Correlations for a set of powders between the charge generation in a pneumatic conveyer and the electrical characteristics measured in a ventilated bed

G. ARTANA, S. SAMMARTINO, M.F. MORIN, G. TOUCHARD

L.E.A., Operation n°9, URA CNRS N°191, Université de Poitiers, 40 Av. du Recteur Pineau, 86022, Poitiers, FRANCE

Abstract

This paper deals with the electrical behaviour of powder materials of the farm-produce industry. Different kinds of experiences have been undertaken to electrically characterise these elements. With our experimental device we have defined a measurement protocol that gives a rather good accuracy and repeatability. So it seems adequate to control parameters as packing and powder humidity. A correlation between the results of our measurements is analysed.

Introduction

Even though powder materials are involved in many industries (chemical, pharmaceutical, farm-produce) their physical properties are not well known and they are rather difficult to characterise. Yet it is not clear which is the set of measurement methods and which are the physical properties that best enable to predict the technological behaviour of powders in some usual industrial processes.
For instance the friction forces between the different particles of a powder or the friction forces between powder and a wall depend on different parameters (water contents, granulometry, etc.). So for two powders of different material or even the same material with different granulometry, neither the angle of the heap nor the sticking of the powder to an inclined plane is usually the same. More, powder materials are difficult to be classified as solids or liquids. When there is no movement of particles the powders have solids characteristics but when they move they behave much more as liquids.
Though much effort has been done in the last twenty years, all these mechanical behaviours are not yet well described by theory. Recently, as powders have specific properties some authors [Jaeger (1992)] have proposed to consider a granular state adding to gas, solid and liquid states.
Concerning the electrical properties of such materials even if a lot of work has already been carried out [Crowley (1986), Jones (1991), Masuda (1977), Glor (1988), Benmadda (1987), Boudalaa (1991)] still the influence of electrical characteristics on mechanical properties remain difficult to predict.

Experimental studies are arduous and very careful processes need to be followed to have reliable and reproducible measurements.
For instance it is well known that the hygrometry plays a very important role in the conduction phenomena of powders, so reproducibility in conductivity measurements may be expected only for very dry products. Even in that case, for rather insulating powders, a contamination as a monolayer of water (very difficult to remove) can drastically change the phenomena and a time dependent electric purification may appear.
Also the electrical conduction phenomena depends on how the particles are disposed and how is the contact between them. So packing of powders must be controlled to avoid nuisances in the experimental study.
Even though all this problems are quite discouraging, there is a lot of interesting fundamental work to be done. Just consider that the surface of powders is enormous when compared with the same volume of a solid materials. As many electrical phenomena depend on the surface or interfaces of the material all these phenomena should be magnified in case of powders.
In this paper we describe the experimental methods we have tested to electrically characterise a set of powders. We study the accuracy and repeatability of the methods as defined in the French norms. Finally, we make an analysis of redundancy that is to say we look for the correlation between two data series.

Powder characteristics

This paragraph gives information about the characteristics of powders we have tested. The set belongs to powders used in the farm-produce industry and it is composed of a vitamin, an antibiotic, an oligo-element and two coccidostatics.
We show in table 1 particle mean diameter ϕ, mass density $\mathbf{d}$, and specific surface $\mathbf{s} = \frac{\mathbf{1}}{\phi\,\mathbf{d}}$ of the powders we have studied.
The density of powders shown in this table is measured after the packing process described in next point.

TABLE 1: **Powder Characteristics**

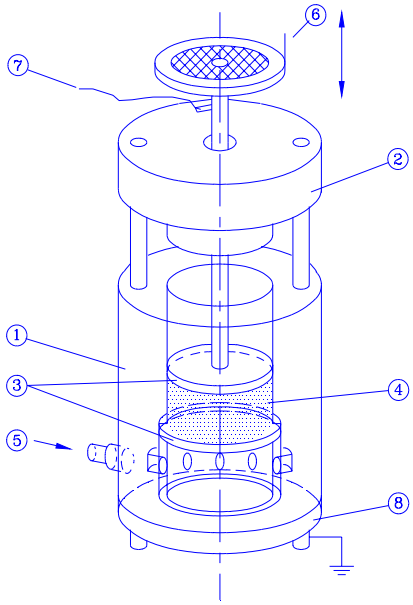
Powder	Mean diameter (µm)	Density (kg/m^3)	specific surface (m^2/kg)
A953	**300**	**1020**	**3.3**
A954	**7-10**	**410**	**243.9-348.4**
A9546	**55**	**880**	**20.7**
A955	**15**	**355**	**18.8**
A957	**12**	**1320**	**63.1**

Measurements in a ventilated bed

We have measured in a ventilated bed the conductivity, the relaxation of charges in a dielectric absorption test and the permittivity of the powders.

Cell description.

Though different cells have been proposed to measure electric powder characteristics [Euler (1978), Whyte (1983)], we have perfected a simple one that enables to have a good control of the powder humidity and of the powder packing.
The cell used in our measurement is shown in figure 1. It is composed of a body made of teflon ① inside which there are two porous planar parallel circular electrodes ③, the lower one being the top of a pressurisation chamber. Powder is placed between these electrodes (electrode diameter is 50 mm) and dry air flows from the chamber through the lower electrode, the powder, the upper electrode and exits to the atmosphere in the upper region through a metallic diffusive screen (not shown in the figure).
For the resistivity measurement the upper electrode is connected to a voltage source and the lower one is grounded through an electrometer. For the dielectric absorption test the upper is connected to a HV source and the lower one is grounded. For the permittivity measurement both electrodes are connected to a capacitor bridge.
In the figure 2, which corresponds to the particular case of the dielectric absorption test, we see that we can press the powder with a constant weight applied to the upper electrode. In all measurements the electrode spacing is measured with a cathetometer ⑦ and an hygrometer ⑥ is placed at the exit of the air flow.
The cell is mounted on a vibrating plate ② and inside a Faraday pail ⑧ in order to avoid external noise during the measurement.



① Body in teflon, ② Guide with ball bearing, ③ Porous electrode, ④ Powder, ⑤ Inlet of air flow, ⑥ Support for weight application, ⑦ Voltage supply connection, ⑧ grounded metallic support connected to the pressurisation chamber and the lower electrode.

Figure 1: **Cell of Measure**

Measurement protocol

The same following chronological procedure was used in the measurements of the conductivity, of charges in the dielectric absorption test and of the permittivity.

1) Packing : by vibration of the cell during one minute with a weight of one kilogram on the upper electrode.
2) Ventilation : by a constant dry air flow of 830 l/h during 10 minutes. During this phase the upper electrode is pushed down by a weight of 5 kg.
3) Measurement : for the same weight and the same air flow.

The mass of powder was measured for each test and was fixed for each powder to obtain after the packing process a volume of around 20 cm^3.
During the packing phase both electrodes are grounded and during the phase of measurement the dry air flows continuously.
To analyse accuracy and repeatability of the measurement this procedure was followed for 12 samples (three per day during four different days) of each powder.

Accuracy and repeatability

An image of accuracy and repeatability of an experiment can be obtained by the quotient between the standard deviation σ_x and the mean value $\bar{x}$ of a data series **X** of a powder. If we consider **N** different powders the following coefficient **p** may be used

$$p = \frac{1}{N} \sum \frac{\sigma_x}{\bar{x}}$$

It is usually accepted that if this parameter does not exceed the value of 0.05 accuracy and repeatability are correct.

Conductivity Measurements

The conductivity measurement has been carried out applying dc voltage difference between the electrodes and measuring the current through a Keithley electrometer 610C.
We apply in each sample a cycle of step voltages **V** (10-20-30-40 V). Current is measured after each step. Then, another cycle of decreasing step voltages (40-30-20-10 V) is imposed and we measure again the current after each step.
For the 5 studied products, current increases linearly with potential difference that means an ohmic behaviour for this field range. So with the current density **j** and the electrode spacing **l,** we obtain the resistivity ρ of our powders by means of

$$\rho = \frac{V}{l\ j}$$

We show in table 2 for our different powders the mean value of ρ and the standard deviation of the twelve measurements**.**

Table 2: **Powder Resistivity**

Powder	mean value	standard dev.
A953	**2.45E+12**	**3. E+10**
A954	**8.86E+11**	**5.1 E+10**
A9546	**1.85E+12**	**1.1 E+11**
A955	**2.35E+07**	**3.1 E+06**

A957	**4.29E+11**	**2.8 E+10**

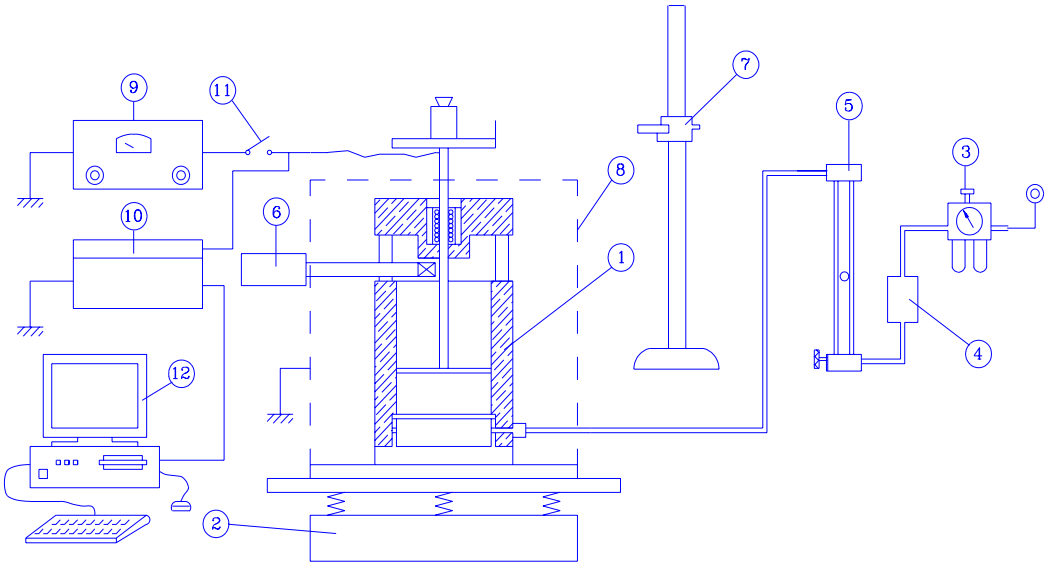
Accuracy and repeatability

The value of the coefficient **p** calculated from our results of resistivity is 0.088. However, usually experimental results when observed in a very large measurement interval are treated in a logarithmic scale. In our case, considering the logarithm values of our measurements the coefficient **p** equals 0.004.
We observe then, that with this protocol resistivity measurements are quite accurate and repeatability is correct in a logarithmic scale.

Dielectric Absorption test

We have measured the presence of free charge carriers in our powder after applying a high electric field with the dielectric absorption test. These charge carriers may be intrinsic (produced by a bulk phenomena) or extrinsic (created at the powder-metal interface and injected in the bulk).

Our study has been undertaken with the device shown in figure 2.

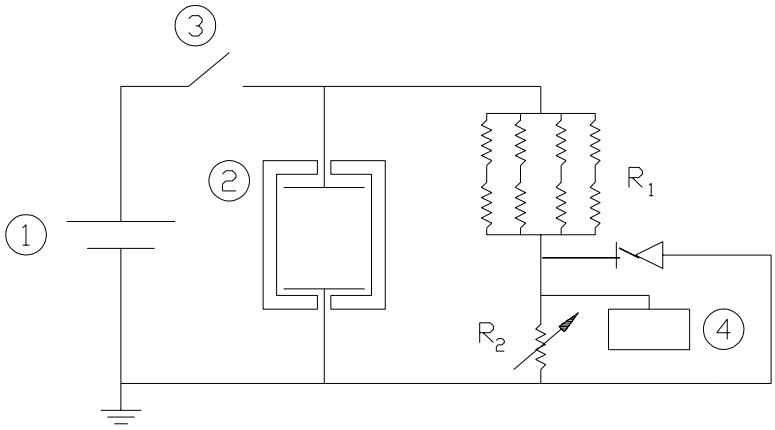


① Cell of measure, ② Vibrating table, ③ Pressure regulator, ④ Dryer, ⑤ Flowmeter, ⑥Hygrometer, ⑦ Cathetometer, ⑧ Faraday pail, ⑨ Voltage supply, ⑩ Discharge circuit, 11 HV-HI electronic switch device, 12 Data acquisition system

Figure 2 : **Device to measure the relaxation of charge of the dielectric absorption test**

We apply a dc voltage between the electrodes during 30 seconds and we open the electronic High Voltage-High Impedance switch 11. Then the powder relaxes its charges and a decreasing current flows through the discharging circuit ⑩. Signal is recorded with a data acquisition system Keithley DAS 1600 12. A descriptive schema of the

discharging circuit is represented in figure 3. The High Voltage-High Impedance electronic switch shown in this figure was constructed with a reed switch. An electronic system (not mentioned in the figure) synchronises the opening of the circuit with the beginning of the data acquisition.



① HV source, ② Measuring cell, ③HV-HI electronic switch device, ④ Data acquisition system

Figure 3 : **Discharging circuit**

When a high electric field is applied between the electrodes, the powder will get polarisation charges and free charges. As the switch is open these charges will relax and fruitful information is obtained measuring the current that flows through the resistors R_1 and R_2.The value of these resistors are much smaller than the resistance of the powder between the electrodes. We show in figure 4 a typical curve of charge relaxation in a neperian logarithmic scale.

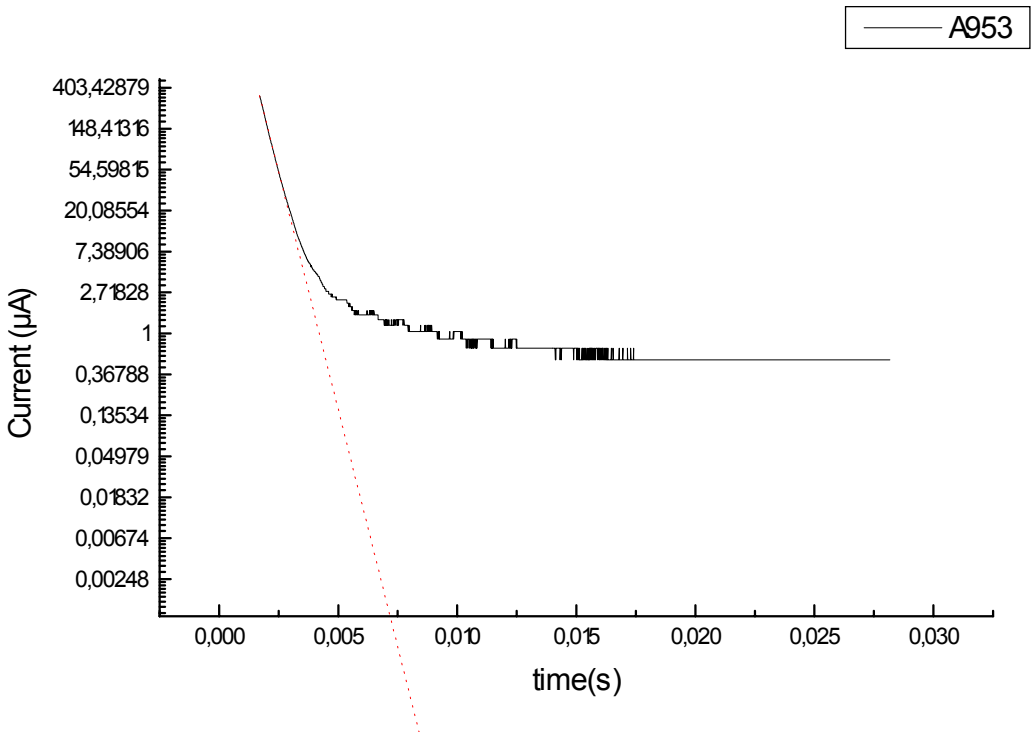


Figure 4 : **Current as a function of time through the discharging circuit**

When the powder has a resistivity high enough, the curves show initially a very clear exponential decay and after some time a residual current. We can identify the first part as corresponding to the relaxation of polarisation charges and the rest to the relaxation of free charges.
Table 3 exhibits the values of free charges we have obtained when using electric fields around 400 kV/m. Values are referenced to the mass of powder used in the test.

Table 3 : **Dielectric Absorption**

Powder	mean value (picoC/g)	standard dev. (picoC/g)
A953	**2984.9**	**576.5**
A954	**135.4**	**45.6**
A9546	**72.0**	**27.7**
A955	**807.5**	**278.4**
A957	**119.0**	**43.9**

Accuracy and repeatability

For our experimental study the value of the coefficient **p** for the dielectric absorption test is 0.326. If we consider the logarithm of the results then this coefficient equals 0.069. We can see from these values that in a logarithmic scale the dispersion of results exceeds slightly the accuracy and repeatability limit, but not so much.

For some dielectric materials a remaining voltage between the electrodes may be detected after the test and this is an important parameter as it indicates the charge kept by the material acting as an electret. For the powders we have tested no signal was detected even in the μV range.

Permittivity Measurements

We have measured the permittivity of powders using our cell connected to a capacitor bridge General Radio 1615-A. The measurement is quite conventional and it consists in equilibrating the unknown capacitor with other capacitors.
Permittivity can also easily be obtained for powders of high resistivity, measuring the time constant τ_1 of the exponential decay of curves described in the previous paragraph. Knowing the capacitance of the empty cell **Co** (measured with the bridge)**,** the time constant τ_0 for the empty cell, and the value of the equivalent resistors **R** through which the relaxation current flows, the dielectric constant **ε** is :

$$\varepsilon = \frac{\tau_1 - \tau_0}{R\, C_0} + 1$$

Using this formula we consider the parasite capacitors in parallel with the cell.
The values of the mean value of **ε** is shown in table 4 for the different powders.

Table 4: **Dielectric Constant ε**

Powder	mean value	standard dev.
A953	**4.08**	**0.10**

A954	**2.03**	**0.02**
A9546	**2.53**	**0.06**
A955	**5.02**	**0.21**
A957	**3.05**	**0.06**

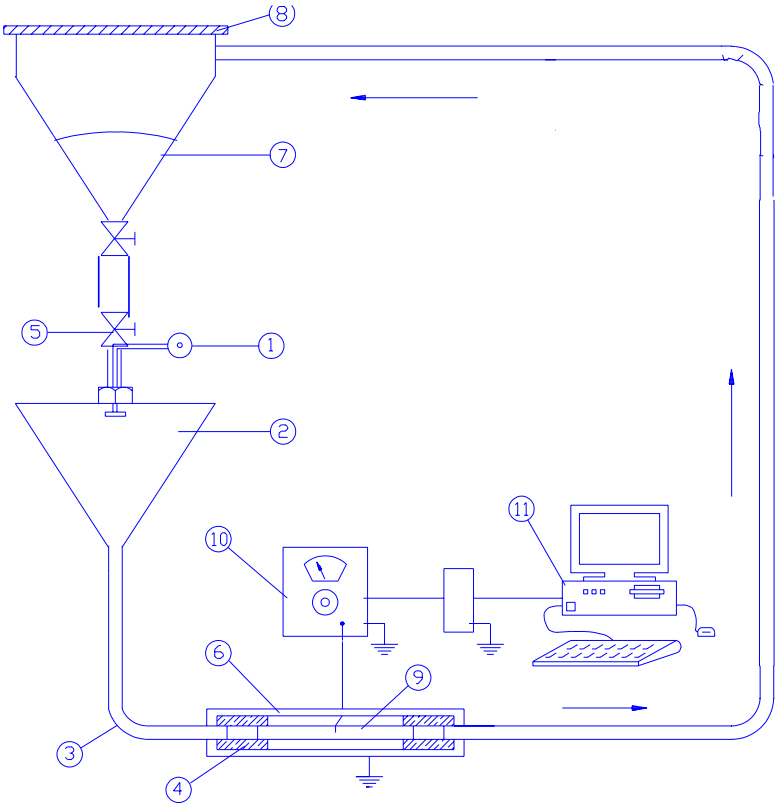
Accuracy and repeatability

For this test the value of **p** equals 0.024. This value confirms that the protocol is good enough to obtain correct results.

Measurement of the powder charging in pneumatic transport

Description of the pneumatic loop.

The measurement of the charge due to the friction with the wall of a metallic tube in a pneumatic transport of powder has been done with the device shown in figure 5.



① Adjustable flow, ② Pressurisation vessel, ③ stainless steel pipe, ④ PTFE insulators, ⑤ Adjustable valve, ⑥ Faraday pail, ⑦ Collecting vessel, ⑧ Porous cover, ⑨ Testing section, ⑩ Keithley Electrometer, 11 Data acquisition system

Figure 5 : **Device to study the charging of powders in a pneumatic transport**

Powder flows from the reservoir ⑦ through an adjustable valve ⑤ to the pneumatic circuit.
Nitrogen flows from the gas reservoir through an adjustable pressure system into the conic vessel ② where powder is incorporated to the flow. Then it passes through

different tubes and exits to the atmosphere through the porous cover ⑧ of the upper reservoir ⑦ where the powder is recovered.
The experiment consists to determine the charge created by the friction of the powder with a stainless steel tube of 4 mm of diameter, 100 mm length and electrically insulated from the rest of the circuit.

Protocol

The electric current is measured with a Keithley electrometer 610C and recorded with a data acquisition system Keithley DAS 1600. The total current obtained in the whole experiment is integrated in time to obtain the total charge. Then we refer it to the mass of powder that flows in the experiment.
We have studied 12 different samples for each powder. All experiences were done for the same air flow rate (1170 l/h) and if no slip between air and particles exists, the mean particle velocity used in the transport is about 25m/s.
Mass flow rates are adjusted so that the transport of powder is in dilute phase.
The different mass flow rates we have used for the different powders and the results we have obtained are shown in table 5.

Table 5: **Pneumatic Charging Test**

Powder	mass flow rate (g/s)	mean value (µC/kg)	standard dev. (µC/kg)
A953	**0.45**	**-4.18**	**0.89**
A954	**0.04**	**-9.94**	**5.21**
A9546	**0.18**	**-6.86**	**0.92**
A955	**0.66**	**4.52**	**0.67**
A957	**0.28**	**26.70**	**4.26**

These values agree with the typical charging level of pneumatic transport 1-100 µC/kg proposed by several authors [Jones (1991),Glor (1988)].

Accuracy and repeatability

For the pneumatic charging test the value of **p** we have obtained is 0.235 and for the logarithm of our measurement is 0.114. These values exceed the limit of 5% of **p** coefficient.
Analysing our results we observe that A954 powder has a great dispersion of results. This powder has a very high specific surface and the mechanical behaviour (even when handling) is quite different from the others. Charging during pneumatic transport depends strongly on the flow particle dynamics and on the surface with which friction occurs. This powder has an important tendency to stick and it is quite possible that the physical phenomena occurring at the walls with this powder will be different from the others.
Excluding this powder the value of **p** is then 0.163 and for the logarithm of our measurement is 0.083, that exceeds the limit of 0.05 but at least in a logarithmic scale the accuracy and repeatability can be considered acceptable.

For this reason, the comparison of results between powders should be done with precaution and we preferred not to consider the results of the A954 in the next point "discussion".

Discussion

In this paragraph we will discuss the correlations that we can find between some parameters obtained in this study. We analyse correlations between the mean values of the inverse of the particle diameter, the conductivity, the dielectric constant, and the mean values of the charges relaxed in the dielectric absorption test and the charge generated in the pneumatic transport test. These last two parameters are multiplied by the mass density **d** to refer our results per unit of volume.
To analyse the correlation between two data series **X,Y** we use the coefficients of correlation $\mathbf{q_{x,y}}$ defined as the ratio of the covariance between both series **cov(X,Y)** and the product of the standard deviation σ_x, σ_y of each data series

$$q_{x,y} = \frac{\mathrm{cov}(x,y)}{\sigma_x \, \sigma_y} = \frac{\frac{1}{n}\sum_i (x_i - \bar{x})(y_i - \bar{y})}{\sigma_x \, \sigma_y}$$

This coefficient enables to determine if both data series vary in a joint way, that is to say if the high values of a series can be associated to the high values of the other (positive correlation) or if the low values of a series are associated to the high values of the other (negative correlation). For the higher correlations this coefficient is 1 or -1 and if there is no correlation this coefficient tends to zero.
In table 6 we show the different coefficients of correlations $\mathbf{q_{x,y}}$ for the different data series.

Table 6: **Coefficients of correlations $q_{x,y}$**

	diel abs. charge *d	**pneum. transp. charge*d**
1/ϕ	**-0,460**	**0,915**
1/ρ	**0,964**	**0,030**
ε	**0,942**	**-0,299**

Correlation of the inverse of diameter with results of the diel. abs. test

The inverse of the diameter of a spherical particle indicates the ratio surface-volume of the particle.
Also for a group of particles, as powders, the inverse of the mean particle diameter is a parameter that indicates roughly the "density of surface" defined as the surface of powder existing per unit of volume.
We can see that the correlation between the "density of surface" and the charge in the dielectric absorption test is not very important. The slight value of the coefficient of correlation and the lower limit of electric field for charge injection phenomena in solids (10 MV/m) [Coelho (1993)] seems to indicate that in some powders of our test the

electric field is not high enough to induce charge injection from the electrodes and only intrinsic carriers may be created.

Correlation of electrical conductivity with results of the diel. abs. test

We observe a high correlation between the electrical conductivity and the charge relaxed from the dielectric absorption test. This is also observed in solids where the worst materials to relax their charges are insulating materials [Sessler (1987)].

Correlation of dielectric constant with the results of the diel. abs. test.

We observe also a high correlation between this both data series. If charge injection exists in our test this behaviour seems to be in agreement with the phenomena observed in space charge limited currents.

Correlation between the charging level of pneumatic transport with the inverse of mean particle diameter

We find a high correlation between this data series. As the charging in pneumatic transport occurs caused by the friction of the powder surface with other surfaces, it is expected that for a powder with high "density of surface" the contact metallic wall-powder will be larger and so the total charge.

Correlation between the conductivity and the dielectric constant with the charging level in pneumatic transport

From our experiments, no clear correlation could be established between these parameters.

Correlation between the dielectric absorption with the charging level in pneumatic transport

The correlation coefficient between both tests is -0.42. From this coefficient it seems that both phenomena are not directly linked.

Conclusions

In this paper we have studied the electrical behaviour of a set of powders of the farm-produce industry in a ventilated bed and during a pneumatic transport in dilute phase.
When using a ventilated bed we observe a very slight dispersion of the conductivity, of the dielectric constant and a greater one of the charge relaxed in dielectric absorption test. Our protocol seems adequate to fix powder packing and powder humidity. Our results seems to indicate that the fields used in our study $\cong$ 0.4MV/m are not high enough to produce high charge injection from electrodes.
We observe high correlations for the dielectric absorption test with dielectric constant and conductivity.
The pneumatic loop considered is suitable to make the measurements but it is not adequate to all kind of powders. It seems that results for powder with very high specific

surface should be treated with precaution. The charging level of these experiments agree with the typical values proposed by other authors.
A high correlation is observed between the charge generated in pneumatic transport and the inverse of the particle mean diameter. No clear correlation of the level of charging in the pneumatic transport with dielectric constant has been observed .

Acknowledgements

This work has been done with the support of TECALIMAN under contract CNRS n°780286.

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