

Article

Influence of Trough Material on Triboelectric Charging and Wall Friction of Microcrystalline Cellulose Powders During Shear Testing

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Abstract

Triboelectric charging can significantly affect the flow behavior of insulating powders on wall surfaces. Microcrystalline cellulose (MCC), a widely used pharmaceutical excipient, is particularly prone to charge accumulation during particle-to-wall contact. In this study, wall friction shear tests were used to examine how trough material affects triboelectric charging and wall friction of MCC under quasi-static flow conditions. Three trough materials were evaluated including stainless steel (metallic), polylactic acid (PLA), and polytetrafluoroethylene (PTFE) by using both standard and layer-by-layer preparation methods. The generated electrostatic charge was measured using a Faraday cup, and the corresponding wall yield loci over a PTFE wall coupon were determined from shear tests. Charge measured after shearing for the standard preparation method revealed that PTFE trough produced charge magnitudes ~16–21 times greater than stainless steel trough (64.5 nC vs. 3.1 nC for MCC 102; 59.7 nC vs. 3.8 nC for MCC 203), while PLA trough produced an intermediate and particularly powder-dependent response, including a polarity reversal for MCC 203 (−38.9 nC vs. +3.8 nC on stainless steel trough). This charging contributed to a direct increase in wall adhesion, with τ_{ad} increasing by 61.6% (MCC 102) and 41.8% (MCC 203) on PTFE relative to stainless steel. The layer-by-layer method amplified charging further for both conductive and insulating troughs. Similarly, adhesion trends were generally consistent across layering preparation methods, with PTFE trough again showing the largest increase relative to stainless steel (40.5% for MCC 102; 55.6% for MCC 203), while PLA trough showed a more variable response, including a slight decrease in adhesion to MCC 102 (−11.7%). PTFE and PLA troughs produced substantially higher charges than the metallic trough, resulting in higher shear stress across the wall yield loci of two MCC powder sizes. The main effect of insulating materials was a non-negligible increase in adhesion. Consequently, the experimental characterization of wall friction via shear testing should be performed with a trough material with an electrical conductivity similar to that of the investigated wall coupon to ensure accurate process replication.

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1. Introduction

Triboelectric charging, also known as tribocharging or triboelectrification, refers to the gain of positive or negative electrostatic charge on a body due to the transfer of electrons or ions during contact between solid surfaces. In industrial units processing powders or granular solids, electrostatic charge may arise when particles come into contact with each other or with the equipment walls, and the charge can be transferred through short- and long-range interactions within the powder bed [1]. Even a short contact time can cause the transfer of electrons or ions between the contacting surfaces, thereby producing oppositely charged regions [2,3]. Tribocharging can also influence the powder flow [4], along with process conditions such as humidity and temperature, which can affect interparticle forces [5,6]. Triboelectric charging plays an important role in powder handling across many industries, including polymer powder bed fusion additive manufacturing [7], powder spreading [8–10], industrial-scale hoppers [11], the food industry [12], and pharmaceutical applications [13]. In the pharmaceutical industry, micro-sized powders are widely used and must exhibit good flowability [14]. Tribocharging can strongly affect both manufacturing efficiency and operational safety during the handling of pharmaceutical powders [15,16], which are often organic materials with high electrical resistivity, limiting charge dissipation [14]. These phenomena can destabilize powder flow and cause problems during transport, feeding, and filling, leading to non-uniform drug content [13] and making the production of homogeneous pharmaceutical blends more difficult [17,18]. During powder handling, electrostatic charging can further induce inconsistencies during tableting, powder adhesion to equipment surfaces [19,20], arching in hoppers, and increased interparticle cohesion, which can promote agglomeration [21,22].

Shear testers are widely used for the characterization of powder flow properties required for the design and operations of powder handling systems and process units [23]. However, the laboratory results often differ from the industrial behavior because of changes in physicochemical interactions at the particle level [24,25]. Lefebvre et al. [26] reported that the rheology and flowability measurements of metal powders can also be affected by tribocharging, depending on the nature of the experimental vessels.

In shear testing of pharmaceutical powders, plastic failure is commonly described by the yield locus, assuming that the forces in the powder bed are mainly mechanical [27]. Humidity and temperature are known to affect yield locus measurements [5,24,28,29], whereas the influence of triboelectric charging generated during shearing is often neglected or treated as an experimental artefact. Several studies have explored the relationship between tribocharging and shear stress [22]. Supuk et al. [30] studied the tribocharging behavior of bulk powders under shear deformation and found that increasing shear led to higher tribocharging and current. Pingali et al. [15] investigated the effects of shear on electrostatics and powder flow in pharmaceutical powders. Their results showed that both the shear strain and shear rate influence the electrical and flow properties of powders, with the shear strain having the stronger effect. Leonard et al. [31] examined torque response during wall friction using an FT4 powder rheometer and observed high data variability for non-lubricated powder, later attributing this variability, particularly for Microcrystalline Cellulose, to electrostatic effects. Liu et al. [18] also reported changes in powder flowability after passage through a twin-screw feeder and suggested that tribocharging generated during feeding was responsible for this behavior.

Despite the growing recognition of tribocharging in powder systems, its effect on the flow behavior of pharmaceutical powders has not been systematically characterized and remains poorly understood. In particular, it is not yet clear how wall–particle contact during shearing may induce charge generation, and how the resulting electrostatic charge may in turn alter the measured frictional and adhesive properties of the powder. The wall

yield locus obtained from wall friction shear tests may be influenced not only by the inherent mechanical properties of the powder but also by triboelectric charging generated through powder–wall and powder–trough interactions during shearing, as well as by the prior charge history of the powder. In addition, the electrical properties of the trough material, whether insulating or conductive, can play a role in determining the extent to which charge is accumulated or dissipated, thereby affecting the measured flow response and its variability. Such dependency underscores the need for a study that isolates the contribution of tribocharging from the purely mechanical behavior of the powder during wall friction testing.

The present work addresses this gap by investigating the wall friction shear behavior of Microcrystalline Cellulose, a widely used pharmaceutical excipient, under controlled shearing conditions. The triboelectric charge acquired during the shearing step is measured for different trough materials and powder preparation methods. The corresponding wall yield loci are compared to assess whether tribocharging influences the measured flow properties and to what extent. The aim is to provide experimental evidence to distinguish electrostatic contributions from the intrinsic mechanical response of the powder, thereby enabling a more reliable interpretation of wall-friction data in pharmaceutical powder characterization.

2. Materials and Methods

2.1. Powder Material

In the present study, two samples of Microcrystalline Cellulose (MCC) powder (Celphere 102, Lot No. 13C2 and Celphere 203, Lot No. 24AC, Asahi Kasei Europe GmbH, Düsseldorf, Germany) were used. In this paper, they will be referred to as MCC 102 and MCC 203, respectively. Their particle morphology and shape were characterized using a field emission scanning electron microscope (FESEM, mod. LEO 1525, Carl Zeiss SMT AG, Oberkochen, Germany). Figure 1 shows the particle shape and morphology. The images reveal that MCC 203 exhibits predominantly oval and spherical shapes, whereas MCC 102, although generally spherical, displays a lower degree of sphericity. The particle size distribution (PSD) was measured by laser diffraction in air using a Mastersizer 2000 instrument (Malvern Instruments Ltd, Malvern, UK). The analysis was based on the Mie theory. The tapped bulk density of the MCC powders was evaluated using a GeoPyc 1365 envelope density analyzer (Micromeritics, Norcross, GA, USA). The moisture content of powders was measured using a KERN DAB 100-3 moisture analyzer (KERN & SOHN GmbH, Balingen, Germany) at 120 °C with a 5 g sample of each powder. All measurements were performed in triplicate, and the averages are provided in Table 1. The PSD and bulk density results for the MCC materials are presented in Table 1. FTIR spectra were obtained using the KBr pellet transmission method (Bruker Vertex 70, DTGS detector, KBr beam splitter, resolution 2.0 cm⁻¹), in which the powder sample is homogeneously dispersed in a KBr matrix. The spectra were recorded from 4000 cm⁻¹ to 400 cm⁻¹ as shown in Figure 2. The FTIR spectra of MCC 102 and MCC 203 have almost the same peak position in the entire spectrum (4000–400 cm⁻¹), indicating that both powders have the same basic chemical structure. The chemical fingerprint remains the same; only the relative intensities of peaks in some regions change due to the particle size difference. The broad absorption at 3437 cm⁻¹ (in the region 3700–2995 cm⁻¹) is assigned to O–H stretching of cellulose hydroxyl groups [32]. The band position and shape are nearly identical for both grades, suggesting similar bulk hydrogen bonding. A substantial difference between the two grades was observed in the O–H bending band (~1643 cm⁻¹), attributed to adsorbed moisture, where MCC 102 showed a higher band intensity than MCC 203 [32,33]. The peak of O–H bending band is much larger in MCC 102, which indicates better moisture retention.

Table 1. MCC powders' physical properties: ρ_B is the bulk density of the powder; d_{10} , d_{50} , and d_{90} are the particle diameters corresponding to the 10th, 50th and 90th percentile of the cumulative particle size, respectively; $d_{3,2}$ is the Sauter mean diameter; $d_{4,3}$ is the volume mean diameter.

Name	Compound	Molecular Formula	PSD (μm)					ρ_B (kg/m^3)	Humidity (%)
			d_{10}	d_{50}	d_{90}	$d_{3,2}$	$d_{4,3}$		
MCC 102	Microcrystalline Cellulose	$(\text{C}_6\text{H}_{10}\text{O}_5)_n$	127	175	241	170	180	889	$5.54 \pm 0.01\%$
MCC 203	Microcrystalline Cellulose	$(\text{C}_6\text{H}_{10}\text{O}_5)_n$	193	263	358	256	270	899	$5.34 \pm 0.04\%$

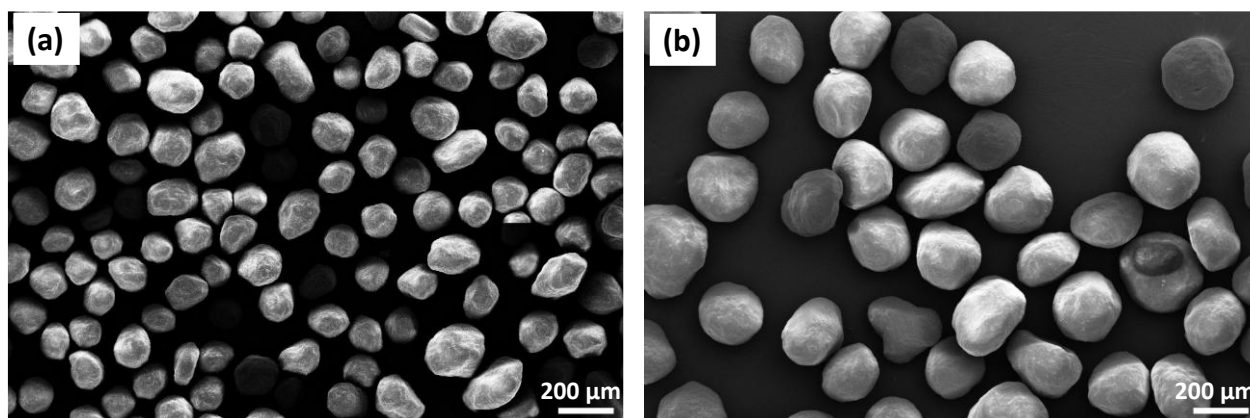


Figure 1. Scanning electron microscope (SEM) images (a) MCC 102 (b) MCC 203.

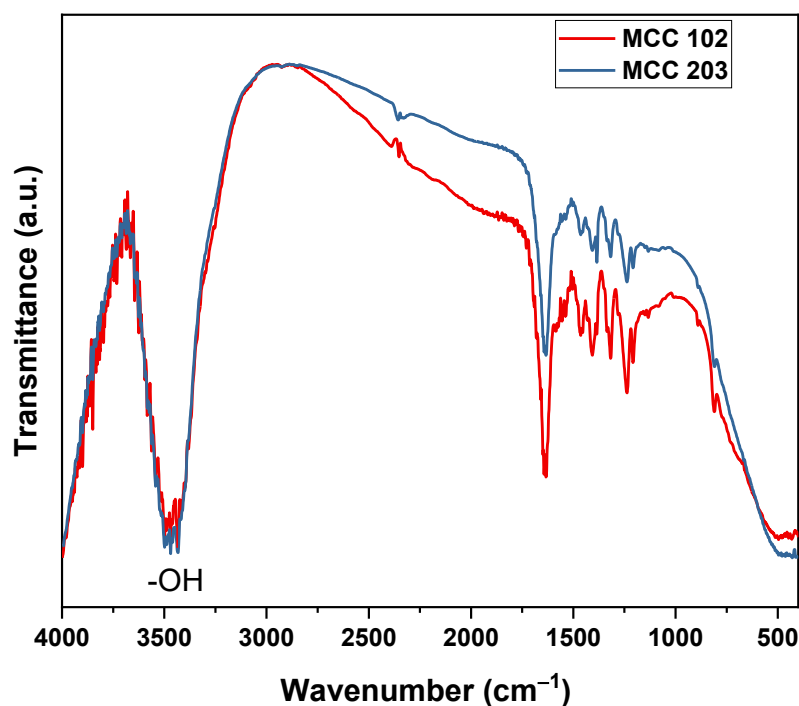


Figure 2. FTIR spectra of MCC 102 and MCC 203.

2.2. Experimental Setup and Methodology

The experimental methodology used in this work was designed to measure the wall friction properties of MCC powders and the electrostatic charge generated during the wall friction test. To evaluate the effect of the container materials that MCC powders may encounter in real applications, wall friction tests were performed using a shear tester with modified shear cell trough materials. All powder handling and shear testing were carried out in ambient conditions with a temperature of 20–24 °C and relative humidity of 55–65%. Full details of the measurement systems, apparatus, and relevant parameters are

provided in Section 2.2.1. particularly in the subsections from “Powder Shear Tester” to “Charge Measurement and Neutralization System”. The experimental procedure is described in detail in Section 2.3.

2.2.1. Experimental Apparatus

Powder Shear Tester

An Anton Paar Shear Cell with an annular cell trough mounted on a Modular Compact Rheometer (Model MCR–302, Anton Paar GmbH, Graz, Austria) was used for the shearing, as shown in Figure 3a,b. The annular cell trough had an internal diameter of 19 mm, an external diameter of 45 mm, and a total internal height of 15 mm. For this study, a dedicated wall friction procedure was developed to quantify the interaction between the powder and the various cell-trough materials. Each sample had undergone a series of shearing phases with progressively increasing normal stresses (σ) of 0.4, 0.8, 1.2, 1.6, and 2 kPa. The metallic lid equipped with a PTFE wall coupon was used to measure the wall friction. A PTFE wall coupon was used in all tests to retain the generated charge and provide a consistent baseline. Conductive coupons would dissipate the charge and prevent reliable accumulation measurements. The torque needed to move the wall coupon over the powder surface was measured for a specific normal stress. During the shearing stage, the measured shear stress reached a steady-state for each normal load applied. The mean steady-state shear stress (τ) obtained from the observed torque was then plotted against the normal stress for each pre-shearing phase to create the wall yield locus. This method allows a direct evaluation of powder–wall interactions under precisely controlled mechanical and electrostatic conditions.

A detailed stage-by-stage analysis of the charge evolution, demonstrating a cumulative but limited increase in the powder charge at each shearing step, is provided in Appendix A. The charge accumulation was found to have a minimal impact across the different shear steps. Therefore, the simplified procedure was retained, as it perfectly aligns with the primary focus of evaluating the broader effects of tribocharging on shear testing under various operating conditions.

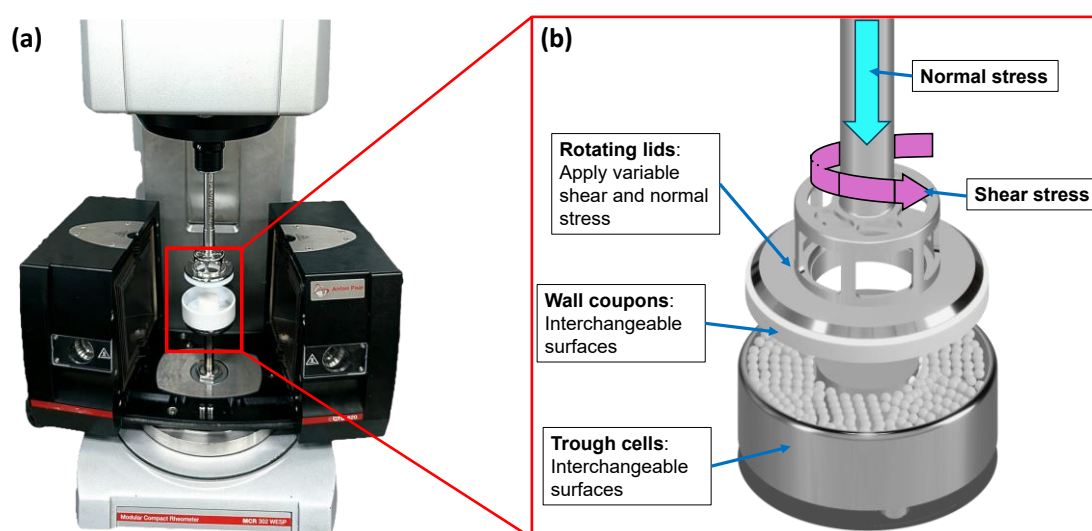


Figure 3. (a) Anton Paar Model MCR–302 rheometer and (b) interchangeable shear cell troughs.

Shear Cell Trough Materials

Three types of shear cell troughs were used in this study:

- i. A standard stainless-steel cell trough, representing the “grounded baseline” (no charge accumulation).

- ii. A custom-made 3D-printed polylactic acid (PLA) cell trough, representing a “moderate insulator” from a common 3D-printing material.
- iii. A custom-machined polytetrafluoroethylene (PTFE) cell trough fabricated using CNC machining, representing an “extreme insulator” with a very high electron affinity.

The use of these three diverse trough materials was intended to systematically assess how material properties affect electrostatic phenomena during shear testing. It has to be recognized that tribocharging phenomena may be affected by the different surface qualities necessarily deriving from the different production processes of the parts. Separating these effects, however, goes beyond the purpose of this paper.

The triboelectric series provides a consistent description of the polarity and strength of the electrostatic charge generated for various materials [34,35]. It lists materials according to their tendency to become positively charged (electron donors) or negatively charged (electron acceptors). In the empirical arrangement of the triboelectric series, materials at the top of the series tend to acquire a positive charge when rubbed against materials lower on the list [36,37]. Similarly, materials lower on the list will acquire a negative charge when rubbed against materials higher on the list [37]. The tribocharging behavior of MCC, the material characteristics and triboelectrification behavior of the powders and trough materials, with their relative positions in the triboelectric series determined by their specific work functions. The electrical properties of the trough material are listed in Table 2.

Table 2. Summary of material characteristics based on material work functions.

Material	Material Type	Work Function (eV)	Dielectric Constant (Relative Permittivity)	Electrical Resistivity (Ω m)
Polylactic Acid (PLA)	Insulating	3.36 [38]	1.7–2.8 [39]	$10^{12} - 10^{19}$ [40,41]
Stainless Steel	Conductive	4.14 [42,43]	-	10^{-7} [44]
Microcrystalline Cellulose (MCC) Powder	Insulating	5.11 [16]	3.11–4.59 [45–47]	$10^8 - 10^{13}$ [48,49]
Polytetrafluoroethylene (PTFE)	Highly Insulating	5.8–6.71 [50–52]	2.0–2.1 [53,54]	$> 10^{16}$ [55]

Charge Measurement and Neutralization System

A Faraday cup (Monroe 284/22B, Advanced Energy Industries, Inc., Denver, CO, USA) and a nanoCoulomb meter (Monroe 284-3, Advanced Energy Industries, Inc., Denver, CO, USA; optional ranges: 2000, 200, and 20 nC) were used to measure the electrostatic charge of the powder sample. The charge was neutralized with an ionizing air gun (Model-KG-5, Suzhou KESD Technology Co., Ltd., Wuzhong District, Suzhou, Jiangsu, China) before and after each experiment. After each test, all components of the experimental setup, including the shear cell trough, lid, trough preparation stand, and Faraday cup, were carefully cleaned with compressed air. The electrostatic charge was then neutralized, and the components were left without further contact for 10 min to allow any residual charge to dissipate. To minimize unintentional charging during powder handling and pouring, a wooden spoon was used. Wooden tongs were also used to handle the shear cell trough before and after each experiment.

2.3. Experimental Procedures

For this study, two methods to prepare the shear cell troughs were used, as shown in Figures 4 and 5.

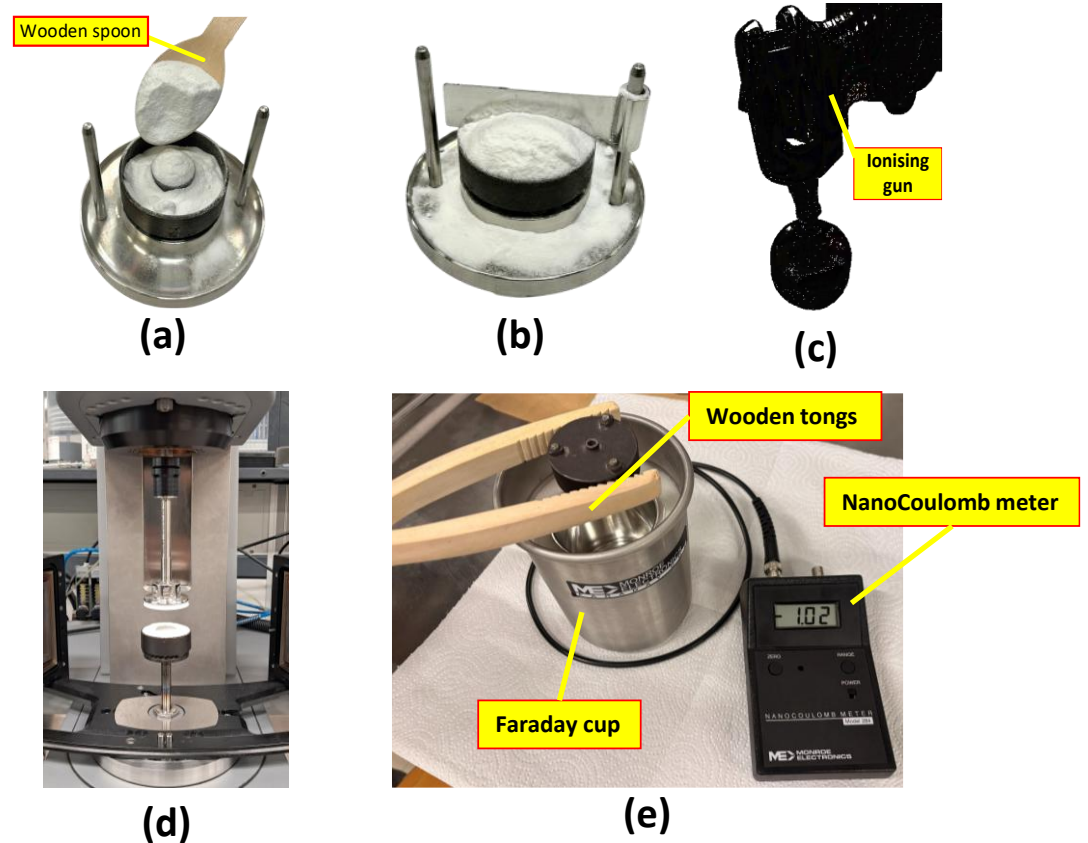


Figure 4. Standard method for preparing shear cell troughs: (a) powder pouring using a wooden spoon, (b) levelling of surface, (c) electrostatic charge neutralization, (d) wall friction shearing test, (e) charge measurement.

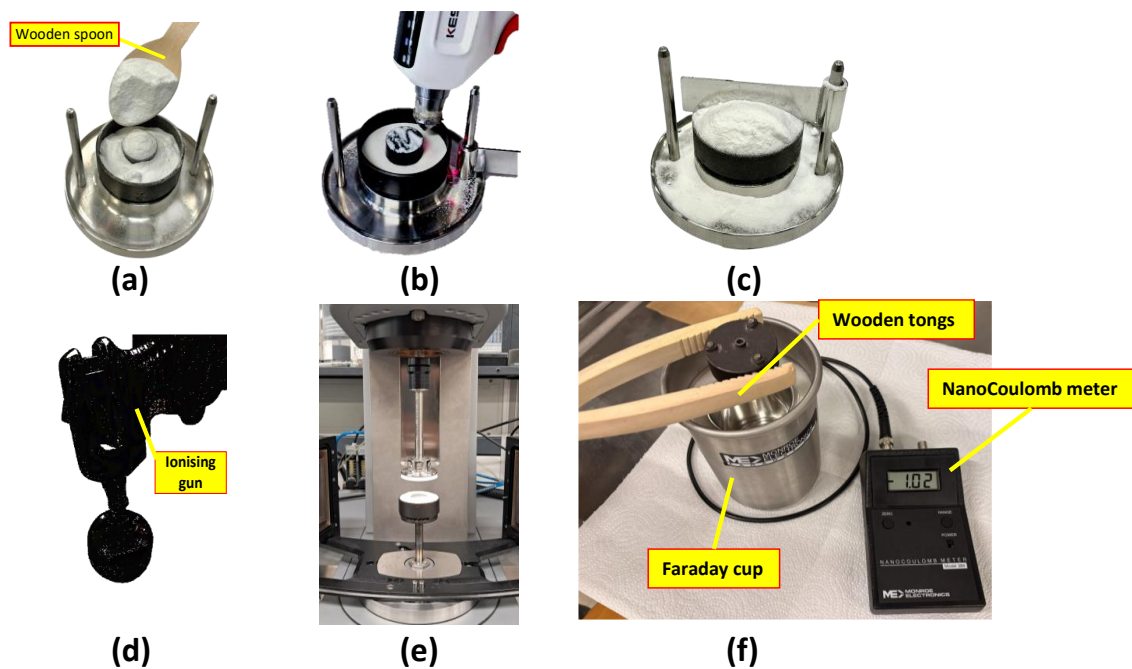


Figure 5. Layering method for the preparation of the shear cell troughs: (a) powder pouring using a wooden spoon, (b) electrostatic charge neutralization of layers, (c) levelling of surface, (d) electrostatic charge neutralization, (e) wall friction shearing test, (f) charge measurement.

2.3.1. Standard Method for Preparing Shear Cell Troughs:

The standard method generally involves preparing a sample as a single uniform unit.

Step 1. The powder is filled into the shear cell trough using a wooden spoon all at once and compacted on a metal workbench to form a uniform sample body (Figure 4a).

Step 2. After filling, the powder is conditioned using a rotating metallic blade. This step levels and redistributes the powder, improving the uniformity of the sample surface, as shown in Figure 4b.

Step 3. Before shear testing, the electrostatic charge generated during the conditioning step is neutralized using an ionizing gun, as shown in Figure 4c. This charge results from friction between the powder particles and the metallic blade, as well as between the blade and the trough wall. The effectiveness of this neutralization procedure has been demonstrated in a previous study [56].

Step 4. The cell is placed onto the rheometer for shear testing, as shown in Figure 4d.

Step 5. The cell is then transferred to the Faraday cup to measure the accumulated charge during shearing process, as shown in Figure 4e. (Figure 4e).

2.3.2. Layering Method for the Preparation of the Shear Cell Troughs

The shear cell trough is prepared using a layer-by-layer incremental-filling procedure, in which thin layers of powder are applied sequentially. To remove the triboelectric charges produced during handling and compaction, electrostatic pre-conditioning involving charge neutralization is also carried out during the layering process. Four layers were chosen as a practical operational limit, not an optimum for complete charge neutralization. Given the shallow trough depth (15 mm), further subdivision (more than four layers) would compromise mechanical feasibility of surface levelling and risk bulk density heterogeneity in the powder bed. The layering procedure was therefore carried out as follows.

Step 1. The powder is added to the shear cell trough in four successive layers using a wooden spoon, rather than in a single filling step. After each layer is compacted, it is neutralized for 1 min using an ionizing gun before the next layer is added, as shown in Figure 5a,b. This procedure reduces charge accumulation and limits charge transfer between the layers. After the fourth layer is added and the required filling height is reached, the procedure follows Steps 2 to 5 of the standard method. The powder is conditioned and levelled, the remaining electrostatic charge is neutralized, the cell is loaded onto the rheometer for shear testing, and it is finally transferred to the Faraday cup for charge measurement. These stages are shown in Figure 5c–f.

3. Results

3.1. Measurement of the Initial Charge of the Powder

The powders used in the current study tended to exhibit a certain residual electrostatic charge before the experiments. The initial charge on the powder is most likely due to frictional interactions with the storage container's surfaces. Additional charge generation could happen while the shear cell trough is being prepared before the shearing process. Therefore, before performing shear experiments, the powder's initial electrostatic charge must be measured. After preparing the cell trough according to both methods described above in Section 2.3, the cell trough was immediately transferred to a Faraday cup using wooden tongs. The Faraday cup was connected to a nanoCoulomb meter to measure the charges. The charge measured on the trough immediately after the powder preparation phase served as a baseline control to separate the charge contributions of the individual components. This initial measurement represents the initial charge generated by the powder interacting with the trough wall. The results are shown in Figure 6a,b for MCC 102 and MCC 203, respectively. From Figure 6a, MCC 102, the results show that the effect

of preparation on the initial charge in the trough and powder together is negligible. The charge generated on the metallic and PLA troughs is positive, while the charge on the PTFE trough is negative and of higher magnitude. Figure 6b shows the initial generated charge for MCC 203. For both preparation methods, the electrostatic charge measured for all three trough materials was nearly identical. This result suggests that all troughs had similar initial triboelectric charge states prior to wall friction shear testing. However, there are noticeable variations in the magnitude of the detected charge across different cell troughs. In addition, the charge polarity on all troughs is negative for MCC 203 in comparison to MCC 102.

The metallic trough has the lowest charge, because it is conductive, which makes it easier for electrostatic charges to disperse. In contrast, the PTFE cell trough acquires the highest charge, followed by the PLA cell trough. This phenomenon results from the insulating properties of PTFE and PLA, which promote the accumulation and retention of electrostatic charges during cell preparation. These findings indicate that the electrical conductivity of the trough material substantially affects the charge accumulation during sample preparation, which may, in turn, impact the interactions between the powder and the wall during shear testing.

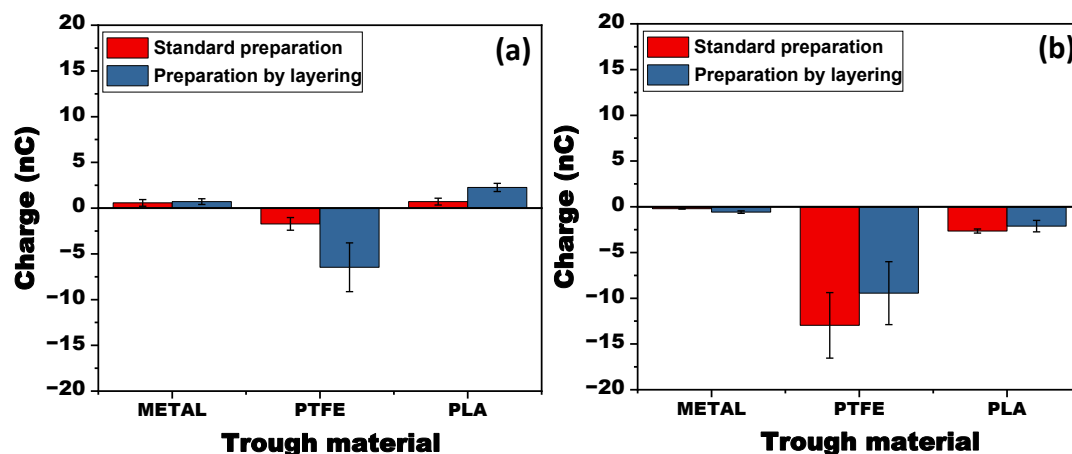


Figure 6. Electrostatic charge generated on the cell trough during cell preparation: (a) MCC 102 and (b) MCC 203.

3.2. Wall Friction Yield Locus for the Standard Preparation Method

The wall friction yield loci obtained using the standard preparation technique for the three shear cell trough materials, namely stainless steel, PTFE, and PLA, are shown in Figure 7a,b for MCC 102 and MCC 203, respectively. The measured electrostatic charge values after shearing are shown in Figure 7c,d for MCC 102 and MCC 203, respectively. The plots show that the relationship between the applied normal stress and the resulting shear stress can be approximated by the affine function for the wall yield locus:

$$\tau = \mu_w \sigma + \tau_{ad} \quad (1)$$

where τ is the shear stress, σ is the applied normal stress, μ_w is the wall friction coefficient corresponding to the slope of the fitted line, and τ_{ad} is the intercept, which represents the adhesion.

Figure 7a reveals some effect of the trough material on the wall yield locus. In particular, for MCC 102, the measured shear stress value is highest for the PTFE trough. It can be observed that the PLA and metallic trough show an almost similar wall yield locus. Consistently, the wall friction coefficient and adhesion are highest for the PTFE trough, while the values for the PLA and metallic trough are similar, as reported in Table 3. From Figure 7b, it can be observed that, for MCC 203, the wall yield loci obtained with the PTFE,

PLA and metallic troughs are substantially overlapped. In this case, the results reported in Table 3 indicate that the adhesion for the PTFE and PLA troughs is slightly higher than for the metallic trough, while the material effect on the wall friction coefficient is less significant. It can be observed for MCC 102 that the insulating nature of the trough material tends to make the measured wall shear stress increase as a combined effect on the wall friction coefficient and the adhesion.

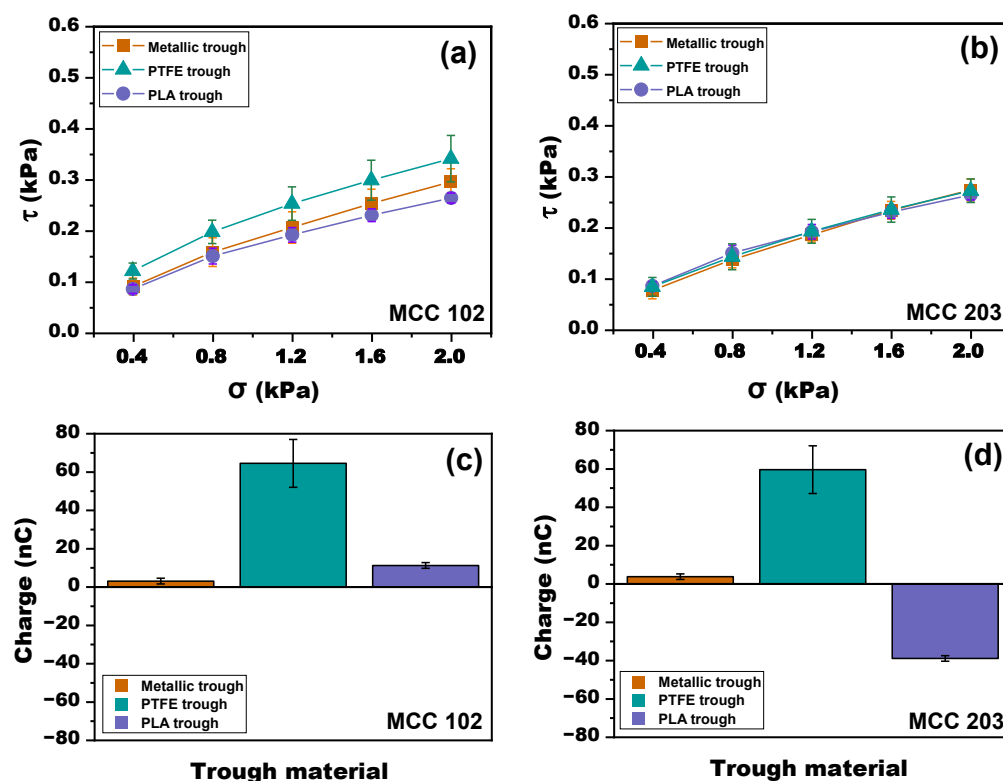


Figure 7. Wall friction yield locus: (a) MCC 102, (b) MCC 203, electrostatic charge measured after shearing (c) MCC 102 and (d) MCC 203 for different cell troughs for standard preparation.

Table 3. Wall friction properties for the standard preparation method MCC 102 and MCC 203.

Trough Material	Powder	Wall Friction Coefficient, μ_w (-)	Adhesion, τ_{ad} (kPa)	Charge on Powder After Shearing (nC)
Metallic	MCC 102	0.1262 ± 0.008	0.0505 ± 0.002	3.11 ± 1.46
	MCC 203	0.1222 ± 0.006	0.0361 ± 0.013	3.82 ± 3.83
PLA	MCC 102	0.1091 ± 0.001	0.0546 ± 0.001	11.26 ± 1.49
	MCC 203	0.1168 ± 0.004	0.0464 ± 0.017	-38.88 ± 2.23
PTFE	MCC 102	0.1351 ± 0.081	0.0816 ± 0.008	64.53 ± 12.47
	MCC 203	0.1083 ± 0.001	0.0512 ± 0.017	59.65 ± 17.69

The values in this table are the average (of at least three measurements) $\pm$ standard deviation.

Figure 7c,d reveal that the highest electrostatic charge generated during shearing is for the PTFE trough for both MCC powders, a result attributable to PTFE's strongly insulating nature. The PLA trough exhibits medium charge generation, whereas the metallic trough generates the lowest charge due to its high electrical conductivity. Notably, however, the charge induced by the PLA trough exhibits opposite polarities between the two powder grades. Comprehensive charge data are summarized in Table 3.

3.3. Wall Friction Yield Locus for the Layering Preparation Method

Figure 8a,b illustrate the wall friction yield loci obtained using the layer-by-layer preparation technique for three trough materials. Figure 8a,b present the results for MCC 102 and MCC 203, respectively. Figure 8c,d report the electrostatic charge generated within each trough cell following the shearing process. For all three trough materials and both powders, Equation (1) was used to fit the relationship between the measured shear stress and the applied normal stress. Table 4 presents the wall yield loci, friction coefficients, adhesion values, and post-shearing powder charges obtained using the layer-by-layer preparation method.

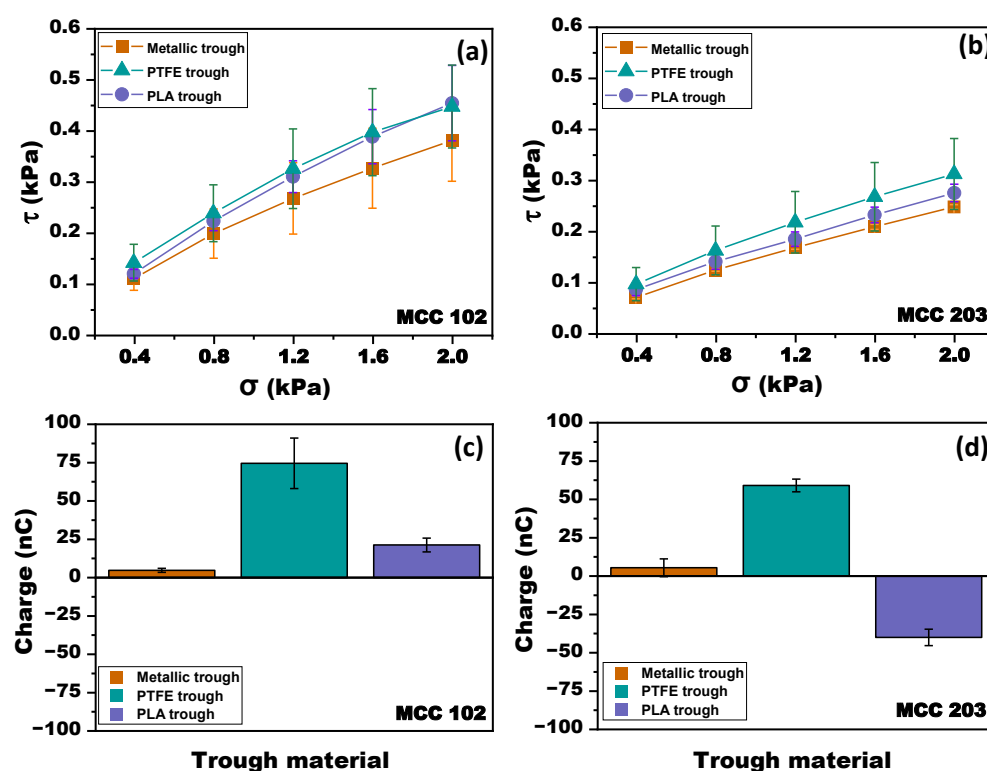


Figure 8. Wall friction yield locus: (a) MCC 102, (b) MCC 203, electrostatic charge measured after shearing (c) MCC 102 and (d) MCC 203 for different cell troughs for preparation by layering (Error bars for MCC 203 on the metallic trough are within the data symbols).

As observed from Figure 8a, the wall yield locus for MCC 102 obtained with the three trough materials appears quite similar, also considering the error bars. Similarly, for MCC 203, the wall yield loci for the PTFE and PLA troughs are shifted slightly upward compared to that for the metallic trough. For all the data points, the error bars are larger than those corresponding to the standard preparation of the sample. This would suggest a lower repeatability for the sample prepared by layering. As reported in Figure 8c,d, the electrostatic charge generated on the powders during the shearing process is comparable for both materials. The PTFE trough exhibits the highest charge generation for both materials, with MCC 102 producing a bit higher charge magnitude than MCC 203. The PLA trough induces opposite charge polarities between the two powders. The details of the measured charge for all three troughs in the layering method are summarized in Table 4.

Table 4. Wall friction properties for the layering preparation method MCC 102 and MCC 203.

Trough Material	Powder	Wall Friction Coefficient, μ_w (-)	Adhesion, τ_{ad} (kPa)	Charge on Powder After Shearing (nC)
Metallic	MCC 102	0.1672 ± 0.047	0.0575 ± 0.025	4.77 ± 1.31
	MCC 203	0.1097 ± 0.001	0.0333 ± 0.001	5.43 ± 5.79
PLA	MCC 102	0.2081 ± 0.052	0.0508 ± 0.022	21.3 ± 4.51
	MCC 203	0.1171 ± 0.004	0.0443 ± 0.011	-40 ± 5.37
PTFE	MCC 102	0.1922 ± 0.041	0.0808 ± 0.044	74.57 ± 16.47
	MCC 203	0.1339 ± 0.027	0.0518 ± 0.03	59.08 ± 4.11

The values in this table are the average (of at least three measurements) $\pm$ standard deviation.

4. Discussion

The effect of the vessel or trough material on particle flow measurements has also been reported by other researchers. Lefebvre et al. [26] observed tribocharging during repeated tests and found that the basic flowability energy (BFE) of titanium powder changed with the rheometer vessel material, including glass, stainless steel and aluminum. However, they concluded that it was difficult to correlate the powder rheology with the charge distribution, and they could not isolate the effect of vessel conductivity. Leonard et al. [31] also observed unexpectedly high variability for non-lubricated MCC, which may have been caused by electrostatic effects. Unlike previous studies that considered only a narrow conductivity range, the present work systematically examines a broad range of materials, from conductive stainless steel to highly insulating PTFE and PLA. It also uses shear testing rather than bulk flow energy indices.

While the relationship was not always perfectly clear in every instance, the overall wall friction tests reveal a connection between the cell trough material and the powder-to-wall frictional and adhesive properties. The experimental data show that the electrical conductivity of the wall actively drives the charge accumulation during the shearing process. Shearing MCC against the PTFE trough generates the highest triboelectric charge. PTFE is highly insulating, which prevents the generated charge during shearing from dissipating. In contrast, the metallic trough allows the charge to dissipate rapidly due to its conductive nature. PLA holds slightly more charge than the metallic trough but sometimes less than PTFE. This confirms that the charge storage capacity of the equipment material directly influences the final electrostatic state of the powder bed.

This accumulated charge, however, does not always show a direct and measurable impact on the macroscopic wall friction parameters, but it varies from one trough material to another. On the whole, the PTFE test resulted in the highest powder adhesion and wall friction values. The physical mechanism behind this behavior might be easily explained. The high electrostatic charge creates a strong Coulombic attraction between the MCC particles and the PTFE wall coupon. This attractive force acts as an additional normal load at the contact points. When the particles are pulled tighter against the wall, the system naturally requires more shear force to initiate movement. This extra resistance explains why the wall yield locus shifts upwards, due to higher shear stress, for the insulated PTFE setup. The metallic troughs provide a solid baseline for comparison in this study. They resulted in the lowest wall friction data and showed much lower charge accumulation overall. Because the electrostatic forces are very weak in these systems, the measured friction represents pure mechanical interaction. The wall friction coefficient values for the PLA trough fell within an intermediate range, closely approaching the values of the PTFE trough. This mechanical behavior corresponds directly with the measured electrostatic charge. Furthermore, an apparent polarity reverse was seen when the powders were sheared against the PLA trough, with MCC 102 charging positively and MCC 203

negatively. This polarity reversal between MCC 102 and MCC 203 at the PLA trough suggests a change in the effective work function of the cellulose particles depending on the particle size and/or moisture content. It is well known that the effective work function of a material decreases with increasing particle size [57,58]. Furthermore, changes in relative humidity and the resulting moisture adsorption cause changes in the surface potential difference, further reducing the work function [59]. The FTIR data strongly supported this finding. The O–H bending band at $\sim 1640\text{ cm}^{-1}$, associated with adsorbed water, was always higher for MCC 102 than for MCC 203. This higher moisture content may lower the effective work function of MCC 102 relative to MCC 203, providing a plausible explanation for the observed polarity reversal between the two grades on the PLA trough. A tentative summary of the relation between the trough conductivity and generated electrostatic charge is reported in Table 5.

Table 5. Triboelectrification behavior of the powder and trough materials, with relative triboelectric positions established using the work function of the materials reported in Table 1.

Material	Material Type	Expected Triboelectric Interaction with MCC (Qualitative Assessment)
PLA	Insulating	Promotes significant triboelectric charging of MCC during shear due to its insulating nature and dissimilar surface properties.
Metallic	Conductive	As a conductive material, stainless steel results in lower triboelectric charging of the MCC powder during shear than PTFE and PLA.
MCC	Insulating	MCC is a highly insulating excipient. Triboelectric charging occurs readily during particle–particle and particle–wall contacts involving MCC.
PTFE	Highly Insulating	Induces the highest triboelectric charging of MCC due to its highly insulating properties.

Another finding concerns the effect of the particle size. As expected, the results show higher adhesion for the finer MCC 102 powder ($d_{3,2} = 170\text{ }\mu\text{m}$) than for the coarser MCC 203 powder ($d_{3,2} = 256\text{ }\mu\text{m}$). Instead, the measured electrostatic charge values for the two powder cuts are very similar, given the differences between the trough materials. This proves that triboelectric charging in this system is mostly governed by the chemical nature of the contact pair. Both samples share the same chemical composition; so, the driving force for charge transfer remains constant. Under the consolidating stress of the shear cell, the true contact area between the compressed powder mass and the wall remains similar for both sizes. This ensures a consistent charge transfer regardless of the individual particle dimensions.

The elevated shear stress observed for insulating troughs in the layering method is not just a consequence of Coulombic attraction. However, the electrostatic mechanisms regulating the particle rheology in this system are significantly more complex. It must be noted that the net charge measured by the Faraday cup is the bulk value. However, the wall adhesion is influenced by highly localized charge density at the powder–PTFE wall coupon. For PTFE and PLA insulating troughs, the charge accumulation may create a heterocharge layer, which induces an image charge within the dielectric wall that electrostatically pins nearby MCC particles to the trough surface and significantly enhances the observed wall friction and adhesion.

Moreover, the charge induces electrostatic repulsion between the particles in the bulk bed, but only between particle of equal size, for which image charge phenomena are less effective. The local charge accumulation might be enhanced by the layer-by-layer preparation procedure. The ionizing gun probably results in partial neutralization of the charge, but unevenly on different levels. Instead of achieving complete electrostatic neutralization, repeated use of the ion gun during layered filling appears to result in localized charge accumulation on individual layers [60]. For charge-retaining insulators such as PLA and

PTFE, as shown in Figure 9, these resulting electrostatic gradients change both the interparticle cohesion and the Coulombic forces at the wall interface and between the particles. Incomplete neutralization or an unbalanced ion stream can lead to regions of net charge deposition in the powder layers (Figure 9), causing internal electrostatic gradients and variations in observed yield locations. In insulating materials such as PLA and PTFE, where the charge dissipation is limited, these local charge differences may persist during shearing, leading to less stable powder–wall interactions. This mechanism is consistent with the pronounced increase in the point-to-point scatter of the yield locus observed for PLA and PTFE under layer-by-layer preparation (Figure 8). The presence of local charges would explain why the average yield locus changes only slightly, while the repeatability of measurements becomes noticeably worse. This increased variability is more meaningful than the small shift in the average yield locus itself. Consequently, the deviations and shifting yield loci observed are a product of these complex cumulative electrostatic interactions.

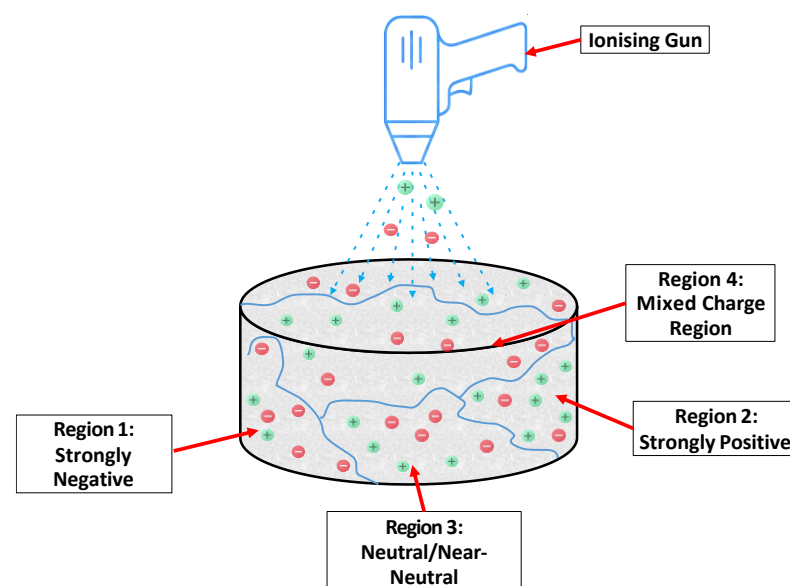


Figure 9. Conceptual illustration of localized charge accumulation during the layer-by-layer preparation process.

Therefore, the main effect of the triboelectric charge observed in the present study may appear to be a systematic shift in the wall yield locus due to an increase of adhesion. The triboelectric charging may change the mean wall yield locus, and it can still affect the measurement's reliability. In most cases, with insulating troughs like PTFE and PLA, the adhesion increased with the electrostatic charge generated. Therefore, triboelectric charging fundamentally alters the flow properties of insulating powders like MCC. The electrostatic effect on adhesion cannot be ignored when testing or during operations in process units with insulating walls like PTFE.

5. Conclusions

This study investigated whether triboelectric charging induced by different trough materials can alter the wall-friction behavior of MCC powders during quasi-static shear testing. The initial assumption was that insulating troughs would lead to higher charge accumulation and significantly shift the wall yield locus. The results on a PTFE wall coupon only partially supported this assumption. The trough material strongly affected the charge generation and also shifted the wall yield locus slightly upwards. Moreover, triboelectric charging limits the repeatability of the measurements, especially for insulating

troughs and for the layering preparation method. In particular, the main conclusions are as follows:

- PTFE generated the highest electrostatic charge, PLA exhibited intermediate charge, and the metallic trough produced the lowest charge for both MCC powders.
- Insulating troughs led to higher adhesion values than the metallic trough, shifting the wall yield loci towards higher shear stress values.
- The layering method for sample preparation introduced higher scatter in the yield locus data, particularly for PTFE and PLA across both MCC powder grades.

Overall, to accurately replicate real process conditions, it is recommended that the experimental characterization of wall friction via shear testing utilize a trough material with an electrical conductivity similar to that of the investigated wall coupon.

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Abbreviations and Symbols

The following abbreviations and symbols are used in this manuscript:

MCC	Microcrystalline cellulose
PLA	Polylactic acid
PTFE	Polytetrafluoroethylene
PSD	Particle size distribution
FTIR	Fourier transform infrared
μ	Wall friction coefficient
σ	Normal stress
τ	Shear stress
τ_{ad}	Adhesion

Appendix A

To evaluate the evolution of charge during the shearing process itself, wall friction tests were conducted for coarser MCC 203 powder ($d_{3,2} = 256 \mu\text{m}$). For this, a PTFE trough was prepared using the standard method, and the powder was subjected to shearing against a PTFE wall coupon. The lowest normal stress of 0.4 kPa of the full wall-friction shearing was considered. The shearing process was separated into three discrete approaches with single, double and triple shearing stages. All performed at a constant normal stress of 0.4 kPa (Figure A1). For each shearing process, fresh powder was used, and

the previous charge was neutralized after trough preparation. The experiments were repeated 3 times.

The materials chosen in these experiments are those corresponding to the highest charge level detected in the process at the lowest normal stress, which minimizes the contact area during shear. These conditions should correspond to the most critical to reach the final charge, and the effect of repeated experiments is expected to be most significant in the measured final charge.

The resulting charge accumulated on the powder after each respective approach is presented in Figure A2. Figure A2 shows that the net charge on the powder increases progressively during each subsequent shearing approach. The mean bulk charge is 100.57 nC for the single shear approach, 126.57 nC for the double shear approach, and 142.87 nC for the triple shear approach. The variance is almost consistent across all three stages. These results indicate a certain dependence of the charge buildup in the tested configuration, which, however, does not affect the comparisons carried out in the paper on the effects of the different trough material tested on the final charge reached and on the limited effects of charging on the shear testing results.

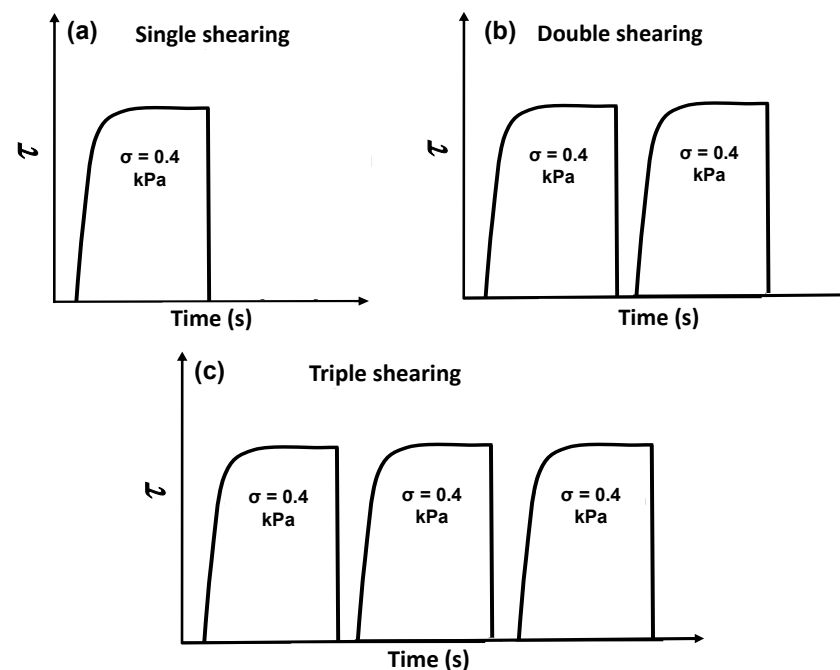


Figure A1. Wall friction shearing process: (a) Single shearing, (b) Double shearing, and (c) triple shearing.

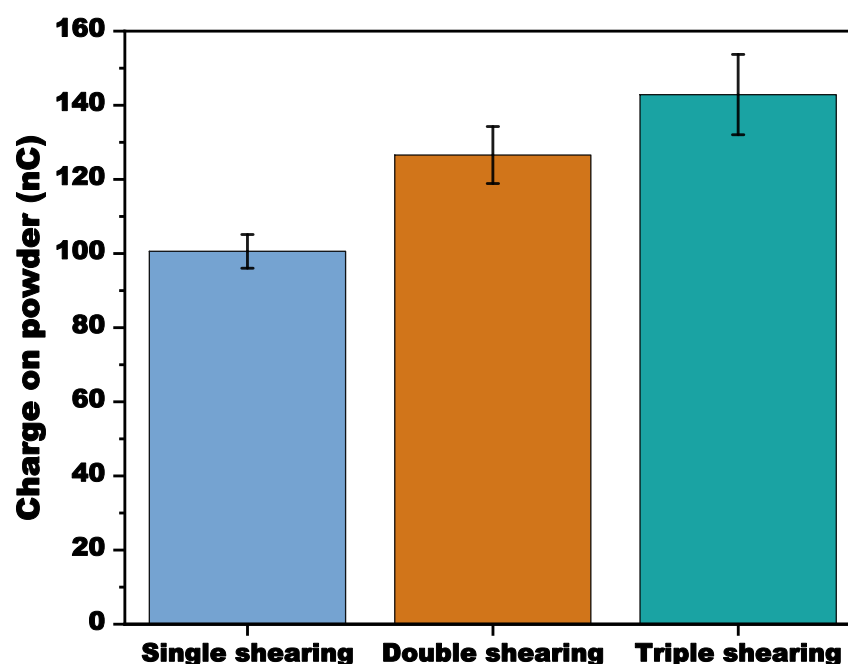


Figure A2. Charge on powder for each shearing approach.

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