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The measurement of the adhesion of film coatings to tablet surfaces: the effect of tablet porosity, surface roughness and film thickness

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The effect of tablet porosity, surface roughness and film thickness on the adhesion of hydroxypropyl methyl cellulose films to placebo tablet substrates have been studied using a specially designed tensile tester (Fisher & Rowe, 1976). There were direct relations between measured adhesion and tablet porosity and also surface roughness and tablet porosity. The effect of film thickness on the measured adhesion is complex with an initial decrease with thicknesses up to 35 μm and then a gradual increase with thicknesses up to 140 μm due to differences in in the stress distibution within the film during testing. A knowledge of these effects is necessary if results from various sources are to be compared. The findings illustrate the potential capability of the extrapolation of measured adhesion results to zero porosity and zero thickness values in order to obtain a measure of the true or intrinsic adhesion at any film/tablet interface without the confusing elements of tablet porosity, surface roughness and residual stresses in the film.

The adhesion of a film coating to a tablet substrate has recently been quantified by measuring the force required to remove the film from a known area of the tablet surface using a specially designed tensile tester (Fisher & Rowe, 1976). However, studies using the apparatus have demonstrated the difficulties in interpreting the results in terms of interatomic binding mechanisms especially when the area of contact at the film/tablet interface is variable due to changes in either the surface porosity (Fisher & Rowe, 1976), roughness (Rowe, 1977) or penetration of the polymer solution during coating (Rowe, 1976).

A similar problem due to inherent surface roughness variations is well documented in studies on the adhesion of polymers to metal substrates where it has been, in part, overcome by the extrapolation of measured adhesion results on different surfaces to a zero roughness value (Reegen & Ilkka, 1962). These authors have also suggested extrapolating measured adhesion results to a zero film thickness value in order to obtain a measure of the true or intrinsic adhesion of a monomolecular layer of a polymer to a specified substrate. The relevance of these concepts and their implications in the study of the adhesion of film coatings to tablet surfaces are discussed in this paper.

MATERIALS AND METHODS

Flat faced placebo tablets (11·1 mm diameter) were prepared by compressing a standard placebo granule consisting of lactose, starch and magnesium stearate at different compression pressures using an instrumented single punch tablet machine (Type F3, Manesty Machines Ltd.) The tablets were coated with a film formulation consisting of a mixture of four parts hydroxypropyl methyl cellulose (Pharmacoat 606, Shinetsu Chemical Co. Ltd., Japan, or Methocel 60HG Viscosity 50, Dow Chemical Co. Ltd, U.S.A.) and one part ethyl cellulose (Grade N7, Hercules Powder Co Ltd, U.S.A.) with 20% w/w glycerol as plasticizer. The solution was applied as a 2·5% w/v solution dissolved in a dichloromethane–methanol (70: 30% v/v) solvent mixture using either a 24 inch Accelacota (Manesty Machines Ltd) or a 6 inch diameter Wurster column. The coated tablets were stored at room temperature (20°) and 50°RH for two weeks before testing. Ten tablets were used for each measurement and the mean and standard deviation calculated. The film thicknesses in excess of 50 μm were measured using a micrometer, those below 50 μm were calculated by extrapolation, knowing the relative amounts of polymer applied.

The mean Ra (arithmethic mean roughness) value was calculated from measurements on ten uncoated tablets using a Surfcom 30B (Ferranti Ltd. Midlothian, Scotland) according to British Standard 1134 (1972).*

* This method of quantifying the surface roughness was chosen since it is the most commonly used and hence the results can be directly compared to those of other workers e.g. Reegen & Ilkka (1962). Another parameter, the Rz (average peak to valley height) value has been used to describe the surface roughness of tablets (Nadkarni, Kildsig & others, 1975). Rz values are generally from four to seven times the corresponding Ra values, the ratio depending upon the shape of the surface profile (British Standard 1134, 1972).

RESULTS AND DISCUSSION

Effect of surface roughness and porosity

An analysis of the surface profiles of placebo tablets prepared from two different formulations compressed to different porosities (specimen traces are shown in Fig. 1) shows that there is a direct relation between the Ra value and porosity (Fig. 2) with correlation coefficients of 0·962 and 0·990 for formulations A and B repectively. This is an important result since it correlates the porosity, a characteristic of the bulk, which can easily be calculated from measurements of the weight and dimensions of the tablet and the density of its constituents, and a complex surface characteristic. As expected, the gradients of the two regression lines are different, but the intercepts are statistically insignificantly different from zero.

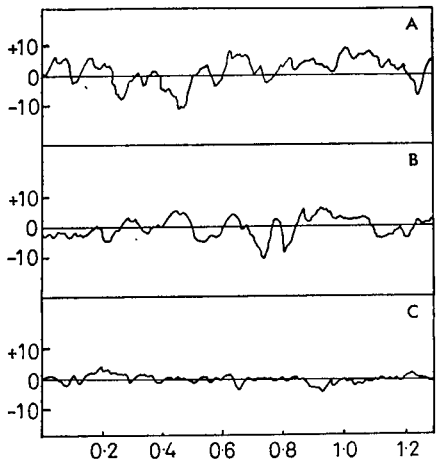

FIG. 1. Specimen traces showing the effect of tablet porosity on the surface roughness profile. A. porosity 24·2%. B. porosity 15·9%. C. porosity 7·8%. Ordinate: Vertical distance (μm). Abscissa: Horizontal distance (mm).

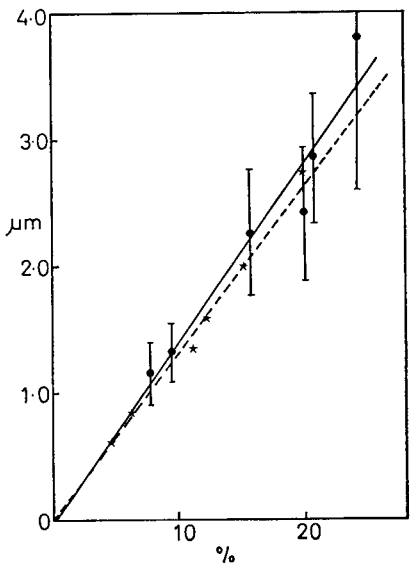

FIG. 2. The effect of tablet porosity (%) on the arithmetic mean roughness (Ra) (μm) for two placebo tablet formulations A (●) and B (★).

A relation between measured adhesion and porosity has already been described by Fisher & Rowe (1976) who found that, after a critical compression pressure, the measured adhesion decreased as the compression pressure increased. Regression analysis on their results shows that above the critical compression pressure of 108MPa there is a direct relation between the tablet porosity and measured adhesion with correlation coefficients of 0·930 for the low viscosity Pharmacoat 606 film formulation and 0·991 for the high viscosity Methocel 60HG viscosity 50 film formulation (Fig. 3). As expected the regression lines show different gradients (± standard error) of 0·821 (±0·188) and 0·586 (±0·079) respectively due to the different rates of penetration of the upper surfaces of the tablet by the two film formulations (Fisher & Rowe, 1976). The differences in the intercepts (± standard error) at zero porosity −23.75 (±2·49) and 24·58 (±1·06) kPa respectively—are statistically insignificant. These values confirm the conclusions drawn by McLaren & Seiler (1949) from their results on the adhesion of polyvinyl acetate polymers to both regenerated cellulose and aluminium substrates in that, providing the failure is adhesive rather than cohesive, adhesion should be independent of the molecular weight of the polymer.

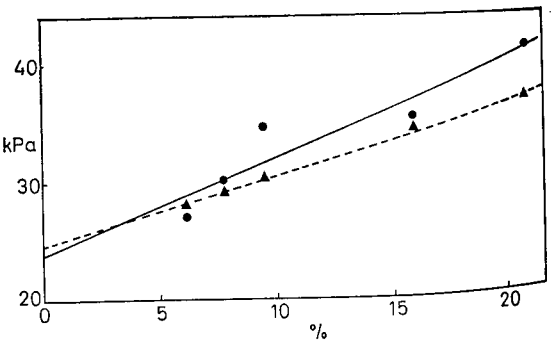

FIG. 3. The effect of tablet porosity (%) on the measured adhesion (kPa) for film formulations containing Pharmacoat 606 (●) and Methocel 60 HG viscosity 50 (▲).

These results illustrate the potential capabilities of the extrapolation of measured adhesion results to a zero porosity value in the comparison of the film/tablet adhesion at any interface without the confusing elements of penetration due to porosity differences in the substrate and viscosity changes in the film formulation, and surface roughness.

Effect of film thickness

The effect of film thickness on the measured adhesion is shown in Fig. 4. In all cases there is a sharp decrease in the measured adhesion as the film thickness is increased to 35 μm (equivalent to an increase in weight of the tablet of approximately 2·5%) followed by a gradual increase as the film thickness increases to 140 μm. Examination of both the coated tablets and the films after removal showed that in all cases there was complete coverage of the tablet surface and that failure occurred at the film/tablet interface, i.e. failure was adhesive rather than cohesive.

It would be expected that, after the initial coverage of a substrate, film thickness should not affect the intrinsic adhesion at the film/substrate interface. However, it appears that the effect of thickness on the measured adhesion is a property of the method of testing, and each testing procedure shows different trends. Simple tensile tests show a decrease in the measured adhesion with increasing film thickness while shear or peel tests show an increase (Gardon, 1967). These apparently anomalous results are due to the differences in the stress distribution within the film during testing. It is well known that, when a film is cast on a substrate, shrinkage occurs on evaporation of the solvent with the creation of stresses within the film. These stresses increase with thickness of film before some limiting value is reached at some defined thickness. During adhesion testing these stresses will either augment or oppose the applied stress and hence affect the measured adhesion (Gardon, 1967). The initial decrease in the measured adhesion obtained in this study (Fig. 4) is a characteristic of the simple tensile test, but the increase in the measured adhesion with very thick films may well be due to further complications in the stress distributions because of the double sided adhesive tape and foam rubber pad used in the design of the apparatus (Fisher & Rowe, 1976).

Extrapolation of adhesion measurements to a zero thickness value as suggested by Reegen & Ilkka (1962) does then provide a method of minimizing the effect of residual stresses in the film. In this case, extrapolation is difficult since the initial decrease in the measured adhesion was not linear and experimental difficulties were encountered in both measuring and testing films below 9 μm thick. A further complication exists in that the measured thicknesses are only mean values and do not take into account the variation that occurs when a film is applied by a spraying technique.

Although from the results (Fig. 4) it would appear that measured adhesion values can be directly compared provided the film thickness is kept constant, extrapolation to zero thickness is necessary if results from various measuring techniques are to be compared and before fundamental studies into the nature and strength of the adhesive bond at the film/tablet interface can be made.

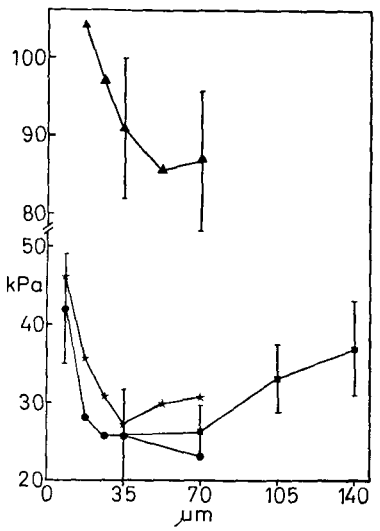

FIG. 4. The effect of film thickness (μm) on the measured adhesion (kPa). Tablet formulation A coated with a film formulation containing Pharmacoat 606 applied using a Wurster Column (●) or 24″ Accelacota (■) and Methocel 60 HG viscosity 50 applied using a Wurster column (★). Tablet prepared from microcrystalline cellulose coated with a film formulation containing Pharmacoat 606 (▲).

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