a decrease at higher concentrations. The different trends were due to the influence of the inherent roughness of the tablet substrate used. The results illustrate the potential of this accurate, rapid, simple and non-destructive technique in the optimization of film formulations during product development.

Over the past two decades there has been a considerable increase in the use of polymeric film formers in the coating of tablets. Since additives in the form of pigments (e.g. aluminium lakes of water soluble dyes), opacifiers (e.g. titanium dioxide), various inorganic materials (e.g. iron oxides, calcium carbonate, talc and colloidal silica) and even pharmacologically active materials (e.g. caffeine and salicylic acid) have been included in film formulations (Pickard & Rees 1974; Friedman et al 1979; Porter 1980), it is pertinent to examine the effect of the particle size of such materials on the surface appearance of the final film-coated tablet. This has been investigated using a stylus instrument normally used to record surface profiles of engineering surfaces, but recently successfully applied to the measurement of the surface roughness of both uncoated and film-coated tablets (Rowe 1978, 1979).

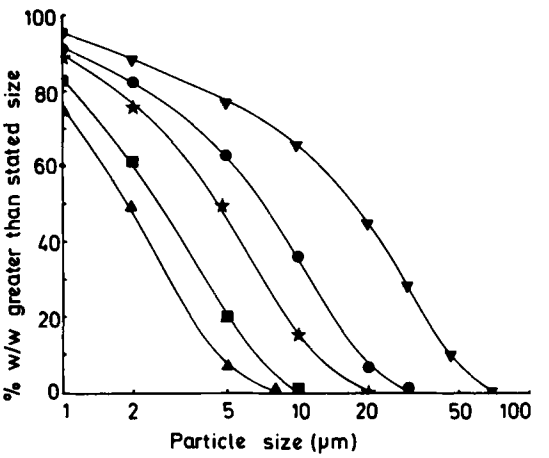

FIG. 1. The particle size distributions of the various grades of dolomite used ▲ Super ■ Extra ★ Medium ● 1 and ▼ 200 (manufacturer's measurements using an Andreasen pipette).

MATERIALS AND METHODS

Because of the wide range of materials added to film formulations it was imperative that a model material be found that was not too dissimilar to materials used in the pharmaceutical industry, that existed in a wide range of particle sizes and that could be dispersed satisfactorily in the coating formulation used. A material—Microdol (A/S Norwegian Talc, Bergen, Norway), was finally chosen. This is normally used in the paint industry and is produced from an extremely white dolomite—a double carbonate containing not less than 99% Ca Mg $(CO_3)_2$- in a wide range of particle sizes (Fig. 1). Specific grades of this material (Super, Extra, Medium, 1 and 200) were added in various concentrations (37·5, 75·0 and 112·5% w/w equivalent to 16, 32 and 48% v/v based on polymer) to a coating formulation consisting of a mixture of four parts hydroxypropyl methylcellulose (Pharmacoat 606, Shinetsu Chemical Co., Japan) and one part of ethylcellulose (Grade N7, Hercules Inc., USA) containing glycerol (20% w/w of polymer) as plasticizer dissolved in a dichloromethane–methanol (70 : 30% v/v) solvent mixture. The coating solution was applied using a 6 inch Wurster column to tablets prepared by compressing a standard placebo granule of lactose, starch and magnesium stearate.

Table 1. The effect of the particle size on the surface roughness of a film coated tablet.

Grade	Mean particle size μm	Concn % v/v	Surface roughness parameters Ra	Rtm μm	Rt	Rp
Super	2·0	16	1·33	7·49	10·59	5·21
		32	1·62	8·64	13·01	6·67
		48	1·56	8·60	11·48	5·07
Extra	2·7	16	1·44	7·84	11·21	4·81
		32	1·56	8·69	11·57	5·94
		48	1·61	9·22	12·21	5·46
Medium	5·0	16	1·29	7·71	10·69	4·61
		32	1·64	9·20	13·73	6·69
		48	1·64	9·61	14·35	6·33
1	8·0	16	1·49	9·24	12·05	5·96
		32	1·52	8·86	12·36	6·10
		48	1·88	10·92	15·21	7·68
200	18·0	16	1·89	11·06	15·41	8·41
		32	1·85	10·47	15·14	7·52
		48	1·61	9·64	14·12	7·22
No additive			1·42	7·06	11·41	5·47

Surface roughness measurements were made on between six and ten individual tablets (11·1 mm flat faced) using a standard surface roughness measuring instrument (Hommel Type T10, Hommelwerke G.m.b.H. West Germany) using a 5 mm traverse length with 0·8 mm cut off. The means and standard deviations of the Ra (the arithmetic mean roughness or centre line average), the Rtm (the average of five peak to valley distances), the Rt (the distance between the highest peak and deepest valley) and the Rp (the distance between the highest peak and the centre line) were calculated from data taken across the diameter of the tablets.

RESULTS AND DISCUSSION

Mean measurements on the coated tablets are shown in Table 1 and Fig. 2. The coefficient of variation was found to be between 6–15% for the Ra and Rtm measurements but higher values (10–30%) were found for the Rt and Rp measurements. This is only to be expected since by definition the former are averages across the surface profile but the latter are single measurements. The ratio of Rtm to Ra was 5·0 for the unpigmented film and between 5·3–6·2 for the pigmented film indicating a slight change in profile.

It can be seen (Fig. 2) that for the three grades with the smallest mean particle sizes (Super, Extra and Medium) graphs of the additive volume concentration versus arithmetic mean surface roughness are sigmoidal, there being no significant difference in the roughness at the 16% level compared with the film with no additive, a significant increase at the 32% level with little or no further increase at the 48% level. For the grade with the intermediate mean particle size (Grade 1) the inflection in the curve is higher occurring at filler concentrations in excess of 32% v/v, while for the grade with the largest mean

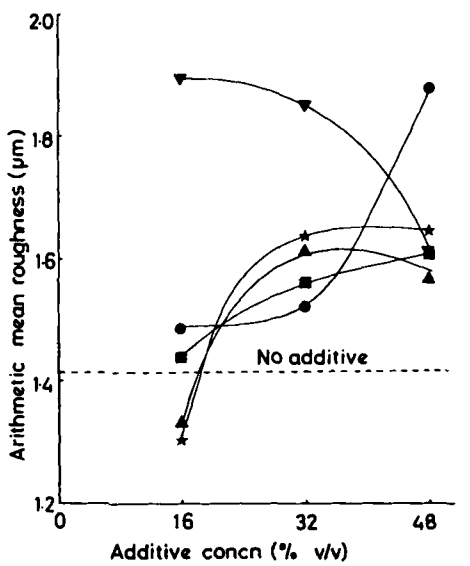

FIG. 2. The effect of additive concentration on the arithmetic mean roughness of a film-coated tablet (symbols as Fig. 1).

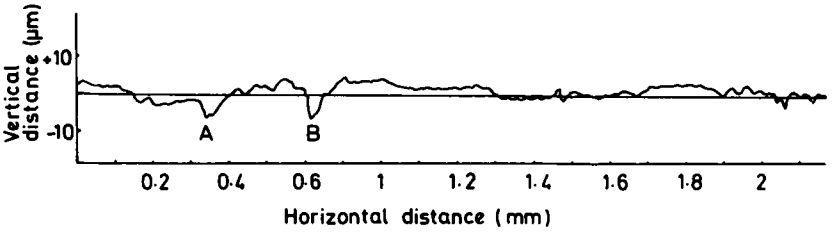

FIG. 3. A specimen profile of the tablet substrate used in this study.

particle size (Grade 200) a complete reversal occurs, there being a marked increase in surface roughness at the lower additive concentrations but at the high concentrations the film becomes smoother. The curves for all the grades except Grade 200 are similar to those given by Asbeck & van Loo (1949) showing the effect of pigment volume concentration on the gloss (a factor closely related to the surface roughness—Hansen 1972) of paint films, and those found in previous experiments (Rowe 1978). Unlike in previous work with a yellow pigment (Rowe 1978) where the differences in the surface finish could be detected visually i.e. from a high gloss at low pigment concentrations to a matt finish at high pigment concentrations, only subtle changes in opacity could be detected in this study.

If the inflection in the roughness/pigment volume concentration graph is related to the critical pigment volume concentration defined as that concentration where there is just sufficient polymer present to cover all the pigment particles, then it would be expected to decrease with increasing particle size as has been shown by Asbeck & van Loo (1949). This does not appear to be the case in this study. However, a further factor, that of the fundamental packing characteristics of the pigment particles, must also be taken into account. In this respect the coarser grades with their wider size distribution will form a more dense packing and this will tend to neutralize the effect of particle size.

The roughness of a coated surface can be regarded as the sum of three components; one due to the coating formulation, one due to the method of application and one due to the irregularities of the substrate used. Since, in this study, only the first of these components has been varied, the others being effectively kept constant, it could be argued that the changes in the roughness of the coated surface must be a consequence of the effect of the filler on the polymer film itself. However, since all the measurements have been made using a tablet substrate and not a smooth flat surface, and since it is generally agreed (Hansen 1972; King & Thomas 1978) that the inherent roughness of the original substrate is the most important component of the three, it is necessary to consider the substrate surface in detail.

A profile of the surface of the tablet substrate is shown in Fig. 3. Unfortunately, because of the differences in the vertical and horizontal magnifications (the former is ten times the latter) the profile is distorted. However, it can be seen that the surface is relatively smooth with some deep valleys, e.g. valleys A and B have the dimensions 70 μm wide, 10 μm deep and 50 μm wide, 15 μm deep respectively. Since a coating applied to a surface naturally fills the valleys first (King & Thomas 1978), it can be seen that, even with polymer present, these valleys could well accommodate all particles with one dimension less than 10–15 μm. This implies that Grade 200 which has a substantial fraction (some 60%) of its particles greater than 10–15 μm (Fig. 1) would be some-what different in its behaviour compared with the rest of the grades—a fact borne out by experiment. However, even this does not explain the decrease in roughness with increasing concentration with this material. Presumably this is due to there being sufficient particles at the highest concentration to pack so as to form an even layer over the tablet surface.

These results have an important practical implication in the film coating of solid dosage forms. Even with a relatively smooth tablet substrate, fillers with particle sizes up to 20 μm (a size in excess of most pigments and opacifiers) can be accommodated at quite high concentrations without unduly affecting the final appearance of the coated tablet. It is possible that if a rougher substrate was used, fillers with even larger particle sizes could be included. The results also illustrate the potential of this accurate, rapid, simple and non-destructive technique of surface roughness measurement in the optimization of film formulations during product development.

Acknowledgements

I wish to thank Mr B. E. R. Hields and Mr M. C. Priestley of Advanced Metrology Systems Limited,

Leicester for assistance in carrying out surface roughness measurements on the tablets.

REFERENCES

Asbeck, W. K., van Loo, M. (1949) Ind. Eng. Chem. 41: 1470–1475

Friedman, M., Donbrow, M., Samuelov, Y. (1979) J. Pharm. Pharmacol. 31: 396–399

Hansen, C. M. (1972) J. Paint. Technol. 44: 61–66

King, M. J., Thomas, T. R. (1978) J. Coating Technol. 50: 56–61

Pickard, J. F., Rees, J. E. (1974) Manuf. Chem. Aerosol News, April 1974, pp 19–22

Porter, S. C. (1980) Pharm. Tech. 3 (9): 55–59

Rowe, R. C. (1978) J. Pharm. Pharmacol. 30: 669–672

Rowe, R. C. (1979) Ibid. 31: 473–474