

## Coating of pharmaceutical tablets: the spray-pan method

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A new method of coating tablets uses the revolving coating pan, but the coating materials are applied in solution or suspension, as a spray from a centrifugal disc atomiser. Addition of coating materials is uniform, controlled, and at a rate which allows even deposition on the tablet. Dry powders need not be used and the process is therefore dust-free. The products of the method compare favourably with tablets coated commercially and with tablets coated by the standard method in laboratory size (16 inch) pans when assessed by uniformity of weight tests, roundness measurement, and radiographic examination.

**T**HE standard method of coating tablets in revolving pans involves addition of powder and syrups alternately, or of suspensions, in varying amounts with drying between each addition. The volumes and manner of addition, and the method of drying yield lumpy surfaces in the early stages of coating and, although this may quickly change the shape to that of a rough oblate spheroid it requires smoothing syrup coats to give an acceptable product. It would facilitate the study of coating and the achievement of a uniform product if coats of any desired thickness could be built up from the commencement of the coating by deposition of smooth layers.

In the present work this has been achieved by applying the coating materials in the form of sprayed droplets and a spray-pan method suitable for laboratory-scale pan coating, and amenable to scale-up is described. The standard coating pan is used to support the tablet bed and the coating materials are sprayed onto the revolving tablets in a controlled manner from a centrifugal disc atomiser.

### Materials and methods

#### SPRAYING APPARATUS

The apparatus is shown diagrammatically in Fig. 1. The spinning disc (4) may be of metal or plastic and is smooth and circular; the laboratory model is 2 inches in diameter and 0.2 inch thick; it is driven anticlockwise by a variable speed motor (Desoutter M 10 x<sup>1</sup>). The speed of the disc was determined ( $\pm 1\%$ ) by a stroboscopic tachometer (Stroboflash 1200E<sup>2</sup>). The disc is located centrally in a rectangular stainless steel or brass shield (5) open towards the coating pan, with an aperture (7) of variable width to allow spraying of fluids with different spray characteristics. The size of this aperture, the position of the disc relative to the pan, and its distance from the tablets are regulated to allow the spray to fall only on the rolling tablets in a line from front to back of the pan. The bottom of the shield is suitably designed to facilitate drainage (80% of spray) which is returned to

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the thermostatically controlled feed reservoir (1) by means of a pump (8).<sup>3</sup> Coating materials are fed onto the centre of the spinning disc through an intermediate glass reservoir (3) which eliminates the pulsatile nature of the flow from the glandless metering pump (2).<sup>4</sup> The metering pump controls the feed rate in conjunction with the size of the nozzle on the intermediate reservoir. In this laboratory study the spraying equipment was used together with a 16 inch bench type coating pan (6),<sup>5</sup> revolving at 30 rpm and receiving 15–20 ft<sup>3</sup>/min air supply at 60°.

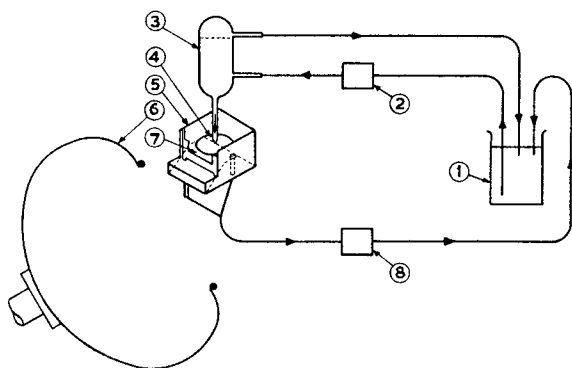


FIG. 1. Flow diagram for spray-pan coating process. 1. Feed reservoir. 2. Metering pump. 3. Intermediate reservoir. 4. Spinning disc. 5. Stainless steel shield. 6. Coating pan. 7. Aperture. 8. Pump.

#### SPRAY CHARACTERISTICS

Paraffin-coated microscope slides were passed through the spray and the adhering droplets were sized microscopically to yield  $D_{av}$  the average drop diameter, and  $D_{sv}$  the volume-surface mean drop size (Friedman, Gluckert & Marshall, 1952) which can also be thought of as the diameter of a drop whose volume to area ratio would be the same as that for the entire spray (Adler & Marshall, 1951).

#### TABLETS

A mixture of icing sugar and lactose, equal parts, was granulated with about 90 ml gelatin solution (20% w/v in water) per kg of sugar-lactose mixture and compressed on a Manesty B3 rotary machine using deep-concave punches 5/16 inch 13/32 inch and ½ inch diameter giving average tablet weights of 0.16, 0.38 and 0.60 g, respectively.

#### COATING MATERIALS

All fluids were applied at 60° to previously warmed tablets (except polishing solution which was used at room temperature). All applications

<sup>3</sup> Multifix Peristaltic Pump, Fisons Scientific Apparatus Ltd., Loughborough, Leics.

<sup>4</sup> Watson-Marlow Flow Inducer type MHRE, Watson-Marlow Air Pump Co., Marlow, Bucks.

<sup>5</sup> Stainless steel pan, BCP 2, Manesty Machines Ltd., Liverpool.

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were made to tumbling tablets without blown air. Coating stages are given in Table 1.

TABLE 1 COATING MATERIALS AND STAGES

Batch number	Coating Stages						
	1	2	3	4	5	6	7
II, III	Acacia syrup suspension A		Syrup		—		—
IV	Titanium dioxide syrup				Syrup		Polishing solution
V	Kaolin syrup				Syrup		Polishing solution
VI	Calcium carbonate syrup				Syrup		Polishing solution
VIII, IX	Syrup				—		—
XI, XII	Gelatin syrup				—		—
IVa, XIa, XIIa	Gelatin syrup and kaolin				Titanium dioxide syrup	Syrup	—
XIIb,	Gelatin syrup and kaolin		Titanium dioxide syrup	Syrup	Polishing solution	—	—
Ia	Gelatin syrup and kaolin	Titanium dioxide syrup	Syrup	Polishing solution	—	—	—
Suspension coating	Titanium dioxide syrup				Syrup		—

Syrup was syrup B.P.; gelatin syrup was syrup containing 1.3% w/w gelatin [170 bloom (Cox)]. Titanium dioxide syrup, calcium carbonate syrup, kaolin syrup and barium sulphate syrup each contained 13.3% w/w of the appropriate solid in gelatin syrup; titanium dioxide was titanium dioxide AE (British Titan Products); the other powders were B.P. quality. Acacia syrup contained 1 g acacia B.P., 10 g sucrose and 10 ml water. Acacia syrup suspension A contained 1 g acacia, 4 g titanium dioxide, 10 g sucrose and 10 ml water; acacia syrup suspension B was similar except that it contained half-quantity titanium dioxide. Polishing solution was 2% w/v white beeswax in carbon tetrachloride. The pan load was always 3 kg (uncoated weight).

### SPRAY-PAN COATING METHOD

For a disc speed of 8,000 rpm and feed-rate of 100–150 ml/min the following operation cycle was used: spray 1–1½ min; tumble only, 1 min without air; blown hot air 1–2 min with tumbling. This cycle was repeated until the desired coated weight was attained. Using this method, tablets could be coated with syrup or gelatin syrup alone; subcoating and dusting powder were not used. Tablets were also coated using titanium dioxide syrup, calcium carbonate syrup, kaolin syrup, barium sulphate syrup or acacia syrup suspension A or B and finished with syrup; dusting powder was not applied.

Tablet coated weight was double the uncoated weight. When suspensions were used, two-thirds of the weight of coat was supplied by the suspension, syrup contributing the remainder.

#### STANDARD COATING METHOD

(a) *Sub-coating*. Gelatin syrup or acacia syrup, 70–100 ml per application, was added alternately with kaolin or titanium dioxide, 250–300 g per application, to a predetermined weight. Tablets were allowed to roll 1–2 min between liquid and powder applications and for 2–3 min after addition of powder, followed by hot air blown in for about 10 min. (b) *Smoothing*. Acacia syrup suspension or one of the powder syrups, 50–100 ml per application, was followed by tumbling (2 min) without blown air, then hot air (5 min) until smoothing was complete. (c) *Finishing*. Syrup, 20–30 ml per application, was used together with tumbling (2 min) without air, and hot air (5 min) to final weight. Syrup at room temperature, with tumbling and no air, was used for about the last four applications. (d) *Polishing*. Polishing solution (100 ml in one application) was added to the tablets in a clean pan and they were left to tumble 6 hr without air application.

#### SUSPENSION COATING

Titanium dioxide syrup, or acacia suspension B, 50–100 ml per application, was applied as in the smoothing operation of the standard coating method, to a predetermined coat weight; finishing and polishing were as in the standard method.

#### COATED TABLETS OF COMMERCE

Samples of coated tablets were obtained from commercial sources together with samples of the same batches taken at different stages in the coating process. Although formulations and methods were not disclosed the stages of coating provided have been named arbitrarily according to appearance of the tablets, thus stage 1 represents sealing; stage 2, sub-coating; stages 3–5 include smoothing, colouring and finishing.

#### UNIFORMITY

*Weight*. Uniformity of weight was tested in the uncoated tablets and after each 0.1 g increase in coat weight during the coating processes. For the test, 20 tablets were weighed individually, and the average weight, standard deviation and coefficient of variation were calculated.

*Coating thickness*. Uniformity of thickness at various stages of coating was tested by means of the Talyrond 2<sup>6</sup> which measures roundness. The measurements were made on the circumferential edge of the tablet together with a reference computer which provides an integrated reference circle, from which departures from roundness are measured to allow calculation of the integrated average departures from the reference circle, designated the mean line average. The graph obtained for each tablet shows the

<sup>6</sup> The Rank Organisation, Rank Taylor Hobson Division, Leicester.

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differences between the circumferential edge and a true circle at the magnification used ( $\times 100$ ). The graph is, therefore, not a pictorial representation of the tablet. The roundness of the core tablets was also measured.

*Radiography of coated tablets.* Tablets were also radiographed at the various stages of coating.

### Results and discussion

The major difficulties in the standard method of pan coating of tablets arise in the method of coat build-up, especially in the sub-coating stage. In laboratory size (16 inch) pans it is difficult to prevent the early occurrence of frank lumpiness which then requires excessive coating material to smooth out the coat. In large pans (36 inch to 7 ft) which are usually rotated at speeds similar to the laboratory-size pan, the grinding action of the weight of the tablet bed (about 30 to 200 kg respectively) and the more efficient mixing and better drying conditions on the greater tablet area exposed, all tend to prevent lumpiness. The difficulties of the standard method were greatly reduced in the 16 inch pan by suspension coating but difficulty in obtaining controlled, uniform addition of coating material remained.

The 16 inch pan is therefore limited in development work using the standard and suspension methods, and results are difficult to scale-up faithfully. Also, this size pan is not entirely satisfactory for research in coating which, at the present time, is concerned not only with depositing certain types of coating but also with the accurate construction of coatings which will provide reproducible rates of release of drugs incorporated in the coat. In large-scale technique, using the standard method, the subjective element, which attends the timing and amount of coating materials added is less than satisfactory for routine production of precisely constructed coats, where reproducibility between, and uniformity within, batches is important. Nevertheless, the revolving pan of the traditional method provides a suitable support for the tablet bed during the application of coatings, and large weights (200 kg) can thus be coated without the necessity of starting with undesirably hard core tablets to minimise the damage which can occur in fluidised beds (Singiser, Heiser & Prillig, 1965).

The revolving pan has been retained in the present spray-pan method, but the means of addition of coating materials has been made more objective by a centrifugal disc atomiser which permits uniform, controlled addition of coating materials at a known rate, without the hand mixing frequently used in the traditional method. Spraying may, of course, be accomplished by pressure, twin-fluid or rotary atomisers. The centrifugal disc is the simplest type of atomiser; it is easily used and cleaned, high pressures are not required, and it can be used with fluids of widely varying properties over a wide range of feed rates. This gives greater possibility of controlled variation in application conditions and of producing sprays of uniform droplet size.

Droplet character from the centrifugal disc type of rotary atomiser depends on the disc (speed, diameter, energy transmission to the liquid

surface), the liquid (flow characteristics, surface tension, density), and on the atmosphere through which the spray passes (Adler & Marshall, 1951; Friedman & others, 1952; Fraser & Eisenklam, 1956; Fraser, Eisenklam & Dombrowski, 1957).

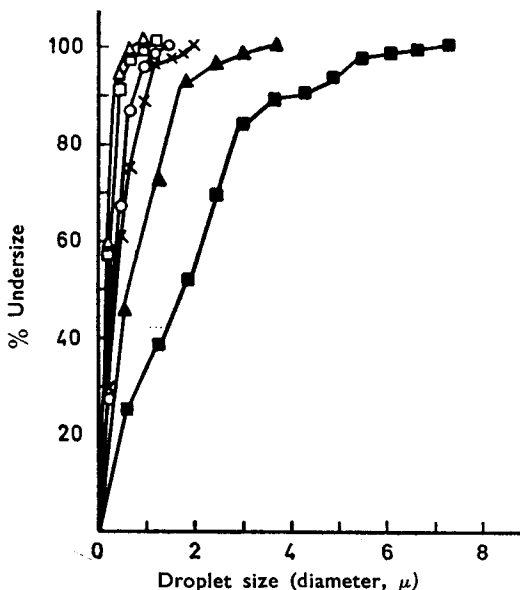


FIG. 2. Cumulative undersize distribution for sprayed coating suspension at a feed rate of 100 ml/min and several disc speeds. Disc speeds (rpm): ■ 2,000, ▲ 4,000, × 6,000, ○ 8,000, □ 10,000, △ 18,000.

To obtain uniform drop size, the centrifugal force should be large compared with the gravitational force, disc rotation should be vibrationless, liquid feed rates should be uniform, and disc surface should be smooth (Walton & Prewett, 1949; Adler & Marshall, 1951). Sufficient centrifugal force was obtained by spraying at a disc speed of 8000 rpm or greater; below this, uniformity of drop size was generally unsatisfactory (Fig. 2). Vibrationless rotation was approached by using an air driven rotor; uniformity of flow rate was achieved by using the metering pump-reservoir combination; the Perspex disc provided a smooth surface.

#### SPRAY CHARACTERISTICS

Fig. 3 shows the variations in  $D_{sv}$  with disc speed.  $D_{sv}$  was chosen to characterise the spray because it is related to the amount of surface created, and creation of new surface is the aim of atomisation. Increasing the disc speed creates more coating fluid surface thus facilitating even spreading and drying on the surface of the tumbling tablets. Increase in feed rate increases  $D_{sv}$ , hence greater feed rates require faster disc speeds for constant  $D_{sv}$ , although the difference between 100 ml/min and 200 ml/min is small (Fig. 3). Figs 2 and 3 indicate that, under the conditions

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of the experiment, 8000 rpm was the lowest speed for reasonable uniformity of spray; speeds greater than this did not greatly decrease  $D_{sv}$ . For increasing disc speed or decreasing feed rate, the mechanism of droplet formation changes from ligament or film formation to direct drop formation which is known to produce sprays with uniform droplets (Walton & Prewett, 1949; Hinze & Milborn, 1950; Adler & Marshall, 1951; Friedman & others, 1952; Fraser & Eisenklam, 1956; Fraser & others, 1957).

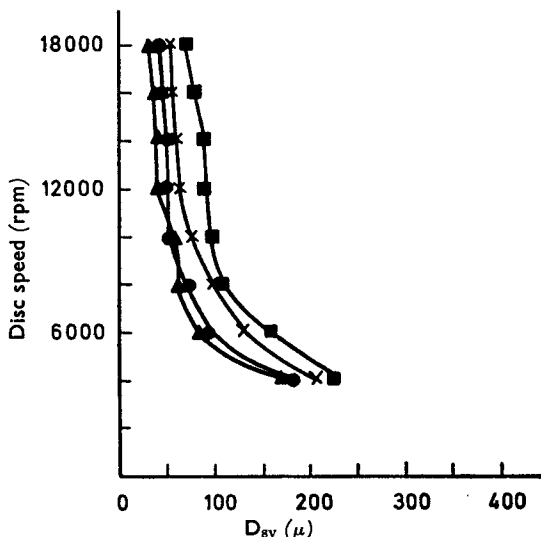


FIG. 3. Variation in volume-surface mean droplet size ( $D_{sv}$ ) with disc speed for two feed rates of a coating suspension (titanium dioxide syrup) and a coating solution (syrup). ▲ Syrup, 100 ml/min, ● Titanium dioxide syrup, 100 ml/min, ■ Syrup, 200 ml/min, × Titanium dioxide syrup, 200 ml/min.

### SPRAY-PAN METHOD

The adjustable aperture (Fig. 1) in the apparatus, while conserving spray, allows determination of the width of horizontal spray falling on the tablets and is, therefore, an additional control of the amount, as well as the position, of the spray. The spraying cycle must be determined for the conditions of the experiment but once determined it is reproducible in inexperienced hands and capable of automatic control. Scale-up is straightforward, whereas in the standard coating method large scale coating is more readily accomplished than laboratory scale coating. The process is also dust-free, and a complete coat can be built up using the spray-pan method without adding any free powder. This is because the uniform addition of liquid in spray form allows a rapid drying rate without sticking or lump formation and the use of suspensions allows rapid coat build-up.

The reasonable constancy of coefficient of variation in weight as the coat builds up in the spray-pan method, contrasts with the picture of coefficient of variation not only for the standard method in the 16 inch pan, but also

for commercial tablets which have been coated in larger pans (Fig. 4D). Figs 4A, 4B and 4C show comparisons between the plots of coefficient of variation versus coating stage for tablets of different core size and show that for a range of core sizes of frequent use the relationship is the same. Batches IVa, XIa and XIIa, coated by the standard method, yield a higher level of coefficient of variation of weight throughout the process, whereas batches II-VI, VIII, IX, XI, XII (spray-pan method) consistently have a coefficient of variation less than 4%, and close to that of the cores.

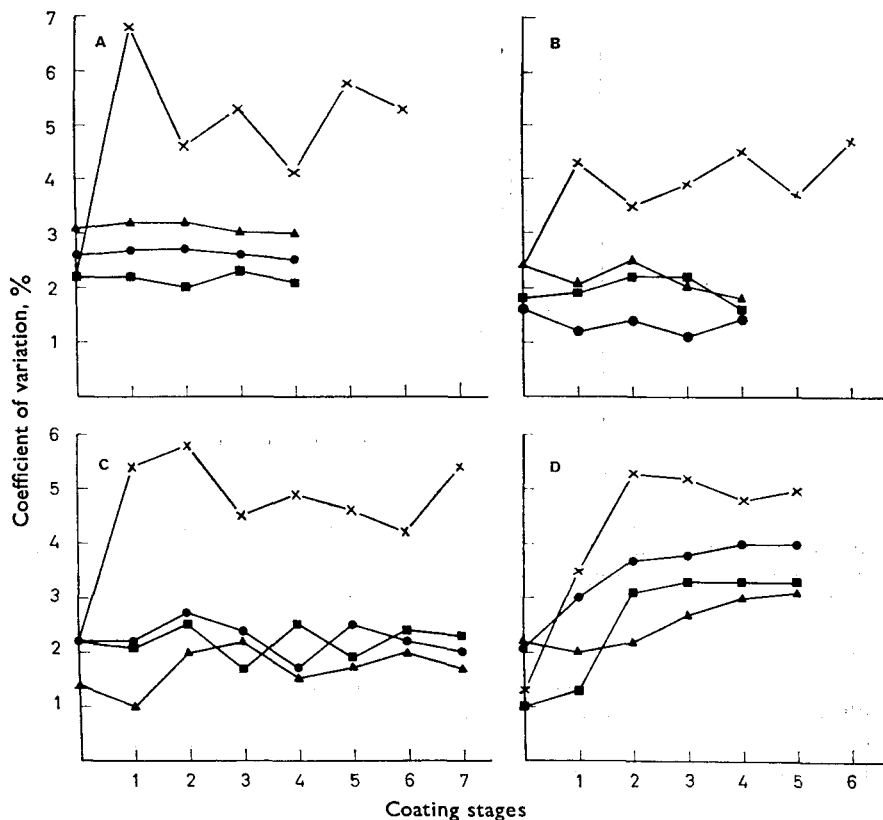


FIG. 4 A-D. Coefficient of variation of weight of tablets at various stages in coating. Batches II-VI, VIII, IX, XI, XII, spray-pan coated; IVa, XIa, XIIa coated by standard method. Other details in Table 1. Diameter of core tablets: A. 5/16 inch; B. 13/32 inch; C. 1/2 inch; D. Commercial, various core diameters. Symbols: A. X = Batch XIIa;  $\blacktriangle$  = Batch II;  $\bullet$  = Batch III;  $\blacksquare$  = Batch XII. B. X = Batch XIa;  $\blacktriangle$  = Batch XI;  $\blacksquare$  = Batch VIII;  $\bullet$  = Batch IX. C. X = Batch IVa;  $\bullet$  = Batch IV;  $\blacktriangle$  = Batch V;  $\blacksquare$  = Batch VI. D. X = Commercial Tablets (1);  $\bullet$  = Commercial Tablets (2);  $\blacktriangle$  = Commercial Tablets (3);  $\blacksquare$  = Commercial Tablets (4).

It is interesting to note that the limit set by the Swedish Pharmacopoeia (1946) of a coefficient of variation of 4.5% for uncoated tablets, is not exceeded even by tablets after coating, when this is done by the spray-pan



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method; indeed, tablets coated by this method are markedly less variable in weight throughout the process of spray-pan coating (Figs 4A–C).

Turning to commercial tablets (Fig. 4D), the effect of subcoating (apparently by a standard method) on the coefficient of weight variation can be clearly seen in the increase occurring in the early stages. Whether the standard method of coating is in 16 inch pans or pans of greater size, it is clear (Figs 4A–D) that the variation in weight distribution during the early (subcoating) stages determines the variation in subsequent stages.

Roundness measurement, performed on the circumferential edge of the tablet by means of the Talyrond, was preferred to surface texture measurement (B.S. 1134: 1961). Examples of Talyrond graphs are in Fig. 5.

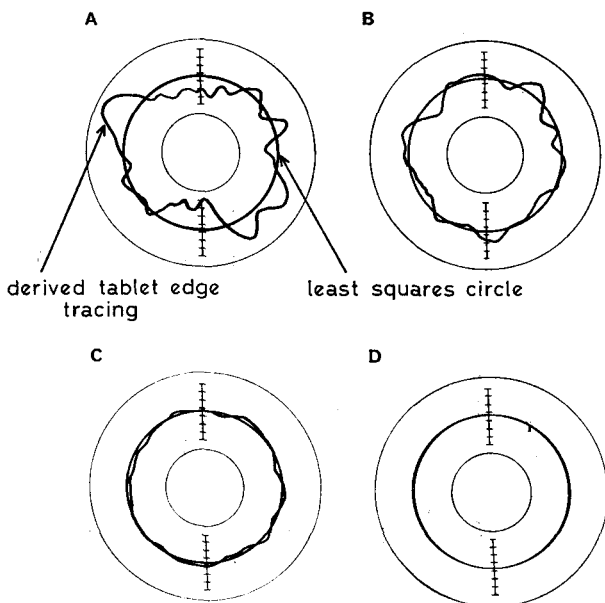


FIG. 5 A–D. Examples of Talyrond graphs. Each figure shows the least squares circle together with the tracing, derived from the tablet edge, which describes the differences between that edge and the least squares circle; superimposed scale (normally 12 such scales with subdivisions appear; these have been reduced for clarity). A. Batch XIa. B. Commercial tablet. C. Batch XI. D. Core.

The roundness measurements made at the various stages of coating are plotted in Fig. 6. They support the results of the uniformity of weight measurements and the findings from radiography (which proved to be a suitable means of visualising coating layers) and show that the spray-pan coated tablets are rounder (smoother) at all stages of coating than the tablets coated by the standard method either in the 16 inch pan or commercially. Fig. 6 shows that the early coats in the coating procedure influence the ease with which a smooth final coat can be obtained. When the mean line average (see p. 792) for the early coats is high, it remains high

throughout the later coating stages, although some commercial tablets did show a slight irregular fall in mean line average in the late coating stages indicating that the smoothing coats were fulfilling, to some extent, the textbook function. High mean line averages after subcoating are important in view of the empirical teaching and practice of the tablet coaters regarding subcoating, any inelegance of which is commonly thought to be readily and completely concealed by the succeeding coats. In the 16 inch pan it was possible to obtain a consistently decreasing mean line average in the later coating stages by decreasing the amount of subcoating (compare batches XIIa, XIIb, Fig. 7). A mean line average value (at the end of the coating) similar to that for suspension coating and spray-pan coating (batch Ia, Fig. 7) was made possible by using only 0.1 g subcoating.

Comparison of roundness measurements with the uniformity of weight results shows that, in the standard coating method, particularly when used on a laboratory scale (Fig. 4A-C batches IVa, XIa, XIIa) but also noticeably on a commercial scale (Fig. 4D samples 1, 2, 3, 4), both departure from roundness and coefficient of variation of weight increase steeply in the

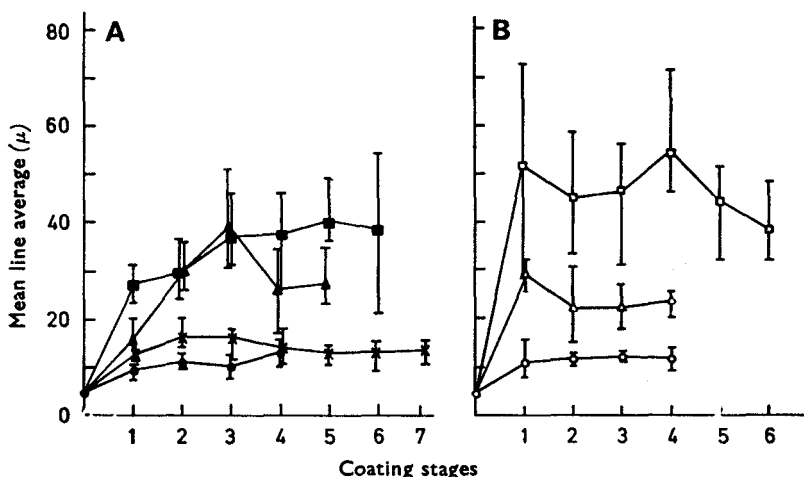


FIG. 6 A—B. Variation in mean line average (uniformity of thickness) as coating proceeds. Each point is the mean of five readings. A. ■ = batch XIIa, ▲ = commercial 1, × = batch IV, ● = batch XII. B. □ = batch XIa, △ = commercial 5, ○ = batch XI.

early stages of coating, the subsequent “smoothing” coats failing to restore the low values for both types of measurement seen in the core tablets. However, in the spray-pan method the coefficient of variation of weight remains constant throughout the process in spite of the small initial increase in departure from roundness of from  $3\mu$  (mean line average) for the core, to  $10\text{--}15\mu$  for the later stages.

This difference between the two methods of coating is caused by the use of free powder in the subcoating stage in the standard method for which

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there is, in many instances, little justification, since the need is better met by using a liquid suspension applied preferably in atomised form.

Other attempts to improve on the pan-coating of tablets have been restricted to automation of the stages in the method (Clay & D'Angelo, 1956; Rieckmann, 1963; Steinberg, 1964) or to the method itself (Lachman, 1966) but the properties of the resulting coat were not described. Kwan (1961) used an automated process to study drying of coatings; Butensky (1961), also using an automated process, confirmed experimentally the experience of tablet coaters that it is the powder-liquid additions and

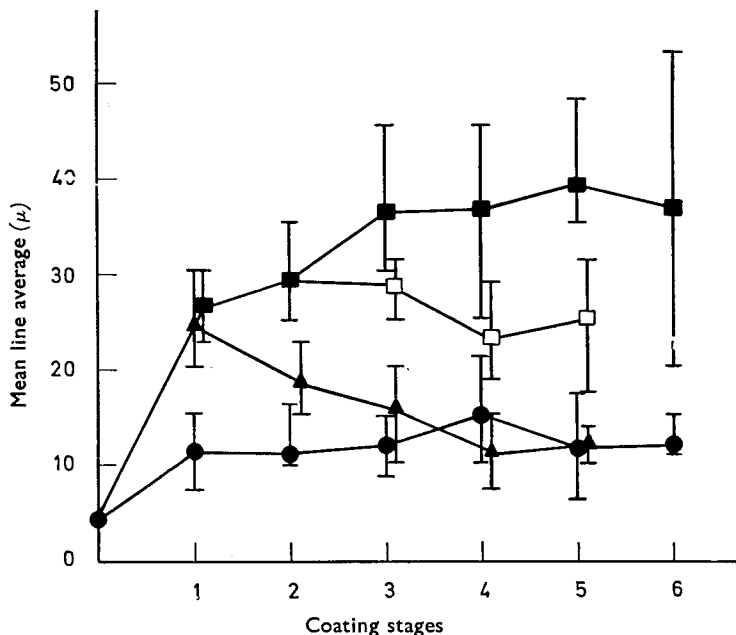


FIG. 7. Variation in mean line average (uniformity of thickness) as coating proceeds. Each point is the mean of five readings. ■ = Batch XIIa. ■ → □ = Batch XIIb. ▲ = Batch Ia. ● = suspension coating (Batches XIIa and XIIb followed a common path to coating stage 2, where the batch was divided; the two batches were thereafter treated separately as shown in Table 1).

ratios, coupled with drying rates, which lead to the difficulties of the standard method. Butensky (1961), who restricted his study to the sub-coating stage, found that, within this stage of tablet coating, the coefficient of variation of tablet weight rose to about 7%, this maximum occurring before the final subcoating application. Although our coefficient of variation is lower, this is in general agreement with our findings for the standard method.

The difficulty in achieving uniformity has been stressed by Mattocks (1958), and this difficulty has hindered development of research in tablet coating. Spray-pan coating provides a method capable of the degree of

standardisation necessary to allow its use in the study of many of the problems in tablet coating which remain.

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