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The adhesion of film coatings to tablet surfaces—instrumentation and preliminary evaluation†

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The strength of the adhesive bond between a film coating and a tablet surface has been studied using a specially designed tensile testing apparatus. The adhesion has been taken as the force require to remove the film from unit area of the tablet surface and has been shown to be dependent on the compression pressure used to prepare the tablet and on changes in the film formulation. Although plasticizers did not show any significant effect on the adhesion of a hydroxypropyl methylcellulose film, a reduction of 45% was found on the addition of 10% w/w titanium dioxide.

Although soluble film formers are now used extensively in the film coating of tablets little research has been done into the problem of the adhesion of the film coating to the tablet surface.

In contrast, the adhesion of thin films to metallic surfaces has received considerable attention and several methods have been used to study this factor. The earliest method employed was the so called 'Scotch tape' test (Strong, 1935), which consisted of pressing a piece of adhesive tape on to the film and observing whether or not the film was removed when the tape was peeled off. However, this method gave no accurate measure of the strength of the adhesive bond. The first quantitative method of note—"the Scratch test'—was developed by Heavens (1950) in which the tip of a hard stylus is drawn across the film. The film adhesion is then related to the critical load required to produce complete detachment of the film from the underlying substrate along the track of the scratch. This method has been applied extensively to films cast on metal surfaces by Brantley, Woodward & Carpenter (1952) but is unsuitable for use on tablet surfaces because of their greater surface roughness. The only known method reported for studying the adhesion of film coatings to tablets used a modified tensile tester to peel a thin section of coating at 90° to the surface of the tablet (Wood & Harder, 1970, Nadkarni, Kildsig & others, 1975). The peel test suffers from several deficiencies in that the peel angle measured at the surface is dependent on the elasticity of the film and the uniformity of adhesion. These variables can lead to wide variations in the results even when comparing similar coatings on similar substrates. In the present instrument an attempt has been made to ensure that the film is removed normal to the interface between the film and substrate thus giving a direct measure of the adhesion.

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MATERIALS AND METHODS

Instrumentation

The method employed to measure the adhesion between the film and a tablet surface consists of pressing a piece of double sided adhesive tape onto the film coated tablet and measuring the force required to remove the film. Fig. 1 shows the main components of the instrument. The tablet is held with a specially designed quick-release collet and holder which can accommodate tablets up to 20 mm in diameter. Separate collets are required for each size of tablet and these are kept in a rack on the cover of the instrument. The other side of the adhesive tape is fixed to a foam backing 12 mm thick which in turn is bonded to an aluminium backing disc fixed

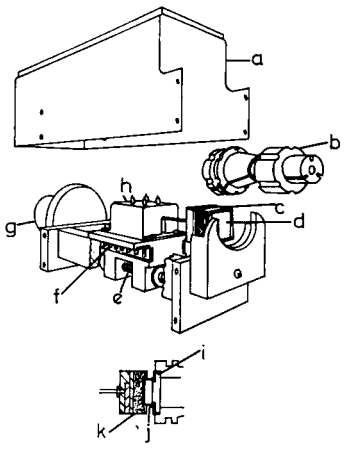

FIG. 1. A diagram of the tablet film adhesion tester showing the main components. a—cover, b—collet holder, c—foam backing, d—double sided adhesive tape, e—screw driving table, f—roller bearings, g—motor, h—dynamometer, i—collet, j—tablet, k—foam.

to the shaft of a small dynamometer (Type UF 1, Pye-Ether Ltd). The foam backing is required to ensure an even compressive force over the whole of the tablet surface when the tape is applied to the coated tablet. The dynamometer is mounted on a table running on linear roller bearings and driving at a speed of 1 mm s^{-1} by means of a screwed shaft driven by a small reversible motor (Fig. 1). Microswitches are mounted under the moving table to prevent overtravel.

Operation and preparation of tablets

In preparation for testing, the film from around the edges of the tablets is removed using a sharp blade. This ensures that the force measured is that required to remove the film from the tablet surface and not that required to tear the coating at the edge of the tablet. The tablet is then mounted in the collet, and the holder fixed into its support. The foam backed disc is removed from the shaft and a piece of double sided adhesive tape (DS adhesive tape, R.S. Components Ltd) fixed to it. The table is driven forward until the tape is firmly adhered to the film (this force is in excess of 10 N). The motor is then reversed and the forces encountered continuously recorded.

A typical trace is shown in Fig. 2; the negative force at point B occurs as the tape is being applied to the coated tablet. The peak force obtained at point D is taken as the force required to remove the film over the whole area of the tablet face. In this study apart from the experiment to study the effect of tablet size, the adhesion has been taken as the force required to remove the film from unit area of the tablet surface.

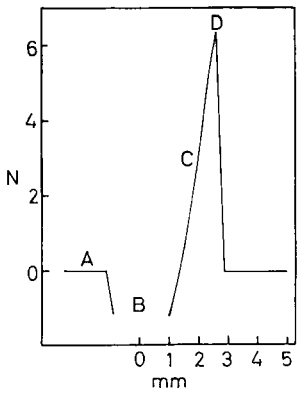

FIG. 2. A typical trace obtained on removing a film from a flat faced tablet, A—zeroforce, B—tape applied to film coated tablet, C—table driving backwards, D—film removed from tablet. y axis—Force required to remove film (N). x axis—Distance travelled by table (mm).

Ten tablets were used for each experiment. These were prepared by compressing a standard placebo granule, consisting of lactose starch and magnesium stearate, using an instrumented single punch tablet machine (Type F3, Manesty Machines Ltd). Flat faced punches were used throughout and, for all experiments other than that used to study the effect of compression pressure, the tablets were compressed at a constant compression pressure of 200 MPa to ensure that the tablet surfaces in any one experiment were identical in porosity. The tablets were coated using either a 6 inch diameter Wurster column or 24 inch 'Accelacota' (Manesty Machines Ltd). The samples were bulked up to the required charge weight for each machine using 8·0 mm normal concave placebo tablets which also served to prevent the flat faced tablets from sticking together. The film formulation consisted of either a mixture of four parts hydroxypropyl methylcellulose and one part ethylcellulose (Grade N7, Hercules Powder Co. Ltd) or hydroxypropyl methylcellulose alone with varying amounts of plasticizer or titanium dioxide. Two grades of hydroxypropyl methylcellulose were used; Pharmacoat 606 (Shinetsu Chemical Co. Ltd, Japan) and Methocel HG 60 viscosity 50 (Dow Chemical Co. U.S.A.), the formulation containing the latter being six times more viscous than that containing the former (both measured at room temperature using a standard U tube viscometer). The formulations were all applied as a 2% w/v solution dissolved in a dichloromethane–methanol (70 : 30% v/v) solvent mixture. The thickness of the film was approximately 30–40 μm. The coated tablets were stored at room temperature and 50°RH for two weeks before testing.

RESULTS AND DISCUSSION

The results in Table 1, show that there is no significant difference in the results for the adhesion measured on either the front or back surface of a tablet indicating that there is minimum disturbance of the film on the back surface of the tablet when

Table 1. *The variation of the adhesion over the front and back surfaces of a tablet coated with four different film formulations (tablet diameter 11·11 mm).*

Film formulation	Adhesion kPa		
	Front surface	Back surface	Mean of both surfaces
A	26·72 ± 4·76	23·93 ± 4·23	24·95 ± 4·65
B	18·61 ± 4·81	17·10 ± 4·28	17·85 ± 4·43
C	17·18 ± 2·51	17·26 ± 1·99	17·34 ± 2·16
D	16·42 ± 2·00	17·97 ± 4·85	17·18 ± 3·70

held in the collet. Therefore, subsequent results are calculated as a mean of twenty readings.

The results of the experiment to study the effect of tablet size (Fig. 3) show that for both film formulations there is a direct relation between the force required to remove the film and the square of the diameter, with correlation coefficients of 0·998 and 0·993 respectively. This result validates the concept behind the design of the apparatus, i.e. the film adheres evenly to the surface of a tablet and the force required to remove the film will be directly proportional to the area of the tablet surface. The coefficient of variation of the force measured for the smallest tablets (6·25 mm diameter) was much higher than that for the largest tablets (15 mm diameter)—33% compared to 18%—due to the difficulties involved in removing the film from around the edges of the small tablets before testing.

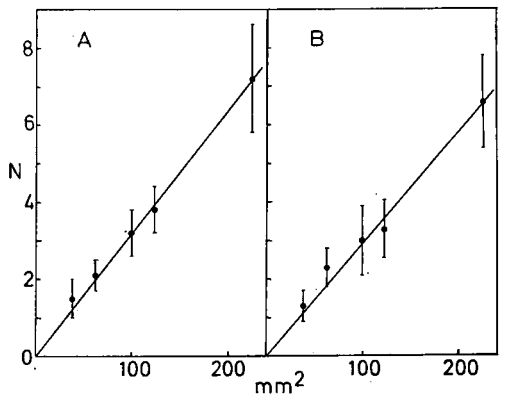

FIG. 3. The effect of tablet diameter on the force required to remove a film from a tablet substrate. A—formulation containing low viscosity grade hydroxypropyl methylcellulose, B—formulation containing high viscosity grade hydroxypropyl methylcellulose. y axis—Force required to remove film (N). x axis—(Tablet diameter)^2 (mm^2).

The effect of compression pressure on the adhesion of two film formulations is shown in Fig. 4. For both formulations the adhesion is at a maximum at a compression of 108 MPa and then decreases as the compression pressure is increased. The latter effect has been reported for other tablet formulations by Nadkarni & others (1975) who suggested that tablets produced at high compression pressures had relatively smoother surfaces than those produced at lower compression pressures resulting in a decrease in the effective area of contact between the film and tablet surface. Further factors must also be considered.

In any coating process some penetration of the partially evaporated coating solution into the upper layers of the tablet surface is inevitable. The rate

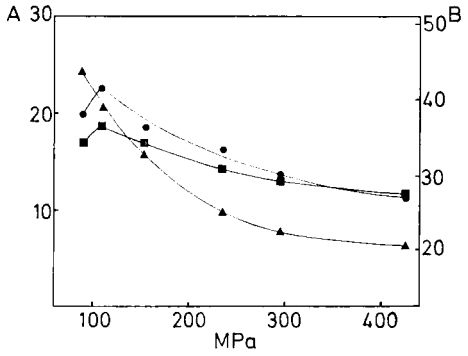

FIG. 4. The effect of compression pressure on the porosity (▲) and adhesion of film formulations containing low viscosity grade (●) and high viscosity grade hydroxypropyl methylcellulose (■). A—Porosity (%). B—Adhesion (kPa). x axis—Compression pressure (MPa).

and depth of penetration will depend on the pore structure of the tablet—the more open the pore structure the faster the penetration—and the viscosity of the coating solution—the more viscous the solution the slower the penetration. The rate of penetration by the film formulation containing the low viscosity grade of hydroxypropyl methylcellulose (Pharmacoat 606) will therefore be much faster than that containing the high viscosity grade and hence the effective area of contact will be higher resulting in higher values for the adhesion. As the porosity of the tablet decreases with increasing compression pressure the rate of penetration for both formulations will decrease until it becomes infinitely slow and the differences in the measured adhesion due to differences in the depth of penetration will become insignificant. For the tablet formulation under test this occurs at compression pressures in excess of 300 MPa with tablet porosities of below 8%.

Such an explanation does not, however, account for the low adhesion values reported for tablets compressed at very low compression pressures. An examination of the films removed from tablets prepared at 91 MPa showed that relatively large amounts of substrate were still adhering to the film while those from tablets prepared at 108 MPa and above showed no evidence of any adhering substrate. It would appear that below this critical compression pressure of 108 MPa the adhesion between the film and tablet is greater than the bonds formed between the powder particles within the tablet resulting in the latter fracturing first under stress.

There have been conflicting reports on the effect of fillers or pigments on the adhesion of polymeric films to various substrates. Brantley (1961) found that the

adhesion of ethylcellulose films cast on aluminium decreased when chalk was added but increased on the addition of talc. Engel & Fitzwater (1962) found that for a polymethyl methacrylate film sprayed on both polymeric and tin substrates the addition of titanium dioxide, ferric oxide and barium sulphate had no effect on the adhesion whereas the addition of mica and talc caused an initial decrease which reverted to the unfilled value at 20% filler concentration. For the film formulation we used, a decrease in adhesion of 45% was produced by the addition of 10% w/w titanium dioxide (Table 2), while the addition of higher concentrations had little further effect.

Table 2. *The effect of the concentration of titanium dioxide on the adhesion of a hydroxypropyl methylcellulose (Pharmacoat 606) film to a tablet substrate (tablet diameter 11·11 mm).*

Concentration of titanium dioxide in film (% w/w)	Adhesion kPa
0	14·45 ± 2·74
10	7·96 ± 1·37
20	8·42 ± 1·98
50	9·18 ± 2·26

The effect of plasticizers and plasticizer concentration on the adhesion of a hydroxypropyl methylcellulose film is shown in Table 3. Although there appears to be a slight decrease in the adhesion on the addition of both glycerol and propylene glycol the effect is not statistically significant. More dramatic results have been reported by Brantley (1961), who found a 50% decrease in the adhesion of a nitrocellulose film when the percentage of dibutyl phthalate was increased to 40%, and by Engel & Fitzwater (1962) who found similar results for a polymethyl methacrylate film containing a variety of plasticizers.

It is generally accepted that the adhesion of a film to a substrate is due to the formation of hydrogen bonds between the polar groups of the film former and substrate constituents. Pigments and fillers will interfere with this interaction by becoming embedded between the polymer film and the substrate and thus cause a reduced adhesion. Plasticizers would also be expected to have some effect on this interaction since they are thought to act by associating with the polar groups of the film former within the film structure. This is especially true with plasticizers such as glycerol and the glycols in cellulose ether films. This would tend to lower the adhesion to a degree dependent on plasticizer and on concentration.

Table 3. *The effect of plasticizer and plasticizer concentration on the adhesion of a hydroxypropyl methycellulose (Pharmacoat 606) film to a tablet substrate (tablet diameter 11·11 mm).*

Plasticizer	Concn in film % w/w	Adhesion kPa
Unplasticized film	—	19·76 ± 4·43
Propylene glycol	10	18·99 ± 4·49
Propylene glycol	20	17·66 ± 3·19
Glycerol	10	18·23 ± 3·62
Glycerol	20	18·24 ± 4·98

The overall results show that differences in the adhesion of film coating to tablet surfaces can be accurately measured using this method. Although the results have been calculated as a force per unit area it is possible to compare a series of related samples in terms of the force required to remove the film provided the tablet size is kept constant. The method offers serveral advantages over the peel test in that normal sized tablets coated using conventional film coating equipment can be used. Although flat-faced tablets have been used in this study, biconvex tablets can be accommodated. However, the film at the edge of the tablet is removed slightly before the film at the centre so the ideal conditions established for flat-faced tablets are not found and a more detailed analysis of the results is required.

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