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One-step measurements of powder resistivity as a function of relative humidity and its effect on charging



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A R T I C L E I N F O

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ABSTRACT

A new one-step technique to measure the effect of ambient humidity on powder resistivity has been previously presented. In this article, we provide more experimental data obtained with five different powders. One-step measurements and traditional multi-step measurements were performed. Also, additional measurements were performed using standard resistivity cell. Results were compared and it could be concluded that the new technique provided meaningful results although significant hysteresis was observed during humidification and drying cycles. Finally, charging of the powder was also measured and it was noticed that it decreases with decreasing resistivity and increasing humidity. © 2015 Elsevier B.V. All rights reserved.

Introduction

Resistivity measurements of powders are important since the resistivity plays a significant role in powder triboelectrification. It is generally thought that decreasing resistivity also decreases charging. This is due to the fact that the charge which is transferred in a contact is able to flow back to original material during separation if the powder resistivity is low. Excess charging can cause a lot of problems in different powder handling operations, such as mixing, filling and transport. Charging experiments can be performed in many different ways, for example, sliding a sample down into a Faraday cup via a pipe, or by charging in a fluidized bed device [1–3]. In general, charging experiments suffer from quite poor reproducibility and are difficult to perform consistently in a normal laboratory without specially trained personnel. Resistivity measurements, on the other hand, are quite easy to perform compared to charging experiments.

It is well known that increasing the humidity of surrounding air decreases powder resistivity due to adsorbed water layer on the particles. Thus, increasing the humidity also decreases charging [4-6]. Thus, humidification is often used to control the charging. However, there are several situations where humidification can

cause additional problems. For example, in the pharmaceutical industry powders are processed in many unit operations and the particles will charge. Humidification could reduce the problems but some of the materials are chemically and/or physically stable only in dry atmosphere. In the chemical industry, large volumes of powders can be transferred pneumatically and high charges may build up. Again humidification would reduce the charging but excess water may significantly increase cohesion and hamper powder flow.

According to the previous discussion, there is a need for a method for determining a humidification level which is high enough for charge control but low enough not to cause additional problems.

Traditionally, the relation between resistivity and humidity is measured in several steps. Dry powder is placed in a resistivity cell, a voltage is applied between the electrodes and the current is recorded. From the recorded voltage and current, resistance can be calculated using Ohm's law. For the calculation of the resistivity of the material, dimensions of the cell need to be known. The volume of a standard resistivity cell is roughly 0.5 L. The electrodes are circular, placed vertically and their radius is 25 mm. Spacing between the electrodes is 5 mm and the electrodes are aligned parallel to each other [7]. Next, the humidity of surrounding air is increased to a certain value, typical increase being approximately 10 - 15 RH%. Water molecules will then diffuse into the powder bed. Depending on the powder properties, particle size and the







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height of the powder bed, the diffusion will last for a certain time. For many powders, the time needed for the system to reach an equilibrium can be several days. The equilibrium has been reached when the current signal has reached a steady state. Humidity is then increased again in a stepwise manner. So, measurement of the whole range of interest can take weeks.

In this work, a one-step method was used together with a new resistivity cell geometry. In this method, humidity was rapidly changed from either low to high, or high to low. The relative humidity of the air surrounding the particles within the powder bed was measured at the height of the electrodes simultaneously with the resistivity. The procedure and electrode geometry are presented in more detail in previous paper [8] and is only briefly discussed here. The electrode maintained at high potential was square and the current electrode was narrow and aligned horizontally. The current electrode was surrounded by a square shaped and grounded guard plate which prevented current which flowed through more humid areas from reaching the current electrode.

Resistivity measurements were performed on five different powders by varying the relative humidity of air from approximately 10 %–90 % (10%–65% on more conducting samples). Results were compared with measurements performed with a standard cell and multi-step humidification. Since the objective of the humidification is to reduce the charging, charging measurements were also performed at different humidities. The charging was performed using a small fluid bed device with closed air loop.

Materials and methods

Materials

Five different powders were chosen for the study. Materials were chosen so that they cover a large resistivity range from 10^5 to 10^{14} Ω m. Two different grades of lactose α -monohydrate were used: Capsulac 60 and Prismalac 40 (Meggle, Germany). Common sugar was also used (Suomen Sokeri Oy, Finland). Monoammonium phosphate (Krista-MAP) was obtained from Yara, Belgium and Sodium chloride from Merck, Germany.

Methods

Resistivity cells

New resistivity cell and the measurement procedure are presented in detail in previous article [8]. Schematic image of the system is presented in Fig. 1. Resistivity ρ of a powder can be obtained from measured resistance *R* across the electrodes using equation $\rho = KR$. Cell constant *K* depends on the geometry of the electrodes so that K is the ratio of the electrode area and the electrode separation. The cell constant of the new resistivity cell was 0.0375 m.

Another resistivity cell was also used and it has been built according to standard [7]. It had circular electrodes with 50 mm diameter and spacing 5 mm which resulted in a cell constant K of 0.393 m. Resistivity cells were placed inside a sealed and grounded chamber. Predetermined relative humidity inside the chamber was obtained using saturated salt solutions and dry air flow. Humidities of the air surrounding the cell and the air within the powder were measured using HMI38-instrument (Vaisala) equipped with two probes, HMP37E and HMP35E (Vaisala). The resistance across the electrodes was measured using an electrometer (Keithley 6517A, Keithley Instruments Inc.). The voltage across the electrodes was kept constant through the whole drying/humidification cycle and it was set to 1000 V for both lactoses and sugar, 100 V for MAP and 10 V for NaCl. Lower voltages were used with MAP and NaCl, because at higher humidities these samples were so conducting that the current would have exceeded the limits of the electrometer if 1000 V had been used. Air humidity inside the powder at the height of the current electrode, humidity of air surrounding the cell, and measured resistance R were recorded simultaneously using a virtual instrument written in LabVIEW and a PC.

One-step measurements were performed so that the resistivity cell containing the sample was placed inside the chamber and the chamber was flushed with dry air. When the relative humidity inside the powder was approximately 10%, a petri dish containing water (or saturated salt solution in some cases) was placed inside the chamber. The voltage was turned on and the values were recorded until the relative humidity of air inside the powder was approximately 90% (65% using MAP or NaCl). Then, petri dish was removed and the chamber was flushed with dry air. Data was recorded during the drying cycle until the powder was again approximately 10% dry. Lower value of high humidity limit was used for MAP and NaCl, because these powders would start to dissolve at higher humidities.

The multi-step measurements were performed in a similar way. Saturated salt solutions were used to obtain a constant relative humidity. Data was recorded until the resistivity signal had reached a steady-state value. Then, another salt solution was introduced into the chamber and the procedure was repeated until the whole humidity range had been covered.

During the measurements it was noticed that the resistivities of dry lactoses and sugar were too high for an accurate measurement. Thus, resistivities above $10^{14} \Omega m$ could not be recorded and have to be considered unreliable.



Fig. 1. Schematic image of the resistivity cell. Figure on the left shows the cross section of the current electrode and the guard plate. Figure on the right represents a side view of the device.

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