J. Pharm. Pharmacol. 1980, 32: 214

COMMUNICATIONS

Rate effects in the measurement of the adhesion of film coatings to tablet surfaces

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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). The instrument has since been used to study the effects of some formulation and process variables on film/tablet adhesion, in particular the effect of some direct compression excipients and lubricants (Rowe 1977) and the effect of surface roughness and film thickness (Rowe 1978). The instrument has been designed to operate at a constant speed of 1 mm s^{-1} and uses a foam padding both to ensure an even compressive force over the whole of the tablet surface and to act as a shock absorber when the double sided adhesive tape is applied to the film. Since it is well known that both the rate of application of stress and stress distribution are important variables in adhesion testing (Gardon 1967), it would seem pertinent to study these effects on film/tablet adhesion using the same mode of removal as in the original work (Fisher & Rowe 1976).

Because of the difficulties in modifying the original instrument, the collet holder was removed and modified such that it could be attached to the crosshead of an Instron tensile tester (Model TM, Instron Limited, High Wycombe, Bucks.). Various foam paddings, chosen for having good shock absorbing characteristics (Foam Engineers, High Wycombe, Bucks.) were bonded to a removable holder attached to the shaft of the tensile load cell. The mode of operation and preparation of the tablets for testing were essentially the same as stated in the original work (Fisher & Rowe 1976).

Two tablet substrates were used in this study; one prepared from microcrystalline cellulose (Avicel PH 101, FMC Corporation, Pennsylvania, USA) lubricated with 1% magnesium stearate—Formulation A, the other prepared from lactose granulated with maize starch paste and lubricated with 1% magnesium stearate—Formulation B. Tablets were compressed using flat faced punches (11·1 mm diameter for formulation A, 15·0 mm diameter for formulation B) on an instrumented single punch tablet machine (Type F3, Manesty Machines Limited) to give porosities of 5 ± 1% for formulation A and 15 ± 2% for formulation B. The tablets were coated with a film formulation consisting of a mixture of four parts hydroxypropyl methylcellulose (Pharmacoat 606, Shinetsu Chemical Company Limited, Japan, or Methocel E 50, Dow Chemical Company, USA) and one part ethylcellulose (grade N7, Hercules Powder Company Limited, USA) with 20% w/w glycerol as plasticizer. This was applied as a 2·5% w/v solution dissolved in a dichloromethane–methanol (70:30% v/v) solvent mixture using a 6 inch Wurster Column or a 24 inch Accelacota (Manesty Machines Limited). For formulation A the solution containing Pharmacoat 606 was applied to give two different film thicknesses of 18 and 70 μm. For formulation B the film thickness was kept constant at 35 μm and the grade of polymer varied. Ten measurements were made on each batch of tablets at each crosshead speed and the mean and standard deviation calculated.

The effect of the rate of crosshead movement on the measured adhesion for all four batches of tablets is shown in Fig. 1. All show a small increase in the measured adhesion from the slowest speed up to a speed

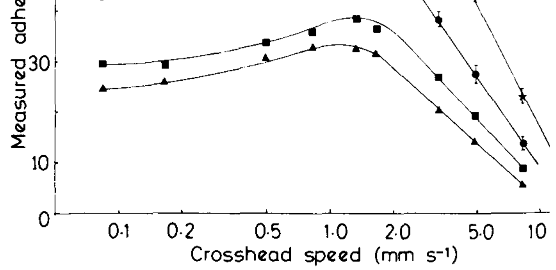

FIG. 1. The effect of crosshead speed on the measured adhesion (★) Formulation A 18 μm thickness film; (●) Formulation A 70 μm thickness film; (■) Formulation B coated with Pharmacoat 606; (▲) Formulation B coated with Methocel E 50.

Table 1. The effect of padding material on the measured adhesion (Formulation B coated with Methocel E 50). Crosshead speed was constant at 1·67 mm s^{-1}.

Material	Adhesion kPa
No padding	20·6 ± 4·8
Expanded nitrile rubber	31·9 ± 4·9
Expanded Polyethylenes	
'Ethafoam'*	25·3 ± 4·9
'Evazote'*	26·1 ± 5·8
'Plastazote'*	26·0 ± 8·7

* These are registered trade marks for different grades of expanded polyethylene.

Table 2. The effect of crosshead speed on the relative changes in the measured adhesion.

Crosshead speed mm s^{-1}	Formulation A 70 μm film: 18 μm film	Formulation B Methocel E 50: Pharmacoat 606
0·083	0·64	0·87
0·17	0·64	0·97
0·50	0·67	0·99
0·83	0·65	0·96
1·33	0·68	0·86
1·67	0·67	0·95
3·33	0·70	0·75
5·00	0·61	0·76
8·33	0·58	0·69

of 1·5 mm s^{-1}, with a decrease at higher speeds. In all cases the measured adhesion or ultimate failure stress was much lower than the tensile strength of the film and failure was observed to be adhesive rather than cohesive. For formulation A it was observed that some adhering substrate had been pulled off with the film.

The effect of rate of crosshead movement is the result of how the rate of deformation can influence, to different extents, the rheological behaviour of the different components in the measuring system, i.e. the foam padding, the double sided adhesive tape and the film itself, and hence how the applied stress is transmitted and distributed at the film/tablet interface. For most polymeric materials the rate of deformation has two main effects. At low rates the materials simply flow while at high rates they behave more as ideal elastic bodies. In all cases their effective moduli increase with increasing rate. Increasing the rate of deformation and hence the effective modulus will result in a more uneven stress distribution at the film/tablet interface and hence a lowered measured adhesion. Decreasing the rate will result in a more favourable stress distribution and hence higher measured adhesions. The effective modulus of the system can be varied by using different padding materials. Table 1 shows the results at a constant crosshead speed of 1·67 mm s^{-1}. There is a gradation in the results, the measured adhesion being highest when expanded nitrile rubber was used (foams prepared from a material with a relatively low modulus $\simeq$10 MPa) and lowest when no padding was used (the effective padding material was steel with a very high modulus $\simeq 10^5$ MPa). Intermediate results were obtained with the three expanded polyethylene derivatives (foams prepared from materials with intermediate moduli $\simeq 10^3$ MPa). However, this explanation does not account for the trend in the results at very low crosshead speeds (below 1 mm s^{-1}). It is possible that at these rates the rupture process is uneven and failure propagates faster than the rate by which the sample is pulled apart i.e. failure occurs by a 'slip-stick' mechanism (Gardon 1967).

Despite these effects the relative changes in the measured adhesions with the two substrates—as shown by the ratios in Table 2—were constant except at the very high crosshead speeds. The trends in the results were the same as those reported previously using the original tensile tester—a decrease in the measured adhesion with increasing film thickness (Rowe 1978) and a small decrease in the measured adhesion on changing the grade of hydroxypropyl methylcellulose (Fisher & Rowe 1976). In the latter case the ratio reported in the original work (0·92) was similar to that calculated in this study.

Although from the results it would appear that the measured adhesion values can be directly compared if the rate of measurement and padding material are kept constant, these are further complicating factors when comparing results from various masuring techniques and in studying, at a fundamenal level, the nature and strength of the bond at the film/tablet interface.

I wish to thank Mr K. D. Barr for technical assistance during this work.

September 17, 1979

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