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AN ANALYSIS OF A SIMPLE TEST DEVICE FOR TRIBO-CHARGING OF BULK POWDERS

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Abstract

A simple device for characterisation of the tribo-charging propensity of powders has been developed at the University of Leeds, where a small amount of powder is placed inside a 10 ml container, which is shaken by reciprocal strokes in a horizontal direction. Several containers with different materials have been made: stainless steel, polytetrafluoroethylene (PTFE) and glass. The charge on the powder is measured using a Faraday cup connected to an electrometer. The charge is measured before and after the shaking process.

The main objective of this work is to analyse the operation of this simple test device by investigating the behaviour of α-lactose monohydrate, hydroxy propyl cellulose (HPC) and a 50:50 binary mixture (by mass) of these two powders with various surfaces that are most commonly used in the pharmaceutical industry. The experiments are carried out in controlled environmental conditions and using different shaking times together with different shaking frequencies of 10, 20 and 30 Hz.

The experimental results show that α-lactose monohydrate and HPC particles have the highest magnitude of charge at 20 Hz frequency against all surfaces tested. This is surprising, as it is intuitively expected that higher charges should be produced at 30 Hz, given other conditions. The dynamic movement of particles within a shaking container vary with frequency. This results in a varied amount of particle-wall contacts which affects particle charging.

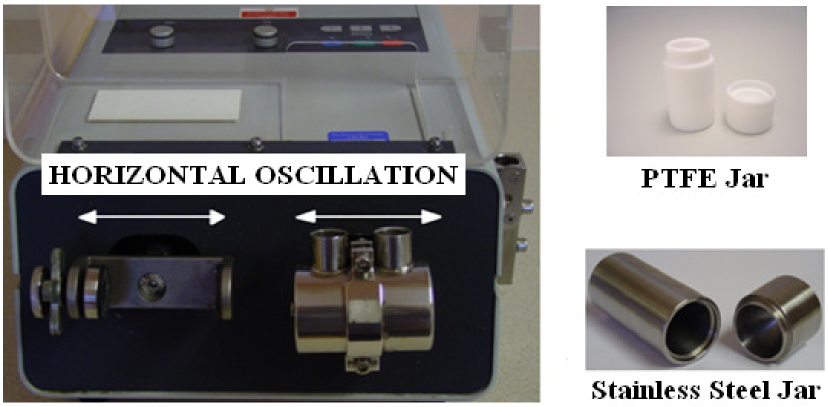
1. INTRODUCTION

In powder handling operations, particles frequently come into contact with each other and with the walls of the processing equipment causing the tribo-electrification of the particles. Pharmaceutical powders are often small in particle size (less than 100 μm), irregularly shaped and have low bulk density. This makes them highly susceptible to electrostatic charging as they normally have a high electrical resistance, preventing charge dissipation, Grosvenor and Staniforth [3]. The electrostatic forces acting on charged pharmaceutical particles dominate in adhesion and deposition of the particles to walls in the case of fine particle systems, such as dry powder inhalers (DPI), Bailey [1] and Balachandran *et al.,* [2].

In the pharmaceutical industry, electrostatic particle charging is a common nuisance as it can cause segregation, dust explosions; Nomura *et al.*, [5], adhesion and deposition or blockage of pipelines; Matsusaka *et al.*, [4], leading to loss of powder and difficulties controlling the powder flow. The ability to control the charging of pharmaceutical powders is essential in improving the quality of the end product and minimising deposition and powder loss.

2. EXPERIMENTAL

To investigate the tribo-electrification of bulk powders due to multiple contacts, a Retsch MM200 shaking machine has been modified at the University of Leeds (Fig.1), where a small sample of powder is placed inside a shaking container of 10 ml in volume. The container performs reciprocal vibrations in a horizontal direction. Due to the inertia of the tribocharging container, the sample material inside is thereby impacted alternately against the rounded ends of the container. The containers made of Stainless Steel, PTFE, Perspex or glass, can be easily replaced to test the surface effect on tribo-electrification of pharmaceutical powders. The intensity can thereby be set precisely between 3 and 30 vibrations per second and a speed control keeps this value constant during the tribo-charging process. The tribo-charging time can be digitally preset from 10 seconds up to 99 minutes. The arm, 8.4 cm in length, rotates 6.12° resulting in an amplitude of the vibrations of 0.89 cm.



PTFE Jar

Stainless Steel Jar

Figure 1. Retsch Shaking Machine and Containers.

To measure the electrostatic charge of the bulk powders, a Faraday cup (Fig. 2) was used which consists of two stainless steel cups isolated from each other by a PTFE spacer. The inner cup is connected to an electrometer (Keithley Model 6514) via a BNC cable, and the outer cup is earthed. The electrometer is connected to a computer and data is recorded using Test Point v6 software. The inner cup can be easily removed to measure weight of the sample poured into the cup. A stainless steel lid, fitted to the outer cup, is used to reduce electric noise. The resolution of the charge measurement is in nano-Coulombs (nC) order of magnitude.

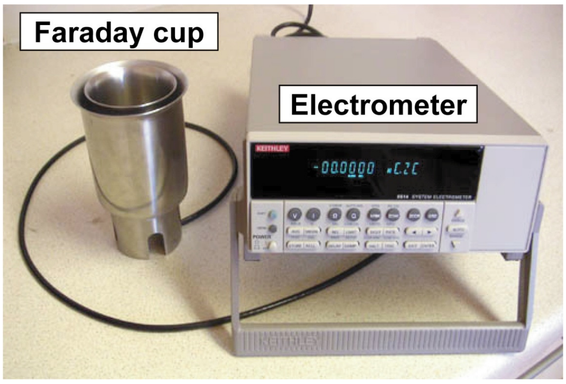


Figure 2. Faraday Cup and an Electrometer.

In order to control the environmental conditions during the test, a Microflow-CIT isolator system of approximately 0.6 m^3 volume capacity was used (Fig. 3). All of the test equipment, including the tribocharging device, electrometer, Faraday cup, humidity, temperature meters and the test materials were placed inside the isolator and sealed. Nitrogen can be supplied through an aperture on the side of the isolator to control the humidity. The edges of the isolator were reinforced using insulating tape to minimise any nitrogen leakages.

The test materials are placed inside the isolator via the side chamber. The humidity and temperature inside the isolator is monitored using a HydroPalm device. By adjusting the amount of gas coming into the isolator, the humidity can be reduced to below 5 %. Another added advantage of a tightly closed system for tribo-charging of pharmaceutical material is the ability of the operator to work with active ingredients with a reduced risk of exposure to dust and associated toxic hazards.

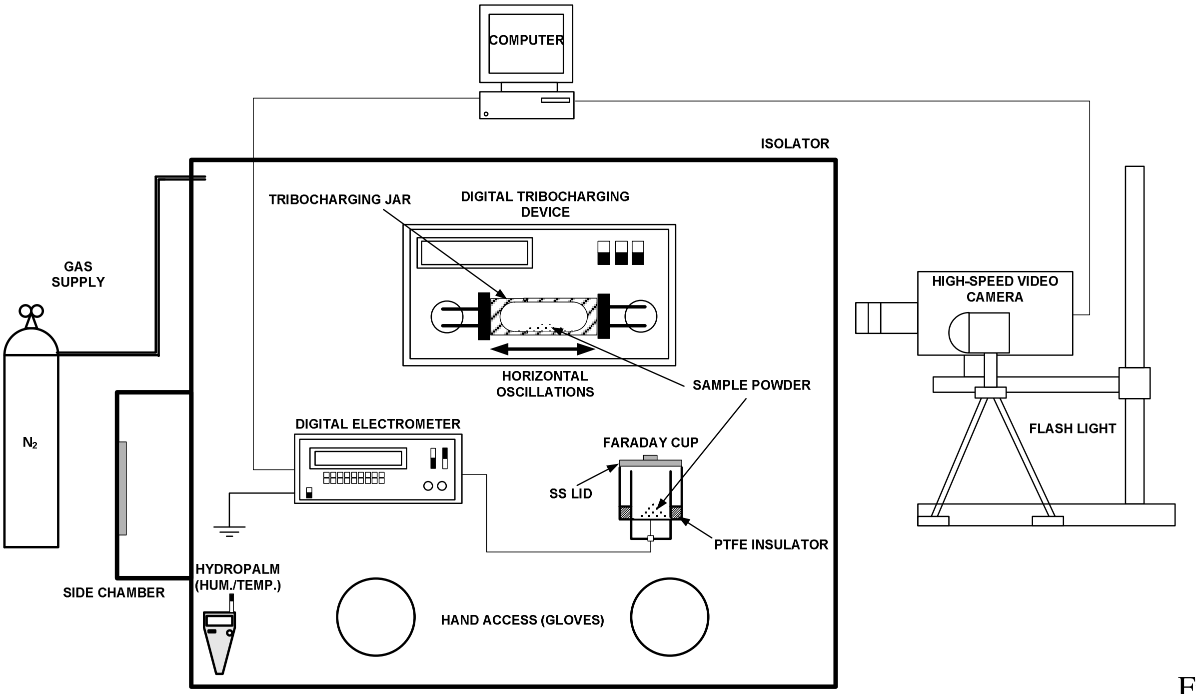


Figure 3. Experimental Set-up of the Bulk Charging Tests.

3. RESULTS AND DISCUSSION

3.1 Equilibrium Charge

In Figure 4, the amount of time that it takes for each powder to reach its maximum charge in a PTFE container is shown. The shaking time for each powder thereby varies depending on how long it takes each powder to reach its maximum charge. It can be seen that HPC and α-lactose monohydrate reached their peaks at 1 and 45 minutes, respectively. The resulting peak charge time for the 50:50 binary mixture is 20 minutes, which is between that of α-lactose monohydrate and HPC (as expected).

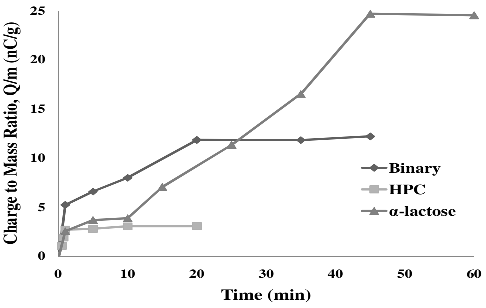


Figure 4. Charge to Mass Ratio at 20 Hz frequency as a function of shaking time for Three Powders against PTFE.

3.2 The Effect of Shaking Frequency

Using the results from Figure 4, the effect of frequency has been studied. Figure 5 show the results of the mechanical shaking tests of α-lactose monohydrate, HPC and a 50:50 binary mixture of the two powders in a PTFE container. The powders were vibrated at a constant frequency of 20 Hz for 45, 1 and 20 minutes, respectively. The vertical axis in Fig. 5 represents the charge-to-mass ratio (in nC/g), and the horizontal axis the shaking frequency. The data are shown as an average values from tests repeated a minimum of three times. The results show higher charge levels against the PTFE surface at a 20 Hz frequency for all three powders, as compared to those generated at 10 and 30 Hz. It can be seen that α-lactose monohydrate has the highest magnitude of charge at all three frequencies compared to the other two powders.

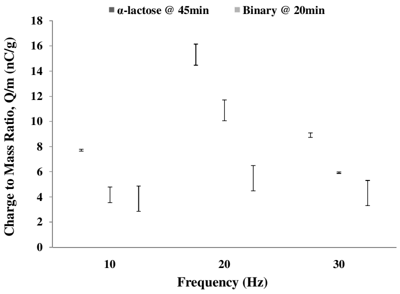


Figure 5. Charge to Mass Ratio at 10, 20 and 30 Hz Frequencies for Three Powders against a PTFE Surface.

3.3 The Effect of Particle-Wall Adhesion

Figure 6 shows the mean percentage of mass loss during tests at three different frequencies within a PTFE container. This was measured by tapping the container several times which dislodged the charged powder into an empty Faraday cup. Figure 6 shows that α-lactose monohydrate has the highest mass loss across the frequencies tested. HPC has the smallest mass loss at all three frequencies and a very low adhesion of particles to walls. The mass loss is thereby the result of powder adhering to the walls of the shaking containers which does not empty into the Faraday cup by gentle tapping on the outside of the container; neither could they be scraped into the Faraday cup as this would additionally charge the powder. The adhesion of particles to walls can have a significant effect on the generated charge. The current hypothesis is that particles which adhere to the wall surfaces may change the charging process from particle-wall contacts to particle-particle contacts which may result in the change of polarity of the overall powder sample.

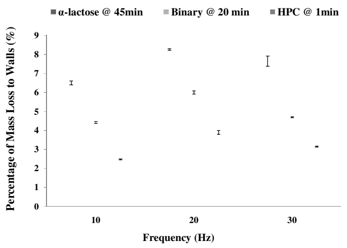


Figure 6. Percentage of Mass Loss During Tests against a PTFE surface at three different frequencies.

3.4 The Effect of Surface Material

During powder handling in the pharmaceutical industry, the powder makes contact with a variety of solid surfaces namely PTFE, stainless steel and glass. It is therefore important to investigate the polarity and level of the charge during tribo-electrification of powders and their surfaces. Figure 7 shows the results of α-lactose monohydrate tested for 2 minutes at 20 Hz against PTFE, glass and stainless steel surfaces. Figure 7 shows that α-lactose monohydrate is charged positively against PTFE surface, whilst it is charged negatively against glass and stainless steel containers.

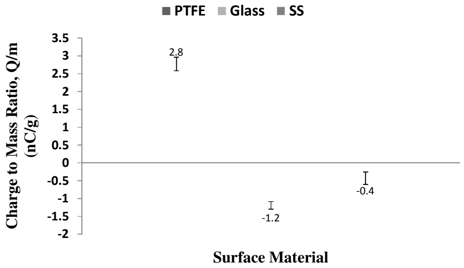


Figure 7. Triboelectrification of α-Lactose Monohydrate against a PTFE, Stainless Steel and Glass Surfaces.

Furthermore, the charging behaviour of a binary mixture is very complex, as our current state of understanding on the charge distribution of each component is simply non-existent. The charge on the blend measured here is obviously the net charge. It may be reasonable to consider in the first instance that the charge on the blend is additive, hence explaining the specific charge on the blend being lower than pure α-lactose monohydrate at all frequencies.

4. CONCLUSIONS

The triboelectrification propensities of α-Lactose monohydrate, HPC and the corresponding 50:50 binary mixture have been characterised by a mechanical shaking test method. The results show that α-Lactose monohydrate has the highest magnitude of charge against a PTFE container at all three frequencies tested.

All three powders tested charge positively against the PTFE container at all frequencies. All sample powders tested have highest powder adhesion and the net charge at the intermediate frequency of 20 Hz. The charge to mass ratio obtained from particles of α-Lactose monohydrate left on the inner wall of the PTFE shaking container carry a much higher magnitude of charge as compared to binary formulation and HPC.

The results also show that it took α-Lactose monohydrate, HPC and the corresponding 50:50 binary mixture approximately 45, 20 and 1 minute, respectively, to attain equilibrium charge at 20 Hz.

The charge generation of α-Lactose monohydrate against PTFE, stainless steel and glass as a result of the tribocharging process was investigated. The results show that α-Lactose monohydrate is charged positively against PTFE and negatively against stainless steel and glass. The magnitude of charge is highest against PTFE and lowest against stainless steel.

The charging processes for the systems investigated in this work are very complicated, as they include multiple particle-wall and inter-particle interactions and space charge effects hence they are difficult to analyse. The knowledge obtained from these multiple particulate systems is useful for comparative evaluations, but lacks generality. Nevertheless, it is reasonable to conclude that the binary formulation tested here becomes charged to a sufficiently high level to cause problems such as the segregation of the components and upset the formulation balance. In future work, a DEM simulation will be used to provide a more detailed and systematic study that is needed to elucidate the mechanisms involved in the process.

5. ACKNOWLEDEGEMENTS

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