DRUG DEVELOPMENT AND INDUSTRIAL PHARMACY, 21(14), 1611-1619 (1995)

ASSESSMENT OF THE THICKNESS VARIATION AND SURFACE ROUGHNESS OF AQUEOUS FILM COATED TABLETS USING A LIGHT-SECTION MICROSCOPE

Twitchell, A.M., Hogan, J.E.* and Aulton, M.E.
Pharmaceutical Technology Research Unit, Department of Pharmacy, De Montfort University, The Gateway, Leicester LE1 9BH, U.K. and *Colorcon Ltd., Orpington, Kent BR5 3QY, U.K.

ABSTRACT

The Light-Section Microscope measures the thickness and surface roughness of transparent film coats without contacting them. It enables the determination of tablet film coat thickness at different regions on a tablet surface and analysis of coat thickness variation. Examination of aqueous film coated tablets using a Light-Section Microscope has shown that the coat application conditions can influence the film density and thickness, film thickness variation and film coat surface roughness. Coat application conditions which give rise to smoother surfaces are shown to produce more dense, thinner coats which may exhibit a more even thickness on smooth substrates but larger variations in film thickness on rough substrates. The potential for film thickness and surface roughness to vary at different positions on the substrate surface has also been demonstrated, with film coats tending to be smoother and thinner at the periphery of tablet faces.

INTRODUCTION

The thickness of polymer films applied to tablets is often determined using a micrometer after film removal from a tablet, or by extrapolation from knowledge of the amount of polymer/coat applied. The former method is destructive and measurements reflect the thickest part of the film. Adhesion of substrate particles may also lead to artificially high thickness values being obtained. With the latter method, accurate values for film density and coating efficiency are required for meaningful thickness calculations to be made. Both methods yield a single value for film thickness and give no indication of thickness variation.

The Light-Section Microscope

The Light-Section Microscope (Carl Zeiss, Oberkochen, Germany) measures the thickness of transparent film coats without contacting them, allowing the determination of tablet film coat thickness at different regions on a tablet surface and analysis of coat thickness variation. Assessment of the coat surface roughness can also be made without physical contact of the tablet surface, as would occur when testing using profilimetry.

The Light-Section Microscope utilises a lamp to illuminate a slit which projects a narrow plane of light at an angle of 45° to the surface being assessed. This band of light is then observed as a series of peaks and troughs through a microscope at the opposite 45° angle after it has been reflected/refracted from the sample (see Figures 1 and 2). The image appears inverted in the eye-piece. A cross-line reticle in the eye-piece can be shifted within the field of view by means of a graduated measuring drum. The required distance values can then be read off the drum with a sensitivity of 0.1 μm. A mechanical stage allows longitudinal or transversal movement of the sample by up to 25 mm by means of micrometeres.

Surface roughness parameters which can be obtained using the Light-Section Microscope include; Rt (the distance between the highest peak and deepest valley, Rtm (the average of five peak to trough distances) and Rw (the average distance between peaks). Calculation of Ra (the arithmetic mean roughness) can only be undertaken after a photographic record has been obtained.

If a transparent film is being analysed, two light bands are seen in the eye-piece, one corresponding to the film surface and the other to the surface of the substrate. Due to the refraction of the light as it penetrates the transparent layer, the distance between the light bands as measured through the eye-piece does not represent the true thickness of the coating (Figure 2). The relationship between the actual thickness of the coat (T) and the apparent thickness (T^o) can be calculated from the formula:

$$T = (2n^2 - 1)^{0.5} \, T^o$$

where n is the refractive index of the transparent layer. For HPMC coatings studied in this work, the value of n was taken to be 1.49 [1] and thus $T = 1.85\, T^o$.

EXPERIMENTAL

Flat-faced 10 mm and 15 mm placebo tablets and scored convex 10 mm placebo tablets were prepared from Microcrystalline cellulose NF (79.25 %w/w), Pre-gelatinised starch NF (20 %w/w) and Stearic acid BPC 1973 (0.75 %w/w). One hundred of each of the three tablet types were added to 12 kg of 8 mm normal convex tablets and coated in a model 10 Accela-Cota with drying air at 65°C, a

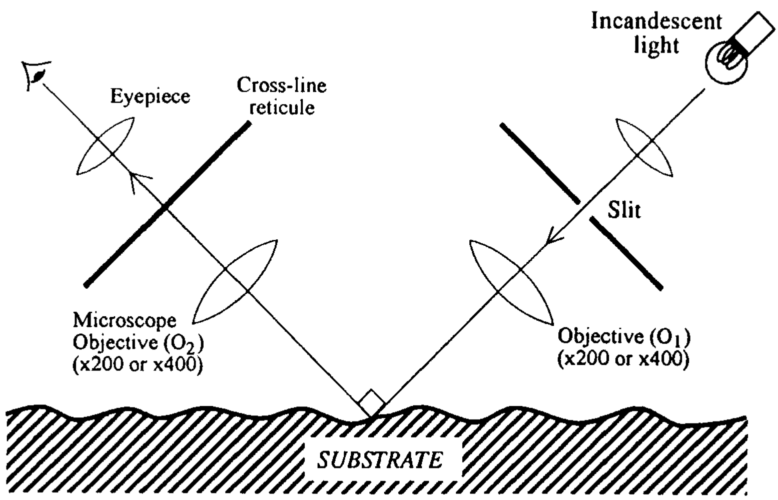

FIGURE 1
Schematic representation of the Light-Section Microscope

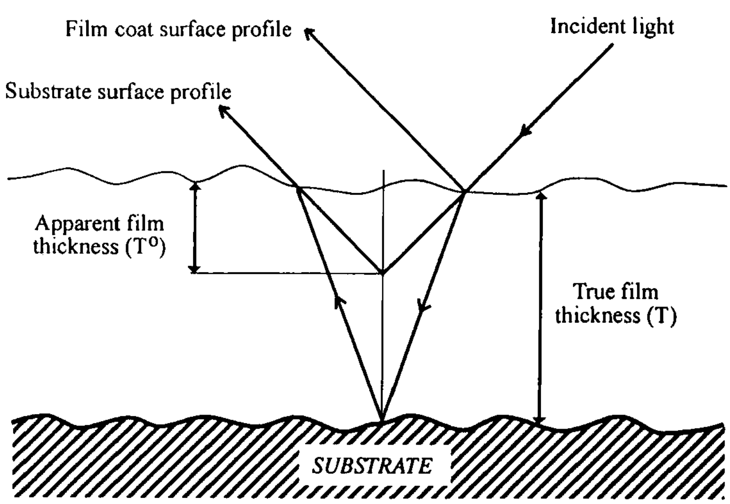

FIGURE 2
Light path through a coated substrate

volumetric air flow rate of 0.13 m^3s^{-1} and a pan rotation speed of 10 rpm. Different spray guns were utilised to apply a 9 %w/w HPMC E5 solution at a rate of 40 $gmin^{-1}$ using various atomizing air pressures, spray shapes and spray gun positions (see Table 1). Sufficient coating solution was applied to give a coat level of approximately 2.25 %w/w (based on uncoated tablet weight). Values of Rtm, Rw and coat thickness for the coated tablets were assessed using the Light-Section Microscope. Measurements were taken from different positions on the tablet surface on at least five different tablets for each coated tablet type studied. For the flat-faced tablets, measurements were taken over the central region of the tablet face and over the peripheral 0.5 mm. With the convex tablets, measurements were taken over the tablet crown, within the score and over the area midway between the crown and the face periphery. Film thickness was calculated both from the coat peak to the substrate (designated T_1) and the coat trough to the substrate (designated T_2) for five consecutive peaks/troughs from each measured region. The ratio of T_2 to T_1 was calculated to give an indication of how evenly the coat was applied.

RESULTS

The data from tablets examined using the Light-Section Microscope are reported in Table 2.

Film roughness and thickness values were found not to be significantly different at different positions on the central region of the face of the flat tablets and average values have therefore been presented. Film roughness and thickness were found however to be considerably lower on the periphery of the face of these tablets compared to the central region are reported separately.

With the 10 mm convex tablets, differences in the roughness and thickness characteristics were noted when measurements were taken on the crown of the tablet, within the score and midway between the crown and the periphery. Roughness values appeared greatest on the crown and lowest within the score. It was not possible to determine roughness values at the periphery of the convex tablets due to the geometry of the tablets and the measuring capabilities of the Light-Section Microscope.

Film surface roughness (Rtm) values were found to be dependent on the coating application conditions used. Increasing the spray gun-to-bed distance, changing the spray shape from a narrow cone to a wide flat spray and decreasing the atomizing air pressure all appeared to produce rougher surfaces. The type of spray gun used did not appear to have a significant effect on surface roughness. The Rtm values obtained from the Light-Section Microscope provided similar information on the effect of these variables as values of Ra obtained from a traditional stylus measuring instrument [2].

TABLE 1
Details of Coating Runs

Coating run number	Spray gun*	Atomizing air pressure (kPa)	Spray shape	Gun-to-bed distance (mm)
1	Walther Pilot	552	flat	180
2	Walther Pilot	276	flat	180
3	Binks Bullows	552	flat	180
4	Schlick	552	cone	180
5	Schlick	276	flat	180
6	Schlick	276	flat	250
7	Schlick	552	flat	250

* Spray gun details
Walther Pilot model WA/WX with a type 0.5-1.5 air cap and a 1.0 mm diameter liquid nozzle
Binks Bullows model 540 with type 63PB air cap and type 66 liquid nozzle (1.8 mm diameter)
Schlick model 930/7-1 with a 0.8 mm diameter liquid nozzle

Values of Rw were also found to be dependent on the coating conditions with smaller values being associated with greater values of Rtm and larger values with smoother surfaces. Thus it would appear that generally those coats which have greater vertical average distances between peaks and troughs also have a greater frequency of peaks/troughs across the tablet surface. Values measured on the 10 mm flat and 15 mm flat-faced tablets were of the same order.

Film thickness readings (T_1 and T_2) showed there to be considerable differences between tablets taken from different coating runs. Similarly differences between the ratio of the two parameters were demonstrated. In every case the value of T_1 was greater than T_2, indicating that the average distance from the coat peak to the tablet surface was always greater than that from the coat trough to the tablet surface.

Lower film thickness values appeared to be associated with smoother film surfaces, with these films also possessing relatively larger variations in film thickness (as evidenced by low T_2:T_1 ratios). When viewed through the Light-Section Microscope, it was apparent that these films did not follow the contours of the substrate, i.e. the peaks in the coat profile did not occur at the point that peaks in the substrate profile occurred. The converse was found for

TABLE 2
Film Coat Thickness and Surface Roughness Values Measured Using a Light-Section Microscope

Coating run	Tablet type (region)	Rtm (μm)	Rw (μm)	Coat thickness T_1 (μm)	T_2 (μm)	T_2/T_1
uncoated	15mm flat (central)	10.5				
uncoated	10mm flat (central)	17.1				
uncoated	10mm convex*	18.3				
uncoated	10mm convex (crown)	30.3				
uncoated	10mm convex (score)	4.1				
1	15mm flat (central)	9.0	155.7	44.1	22.2	0.503
1	15mm flat (periphery)	1.4		29.0	17.0	0.586
1	10mm flat (central)	9.9	175.5	42.4	28.7	0.677
1	10mm flat (periphery)	7.0		32.5	19.0	0.585
1	10mm convex*	10.5		50.2	35.4	0.705
1	10mm convex (score)	3.6		41.4	38.5	0.930
2	15mm flat (central)	12.4	187.3	56.2	42.0	0.747
2	15mm flat (periphery)	2.5		47.0	38.6	0.821
3	15mm flat (central)	10.7	239.5	46.7	35.6	0.762
3	15mm flat (periphery)	6.8		40.5	30.6	0.756
3	10mm flat (central)	10.7	251.7	58.7	45.1	0.768
3	10mm flat (periphery)	8.1		45.2	31.9	0.706
3	10mm convex*	10.5		58.8	48.9	0.832
3	10mm convex (score)	1.3		53.1	51.5	0.970
4	15mm flat (central)	6.2	278.1	42.4	22.6	0.533
4	10mm flat (central)	7.2		45.1	25.7	0.569
4	10mm convex*	5.9		46.2	25.4	0.550
4	10mm convex (crown)	9.1		56.3	12.3	0.218
4	10mm convex (score)	2.3		38.0	34.4	0.905
5	15mm flat (central)	11.6	123.8	55.1	43.6	0.791
5	15mm flat (periphery)	4.3		41.8	36.7	0.878
6	15mm flat (central)	14.9	127.8	58.1	46.9	0.808
6	10mm flat (central)	15.3	120.1	51.0	37.1	0.727
6	10mm convex*	12.1		55.2	47.0	0.851
6	10mm convex (score)	6.5		55.4	49.0	0.884
7	15mm flat (central)	15.4	132.5	52.6	43.9	0.835
7	10mm convex*	12.5		51.0	45.1	0.884
7	10mm convex (score)	6.0		51.2	49.2	0.961

* region midway between crown and face periphery

relatively rough film surfaces, with the contours of the film tending to correspond to those of the tablet.

With the 10 mm convex tablets the variation in film thickness was found to be greatest on the crown of the tablet where the tablet surface was roughest. Within the tablet score (where the tablet surface was very smooth), little thickness variation was apparent.

DISCUSSION

Theoretical film thickness values calculated from the tablet dimensions, the amount of polymer applied and a film density of 1.2 $g\,cm^{-3}$ (cast film density value [3]) were found to be approximately 24 µm. The average film thickness values determined from the Light-Section Microscope data were however considerably greater than the theoretically calculated values and varied markedly between tablets from different coating runs. This effect was not thought to be due to differences in coating efficiency (ratio of the total weight of coating on the tablets to the total weight of solids applied) since those coating runs where the efficiency was likely to have been lower (e.g. where increased gun-to-bed distances were employed or flat sprays used) did not produce thinner coats.

The observed differences in film thickness, both from the theoretical values and between tablets from different coating runs are thought to have arisen predominately from variations in film density caused by the use of different application conditions. Similar conclusions could be drawn from photographic evidence of sprayed HPMC films [4]. The potential for spray application conditions to influence film density has also been reported for free films generated from solutions of cellulose acetate in acetone [5]. Coated tablet film density will be dependent upon the degree of air entrapment within the film. This air may be trapped either within spray dried droplets or between film layers due to insufficient droplet spreading and coalescence on the tablet surface. Thus factors which enhance spreading (increased atomizing air pressure or positioning of the gun closer to the tablet bed) or reduce the tendency to spray dry (use of a narrow spray angle) would be expected to produce thinner, more dense films. This is substantiated by the results in Table 2.

Theoretical film thickness values are only likely to be produced if the application conditions produce films which have a similar density to cast films. This may occur with solutions applied using conditions which maximise droplet spreading and coalescense and minimise spray drying.

Variation in film thickness at different regions on the tablet surface was particularly apparent with coating conditions which produced smoother, more dense, thinner films. Since these conditions enhance droplet spreading, the droplets may preferentially "fill in" irregularities in the substrate surface [6]. This was supported by the image seen through the Light-Section Microscope, which showed the thinnest areas of coat to exist at the peaks of the uncoated tablet surface. Application conditions which produced relatively thick coats tended to form films with a much more even thickness, the films more closely following the contours of the uncoated tablet surface.

With the 10 mm convex scored tablets, differences in uncoated tablet surface roughness at different regions on the tablet surface would contribute to the film thickness variation. Within the score where the tablet was very smooth, there were few irregularities to fill in and therefore the potential for film thickness variation was low. On the crown where the surface was much rougher, a large proportion of the coat may have been used to fill in irregularities before the coat was built up over the peaks of the uncoated tablet surface, this giving rise to a greater likelihood that large differences in film thickness will exist.

The lower average film thickness at the tablet face periphery was probably due to the greater attrition forces experienced here during tumbling in the coating pan. This would also account for the smoother surfaces at the face periphery.

Variation in film thickness at different points on the tablet surface may be important if the coat is to be used to modify drug release, since the drug retarding properties of the coat may depend on the thinnest parts of the film coat. In these circumstances it may be possible to reduce the level of coat required to confer the desired properties by producing a coat of even thickness e.g. by ensuring the substrate has a low surface roughness and the application conditions produce a smooth coat. The widest variation in film thickness is likely to occur with rough substrates and coating conditions which produce a low surface roughness.

The occurrence of variations in film thickness across the tablet face also highlights the potential inaccuracy of determining film thickness values using a micrometer, since this instrument is likely to only measure the thickest part of the film. Similarly calculation of the theoretical thickness values from cast film density measurements may only be applicable if the coating conditions produce films with a density similar to that of the cast film.

It has been reported previously that the film coat surface roughness within a tablet intagliation can be 2 - 3 times higher than on the tablet body [7]. It was suggested that on the exposed surface the shear forces arising from mutual rubbing were high enough to smooth out the partially gelled coating formulations, whereas within the intagliation only small surface tension forces existed and little levelling occurred. The data in Table 2 however do not support this theory, since in each case studied, the roughness in the score mark of the 10 mm convex tablets was considerably lower than on the rest of the tablet. One factor which is likely to have contributed to this effect was the very low initial roughness within the score, although this was also the case in the earlier study [7]. The reasons for the discrepancy between the findings of this study and those reported previously [7] are difficult to determine due to the lack of coating process details reported in the latter case. It could be that fundamental differences in the way the films are formed within the intagliations and score marks may be responsible. Alternatively, differences in the methods of determining the surface roughness may have contributed. The Light-Section Microscope used in this study measured the surface roughness of the film itself, whereas the stylus instrument [7] could not discriminate between the film and any surface debris (e.g. spray dried droplets which are often visible in intagliations) and may therefore have yielded artificially high values of Ra.

CONCLUSIONS

This study has illustrated the potential uses and advantages of the Light-Section Microscope for investigating film coats and the film coating process. The information generated has demonstrated that changing the coating application conditions can markedly affect the film surface roughness, film density, film thickness and thickness variation. In addition film thickness has been shown to vary at different positions on the substrate surface. Film thickness and density variation may have important implications if the coat is used to modify drug release since in these circumstances the functionality of the coat may depend not on the average film thickness but on the thinnest part of the film coat.

A coat of even thickness is likely to be produced if the substrate has a low surface roughness and the application conditions produce a smooth coat. The widest variation in thickness is likely to occur with rough substrates and conditions which produce a smooth coat.

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