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International Journal of Pharmaceutics 195 (2000) 7–11

international journal of pharmaceutics

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Communication

Effect of contamination of pharmaceutical equipment on powder triboelectrification☆

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Received 18 August 1999; received in revised form 6 September 1999; accepted 11 October 1999

Abstract

Triboelectrification of pharmaceutical powders may cause problems during processing and manufacture due to adhesion/cohesion effects. The aim of this work was to investigate the role of adhered particles and moisture as contact surface contaminants on the electrostatic charging of size fractionated lactose, following contact with a surface, i.e. stainless steel, typically used in pharmaceutical process and manufacturing operations. Replicated experimental runs without cleaning the contact surface showed a successive decrease in the net electronegative charge due to adhered lactose particles. Removal of these contaminating particles by different cleaning methods had a considerable effect on the charge after triboelectrification. The charge on the lactose samples was found to decrease when humidity in the cyclone apparatus was increased from 2 to 100% relative humidity. These results clearly demonstrate that moisture, particulate contamination and method of cleaning of processing equipment during pharmaceutical manufacturing operations may influence the electrostatic behaviour of powders. © 2000 Published by Elsevier Science B.V. All rights reserved.

Keywords: Triboelectrification; Electrostatic charge; Contamination; Lactose; Moisture

1. Introduction

Triboelectrification of pharmaceutical powder systems during processing operations may give rise to powder cohesion and particle adhesion to the surface of processing equipment. Interparticulate or particle/substrate collisions lead to charge accumulation which is influenced by particle size and shape (Carter et al., 1998), nature and work function of the contacting materials (Elsdon and Mitchell, 1976; Bailey, 1984), contact area and frequency (Lowell, 1990), contact surface roughness and contamination (Eilbeck et al., 1999) and atmospheric humidity (Nguyen and Nieh, 1989; Mackin et al., 1993). The aim of this work was to investigate the role of adhered particles and mois-

☆ Presented in part at UKaps Annual Conference, Manchester, UK, 28–30 June, 1999.

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PII: S0378-5173(99)00351-8

ture as contact surface contaminants on the electrostatic charging of size fractionated lactose after triboelectrification with stainless steel.

2. Materials and methods

Alpha lactose monohydrate (Lactochem) size fractions in the range 90–212 μm were prepared by a standardized sieving method using BS test sieves (Endecott), followed by air jet sieving (Alpine LS320) of each fraction for 10 min on a 45-μm sieve. Microscopic examination showed this to be an effective technique for removing unwanted fine particles.

Particle size and shape of the size fractions were determined using optical microscopy incorporating image analysis (VIDS V, AMS) providing mean x-Feret diameters, area/perimeter^2 (A/P^2) and length/breadth (L/B) ratio distributions.

The cyclone apparatus described previously (Carter et al., 1998) (Fig. 1) was used for triboelectrification of 1.0-g samples of powder against the stainless steel contact surface of the inner wall of the cyclone separator. The samples were pneumatically conveyed through the apparatus at a range of air velocities (6–11 m s^{-1}) with controlled relative humidity (rh). A polonium-210 radioactive charge neutralizer was used to establish a baseline charge on the powder before triboelectrification. The specific charge (nC g^{-1}) on the powder was determined by the Faraday well connected to an electrometer (Keithley 610C) and force compensation load cell (Precisa 400M) for real time measurement of charge and mass respectively, as the particles dropped from the cyclone.

The effect of particulate contamination of the contact surface on the charging of the lactose size fractions was determined at $<10\%$ rh and after each experimental run ($n = 5$ at each selected gas velocity) the cyclone was not cleaned and contact surface contamination occurred. Two cleaning methods for removal of particulate contamination from the inner cyclone were compared. Firstly, surface rinsing with water (40°C) then rinsing with acetone and allowing to dry under ambient

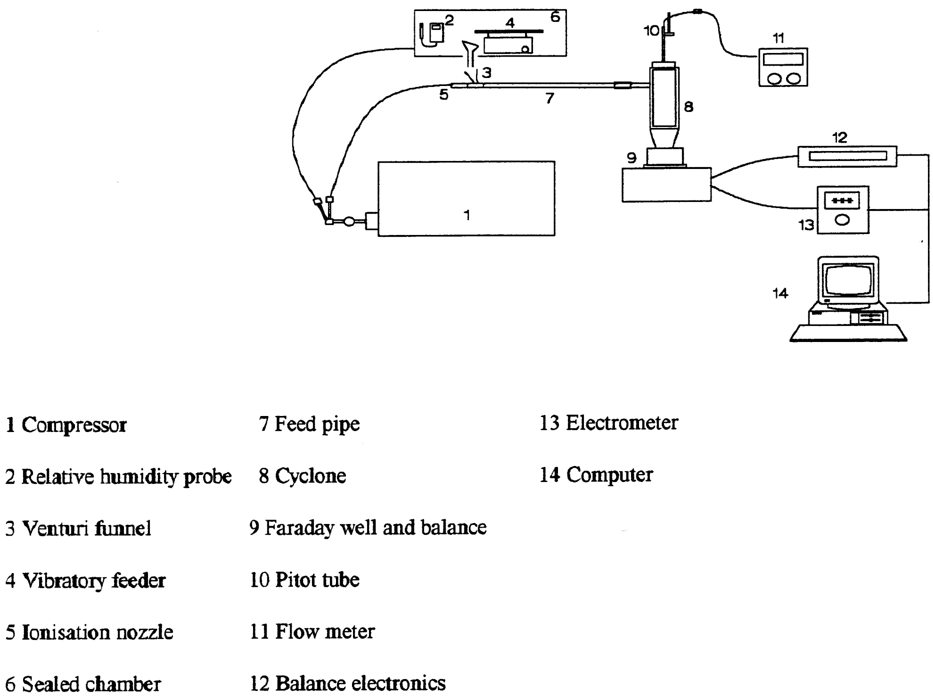

Fig. 1. Cyclone apparatus for charge measurement.

Table 1
Characterization of fractionated lactose[a]

Sieve fraction (μm)	Mean size (μm)	Mean A/P^2	Mean L/B
90–125	124 (23)	0.061 (11)	1.67 (21)
125–150	153 (18)	0.062 (10)	1.43 (20)
180–212	233 (17)	0.059 (11)	1.34 (17)

[a] Percent coefficient of variation in parentheses.

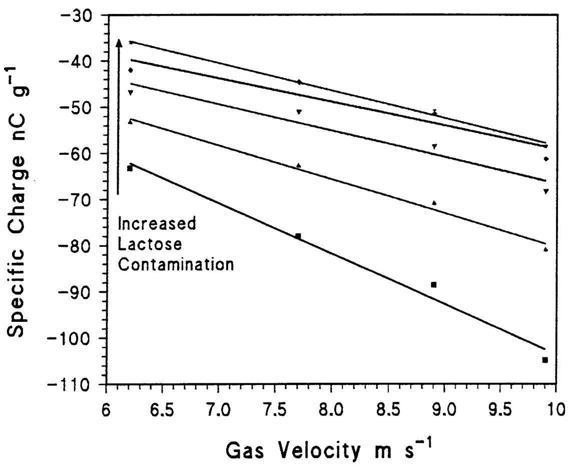

Fig. 2. The effect of contaminating lactose particles on charge of lactose 90–125 μm size fraction following triboelectrification at different gas velocities with a stainless steel surface.

conditions. Secondly, placing the cyclone in an ultrasonic bath (Bandelin Sonorex RK 1065) containing dilute (1 in 40) ammonia solution 35% w/v then rinsing with water (40°C) before drying under vacuum with dessicant (calcium sulphate) crystals. The effect of moisture contamination of the contact surface on the charging of the lactose size fractions was determined for each fraction ($n = 5$ at each selected gas velocity) at 2 and 100% rh. After each experimental run, contaminated lactose particles from the inner cyclone surface were removed using the ammonia solution method.

3. Results

Table 1 shows particle size and shape values for the lactose fractions. The values for L/B suggest that the smaller particles were more elongated than the larger particles and this may provide an explanation for the higher than expected mean particle sizes for the smaller sieve size fractions. The results in Fig. 2 have been selected from the data and show the average specific charge calculated from the final net charge and mass values, versus gas velocity, for the 90–125 μm fraction and shows the effect of replicated experimental runs without cleaning the contact surface. Fig. 3 shows average specific charge versus gas velocity, for the 125–150 μm fraction and the effect of contact surface cleaning conditions for the removal of adhered particles. Fig. 4 shows average specific charge versus gas velocity, for the 180–212 μm fraction and the effect of 100 and 2% rh conditions.

4. Discussion

The effect of particle contamination is shown clearly in Fig. 2 through replicated experimental runs carried out without cleaning the contact surface. Although this contamination was not quantified, qualitative evidence showed a clear increase in the extent of powder adhesion after

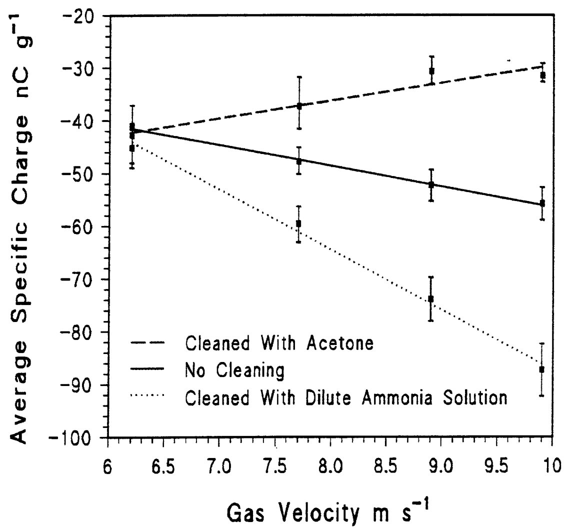

Fig. 3. The effect of contact surface cleaning conditions on charge of 125–150 μm size fraction following triboelectrification at different gas velocities with a stainless steel surface.

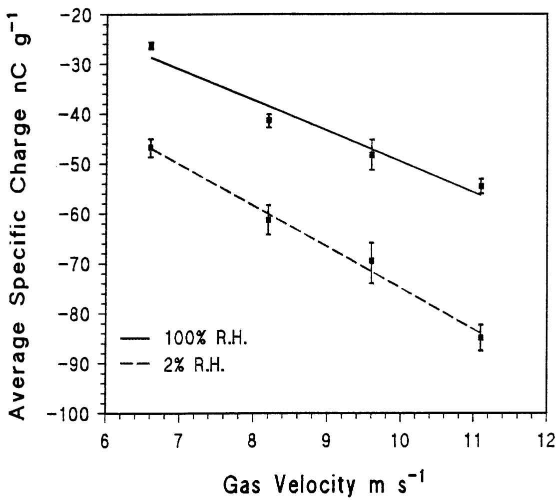

Fig. 4. The effect of relative humidity on charge of lactose 180–212 μm size fraction following triboelectrification at different gas velocities with a stainless steel surface.

each successive experimental run. Increased particulate contamination led to a successive decrease in charge and this effect was more pronounced for the smaller size fractions. For example the charge of the 90–125 μm size fraction changed from -62 to -35 and -105 to -55 nC g^{-1} for gas velocities of 6.2 and 10 m s^{-1}, respectively, after five runs. Whereas, in the case of the 180–212 μm size fraction, the change in charge was from -50 to -38 and -94 to -68 nC g^{-1} for 6.2 and 10 m s^{-1}, respectively. Carter et al. (1998) showed that smaller lactose size fractions acquired a lower net charge than larger size fractions within the range 45–125 μm. This was attributed to particle adhesion to the contact surface with increased particle–particle interactions and reduced particle-contact surface collisions. It was envisaged that these conditions lead to a complex bipolar system which affected the net charge and made it difficult to predict the effect of particle size. In this work with size fractions in the range 90–212 μm, there was a gradual increase in particle adhesion to the contact surface due to replicated runs without cleaning and therefore charge acquisition through particle-contact surface collisions was decreased due to contact surface contamination. This effect was less pronounced with the larger size fractions due to reduced adhesion. The results show that the intensity of triboelectrification increased with greater gas velocity and the magnitude of charge increased with decrease in particle size. Eilbeck et al. (1999) suggested that higher velocities may lead to increased particle deformation, higher collision frequencies and reduced particle-substrate separation time, however the parameters of the contact event, i.e. pressure, frequency and area of contact are difficult to quantify and relate to the charge accumulation/transfer process. Increase in net charge with decrease in particle size is thought to be due to the increased surface area available for charge transfer. These results show that processing conditions and particle properties affect the degree of surface contamination and hence the triboelectrification process and indicate the importance of investigating cleaning processes prior to triboelectrification experiments. Fig. 3 shows differences in charge arising from different preparation conditions of the contact surface prior to triboelectrification. The decrease in electronegative charge with increased gas velocity observed with the acetone treated surface suggests that a film of lactose and/or solvent was deposited to provide greater coverage than that from adhered particles. The surface cleaned with dilute ammonia solution in which lactose is freely soluble gave higher charge values than from the surface contaminated with adhered particles, whereas the acetone rinsed surface gave the lowest charge values.

Fig. 4 shows differences in the charge due to differences in humidity conditions with high rh giving lower electronegative values than low rh. These results show that moisture uptake of the powder, in addition to contamination of the contact surface are important considerations when undertaking triboelectrification measurements. Moisture may limit surface contact, alter the bulk properties of powders, change the conductivity of materials and enhance the neutralization of charges (Nguyen and Nieh, 1989). Equilibrium moisture values for alpha-lactose monohydrate are $< 0.5\%$ w/w up to $\sim 80–85\%$ rh above which there is an increase to $\sim 7\%$ w/w and Mackin et al. (1993) showed charge values for alpha-lactose monohydrate did not change with increase in rh up to 80%.

The results in Figs. 2–4 show and confirm the importance of controlled experimental conditions, e.g. surface contamination in triboelectrification experiments, and suggest moisture and particulate contamination of processing equipment during pharmaceutical manufacturing operations may influence the electrostatic behaviour of powders and hence processing conditions.

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