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Electrostatic Charge Generation due to Shear Deformation of Pharmaceutical Powders

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ABSTRACT

In powder handling operations, such as mixing, conveying, sieving and milling, particles frequently make contact with each other and the walls of the processing equipment. During these interactions, the electrification of particles takes place, commonly known as tribocharging. This paper focuses on investigating the tribocharging characteristics of bulk powders under shear deformation. In particular, the effect of shear strain and shear rate on the charge generation is analysed. An annular shear cell, modified for electrostatic charging, is used for applying a shear strain to a bed of pharmaceutical powders.

α-lactose monohydrate particles, widely used as an excipient for pharmaceutical formulations, is used as a model system. It is shown that the charge on the particles increases with the applied shear strain.

1 INTRODUCTION

Electrostatic particle charging is a common nuisance in industrial process operationsas as it can cause segregation, dust explosion (Nomura *et al.*, 1992), adhesion and deposition or blockage of pipelines (Matsusaka *et al.*, 2001) leading to a loss of powder and difficulties controlling the powder flow. In the pharmaceutical industry, the problem extends further in affecting the quality of the end product.

Tribocharging of powders can cause poor tablet content uniformity. An important manifestation of this problem is a susceptibility to a change in drug formulation in various processes such as tabletting. The pharmaceutical powders are usually semiconductors or insulators of small particle sizes and low bulk densities, providing ideal conditions for electrostatic effects.

The importance of understanding charging propensities can be seen from the SEM image in Figure 1. The net charge on Drug A alone is -4 nCg^{-1} and that of lactose being -5 nCg^{-1}. The combination of these two negatively charged materials has resulted in certain regions containing only the agglomerate of Drug A. These types of interactions between the active components of pharmaceutical formulation and the excipient can lead to variations in the end product composition.

On the other hand, tribocharging of powders that results in selected polarity and magnitude is a desirable feature, which has been positively utilised in many applications, such as electromechanical operation (Ghadiri *et al.*, 1992), electrostatic powder coating (Kleber *et al.*, 1998) and tomography (Machida *et al.,* 1998). In other processes, such as electrostatic separation of powder, tribocharging phenomena are used to charge a binary mixture with opposite polarity which is then separated in an electrostatic field.
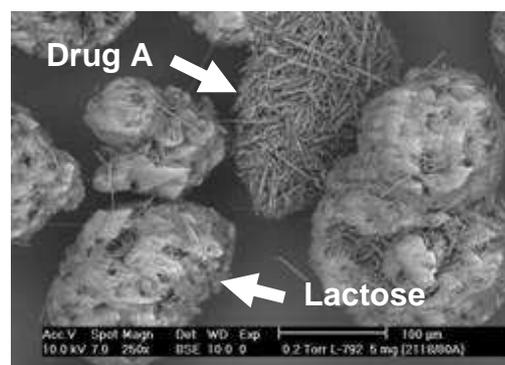

Figure 1: Interactions of negatively charged ingredients with lactose and active pharmaceutical particles (Pitt, 2005).

It is therefore paramount for the industry to have a complete understanding of the charging propensities in all powder handling processes.

Tribocharging due to single impacts has been investigated extensively (Masuda *et al.*, 1978, Matsuyama *et al.*, 1989, Matsusaka *et al.*, 2000 and Watanabe *et al.*, 2004). However, for a number of cases associated with powder flow, the bulk powder is in a dense state and can only flow if it is sheared, a situation which can also cause tribocharging. However, little work has been reported in the literature on this area. The focus of the present work is on studying tribocharging characteristics of bulk powders under shear deformation. Electrostatic charge generation will be achieved by applying a shear strain to a bed of α-lactose monohydrate particles in the size range of 250-300 µm using an annular shear cell. α-lactose monohydrate is chosen as a test material as it

is commonly used in the pharmaceutical industry as an excipient or carrier having wide interacting capabilities with actives and other excipient ingredients. The shear cell was electrically isolated and the electric current through the earth was measured by an electrometer. The ongoing work on the analysis of current as a function of the applied normal load and shear strain will be presented below.

2 EXPERIMENTAL

A Faraday cup is used to measure the initial and final charge levels on the particles before and after shearing. It consists of a stainless steel cup which is insulated by polytetrafluoroethylene (PTFE) and surrounded by another stainless steel cup rigidly constructed to avoid the induction of current by mechanical vibrations. The inner cup (which is removable to allow the powder to be emptied with ease) is connected to an electrometer. The electrometer (type 6514, Keithley Instruments, Cleveland, USA) is used for charge and current measurements.

A sample of powder is sheared using a modified Schulze shear cell (Schulze, 1994) and the current flowing through the bed of powder is measured by an electrometer which is connected to the bottom ring of the shear cell forming the lower electrode (Figure 2). The modified shear cell is made out of PTFE and has a hole (1 mm in diameter) in the wall. This allows a wire to pass through so that the lower electrode can be connected to the electrometer for current measurement during testing. This shear cell is a modification of the original shear cell that was made out of aluminium with inner and outer diameters being 32 and 64 mm respectively.

The shear cell is filled with the powder in such a way that the powder surface is flush with the upper edge of the shear cell. The filled shear cell is then weighed and placed on the Ring Shear Tester. The mass of the filled shear cell is required by the control software for the calculation of the bulk density. The lid is then placed on the powder and the loading rod inserted followed by attaching the tie rods to the lid. The sample is then sheared at normal load of 20 kPa and the current recorded continuously by the method described above.
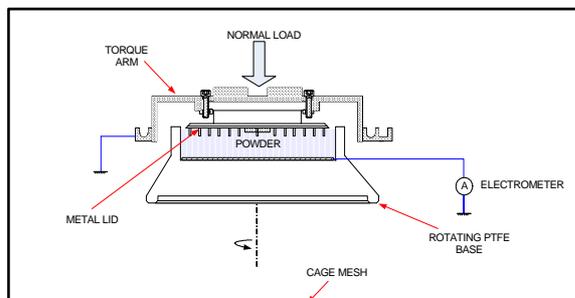

Figure 2: Measurement of charge generation as a result of shearing.

3 RESULTS AND DISCUSSION

Samples of α-lactose monohydrate in the particle size range of 250-300 μm were sheared under the normal load of 20 kPa at two different shear rates. To investigate the effect of shear strain on electric current generation, the samples were tested at shear rates of 0.0021 s^{-1} and 0.0057 s^{-1}. The tests were carried out under controlled surroundings to minimise the effects of varying atmospheric conditions. During tests, the relative humidity ranged from 35.2-39.4% and temperatures between 22.8-23.7°C. The amount of powder that was used for each shearing test varied between 20.3 and 24.6 grams, depending on the particle size range chosen.

Prior to carrying out shearing tests, i.e. without the application of stresses, an initial charge on the powder sample was measured by pouring the sample powder out of the stainless steel cup into the Faraday cup that was connected to an electrometer. This indicated the polarity and magnitude of the initial charge on the sample powders prior to the shearing test.

The preconsolidation normal load set to 20 kPa and the sample powder was subjected to shearing. The variation of the shear force, divided by the annulus area, represents the average shear stress, τ. The shear stress versus time reaches a peak and then follows a descending trend reaching a steady-state level. The electric current in the powder during the test is measured simultaneously.

The shearing is then stopped and the powder sample is relieved from the normal load. The powder sample is then poured into the Faraday cup connected to the electrometer for final charge measurements. This procedure is repeated for each powder sample at various shear rates and strains with current measured simultaneously. The results obtained are presented in the following figures.

In Figure 3, the results of the sample of α-lactose powder, sheared at a rate of 0.0021 s^{-1} are presented. The normal load was preset at 20 kPa. The initial charge on the powder prior to shearing was -2.181 nC. The shear rate (γ) in the figure caption is defined as

$$\gamma = \frac{\omega(r_i + r_o)}{2h}$$

where ω is the angular speed, h is the bed height, r_i and r_o are inner and outer radii of the annular space. The shear strain (ε) is defined as

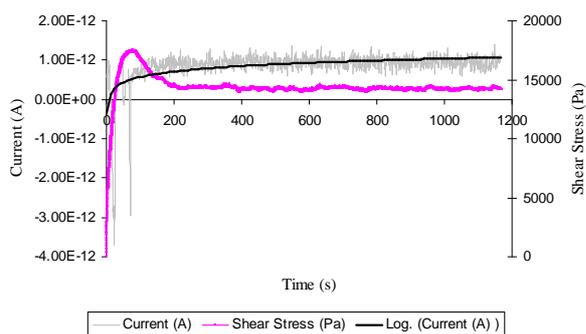$$\varepsilon = \gamma \times t$$

where γ is the shear rate and t is the shearing time.

Figure 3: Effect of shearing on the electric current for α-lactose (250-300 μm) powder at 20 kPa normal load and shearing rate (γ) of 0.0021 s^{-1}.

The particles were loosely packed (packing fraction 0.57) at the beginning of the shearing stage. This accounted for the low initial current as the inter-particle friction forces are small at the beginning. However, particles can move against each other and rotate and slide, causing a general increase in the current as the shear strain is increased. The charge measurement at the end of the test was 2.360 nC which indicated a large increase in the magnitude of charge, in addition to a change in the polarity after the sample of α-lactose was sheared.

Figure 4 shows the current generation when α-lactose (250-300 μm) powder was tested at a higher shear rate of 0.0057 s^{-1}. The net charge on the powder was 2.981 nC. An increase in current is generated as the sample powder is sheared at these conditions, and can be seen in Figure 4. The current is averaging approximately 2×10^{-12} Amps and is further increasing in comparison to tests at shear rate of 0.0021s^{-1} where current was averaging approximately 1×10^{-12} Amps.
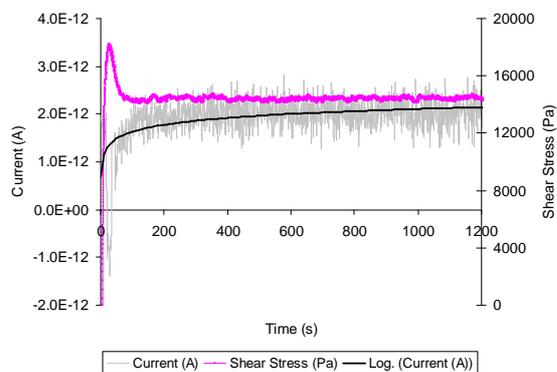

Figure 4: Effect of shearing on the electric current at a higher shear rate of 0.0055 s^{-1}.

The results obtained during the shearing process for α-lactose (250-300 μm) suggests an occurrence of tribocharging in the powder due to shear deformation, which is verified by a significant increase in current generated when the shear strain was increased up to 6.84. There was also a significant difference on the amount of charge generated on the powder at higher shear strain (see Figure 5).

When increasing the shear strain on a bulk powder, the powder becomes increasingly compact and a general increase in the current generated is observed in comparison to the same powder with a lower shear strain. This may be due to the fact that the particles are pressed against each other and thus the frictional forces increase giving rise to tribocharging. Finally, the frictional forces between the particles are fully mobilised so that the maximum possible friction force and charge generation occur.

A common feature of all the experiments were a positive current measurement during the shearing stage. Positive current to earth implies that the particles become negatively charged due to preservation of charge. However, in these tests the net charge of the powders changes from negative to positive indicating that the net charge was produced by contact between particles and the PTFE.

The net charge measurements for α-lactose (250-300 μm) at various shear strains are shown in Figure 5, and confirm an increase in charge as the shear strain is increased. The results can be attributed to higher particle activity at a higher shear strain resulting in a large number of particle contacts.
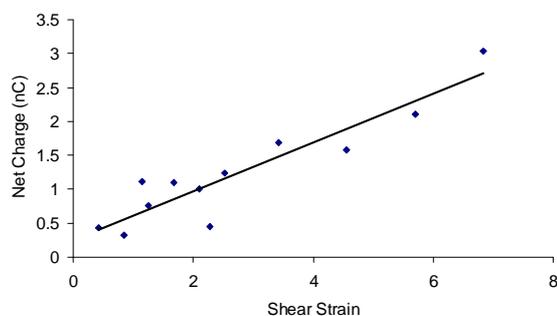

Figure 5: Relationship between shear strain and net charge for α-lactose (250-300 μm) powder.

Surface contact effects, including surface roughness and contact force, may also influence the current generation. In the case of α-lactose powder in Figure 6, the particles do not have a defined shape therefore come in a variety of shapes some of which are less spherical compared to some other pharmaceutical excipients, such as sugar granules.
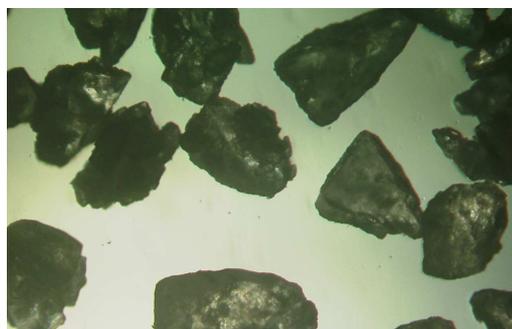

Figure 6: Image of a sample of α-lactose, captured using an optical microscope, in the particle size range between 300-355 μm.

The connecting wire between the electrometer and a bottom electrode of the shear cell (see Figure 2) is a limitation on the length on the shearing time. As the shear cell rotates, the wire becomes shorter by wrapping itself around the cell.

Therefore to eliminate this problem, the shear cell will further be modified by incorporating a metal strip on the outside of the cell. A set of brushes will then be used to contact the outer strip allowing the shear cell to rotate infinitely and achieving larger shear strains.

In future work to follow, binary mixtures will be sheared, the shearing times will be increased and tests will be carried out at various normal loads. This may result in the net charge being produced by the inter-particle contacts instead of the particle-to-wall collisions.

4 CONCLUSIONS

Application of normal and shear stresses that bring about shear deformation in a bed of selected pharmaceutical powders using an annular shear cell, gives rise to tribocharging. An increase in the shear strain in powder samples, results in an increase of the magnitude of current and charge generated as particles are rubbing and colliding with each other and with the walls of the shear cell.

The results presented above are for α-lactose (250-300 μm) powder under specific conditions. It is therefore essential that shearing of different powders is also studied as physical and environmental factors such as the particle size, shape, relative humidity, temperature and many other factors may affect the tribocharging process.

ACKNOWLEDGEMENTS

REFERENCES:

Ghadiri, M., Martin C.M., Morgan, J.E.P. and Clift, R. (1992) An electromechanical valve for solids, *Powder Technology*, **73**, 21-35.

Kleber, W. and Makin, B. (1998) Triboelectric powder coating: a practical approach for industrial use. *Pariculate Science Technoogy*, **16**, 45-53.

Machida, M. and Scarlett, B. (1998) Development of displacement current tomography system. *Particle Characterisation*, **15**, 36-41.

Masuda, H. and Iinoya, K. (1978) Electrification of particles by impact on inclined metal plates. *AIChE Journal*, **24**, 61.

Matsusaka, S., Ghadiri, M. and Masuda, H. (2000) Electrification of an elastic sphere by repeated impacts on a metal plate. *Journal of Physics D: Applied Physics*, **33**, 2311-2319.

Matsusaka, S., Theerachaisupakij, W., Yoshida, H. and Masuda, H (2001) Deposition layers formed by a turbulent aerosol flow of micron and sub-micron particles. *Powder Technology*, **118**, 130-135.

Matsuyama, T. and Yamamoto, H. (1989) Charge transfer between a single polymer particle and a metal plate due to impact. *Kona,* **7**, 15-21.

Nomura, S. and Tanaka, T. (1992) Theoretical analysis of dust explosions. *Powder Technology*, **71**, 189-196.

Pitt, K. (2005) Pharmaceutical industrial challenges of the electrostatic phenomena, *Electrostatic Phenomena in Particulate Systems Seminar*, Leeds, UK.

Schulze, D. (1994) Development and application of a novel ring shear tester, *Aufbereitungstechnik 35*, **10**, 524-535.

Watanabe, H., Samimi, A., Ding, Y., Ghadiri, M., Matsuyama, T. and Pitt, K. G., (2004) Measurement of tribo-charging by particle impact, *9th International Conference Multiphase Flow in Industrial Plants*, Rome, Italy.