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# The effect of frictional charges on flow properties of direct compression tableting excipients

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## Summary

An electronic flow balance method was used to study flow properties of 4 pharmaceutical excipients. The technique yielded quantitative information on flow rates and flow uniformity.

An air cyclone method was used to charge powders by friction prior to determination of flow properties. It was found that increased frictional charging markedly reduced flow rates and uniformity of normally free-flowing powders. Following charging, the powders exhibited properties usually associated with cohesive materials including increased angles of repose.

The method of Jones and Pilpel was used to derive theoretical flow values and these agreed reasonably well with those obtained experimentally prior to charging. Following electrification, however, it was found necessary to add an empirical function to account for increased interparticle attraction.

## Introduction

Several different methods of assessing the flow properties of direct compression tableting excipients have been used in order to predict or evaluate the performance of powders in the tablet compression process (Ho et al., 1977).

Characterization of powder flow rates is necessary in cases where poor drug-content uniformity presents a problem in tablet production or in other processes where unacceptable solids flow occurs. Assessment of powder flow has been carried out using methods such as determination of angles of repose, percentage compressibility of bulk powders and factors derived from shear cell measurement, but these do not necessarily relate directly to powder flow properties found during normal processing.

Gold et al. have reported a method for direct assessment of powder flow using a 'recording flowometer' (Gold et al., 1968). An adaptation of the flowmeter technique has been used in the present study to assess flow rates before and after powder charging.

The charging method used was intended to simulate extremely efficiently, the type and magnitude of powder charging likely to occur during normal processing. Powders become charged whenever particles move relative to one another or a containing surface; this occurs by frictional and contact electrification known collectively as triboelectric charging (Staniforth and Rees, 1981).

## Experimental

### *Materials*

Three free-flowing direct compression tableting excipients were studied: Emdex, a spray crystallized dextrose (Edward Mendell, New York, U.S.A.); Di-Pac, a co-crystallization of 97% sucrose and 3% modified dextrins (Amstar, New York, U.S.A.) and Elcema G250, a coarse grade of microfine cellulose (Degussa, Frankfurt, F.R.G.) and one cohesive excipient, Avicel PH101, a fine-particle form of microcrystalline cellulose (FMC, Philadelphia, U.S.A.).

Emdex (E) and Dipac (D) had similar particle size distributions with approximate median sieve diameters of 250 μm, Elcema (EG) particles had a smaller median diameter of 100 μm and the cohesive Avicel (A) powder, 5 μm.

### *Procedure*

The flow properties of each excipient were evaluated using a glass hopper with a 10 mm orifice diameter mounted over an electronic balance (Oertling, London) as

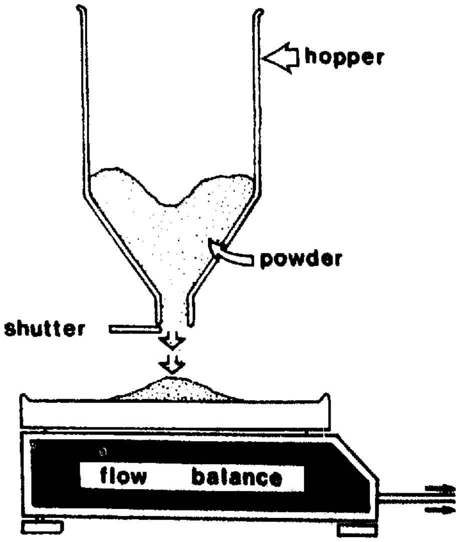


Fig. 1. Flow properties of each excipient were evaluated using a glass hopper with a 10 mm orifice diameter mounted over an electronic balance connected to a chart recorder.

shown in Fig. 1. As powder flowed on to the electronic balance the increasing mass was integrated against time using a chart recorder (Pye Unicam, Cambridge) connected to the balance output. An advantage of using a flow balance arrangement in preference to simply measuring the flow time of a known mass of powder is that in addition to obtaining an average flow rate value a measure of flow uniformity can be derived. This was achieved by constructing a straight line between the start and finish of flow on the chart printout. By comparing the length of this line, $x$, with the length of the curve obtained experimentally, $y$, a measure of flow uniformity can be derived:

$$F_u = y/x \times 100 \qquad (1)$$

where $F_u$ is the flow uniformity coefficient. For powders exhibiting perfectly regular flow the flow uniformity coefficient will take a value of 100. In powders with non-uniform flow properties, $F_u$ will decrease as flow becomes more irregular.

Flow rates and uniformity coefficients were determined following storage of powders under ambient conditions and also following charging in an air cyclone (Staniforth and Rees, 1981). The powders were fed to the cyclone in air suspension and after frictional contact with the cyclone the charge magnitude on the particles was measured using a Faraday well connected to an electrometer (Model 610C, Keithley Instruments, Cleveland, U.S.A.). Charged powders were fed into the glass hopper and flow rate measurements were carried out within 15 min of triboelectrification which was within the half-period for charge decay in the powders studied. The air cyclone was used as an efficient method of particle charging although similar effects could be produced by dry mixing, fluidization or pneumatic transfer.

Angle of repose measurements were carried out on the 4 excipients using the fixed-height cone method. Powder was released from a funnel using a simple shutter device and the angle $\alpha$, was calculated as the mean of 4 separate determinations.

## Results and Discussion

The increase in charge magnitude of the 4 excipients following triboelectrification is shown in Table I. It was found that although the powder became charged by friction against the hopper wall, the charge magnitude increased by a factor of between 30 and 100 following triboelectrification (Table I).

Of the powders tested prior to charging, Emdex and Dipac showed good flow properties as did Elcema G250; only Avicel exhibited cohesive properties (Table II), and flow was only achieved using a chromatograph column vibrator acting on the hopper wall. Following charging there was a very marked decrease in flow rates of the coarse excipients Emdex, Dipac and Elcema G250 and a slight reduction in Avicel (Table II). The sample flow profiles (Figs. 2–5) show the change from non-cohesive, free-flowing powders to ones showing evidence of bridging and intermittent flow typical of cohesive powders. Flow uniformity coefficients, $F_u$, also show a change from extremely regular flow in Emdex and Dipac and fairly regular

TABLE 1

ELECTROSTATIC PROPERTIES OF POWDERS BEFORE AND AFTER FRICTIONAL CHARGING

| | Charge after flow through hopper ($C \cdot g^{-1}$) | Charge after frictional charging ($C \cdot g^{-1}$) |
|---|---|---|
| Emdex | $-0.566 \times 10^{-9} \pm 0.11$ | $-329 \times 10^{-9}$ |
| Dipac | $-0.93 \times 10^{-9} \pm 0.04$ | $-98.9 \times 10^{-9}$ |
| Elcema G250 | $-3.47 \times 10^{-9} \pm 1.61$ | $-248 \times 10^{-9}$ |
| Avicel PH 101 | $-7.23 \times 10^{-9} \pm 0.70$ | $-385 \times 10^{-9}$ |

TABLE 2

FLOW PROPERTIES OF POWDERS DETERMINED USING FLOW BALANCE METHOD

| | Flow rate before charging ($g \cdot s^{-1}$) | Flow rate after charging ($g \cdot s^{-1}$) |
|---|---|---|
| Emdex | 3.123 | 0.717 |
| Dipac | 3.011 | 0.971 |
| Elcema G250 | 1.330 | 0.216 |
| Avicel PH 101 | 0.315 * | 0.250 * |

* Flow achieved only after vibration.

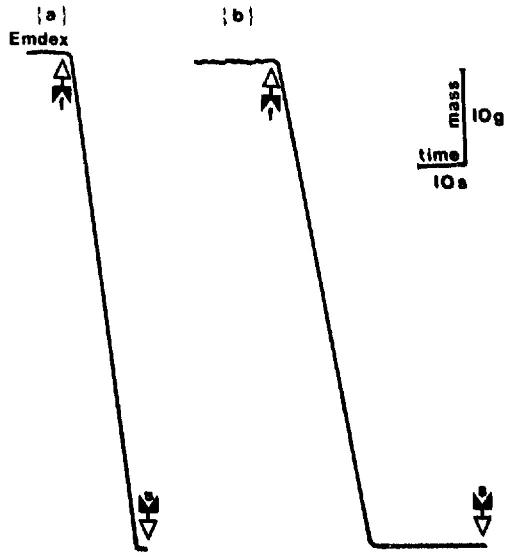


Fig. 2. Sample profiles for Emdex powder before (a) and after (b) charging showing change from non-cohesive free-flow to delayed flow caused by bridging. s = start of flow; f = end of flow.

TABLE 3

FLOW UNIFORMITY COEFFICIENTS ($F_u$) AND FLOW CHARACTERISTICS OF POWDERS BEFORE AND AFTER TRIBOELECTRIC CHARGING

| Excipient | Uncharged | | Charged | |
|---|---|---|---|---|
| | $F_u$% | Description | $F_u$% | Description |
| Emdex | 98.9 | Very free flowing | 86.4 | Initial bridging delayed flow |
| Dipac | 99.4 | Very free flowing | 84.4 | Initial bridging delayed flow |
| Elcema G250 | 88.8 | Fairly free flowing | 83.2 | Intermittent flow |
| Avicel * | 83.8 | Very cohesive | 82.9 | Very cohesive |

* Vibration-assisted

flow in Elcema G250 prior to charging to irregular or intermittent flow following charging (Table III).

In addition to reducing flow rates and uniformity, frictional charging also increased poured angles of repose (Table IV). The observed change in angles of repose followed a similar pattern to other results such as flow rate and uniformity measurements, indicating increased interparticle cohesion following charging.

Jones and Pilpel (1966a, b) developed an expression for determining theoretically the flow properties of powders based on physical characteristics such as size, shape

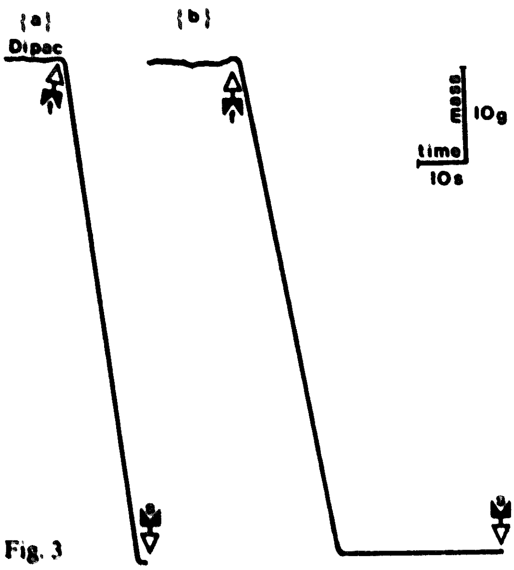


Fig. 3

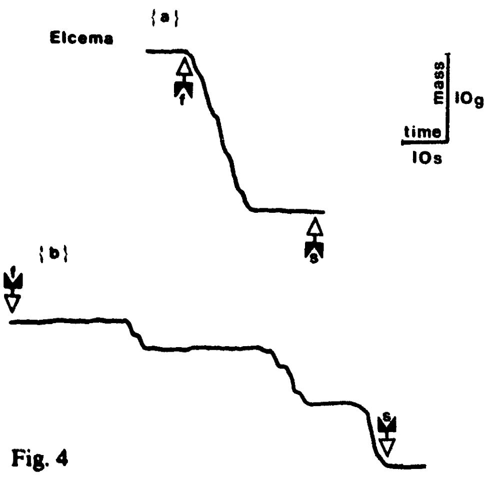


Fig. 4

Fig. 3. Sample profiles for Dipac powder before (a) and after (b) charging showing change from free-flow to delayed flow caused by bridging. s = start of flow; f = end of flow.

Fig. 4. Sample profiles for Elcema G250 showing change from free-flow before charging (a) to intermittent flow after charging (b). s = start of flow; f = end of flow.

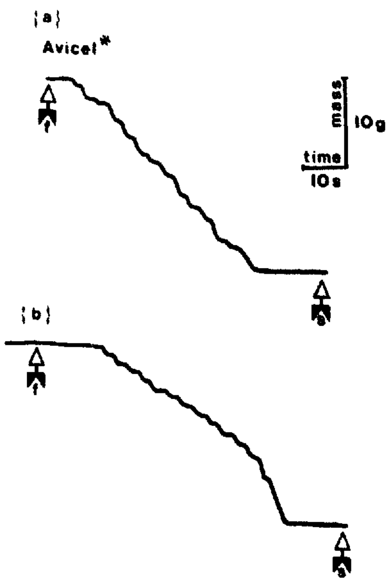


Fig. 5. Sample profiles for Avicel PH101 showing cohesive flow before (a) and after (b) charging. *Flow was vibration-assisted. s = start of flow; f = end of flow.

and density (Eqn. 2)

$$D_0 = A\left[\frac{4W}{60\pi\rho\sqrt{g}}\right]^{\frac{1}{n}} \tag{2}$$

where W is flow rate in $g \cdot s^{-1}$, A and n are empirical functions of D; $\rho$ powder density; $D_0$ orifice diameter and g, acceleration due to gravity.

Table 5 shows values of theoretical flow rates calculated for the 4 excipients which were comparable with flow rates obtained experimentally. However, flow rates were so reduced following charging that the theoretical values were no longer representative of measured flow values.

A similar problem was found using the flow model proposed by Carstensen and

TABLE 4

EFFECT OF FRICTIONAL CHARGING ON POURED ANGLE OF REPOSE FOR 4 DIFFERENT EXCIPIENTS

| | A | EG | E | D |
|---|---|---|---|---|
| Poured angle before charging | 35° | 30° | 17° | 26° |
| Poured angle after charging | 40° | 35° | 24° | 41° |

TABLE 5

THEORETICAL FLOW RATES OF MATERIALS BEFORE FRICTIONAL CHARGING CALCULATED ACCCORDING TO JONES AND PILPEL(1966a, b)

| | Flow type | Theoretical flow rate ($g \cdot s^{-1}$) | Difference from measured flow rate (%) |
|---|---|---|---|
| Emdex | I | 2.646 | −15 |
| Dipac | I | 2.634 | −12 |
| Elcema G250 | II–III | 1.064 | −20 |
| Avicel PH 101 | IV | Theoretical relationship not applicable to type IV powders | |

Laughlin (1981) shown in Eqn. 3:

$$W_s = \frac{\pi\rho\sqrt{g}}{4}\left[\frac{D_0}{1.65 + 2.34d}\right]\left[\frac{1}{0.24 - 0.038Lnd}\right] \quad (3)$$

where $W_s$ denotes static flow rate and d, particle diameter. Prior to charging, theoretical and experimental values were comparable whereas following charging the two sets of values differed by more than 60%.

The differences between measured flow rates and those calculated theoretically may be partly due to wider particle size distributions of materials used in these experiments as compared with those studied by Jones and Pilpel. However, the main reason for the apparent discrepancies is probably due to increased interparticle cohesion and wall-particle adhesion not accounted for in the theoretical models. A further function is required to represent the cohesive influence on flow; this can be achieved either from a consideration of internal friction differences or from charge magnitude differences which produced increased friction. For example, in the

TABLE 6

THEORETICAL FLOW RATES OF MATERIALS AFTER FRICTIONAL CHARGING CALCULATED ACCORDING TO:

$$D_0 \approx A \cdot \sqrt[4]{C}\left[\frac{4W}{60\pi\rho g}\right]^{1/n}$$

| | Theoretical flow rate ($g \cdot s^{-1}$) | Difference from measured flow rate (%) |
|---|---|---|
| Emdex | 0.622 | −15 |
| Dipac | 0.837 | −16 |
| Elcema G250 | 0.268 | +24 |

expression of Jones and Pilpel (1966a, b) an empirical function, $\sqrt[4]{C}$ can be introduced to account for the increase in particle charge following triboelectrification:

$$D_0 = A\sqrt[4]{C}\left[\frac{4W}{60\pi\rho g}\right]^{1/n} \tag{4}$$

where C is the difference in charge magnitudes before and after triboelectrification.

This modified equation yields theoretical flow values for charged particles (Table VI) which again approximate to those obtained experimentally. Alternatively, angles of repose determined in radians can be used to derive an empirical function based on differences in interparticle attractions rather than charge differences. Further work is required to define accurately a universal relationship between particle charge or interparticle attractions and powder flow, but if functions such as those described above are generally applicable it appears that a 10-fold increase in charge can almost halve powder flow rate.

## Conclusions

The results show that normally free-flowing powders exhibit cohesive properties following frictional charging. After triboelectrification, flow rates were reduced and flow became more irregular as indicated by a decrease in the flow uniformity coefficient. In addition, angles of repose increased, suggesting that interparticle cohesion was stronger and probably produced the measured alterations in flow properties.

The models of Jones and Pilpel (1966a, b) and Carstensen and Laughlin (1981) were found to produce theoretical flow values close to flow rates determined experimentally prior to charging. Following electrification the models no longer accurately represented measured flow rates. Empirical functions based on charge and cohesion differences were added to the model of Jones and Pilpel (1966a, b) and the new theoretical values were found to compare with rates obtained after frictional charging.

Since frictional charging occurs each time a powder is moved, the above results suggest that reducing the duration or number of operations such as milling, fluidization or pneumatic transfer should prevent deterioration of powder flow properties.

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