

An evaluation of the air stream Faraday cage in the electrostatic charge measurement of interactive drug systems

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Summary

An air stream Faraday cage was used to measure the electrostatic charge generated when adhered particles were detached from an interactive unit interface. Theoretical and practical considerations in the design of the apparatus were discussed. Average charge to mass ratios in the range $1.7-7.6 \mu\text{C} \cdot \text{g}^{-1}$ and their polarities were determined for a number of drug materials in relation to a polymer-coated carrier. Spurious charging due to the surrounding electrical field, air streaming and carrier-carrier or carrier-cage interaction was less than 1% of the total charge measured and was not a significant consideration under the charge-measuring conditions used in this study. The electrostatic charge produced on drug particle detachment was not altered by further drug-carrier or drug-cage interaction in the interactive systems studies. However, when highly triboelectrified interactive systems were used, drug-cage interaction occurred negating some of the electrostatic charge and providing average charge to mass ratios which were not representative of all of the particles in the interactive system. Incremental air flow charge measurements allowed the determination of the interactive systems' charge distribution.

Introduction

Particle adhesion has important consequences in the processing and formulation of solid dosage forms. The flow properties of powders, drug homogeneity in mixing processes, uniformity of powder filling procedures, loss of drug due to particle interaction with processing equipment and the solid dosage form properties including compressibility and dissolution will be influenced by the degree of adhesion of powders in solid systems (Stewart, 1986). Particle adhesion occurs when the

interaction forces between a particle and a surface exceed the detachment forces exerted on the particle. While the detachment forces are usually gravitational or accelerative in nature, particle adhesion occurs due to capillary, contact potential, coulombic and intermolecular interactions and solid bridge formation (Zimon, 1982; Wake, 1976; Hendricks, 1973; Staniforth and Rees, 1982). The conditions during the processing of solids will dictate the relative contribution of the adhesion forces that occur in interactive systems. For example, high humidity conditions will favour capillary interaction and high impact forces will cause particle deformation with closer contact between contiguous surfaces and a higher intermolecular interaction component. However, in most processing

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conditions, the relative movement of particles and subsequent collisions between particles provides an ideal environment for the development of contact potential or electrostatic interactions. Such electrostatic charging occurs by frictional contact between dissimilar contiguous particle surfaces with particles remaining charged after separation. These forces have been shown to contribute significantly in the adhesion of small particles to solid surfaces (Donald, 1969; Aleinikova et al., 1968).

The measurement of the degree of electrostatic interaction is therefore of considerable importance in pharmaceutical processing. This study reports the application of an air stream Faraday cage (Schein and Cranch, 1975; Lee and Weser, 1979) to the measurement of electrostatic charge in pharmaceutical systems.

Materials and Methods

Materials

Solid glass spheres (500 μm), coated with hydropropyl methylcellulose phthalate (HP55; Shin-Etsu Chemical Co., Japan) by an air suspension technique, were used as the carrier particles. Sulphapyridine, sulphamerazine, succinylsulphathiazole (Sigma Chemical Co., U.S.A.), ascorbic acid (David Craig, Aust; B.P.) and phenacetin (Evans Medical, U.K.; B.P.) were used as adherent powders. Fractions less than 45 μm were classified by sieving (Sonic Sifter, model L3P, ATM Corporation, U.S.A.).

Apparatus

Dry compressed air (Atlas Copco, Australia; 5.8 atm at 12.7 litres $\cdot \text{s}^{-1}$; RH = 10%) was filtered (Pall Filter) and directed through the Faraday cage (stainless steel; 5.8 cm in diameter and 1.5 cm in length) via a solenoid valve (3-way normally open; Goyen Control, Aust.). The valve was controlled by an electric twin timer (Omron model TDV-2; Omron Tateisi Electronics Co., Japan). The air velocity was determined using a pitot static tube connected to a calibrated manometer (type 4/5 air flow test set; Air Flow Development, U.K.). The charge and polarity of the cage were

measured using an electrometer (Keithly, model 616; Keithly Instruments, U.S.A.). Insulation of the Faraday cage was achieved using PTFE blocks and tape.

Methods

Charge measurements were performed by accurately weighing the Faraday cage ($\pm 10^{-4}$ g). A sample of the powder mixture (1–6 g) was transferred to the cage and weighed. The Faraday cage was assembled into the system and the intermittent compressed airflow at known velocity directed through the cage to cause detachment of the adhered particles. When the electrometer measured the peak charge acquired by the cage, its magnitude and polarity were noted. The cage was removed from the system and reweighed to determine the weight of adhered particles detached.

Formation of the interactive mixture of polymer-coated glass beads and adhered material occurred by blending in a glass jar (10–20 g; CV < 1%). The interactive system was confirmed by SEM.

Samples from this mixture were taken for charge measurement. Powders and interactive mixtures were stored under controlled humidity conditions throughout the study (desiccator containing silica gel; RH < 10%).

Particle size distributions of the adhering materials were determined by automated laser diffraction (Malvern, model 3600; Malvern Instruments, U.K.).

Results and Discussion

Theoretical considerations

Contact potential (or electrostatic) interaction occurs between uncharged dissimilar contiguous solids. The work function of a metal (ϕ) is the difference in the energy states between the outermost conduction band of electrons (i.e. the Fermi level) and some outside reference (i.e. the vacuum energy level). On contact between metals possessing different work functions, transfer of electrons occurs until the Fermi levels are the same (Fig. 1). A contact potential which is the difference be-

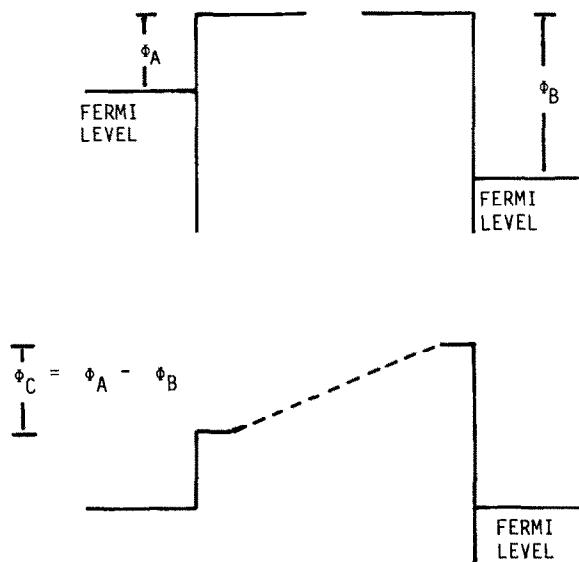


Fig. 1. The development of a contact potential between contiguous surfaces.

tween the work functions is produced (ie $\phi_C = \phi_A - \phi_B$).

Most pharmaceutical powders are organic materials and are semiconductors or insulators. The concept of work function for organic materials is more complex than for metals and involves the Fermi level passing through a "forbidden" energy zone situated below the conduction band (Schnabel, 1968; Krupp, 1971). Conceptually, the same type of electrostatic charging behaviour occurs as for metals, although the development of contact potentials is more complicated and less well defined (Gallo and Ahuja, 1975). Pharmaceutical powders possessing different work functions therefore will interact developing contact potentials. On separation, the particles will possess an equal but opposite surface charge which will be proportional to the contact potential and which will exist diffusely to about a 1 μm depth.

Air stream apparatus has been used to measure the surface charge developed on particle separation. Such an apparatus (Fig. 2) consists of a specially designed Faraday cage enclosed by mesh at both ends of the cylinder. An interactive mixture consisting of drug particles adhered to a larger carrier particle can be introduced into the

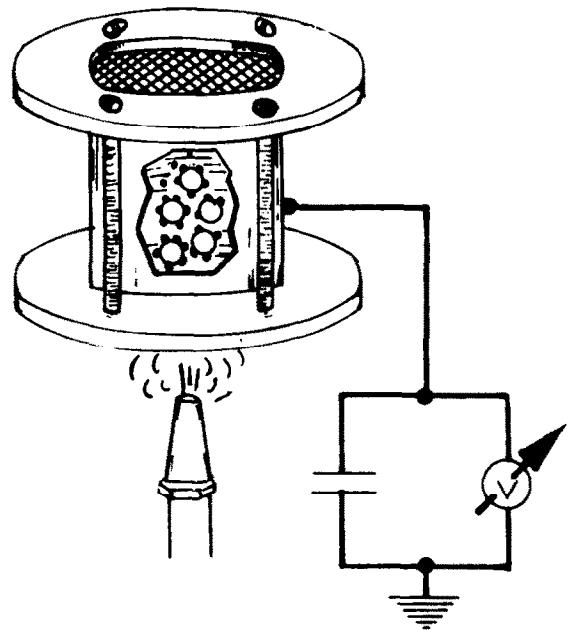


Fig. 2. Particle detachment in the air stream Faraday cage.

cage. Compressed air, which was directed through the cage removed the adhered drug particles from the carrier inducing equal but opposite charges on the carrier and drug particle, respectively. The drug particle was removed from the cage through the mesh; however, the large carrier particle remained within the cage, and transferred the electrostatic charge to the cage. Measurement of the charge's magnitude and polarity can be effected using an electrometer.

The total design of the airstream Faraday cage is shown in Fig. 3. Important considerations in the design included: (a) the compressed air should be filtered to remove traces of oil and moisture which would contaminate the cage and contents; and (b) an intermittent air flow prevented blockage of the mesh by the large carrier particles.

Average charge-to-mass ratio

The following parameters can be calculated from the data.

(a) The triboelectric property of an interactive mixture was expressed as average charge-to-mass ratio or tribo (T) in microcoulombs per gram

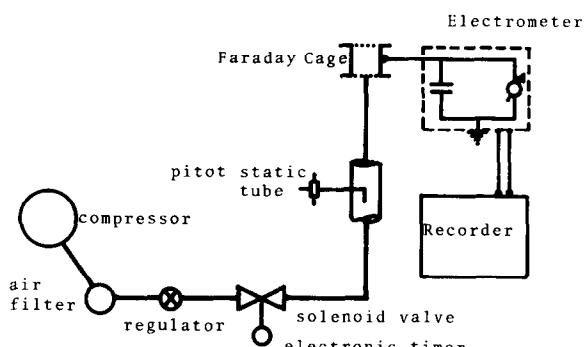


Fig. 3. Total design of the air stream Faraday cage apparatus for measuring electrostatic charge at interactive unit interfaces.

($\mu\text{C} \cdot \text{g}^{-1}$) of adherent powder:

$$T = Q/M$$

where Q is charge magnitude measured and M is the mass of powder detached.

(b) The concentration of powder detached ($C\%$) is expressed as

$$C\% = 100M/W$$

where W is the weight of mixture after powder detachment.

Examples of average charge-to-mass ratios or tribos of 5 drugs determined in relation to a coated carrier are presented (Table 1). The polarity observed for these compounds was in relation to the carrier used in this study; other interactive systems may produce different polarities on drug detachment due to the difference in work function

between two contiguous bodies. Powder which gained negative polarities possessed greater work functions than the carrier and vice versa.

Charge measurements of the same mixture showed the good reproducibility which was attainable for all types of the mixtures observed. The coefficient of variation of replicated determinations of the average charge-to-mass ratio was less than 10% and was satisfactory by comparison with the powder charge by conventional method of pouring the powder from the container, e.g. variation up to 20% (Boschung and Glor, 1980) or between 50% and 90% in some determinations (Staniforth and Rees, 1982). The concentrations of powder detached ($C\%$) among the samples taken from the same mixture were also in good agreement.

System validation

Spurious, undesired charging associated with the cage and carrier during the detachment procedures would invalidate the charge measurement. Validation of the system included an investigation of extraneous charge production due to the Faraday cage configuration, carrier charging caused by carrier-carrier or carrier-wall interaction, and the drug particle interaction with other carriers, cage wall, or screen.

(a) Electrostatic charging in the empty cage

The degree of interference of the electric field from the surroundings was observed by assembling the empty cage in the system and measuring charge development with time. Observation of

TABLE 1
Triboelectric properties of powder drug-carrier mixture

| Powder | Sample size (g) | $C \pm \text{S.D.}$ (%) | $T \pm \text{S.D.}$ ($\mu\text{C} \cdot \text{g}^{-1}$) | Polarity | |
|-------------------------|--------------------|----------------------------|--|----------|--------|
| | | | | Carrier | Powder |
| Sulphapyridine | 1 | 0.75 ± 0.05 | 4.54 ± 0.32 | — | + |
| Succinyl sulphathiazole | 1-2 | 0.38 ± 0.05 | 2.49 ± 0.26 | + | — |
| Sulphamerazine | 1 | 0.62 ± 0.04 | 7.62 ± 0.70 | + | — |
| Phenacetin | 1-2 | 0.57 ± 0.08 | 4.20 ± 0.39 | — | + |
| Ascorbic acid | 1 | 0.74 ± 0.03 | 1.67 ± 0.15 | + | — |

Average of 4 determinations at air velocity of 7.2 m/s.

charge accumulation from the surrounding electrical interference during 8 min on 3 different days showed that the gain was negligible (i.e. up to 2.2×10^{-11} C which was less than 0.01% of charge during detachment measurements).

The degree of charge build-up by the compressed air stream through the empty cage was examined. Undesired charging can occur in two ways: (i) charge build-up due to vibration of the cage; and (ii) exchange between cage wall and air stream. Charge development during air flow through the cage for up to 8 min at the air velocities typically used in charge measurement was negligible compared to the charge measured during detachment (i.e. up to 9.9×10^{-11} C which was about 0.1% of the charge developed during average charge-to-mass ratio measurements).

(b) *Carrier interaction*

Carrier charging can occur in a number of ways: (i) interference of surroundings; (ii) mechanical vibration of the cage at high air velocity

causing charge generation at the connection point of the cage to the air flow hose; (iii) charging by high impact of the carrier to the cage wall; and (iv) piezoelectric effect due to high stress between carrier particles or between carrier and the cage wall.

Charge exchange between cage and carrier was tested by blowing compressed air through 2 g of the carrier at various air velocities (Table 2). The degree of charge build-up increased with increasing time of air streaming and velocity. However, particle detachment time during charge measurement of the interactive powder mixtures was not more than 1 min and it was generally shorter for low triboelectrified powder or for incremental detachment charge determinations. During this time period (about at 15 intermittent cycles), the magnitude of charge gained at air velocity of $7.2 \text{ m} \cdot \text{s}^{-1}$ (the maximum air velocity used in experiments) was 5×10^{-10} C. These values were less than 1% of the charge measured from the mixtures.

TABLE 2

Observation of charge exchange between cage and carrier at different air velocities

| Observation | No. of intermittent air flow cycles | Air velocity ($\text{m} \cdot \text{s}^{-1}$): | Charge, $\times 10^{-7}$ C | | | |
|-------------|--|--|----------------------------|---------|---------|---------|
| | | | 3.0 | 5.0 | 7.2 | 8.5 |
| 1 | 5 | | 0.00030 | 0.00057 | 0.00209 | 0.00840 |
| | 10 | | 0.00045 | 0.00076 | 0.00365 | 0.01100 |
| | 15 | | 0.00055 | 0.00104 | 0.00524 | 0.01762 |
| | 20 | | 0.00054 | 0.00154 | 0.00721 | 0.01985 |
| | 25 | | 0.00070 | 0.00199 | 0.00952 | 0.02810 |
| | 30 | | 0.00094 | 0.00241 | 0.01095 | 0.03380 |
| 2 | 5 | | 0.00013 | 0.00068 | 0.00109 | 0.00690 |
| | 10 | | 0.00068 | 0.00079 | 0.00246 | 0.00990 |
| | 15 | | 0.00085 | 0.00087 | 0.00389 | 0.01379 |
| | 20 | | 0.00098 | 0.00100 | 0.00526 | 0.01829 |
| | 25 | | 0.00109 | 0.00132 | 0.00637 | 0.02280 |
| | 30 | | 0.00121 | 0.00155 | 0.00774 | 0.02800 |
| 3 | 5 | | 0.00005 | 0.00068 | 0.00135 | 0.00890 |
| | 10 | | 0.00007 | 0.00074 | 0.00249 | 0.01386 |
| | 15 | | 0.00013 | 0.00080 | 0.00370 | 0.01796 |
| | 20 | | 0.00019 | 0.00091 | 0.00488 | 0.02300 |
| | 25 | | 0.00023 | 0.00109 | 0.00632 | 0.02710 |
| | 30 | | 0.00029 | 0.00130 | 0.00746 | 0.02980 |

13 intermittent cycles equals 1 min air flow.

(c) *Drug and particle interactions*

The true electrostatic charge produced on detachment from the carrier may be altered during charge measurement in the air stream Faraday cage (Schein and Cranch, 1975). When the air stream detaches particles from the carrier surface, true charge measurement relies on the drag force carrying the particles out of the cage without contact with other surface. Contact with the cage wall, screen or other carrier particles would cause charge exchange and produce spurious total charge measurements. The following experiments were carried out to indicate whether the powder charges were altered. The tests have been made using sulphapyridine-carrier interactive mixtures.

(i) *Effect of sample size.* The effects of particle charge exchange with the other carriers during detachment were tested by varying the sample size of the mixtures taken for charge measurement (Table 3). Triboelectric properties of 3 different mixes (A, B and C) were measured by loading the cage with the sample between 1 and 2, 1 and 3, and 2 and 6 g, respectively. The resultant charge measurements showed good agreement among the different cage loadings for 3 mixtures. The difference in average charge-to-mass ratio between 3 mixtures reflected the different triboelectrical charging during interactive mixture preparations and charge decay before the measurements were taken. On the assumption that increasing the number of carrier particles in the cage (by increas-

ing cell loading) would increase the number of contact between detached particles and other carriers, particle charges might be altered in proportion with the cage loadings. No dependence of charge measurements on the sample size was observed (*F*-test, $P = 0.05$) indicating that no powder charge exchange with other carriers occurred. However, at increasing sample size, if the blockage of powder leaving the cage arises, significant error of charge measurement might be observed.

(ii) *Effect of screen size.* The possibility that powder can charge exchange with the screen during removal from the cage was tested by using the different screen sizes on the cage. It was assumed that the smaller screen size could increase the area contact with the powder particles so that if charge exchange occurred, the results of charge measurement of the same mixture would be altered. Table 4 shows the charge measurement results for the sulphapyridine-carrier interactive mixture when the screen sizes were varied between 160, 180 and 250 μm . No statistically significant effect of charge exchange between particles leaving the cage and the screen was observed (*F*-test, $P = 0.05$).

(iii) *Effect of cage lining.* The effect of charge exchange between powders and the cage wall was examined by lining the inner cage wall with different materials. The charge measurements of the sulphapyridine-carrier interactive mixture were performed in an unlined cage and in cages lined with aluminium sheet and plastic tape. The results (Table 5) showed no change of powder charges with different cage wall properties (*F*-test; $P = 0.05$) indicating the sulphapyridine particles did not interact significantly with the cage wall.

TABLE 3

Triboelectric values of sulphapyridine powder-carrier mixture at different cage loadings

| Mix | Sample size (g) | $T \pm \text{S.D.}$ ($\mu\text{C} \cdot \text{g}^{-1}$) | $C \pm \text{S.D.}$ (%) |
|-----|-----------------|---|-------------------------|
| A | 1 | 5.08 ± 0.44 | 0.69 ± 0.07 |
| | 1.5 | 4.86 ± 0.57 | 0.76 ± 0.07 |
| | 2 | 5.02 ± 0.30 | 0.78 ± 0.03 |
| B | 1 | 3.33 ± 0.16 | 0.84 ± 0.06 |
| | 3 | 3.33 ± 0.19 | 0.92 ± 0.04 |
| C | 2 | 3.89 ± 0.16 | 0.79 ± 0.04 |
| | 4 | 4.10 ± 0.30 | 0.84 ± 0.03 |
| | 6 | 4.24 ± 0.17 | 0.83 ± 0.03 |

Average of 4 determinations.

TABLE 4

Triboelectric values of sulphapyridine powder-carrier mixture when placing different screen sizes on Faraday cage

| Screen size (μm) | $T \pm \text{S.D.}$ ($\mu\text{C} \cdot \text{g}^{-1}$) | $C \pm \text{S.D.}$ (%) |
|-------------------------------|---|-------------------------|
| 160 | 5.45 ± 0.60 | 0.68 ± 0.05 |
| 180 | 5.54 ± 1.00 | 0.63 ± 0.08 |
| 250 | 5.64 ± 0.90 | 0.63 ± 0.08 |

Average of 4 determinations.

TABLE 5

Triboelectric values of sulphapyridine powder-carrier mixture when lining the inner cage wall with different materials

| Lining material | $T \pm S.D.$ ($\mu C \cdot g^{-1}$) | $C \pm S.D.$ (%) |
|-----------------|--|---------------------|
| Unlined cage | 3.83 ± 0.03 | 0.76 ± 0.03 |
| Aluminium sheet | 3.71 ± 0.12 | 0.82 ± 0.03 |
| Plastic tape | 3.69 ± 0.31 | 0.79 ± 0.05 |

Average of 4 determinations.

Limitations of average charge-to-mass ratio measurements

The average charge-to-mass ratio recorded in Table 1 reflected the triboelectric property of the powder mixtures after storage of the mixture under constant conditions for a period of time. On storage, charge decay occurred with a resultant decrease in interactive force between the adherent particle and carrier. During charge measurement, therefore, most of the adhered powder was easily removed from the Faraday cage.

The phenomenon was well illustrated using a sulphapyridine-carrier interactive mixture stored at controlled temperature and humidity (Table 6). Freshly prepared samples showed a high average

TABLE 6

Relationship between intensity of triboelectric charges of sulphapyridine powder-carrier mixture and detachment capability of air velocity at $7.2 \text{ m} \cdot \text{s}^{-1}$

| Time (days) | Tribo ($\mu C \cdot g^{-1}$) | C (%) | % amount retained on carrier | Estimated % amount adhering on the cage |
|----------------|-----------------------------------|------------|------------------------------------|--|
| 0 | 12.46 | 0.43 | 0.18 | 0.28 |
| | 13.24 | 0.44 | 0.14 | 0.31 |
| 2 | 6.49 | 0.75 | 0.03 | 0.11 |
| | 6.47 | 0.77 | 0.01 | 0.11 |
| 3 | 4.70 | 0.79 | 0.02 | 0.08 |
| | 4.35 | 0.81 | 0.03 | 0.05 |
| 4 | 4.17 | 0.88 | 0.02 | — |
| | 3.90 | 0.89 | 0.03 | — |
| 5 | 3.67 | 0.89 | 0.01 | — |

Nominal weight of sample = 1.5 g.

charge-to-mass ratio (about $13 \mu C \cdot g^{-1}$) which decayed to $3.67 \mu C \cdot g^{-1}$ over a 6 day period.

Two major difficulties arose in the charge measurement of freshly prepared interactive mixtures using commercial powder samples: (a) the powders contained a high proportion of small particles; and (b) the powders were highly triboelectrically charged during interactive mixture preparation.

The aerodynamic force (F_{drag}) directed on adherent particles will be determined by its particle size and air velocity employed according to the relation (Zimon, 1982)

$$F_{\text{drag}} = c_x D V^2 \pi r^2 / 2$$

where c_x is the drag coefficient of particle, D is the density of the air, r is the radius of particle, V is the air flow velocity.

For a given air velocity, smaller particles were subjected to less action of air drag force of detachment than are larger size. Very high air velocities were therefore needed to detach the interacted small particles with a resultant potential error due to spurious charge induction by mechanical vibration of the cage and high impact forces on the mixture.

In addition, for highly triboelectrified mixtures, some particles which were detached, were not removed from the cage and interacted with the cage material. Charge neutralization occurred with a subsequent underestimate of the average charge-to-mass ratio. The interaction also was reflected in the low $C\%$ values.

For example, for a freshly prepared sulphapyridine-carrier interactive mixture, the $C\%$ value was about 0.43% with about 30% of the highly charged sulphapyridine adhering to the cage wall. As charge decay occurs and the electrostatic charging decreases, most of the detached particles were removed from the cage with little interaction.

Electrostatic charge distribution

Incremental particle detachment can be performed using the air stream Faraday cage and can lead to the determination of a charge distribution. Instead of removing the powder with a single high air velocity, the powder was detached at several increments of air velocity. Charge-to-mass ratios

at each incremental air velocity will represent the powder charge distribution.

At each incremental air velocity (V), the mass removed (M_i) and charge measured (Q_i) were used to calculate following parameters.

(a) Fraction mass (FM_i) which describes the percentage fraction of powder removed at each incremental air velocity is:

$$FM_i (\%) = 100 \cdot M_i / M$$

where M is the total mass removed from the mixture.

(b) Cumulative concentration (CC) of powder which describes total amount of powder removed up to that incremental air velocity is:

$$CC (\%) = \sum M_i / W$$

where $\sum M_i$ is the cumulative amount of powder removed up to that increment, and W is final weight of mixture after total powder detachment.

(c) Intermediate tribo (IT_i) which represents average charge-to-mass ratio of powder removed at each increasing air velocity is:

$$IT_i = Q_i / M_i$$

(d) Average tribo ($A_i T_i$) which represents total average charge-to-mass ratio up to incremental air velocity used is:

$$A_i T_i = \sum Q_i / \sum M_i$$

where $\sum Q_i$ is the cumulative charge registered on electrometer up to that increment. The $A_i T_i$ value at final increment will correspond to average charge-to-mass ratio determined by a single detachment procedure.

Duplicate incremental detachment measurements of both sulphapyridine and sulphamerazine interactive powder mixtures were obtained (Tables 7 and 8). Tribo distribution of the two replications of the same mixture were consistent. The results of the two procedures, incremental detachment and total detachment at single air velocity of $7.2 \text{ m} \cdot \text{s}^{-1}$, were compared for the same sulphamerazine-carrier interactive mixture (Table 8). Total tribo obtained by both procedures was in good agreement. This suggested that the procedure of incremental detachment did not alter the powder charges and no dependence of powder charge on air velocity was observed. However, the C value of incremental detachment was less than that for total detachment. The lower C value might be due to the fact that the lower air velocities could not expel some particles because of weak air drag force acting on them. These particles were likely to deposit on the surface of the cage wall or screen and would be difficult to remove because of the boundary layer effect. The difference in $\%C$ does not have any significant effect on total tribo measured.

Two explanations have been discussed on the cause of charge distribution of powders. Schein and Cranch (1975) demonstrated that the charge distribution of powder mixtures was particle size

TABLE 7

Triboelectric properties of sulphapyridine-carrier mixture detached at incremental air velocity

| Replicate | Air velocity ($\text{m} \cdot \text{s}^{-1}$) | FM_i (%) | IT_i ($\mu\text{C} \cdot \text{g}^{-1}$) | CC (%) | $A_i T_i$ ($\mu\text{C} \cdot \text{g}^{-1}$) |
|----------------|--|---------------|---|-------------|--|
| 1 ^a | 2.0 | 21.8 | 2.78 | 0.18 | 2.78 |
| | 4.0 | 51.5 | 6.47 | 0.62 | 5.37 |
| | 6.0 | 26.7 | 8.18 | 0.85 | 6.12 |
| 2 ^b | 2.0 | 21.2 | 3.13 | 0.16 | 3.13 |
| | 4.0 | 57.6 | 6.67 | 0.61 | 5.71 |
| | 6.0 | 21.2 | 8.75 | 0.77 | 6.36 |

^a Sample wt. of replicate 1 = 1.9602 g.

^b Sample wt. of replicate 2 = 1.9717 g.

TABLE 8

Triboelectric properties of sulphamerazine powder-carrier mixture detached at incremental air velocities

| Replicate | Air velocity ($\text{m} \cdot \text{s}^{-1}$) | FM_i (%) | IT_i ($\mu\text{C} \cdot \text{g}^{-1}$) | CC (%) | $A_i T_i$ ($\mu\text{C} \cdot \text{g}^{-1}$) |
|----------------|--|---------------|---|-----------|--|
| 1 ^a | 2.0 | 32.0 | 2.42 | 0.16 | 2.42 |
| | 4.0 | 31.1 | 7.50 | 0.32 | 4.92 |
| | 6.0 | 29.1 | 16.33 | 0.47 | 8.53 |
| | 7.2 | 7.8 | 31.25 | 0.51 | 10.29 ^c |
| 2 ^b | 2.0 | 40.2 | 2.09 | 0.21 | 2.09 |
| | 4.0 | 31.8 | 8.53 | 0.37 | 4.94 |
| | 6.0 | 21.5 | 20.43 | 0.48 | 8.17 |
| | 7.2 | 6.5 | 27.14 | 0.51 | 9.27 ^c |

^a Sample weight of replicate 1 = 2.0294 g.^b Sample weight of replicate 2 = 2.0944 g.^c Tribo of the same mixture by total detached at air velocity of $7.2 \text{ m} \cdot \text{s}^{-1}$. $T = 10.60 \mu\text{C} \cdot \text{g}^{-1}$, $C = 0.54\%$. $T = 9.902 \mu\text{C} \cdot \text{g}^{-1}$, $C = 0.61\%$.

distribution dependent. The charge on similar-sized powder particles was considered to be uniformly distributed. In contrast, Harpavat and Orr (1975) suggested that the number of rubbing contact events between powder particle and carriers during blending was random, and all particles were unequally charged. Hence charge distributions were obtained.

It was considered that both causes could occur depending on concentration of powders in mixtures. Total tribo was a function of the amount of powder on the carriers. As the powder concentrations increased reduction of tribo values occurred (Lee, 1978). This sort of drop in tribo was related to reduction of surface charge density (charge magnitude per surface area) of particle due to the formation of multilayer adherent particles and to the lack of direct contacts between powder and carriers (Dravin and Belousov, 1972; Narusawa et al., 1978). Consequently, random contacts were likely to predominate in high powder concentration mixtures because of competition among particles to make contact with the carriers. Difference in contact events resulted in unequal charging of particle; hence charge distribution occurred even for similar-sized particles. For low concentration powder mixtures, all powder particles could have the same chances of contact with the carriers during blending so that a more uniform charge

distribution of particles was obtained.

Incremental detachment provided more detailed information on the distribution of triboelectric properties of the powders while total detachment measurement gave the only single value property of tribo. Powder mixtures which possess similar total average tribo may be different in tribo distributions.

Conclusions

The use of the air stream Faraday cage allowed the measurement of the average charge-to-mass ratio generated on the detachment of a drug particle from a carrier surface. The measurements were relatively precise and the system was validated using interactive powder mixtures which were not too highly triboelectrified. The air stream method of measuring electrostatic charge had some distinct advantages.

(a) The method allowed the measurement of a relatively high electrostatic charge value minimizing the background effect of spurious electrostatic fields and carrier-cage charging.

(b) The method allowed the measurement of contact potential charge in relation to a particular drug particle-carrier interface. The value of the charge generated on detachment therefore in-

dicated the stability of the interactive system to drug segregation.

(c) The method measured the particulate charge upon detachment from an interactive system which typified such systems found in pharmaceutical processing. The degree of contact potential interaction between the drug particle and a substrate will depend on the manner in which the mixture was formed. In most pharmaceutical processing, e.g. mixing, particle size reduction filling, etc., particle interaction will involve shear, impact, accelerative and centrifugal forces. The mixing procedure used in this study to form the interactive system therefore provided a more realistic model to study particle detachment than other methods which rely on simply dusting powders onto substrate surfaces.

(d) The method allowed the measurement of a charge distribution to characterize the interactive system.

The major limitation of the method was its inability to measure highly triboelectrified systems. The detached particles interacted with the cage negating the charge measurement for the particular particle.

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