

# Investigation of particle electrification in mixers – effect of particle size, mixer type, rotation speed

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## Abstract

In the manufacturing of pharmaceutical product, mixing is one of the very important processes for preparing active pharmaceutical ingredient (API) with excipients to form a homogeneous mixture. Electrostatic charges generated and accumulated during the mixing process not only affect the product homogeneity, but also influence the processes handling these mixtures and eventually the drug delivery efficiency. Understanding particle electrification in the mixer is then very important for optimizing the formulation for dry powder inhalation drug delivery. In this work, particle electrification in the Turbula and horizontally oscillating mixers were investigated for adipic acid, microcrystalline cellulose (MCC), and glycine particles. MCC and glycine particles acquired positive electrostatic charges, while adipic acid particles attained negative charges in both mixers. Adipic acid (of sieved size larger than 500 $\mu$ m), MCC, and glycine particles were monotonically charged to saturated values, and had a negligible wall adhesion. On the contrary, the adipic acid particles containing fine particles firstly acquired charges up to a maximum value, and then the charges slowly reduced to a lower saturated value with increasing mixing time. For these adipic acid particles, particle wall adhesion was found to be significant and increased with increasing mixing time. Horizontally oscillating mixer was found to charge more efficient than the Turbula mixer, and the rotation speed was shown to have negligible influence on the saturated charge value taking the measurement uncertainty into account. The results obtained from the current work suggested that electrostatic force enhanced particle–wall adhesion, and the adhered particles can have a significant impact on particle electrification.

## Introduction

Electrostatic charges can be generated when two different materials are brought together and then separated<sup>1</sup>. Particles can be charged when they are processed in gaseous environments. Particle electrification is commonly a nuisance and may cause dust explosions<sup>2,3</sup>. On the other hand, particle electrostatic charges can also be exploited for example in powder flow measurement<sup>4,5</sup>, powder separation<sup>6</sup>, electrophotography in photocopiers and laser printers, and dry powder coating<sup>7</sup>. For the safe operation of particle handling systems and optimization of the performance of particulate systems, it is of great importance to understand the particle electrification behavior in particle processing systems.

Mixing of particles is important in many pharmaceutical operations. In many situations, a better mixing process could tremendously increase the quality of product. Electrostatic charges are generated and accumulated as a result of continuous collisions between particles and mixer container as well as between particles. The generated electrostatic charges affect binding between particles and the container wall as well as between particles, and hence influence the quality of final products. However, charge generation and accumulation during particle mixing is still not clear.

In the current work, particle electrification of adipic acid, microcrystalline cellulose (MCC), and glycine particles in mixers was investigated. The main purpose of the current work was to evaluate the influence of the mixer type, rotation speed, and particle size on the particle electrification.

### Materials and Methods

Table 1. presents the physical characteristics of particle materials used in the current work. Adipic acid (Lot No: S11343-124) and MCC (Batch #07021PA) purchased from Sigma-Aldrich were used as supplied. Glycine was re-crystallized from aqueous solution by a cooling method. Adipic acid sample was mechanically sieved into six different size fractions as follows: a) >500  $\mu\text{m}$ ; b) 250~500  $\mu\text{m}$ ; c) 180~250  $\mu\text{m}$ ; d) 150~180  $\mu\text{m}$ ; e) 125~150  $\mu\text{m}$ ; f) 90~125  $\mu\text{m}$ . The particle size distribution of these particles are shown in Table 2. MCC particles of particle size larger than 125  $\mu\text{m}$  were also obtained from the purchased samples by mechanically sieving out the fines.

**Table 1.** Physical characterization of particle materials

<b>Material</b>	<b>True density (kg/m<sup>3</sup>)</b>	<b>Tapping density (kg/m<sup>3</sup>)</b>	<b>d (0.1) (<math>\mu\text{m}</math>)</b>	<b>d (0.5) (<math>\mu\text{m}</math>)</b>	<b>d(0.9) (<math>\mu\text{m}</math>)</b>	<b>AR (-)</b>
<b>Adipic acid</b>	1330	810	35.9	289.9	535.8	0.71
<b>Glycine</b>	1570	880	267.1	567.0	929.2	-
<b>MCC</b>	1560	350	83.2	142.7	236.4	0.56

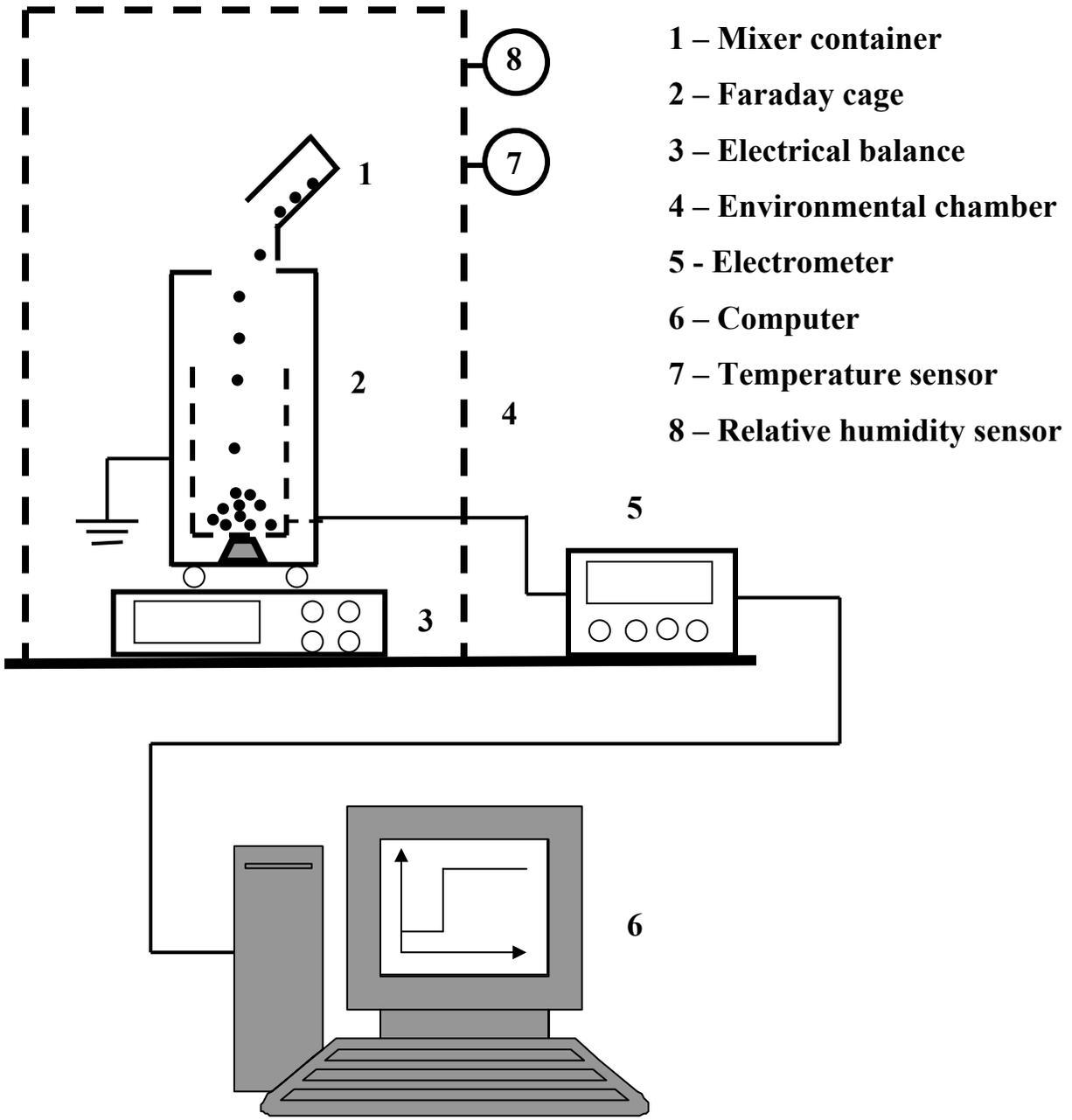
The mixer container was made of a cylindrical stainless steel tube. During an experiment, the bottom cap of the container was fixed, while the top cap was removable for powder loading/unloading. A knob made of electric insulating material was attached to the container to minimize the electrostatic disturbance during the transport of the container. Precautions were taken to clean the mixer container well before conducting any experiment. Prior to each measurement the inner wall of the stainless steel container was washed with deionized water, scrubbed with a metal brush, rinsed with pure ethanol, and then blow dried using compressed air. The container was then placed in the humidity chamber to equilibrate at the environment conditions desired for the next experiment.

**Table 2.** Particle size distribution analysis by Malvern particle sizer, sieve, and image analysis

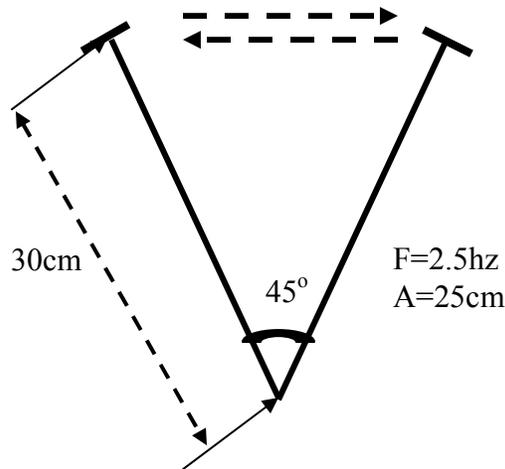
Size fraction ( $\mu\text{m}$ )	Volume size distribution by Malvern particle sizer			Image analysis	
	d (0.1) ( $\mu\text{m}$ )	d (0.5) ( $\mu\text{m}$ )	d (0.9) ( $\mu\text{m}$ )	d (ave) ( $\mu\text{m}$ )	AR (-)
a (>500)	305.6	604.5	1130.8	588.7+/-170.0	0.65+/-0.14
b (250-500)	218.1	399.9	672.5	392+/-74.6	0.62+/-0.14
c (180-250)	97.3	223.0	395.5	279.8+/-57.3	0.64+/-0.17
d (150-180)	49.2	139.0	266.3	216.7+/-38.7	0.70+/-0.15
e (125-150)	20.7	81.0	168.7	160.7+/-29.9	0.71+/-0.13
f (90-125)	13.8	57.1	129.4	90.3+/-28.1	0.71+/-0.15

All the experiments were carried out in the controlled environmental chamber with the temperature set at 25°C. A Faraday cage was used to measure electrostatic charges carried by powders through a charge induction method. The Faraday cage was placed within the environmental chamber, and connected to an electrometer (Keithley 6517, Keithley Instruments, USA) which was then connected to a computer through a GPIB interface. Fig. 1 presents the schematic diagram of experimental setup. The electrometer was switched on for at least two hours before conducting experiments to minimize any electrical drifting. Powder samples were laid out as thin layers on earthed metal plates to equilibrate inside the environmental chamber (Model 518, ETS, USA) at the temperature of 25°C and chosen relative humidity for at least 48 hours before experimentation. A measured quantity of powder was loaded into the container with a spatula made using insulating material. The container was sealed with the top cap and mounted on the T2F Turbula mixer. After mixing for the required duration, the mixer was stopped, the container was removed, and powder was poured into the Faraday cage to measure the particle charges. The weight of particles inside the Faraday cage was measured and the specific charge (nC/g) was then calculated. Particles adhering to the container wall were also estimated from the difference in weights of powder originally loaded and that received in the Faraday cage.

The experimental procedure for measuring electrostatic charges of adipic acid, MCC, and glycine particles in the T2F Turbula mixer was carried out as previously described and for various mixing durations. The runs were continued until the measured specific charge did not change with further increasing in mixing time. Experiments were repeated to explore the influence of mixer rotation speed and relative humidity on particle electrification.



**Fig.1 Schematic diagram of experimental setup for measuring electrostatic charges.**



**Fig.2 Schematic diagram of horizontally oscillating mixer**

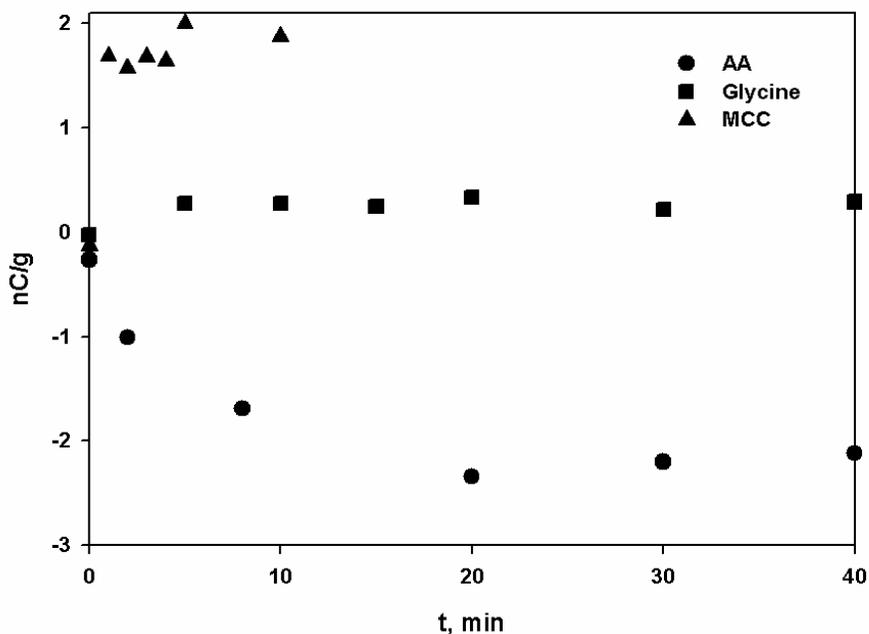
In addition to the Turbula mixer, particle electrification tests in a horizontally oscillating mixer for adipic acid, MCC, and glycine particles were also conducted. In contrast to the 3-D motion in the Turbula mixer, the horizontally oscillating mixer has a lever of 30 cm with a sample holder at the top end for attaching the mixer container, and the bottom end is fixed to an axis which rotates at the frequency of 2.5Hz and the rotation angle of 45°. Fig. 2. shows the schematic diagram of the mixer. The experimental procedure for the electrostatic charge measurement was the same as that for the Turbula mixer.

## Results

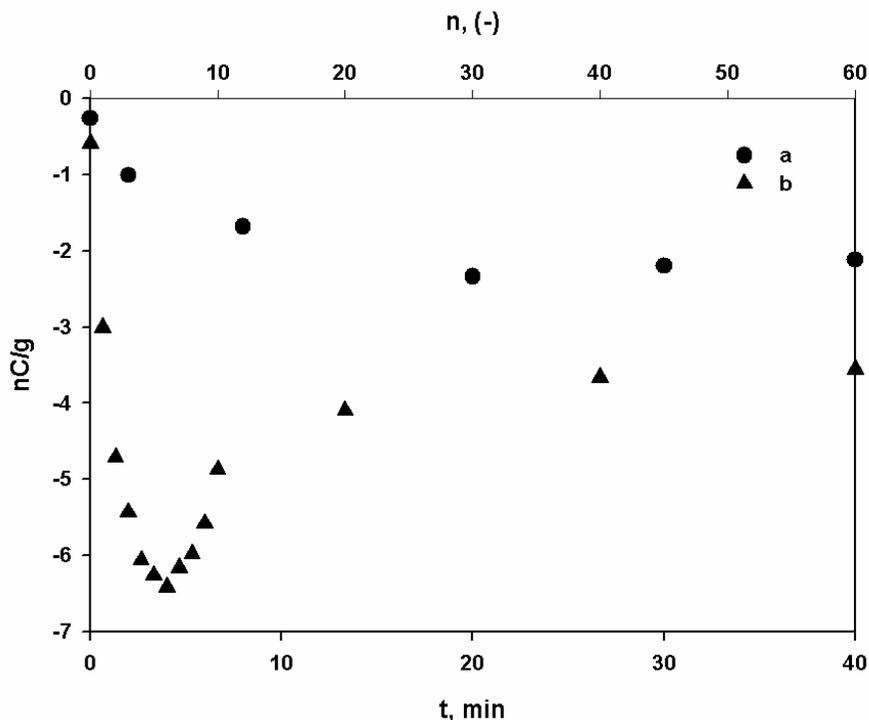
Fig-3 presents the electrostatic charges acquired in the stainless steel container in the tabular mixer operated at 49 rpm at the temperature of 25°C and relative humidity of 25% for MCC, adipic acid, and glycine materials. All materials obtained electrostatic charges during mixing as a result of particle and container wall collisions. Adipic acid particle acquired negative charges, while glycine and MCC were positively charged under the experimental conditions. Compared to MCC and glycine particles, adipic acid particles took longer time to reach a saturated charge level.

Charging behavior of MCC and glycine particles in the horizontally oscillating mixer was found to be similar to that in the turbula mixer. Fig. 4 shows the charging behavior of adipic acid particles in turbular mixer (a) and horizontally oscillating mixer (b). Dramatic difference in the charging curve was found for adipic acid particles, unlike MCC and glycine particles. Relative motion of particles and the mixer container wall may be different due to different mixing mechanisms for different mixers, and the difference in the relative motion may influence the charging behavior of particles in the mixers. Compared to the charging in turbular mixer in which adipic acid particles acquired electrostatic charges with time until reaching a saturated value after mixing about 30 mins, adipic acid particles acquired charges dramatically at first several mixing cycles, then decreased its charges upon further mixing, and finally reached a saturated value around 30 mixing cycles. Substantial adipic acid particles were

found to stick to the inner container wall during mixing operation in the horizontally oscillating mixer, and this particle wall adhesion was believed to be responsible for the observed peculiar charging behavior.



**Fig. 3** Electrostatic charges acquired with mixing time in a stainless steel container in the T2F Turbula mixer operated at the rotation speed of 49rpm, at the temperature of 25°C, and the relative humidity of 25% for adipic acid, glycine, and MCC.



**Fig. 4** Electrostatic charges acquired at with mixing time in a stainless steel container at the temperature of 25°C and the relative humidity of 25% for adipic acid particles (a) T2F Turbula mixer; (b) horizontally oscillating mixer.

Table 3 shows the surface charge density of adipic acid particles mixed in the stainless steel container in turbular mixer operated at various rotation speeds at temperature of 25°C and relative humidity of 25% for various particle size fractions. Adipic acid particles having larger size fractions had larger surface charge densities. This may be due to combined effect of larger particles having larger collision velocity and less particle wall adhesion. Rotation speed, however, seemed to have negligible effect on particle electrification behavior in the turbular mixer under the current experimental condition.

**Table 3. Saturated surface charge density of adipic acid particle in the tabular mixer operated at various rotation speed under 25°C and 25% RH.**

Mixer speed	Surface charge density nC/m <sup>2</sup>					
	500 μm	250 μm	180 μm	150 μm	125 μm	90 μm
49 rpm	-199.12	-121.76	-54.78	-24.14	-26.51	-33.84
72 rpm	-201.98	-132.52	-50.08	-23.83	-25.80	-19.28
101 rpm	-166.26	-94.43	-37.71	-19.63	-31.08	-22.48

## Conclusions

The electrification of adipic acid, MCC, and glycine particles in the stainless steel container in Turbula and horizontally oscillating mixers was measured using a Faraday cage. MCC and glycine particles acquired positive charges with time until reaching saturated charge levels in both turbula and horizontally oscillating mixers. Unsieved adipic acid particles were also found to charge to saturated charge level with increasing mixing time but with a slower charging speed and of negative polarity. However, very different charging behavior was found for these adipic acid particles in the horizontally oscillating mixer, and the difference could be due to the adhesion of adipic acid particle to the mixer container wall. Sieved adipic acid particles with larger size fraction was found to have larger surface charge density upon reaching saturated charge level after mixing in the stainless steel container in the turbula mixer. Negligible difference in the adipic acid particle saturated charge density was observed in the current experimental conditions for different rotation speeds. The results of the current work suggest particle wall adhesion have significant influence on the particle electrification.

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