Dielectric measurements for the examination of electrostatic charging of powders

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Abstract. The electrostatic charge accumulation of extremely pure substances usually makes the manufacturing technology more difficult. The electrostatic behaviour of the materials can be examined by the measurement of charge decay time, but the conductivity of pure materials is very low, therefore they show very long charge decaying times. Hence, this quantity is not applicable for reliable electrostatic characterisation of pure powders. In this investigation, dielectric and charge accumulation measurements were executed on extremely pure powders. The results of leakage current and voltage response methods show significant difference between the powders according to their electrostatic behaviour. The result of dielectric measurements is confirmed by the charge accumulation measurement. The findings reveal the importance of the investigation of slow polarisation mechanisms in the charge accumulation processes of powders.

1. Introduction
Several companies in many industries have technological problems with extremely pure powders provided by different suppliers. The necessity of using of high purity powders increases the problems caused by the changed electrostatic behaviour of the powders [1]. The charging of powders depend on many factors [2] and can cause hazards or serious problems in the processing. Besides this the small particle diameter of the handled material means high surface to volume ratio, which implies a high charge to mass ratio of the powder. Several methods are developed to measure and characterise the charging intensity and charging processes of these fine powders but in cases of bipolar charging these methods has own difficulties to correctly predict the behaviour of particles [3].

By the charging of powders there can be several scenarios discerned. In the case of powder mixtures, the charging process is characterised by the collision of materials with different chemical and electrical properties. These can be the two components of the powder or the powder particles and the wall of the container or pipeline. In the case of only one material, it would be assumed that only the particle and wall collisions play the only role in the charging process. In fact it is proven that the contact and separation of particles with the same properties also generate a rather high charge separation and so a bipolar charging [4].

In the practical measurement methods, one of the biggest challenge is to distinguish between the charging caused by the collision of the walls and the particles and the charging caused by
the collision between particles [5]. An other big issue is that the overall charge of the powder amount could be measured for negligible small, whereas the particles seen in a much smaller scale are charged positively and negatively in the same amount. Hence the measurement of the overall charge is misleading and the correct amount of positive and negative charge distribution is needed to characterise the charging of the powder. The use of the Faraday pail method produces correct results for dust which has unipolar charge accumulation, but can not provide accurate results by bipolar charging. A method was supposed with a fluidised bed of particles to eliminate the charging at wall and then an electrostatic separator collects the differently charged particles [6], so overcoming the difficulties from other methods.

In this work, four powder samples with different electrostatic behaviours were investigated because different electrostatic behaviour are experienced depending on the manufacturer. Electrostatic charging of a powder of a given manufacturer causes a technological problem because of the adhesion of particles. The chemical analysis of the samples does not show difference between the substances. Moreover, the standardised measurement of the time constant of charge decaying was carried out, but the results were between 3860 s and 4560 s. These values did not provide acceptable explanation for the different electrostatic behaviour of the samples.

Since, the contamination and impurities in non-conducting materials increase the density of charge carriers (e.g. trapped electrons below the conducting band and ions) and the dielectric properties are strongly depended upon the number of charge carriers, the dielectric properties were measured and compared with the charge accumulation measurement. In this paper, the results and evaluation of the measurements are introduced.

2. Measurement of dielectric properties

To investigate the dielectric behaviour of the powder samples two measurements were used namely, the leakage current and the voltage response measurement.

For the measurements, parallel-plate probe was prepared (see Figure 1) [7]. The effective area of plates were 5650 mm$^2$ and the distance between them was 5 mm. Before the measurements the powders samples were preconditioned for 24 hours in the air conditioned laboratory, where the temperature and the relative humidity were 24.9–26.1°C and 40.6–56.0%, respectively. The environmental parameters were the same during the measurements.

To ensure the homogeneous filling of the probe, the powder was continuously compressed during filling processes. This ensures the filling mass was between 17.924 g and 19.770 g. Considering the volumetric mass density of the powder, the filling ratio of the probe was 41.781-48.693% in all cases.

The leakage current measurements were carried out by an Agilent 4339B high resistance meter. The test voltage and time were 1000 V and 1000 s, respectively. The sampling time for recording the insulation resistance values was 10 s. From the measurement results, leakage current as a function of charging time curves were calculated and plotted. The result of the measurements are in Figure 2.

The results show the powder samples can divide into two groups according to their leakage current curve. The group with higher leakage current has higher conductivity this sample contains higher contamination or impurities.

The voltage response measurement is developed for condition monitoring of high voltage apparatus [8], but it is successfully used for material characterisation, recently [9, 10]. The decay voltage can be measured after long charging period of an insulation, while the return voltage appears after a few seconds shorting of the charged arrangement. The initial slope of the decay voltage ($S_d$) is directly proportional to the specific conductivity of the material and the slope of the return voltage ($S_r$) is directly proportional to the intensity of polarisation processes having time constant higher than the discharging time. The detailed description of the method and the relationship between the measured parameters and the dielectric properties
can be found elsewhere [11].

The S_d was measured after 2000 s long charging of powder samples by 1000 V then S_r was measured after 1 s discharging time. The results of the measurements are in Table 1.

**Table 1.** The results of voltage response measurement.

| Sample   | S_d [V/s] | S_r [V/s] |
|----------|-----------|-----------|
| Sample 1 | -0.011    | 8.293     |
| Sample 2 | -0.028    | 8.361     |
| Sample 3 | -0.031    | 2.934     |
| Sample 4 | -0.013    | 3.267     |

However, the slopes of decay voltages do not show significant difference between the samples, by slopes of return voltages they can be divided into two groups. The S_r values of Sample 1 and 2 are higher by almost three times than that of Sample 3 and 4. This suggests the higher intensity slow polarisation processes, which can be related to the space charge or interfacial polarisation. The dielectric measurements suggest that Sample 1 is identical to Sample 2 and Sample 3 is identical to Sample 4, as well. This is confirmed by the manufacturer.

3. Measurement of charge accumulation

After the dielectric measurements the charge accumulation was measured on the samples.

The test arrangement was similar to that published by Peltonen et al. in [12, 13] (Figure 3). Since, the purpose was to simulate realistic conditions, the pipe is taken from the production line and it was grounded.

After some measurement it was noticed that the powder started to adhere to the pipe. Therefore the test setup was modified. The angle of the sliding pipe is increased to 60°, the powder samples were dropped in 3...5 g portions into the Faraday cup and the Faraday cup was grounded after dropping of each portion. From the measured voltage, the charge accumulation of a given portion of the powder were calculated. This method was chosen to investigate the
real manufacturing conditions such as the powder is covering the inside of the pipeline and its effect on the charging properties. In spite of that the each samples were measured separately, the results of Sample 1 and Sample 2 were evaluated together and Sample 3 and 4 were also did. Measurement results are in the Figure 4.

![Test arrangement after [12]](image)

**Figure 3.** Test arrangement after [12]

![Results of cumulative charging measurement](image)

**Figure 4.** Results of cumulative charging measurement

The result of the measurement shows that the charge accumulation of both powders is almost the same in the beginning of the tests. The specific charges decrease by increasing of cumulative mass in case of both powders. It is caused by the adhesion of particles onto the inner surface of pipe. Hence, in the beginning of the test, the metal-powder contacts lead to higher charge accumulation, while in the later phase, powder-powder contacts are the more dominant processes in the charge accumulation. Similar observations have been reported by Peltonen et al. in [12, 13]. In case of Sample 1 and 2, the final value of the specific charge measurement shows higher value around 8 pC/mg, while this value of Sample 3 and 4 is below 6 pC/mg.

4. Granulometric data
Since the size distribution has effect on the charging [14, 15] and dielectric behaviour of the powders, therefore the size distribution was provided by the manufacturer. The granulometric data of samples are in the Table 2.

| Sample | Sample 1 and 2 | Sample 3 and 4 |
|--------|----------------|----------------|
| d (10) | 0.6 µm (±2.3%) | 0.3 µm (±2.0%) |
| d (50) | 2.1 µm (±0.7%) | 2.2 µm (±0.5%) |
| d (90) | 5.4 µm (±0.4%) | 5.4 µm (±0.6%) |

The granulometric data does not show significant difference between the particle size distribution of the samples.
5. Discussion
The dielectric measurements show more intensive slow polarisation processes in case of Samples 1 and 2 (see Table 1). The final specific charge values of charge accumulation measurements are also higher for Sample 1 and 2 than Sample 3 and 4, therefore higher charge can be accumulated with the application of Sample 1 and 2 in the manufacturing process. The manufacturer also confirmed that Sample 1 and 2 exhibit poorer electrostatic behaviour. Since, the granulometric data does not show significant difference between the samples, it can be assumed that Sample 1 and 2 are less pure, this substance contains more foreigner molecules. Therefore more intensive space charge or interfacial polarisation processes can be occurred in this substance, which result in higher charge accumulation.

6. Conclusion
The leakage current and voltage response measurements show significant difference between the powder having different electrostatic behaviour. The charge accumulation test also show different charging capabilities. Since both dielectric tests measure the slow polarisation processes having higher time constant than one second, the results of the examination suggests the relationship between the interfacial polarisation processes and the charge accumulation property of powders. The cause could be the difference between the contamination content of the samples, which is sensitively detected by the dielectric measurements.

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