The Measurement of Charge Response of Powders Exposed to Ion Bombardment

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Synopsis

A system has been designed and developed to subject a sample of powder on a grounded metal plate to charging by ion bombardment and to measure the charge accepted by the sample. Observation of the charge decay from the sample was also possible. A method of sample preparation was evolved which made it possible to obtain samples of known particle size and number of particles. The sensitivity of measurement and the noise levels were investigated and found to be acceptable. Calibration tests were made to determine the validity of the proposed equation relating the measured voltage, circuit capacitances, and the ratio of probe height to net charge location. It was found that for a wide range of values of the ratio of probe height to particle diameter, the linear equation was a good description of the process and could be used to accurately determine the charge on a sample. Some measurements were made on typical powders to illustrate the feasibility of the method and equipment to characterize variable charging properties.

INTRODUCTION

The application of electrostatic charge to powders as a means of controlling the movement of the particles has been used in many processes. Currently many new applications are being found. Three basic processes that may be used to charge particles are: (*1*) ion bombardment, (*2*) induction, and (*3*) triboelectric.

Experience has shown that the equations derived earlier for powders of moderate resistivity are adequate for describing the charging process, but are inadequate when powders of extremely high resistivity are involved. The authors chose to develop a new technique and equation and to appraise the feasibility of measurement of the charge acquired by a plastic powder when subjected to ion bombardment charging.

It was intended that the method should enable the determination of average values of charge per particle with a known accuracy. The technique was also required to allow measurement of the rate of decay of charge from the sample after charging ceased.

No attempt has been made to investigate the location and distribution of charge on a particle. Whether it is a surface, volume, or some other relationship is unknown.

PROCEDURE

One way to meet the objectives was to design a device which would allow the sample to be exposed to charging for a short period at regular intervals. Measurement of the charge on the sample then could be made between each charging period.   The simplest device for doing this is a rotating table which carries the sample successively beneath a charging electrode and a capacitance-measuring probe.   This method has been used previously.[5]   The most desirable feature of this approach is that leakage of the probe signal to ground through the input resistance of the measuring instrument is very small, as the exposure of the sample to the measuring probe is of short duration.   The reading obtained is therefore a true value.   A reading is obtained at each revolution of the turntable so that a series of point values can be recorded.   This is of more use than a continuous curve, which includes a decay due to leakage of the input signal to ground.

A phonograph turntable with base and mounded plastic dust cover was
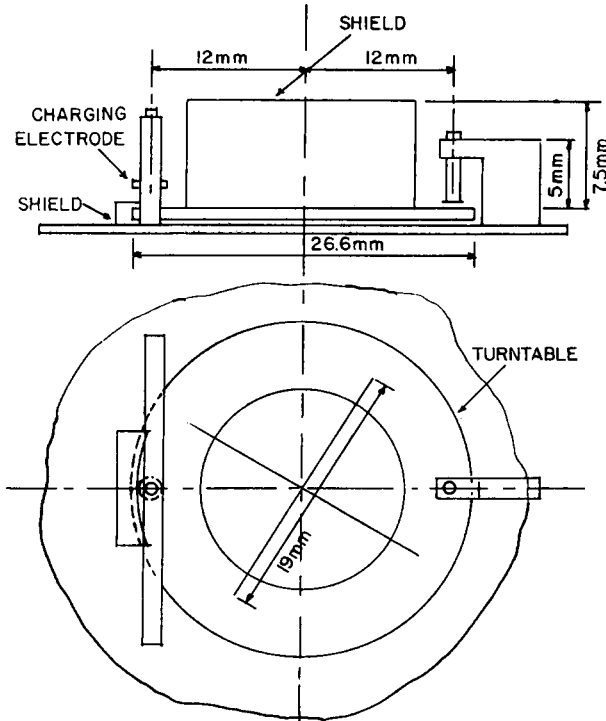

Fig. 1.   Drawing of the modified turntable.

acquired and modified as shown in Figure 1.   The important features are: (1) charging electrode, (2) interprobe shielding, (3) turntable edge shielding, (4) removal of paint, etc., from areas likely to be subject to charging, (5) brush to ground turntable, (6) capacitance probe, (7) sample holder, (8)

switch to ground electrometer input, (9) switch to trigger oscilloscope trace, (10) access *door* in dust cover, and (11) dry-air inlet and desiccant.

The simplest way of producing ion bombardment of a sample of powder on a grounded metal plate is to mount an electrode above the plate and apply a negative dc potential to the electrode. At a certain potential a corona discharge develops at the electrode and the electric field causes the ions released to travel to the grounded plate, some of them colliding with the powder particles. The energy level of the ions as they arrive at ground is a function of the electric field strength at the grounded surface. The electric field strength is in turn a function of the voltage applied to the electrode, the geometry of the probe, and its surface and the electron cloud at the electrode due to corona. For most systems the electric field strength at any specific location cannot be determined.

For our system, the charging electrode is a needle point located 14 mm above the surface of the grounded sample plate. This needle is connected to the negative side of a variable-voltage dc power source. The positive side of the power source is grounded. To prevent the applied potential from directly influencing the measuring probe, a shield is mounted on the turntable and grounded via the turntable bearings and a grounding brush. The dimensions are shown in Figure 1. It was also found necessary to fit a small shield near the charging probe to prevent charge accumulation on stray dielectric materials on the edge of the turntable.

The method of measuring the charge described in ref. (1) requires that the sample be spread uniformly over the grounded sample holder. The equations presented are descriptive of this system, but do not describe our system where sensitivity is sufficient to permit the examination of the charge on relatively few particles spaced so as to have little if any interaction. The details of the sample holder and the equivalent electrical circuit are shown in Figure 2. The symbols used in Figure 2 and in the following equations are: U = electrometer reading (voltage), C_1 = capacitance between charge location and sample plate surface, C = capacitance between charge location and probe surface, C_2 = capacitance between probe surface and sample plate surface, C_3 = electrometer input capacitance including input lead, h_1 = distance between average charge location and sample plate surface, h_2 = distance between probe and sample plate.

Assume that a charge, Q, is placed on C_1 and it is then moved under the test electrode forming the circuit shown in Figure 2. Q will then be distributed between C_1 and the series-parallel network consisting of C', C_1, C_2 and C_3, in proportion to the capacitance of C_1 and that of the network, C_x:

$$Q_1 = Q \frac{C_1}{C_1 + C_x} \text{ and } Q_x = Q\frac{C_x}{C_1 + C_x}.$$

The circuit potentials are

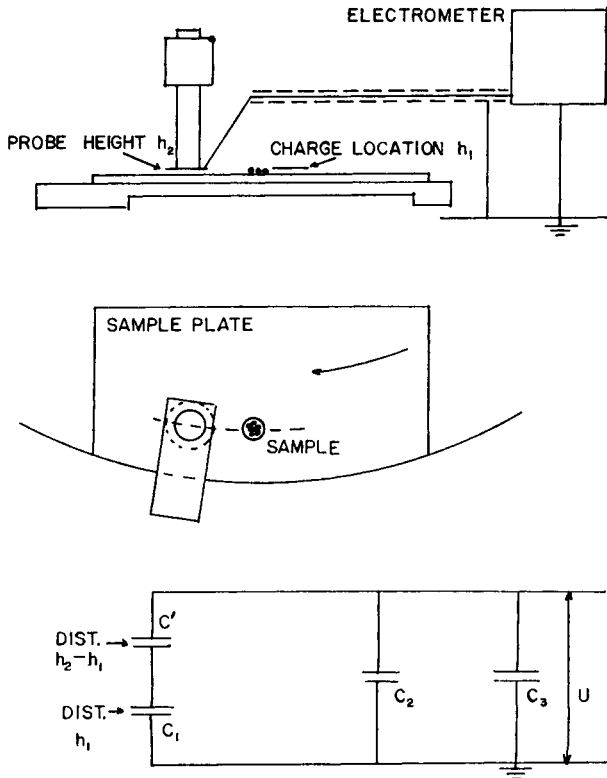$$\frac{Q}{C_1 + C_x} = \frac{Q_1}{C_1} = \frac{Q_x}{C_x}. \tag{1}$$

Fig. 2. Measuring probe and sample plate details and equivalent circuit diagram.

Since the charge on each capacitance of a series network is equal to the total charge on the network,

$$Q_x = Q' = Q_{2+3}, \tag{2}$$

then

$$U = \frac{Q_{2+3}}{C_2 + C_3} = \frac{Q_x}{C_2 + C_3}$$

and

$$\frac{Q_x}{C_x} = \frac{Q'}{C'} + \frac{Q_x}{C_2 + C_3} = \frac{Q'}{C'} + U. \tag{3}$$

Substituting in eq. (1), we obtain

$$\frac{Q}{C_1 + C_x} = \frac{Q'}{C'} + U. \tag{4}$$

From eqs. (2) and (3) we obtain

$$\frac{Q}{C_1 + C_x} = \frac{QC_x}{C'(C_1 + C_x)}. \tag{5}$$

Combining eqs. (4) and (5), letting $C'/C_1 = h_1/h_2 - h_1$, and simplifying, we obtain

$$Q = U\left[C_1 + (C_2 + C_3)\frac{h_2}{h_1} \right] \tag{6}$$

If eq. (6) is valid, then to calculate Q requires the knowledge of $(C_2 + C_3)$, h_1, h_2, and C_1. For the purpose of this investigation, it is assumed that h_1 equals the radius of the particles. Due to the ion bombardment being from one direction, it is unlikely that the charge distribution is uniform, therefore one would expect the net charge location to lie above the half-height of the particles. If the true location is at 70% of the particle height, then the results calculated on the basis of the above assumption would be 40% too large for the largest powder particles used.

We have no way to estimate C_1, however, if $(C_2 + C_3)$ is large enough, the effects of C_1 on the calculation of Q can be quite small. In all of our calculations C_1 is neglected. This results in the calculated values of Q being too small by an indeterminate amount. It should be noted that this source of error is in the opposite direction to that resulting from an underestimate of the charge location height.

To determine the effective input capacitance of the electrometer, a charge was measured twice, the input capacitance being different for each measurement. The difference in input capacitance, K, was known so that the equation $U_1(C_2 + C_3) = U_2(C_2 + C_3 + K)$ could be solved for $(C_2 + C_3)$. This was done three times using powders having different charging characteristics and different mean diameters. K had a value of 97 pf. The average value calculated from the measurements for $(C_2 + C_3)$ was 20.67 pf $\pm$ 0.8 pf. The error term includes an estimate of the 95% confidence limits for the three measurements plus our best estimate of the tolerance of K. The value of the input capacitance of 20.67 pf was obtained with $h_2 = 1.32$ mm. Values of 20.47 pf and 20.27 pf were obtained for distances h_2 of 1.98 mm and 2.68 mm, respectively.

The capacitance probe was connected to the electrometer input by a short length of low-capacitance shielded cable and a coaxial connector. A bronze mesh screen was fitted over the capacitance probe and the exposed leads near it to eliminate pickup of stray electric fields. A small brush was arranged to ground the turntable directly in case the resistance through the bearings was too high. The rim of the turntable was profiled so that, in conjunction with a contact from the probe, it formed a switch which grounded the electrometer input for part of each revolution of the turntable. This switch was timed so that the electrometer input was ungrounded shortly after the leading edge of the sample plate passed under the probe and regrounded as the trailing edge of the sample plate passed under the probe. The input remained grounded during the rest of the cycle. This system ensured that zero voltage was impressed on the electrometer input until approximately 0.2 sec prior to the charge measurement. Another contact and a 1.5-V dry cell were used to give a signal to trigger the os-

cilloscope trace. This signal and the trace sweep time were adjusted so that the trace moved across the screen as the sample plate passed under the probe. The electrometer reading of the charge appears as a flat-topped peak in the center of the screen. The zero reading could be observed either side of the peak. The electrometer used had an input impedance greater than 10^{14} ohms shunted by 10 pf. Full-scale ranges from 0.001 V to 1.0 V produced an output signal to the oscilloscope of ± 10 V.

The system sensitivity was such that a probe voltage of 2 mV equaled the system noise. With a probe height, h_2, of 1.32 mm and a sample consisting of 100 particles of 0.0895 mm mean diameter, d, the minimum charge per particle, q, that could be detected was 0.0122×10^{-12} coulomb. This is equivalent to 7.68×10^4 elementary ionic charges. In order to achieve this sensitivity, it was necessary to control the humidity in the test area to a low value. A supply of controlled humidity air was continually introduced into the test space. No study was made of the effects of varying the humidity. All tests were made at relative humidities between 20% and 25%.

VERIFICATION OF EQ. (6)

Equation (6) requires that U be a linear function of the ratio h_2/h_1. This was checked by measuring the charge, Q, accepted by two samples each of two different powders. Each sample was tested for charge acceptance at two values of charging voltage and with three different probe heights. Each test was repeated three times. This resulted in 72 individual measurements from which the mean charge acceptance values and their 95% confidence limits were calculated. The results are tabulated in Table I and plotted in Figure 3. These show that, for the range tested,

TABLE I
Measured Charge on Samples With the 95% Confidence Limits
(Units are Coulombs $\times 10^{-12}$)

| Identification	Charging potential, kV	Probe height, h_2		
		1.32 mm	1.98 mm	2.68 mm
Sample (d) (polyethylene, mean d 0.1795 mm)	10	98.8 ± 4.0	99.8 ± 3.2	103.0 ± 4.2
	15	124.6 ± 4.0	123.4 ± 5.4	130.0 ± 7.8
Sample (e) (polyethylene, mean d 0.0895 mm)	10	74.2 ± 4.2	81.4 ± 2.8	81.4 ± 3.6
	15	119.6 ± 9.6	113.2 ± 6.8	112.6 ± 8.4
Sample (f) (epoxy, mean d 0.1795 mm)	10	136.8 ± 8.0	132.8 ± 5.8	132.4 ± 4.2
	15	172.4 ± 8.0	169.8 ± 5.8	168.2 ± 7.8
Sample (g) (epoxy, mean d 0.0895 mm)	10	198.8 ± 8.0	202.4 ± 6.4	196.2 ± 8.4
	15	261.2 ± 8.0	250.0 ± 1.8	253.6 ± 8.4
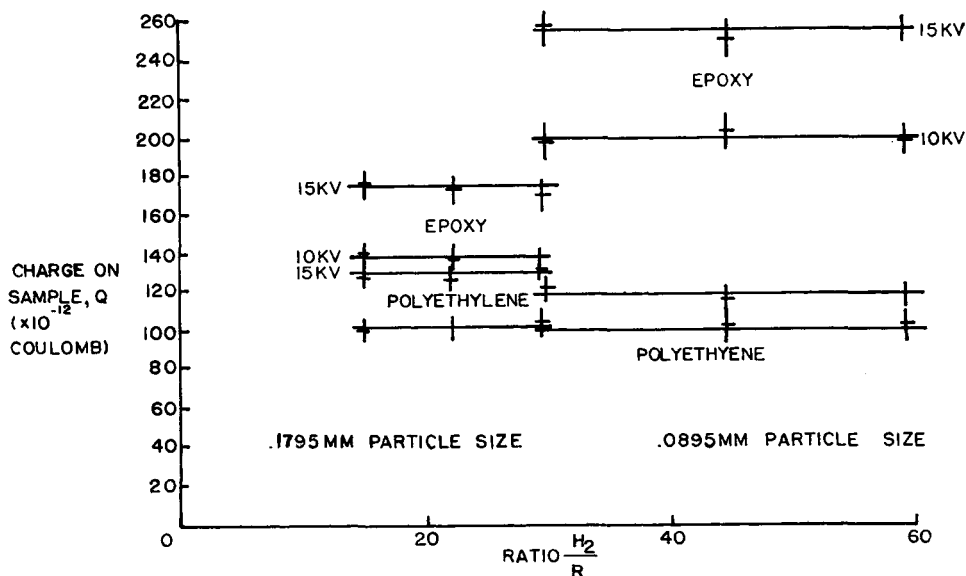
Fig. 3. Verification of eq. (6).

there is no indication of nonlinearity between U and h_2/h_1. In only one of the eight curves was it not possible to draw a straight line through each of the calculated values of Q or its 95% confidence limits. The 95% confidence limits on the experimental data were approximately 4% of the calculated charge value. The assumption that the system was linear, therefore, was not a limiting factor in the accuracy of the method.

Our results are, however, all low by the charge represented by the factor UC_1. We can only make an estimate of its magnitude relatively to the factor $U(C_2 + C_3)h_2/h_1$. With $(C_2 + C_3)$ equal to 20.67 pf and h_2/h_1 equal to 10, C_1 would require a value of 200 pf to result in a 100% error. We feel that it is unlikely that C_1 is greater than 10 pf which would result in all of our charge values being 5% low.

MEASUREMENT OF SOME POWDER PROPERTIES

To illustrate the uses of the technique, some measurements were made to determine the charge acceptance per particle when subjected to specified charging conditions. Both powder material and particle size were varied so that comparisons could be made. Also, observations were made of charge decay as a function of time for a few samples. The samples used are described in Table II. When the samples were prepared and examined under a microscope, it was found that the most typical particle dimension was quite close to the mean of the sieve openings passing and not passing the sample. The shape of the particles was found to vary considerably. In the case of the larger sizes, an effort was made to select the most nearly spherical particles. It was not practical to do this for the smaller size frac-

TABLE II
Tests on Various Materials

Sample No.	Size, mm	Charge per particle, 10^{-12} coulomb	Decay time, sec
	PVC with ditridecyl phosphate plasticizer		
1(a)	0.0895	0.368	9
1(b)	0.0895[a]	0.870	7
1(c)	0.1795[a]	1.256	16
1(f)	0.1795[a]	0.940	10
1(g)	0.1795[a]	0.452	7
	PVC with trioctal phosphate plasticizer		
2(a)	0.1975[a]	0.010	too short to be observed
	PVC with diethyl phthalate plasticizer		
3(a)	0.0895	0.638	70
3(b)	0.0895	0.320	64
3(c)	0.1795[a]	0.986	72
3(d)	0.1795[a]	0.620	75
	PVC with tricresyl phosphate plasticizer		
4(a)	0.0895	0.204	83
4(b)	0.1795	0.230	128
	Diamond Drysol, K 408 Vinyl		
5(a)	0.1795[a]	<0.006	—

[a] Group diameters where particles were composed of groups of smaller particles.

tions. The tolerances on particle size in the graphs is arbitrary and is shown to prevent the reader from placing undue confidence in the curves. The estimates of charge are also dependent on the accuracy of particle size determination, and this source of error has not been included in the results.

Other sources of error not accounted for in these illustrations are: (1) errors in particle count, (2) deviations of net charge location from the assumed location at h_1 equal to the particle radius, and (3) the quantity UC_1 was neglected. Errors due to (1) can be controlled to acceptable levels (1% or less) in samples of 100 particles or less. Errors due to (2) have not been investigated and could be serious. As indicated earlier, it is unlikely that they would exceed 40%. The difference in error between samples would be much less, probably about 10%. Errors due to (3) have been discussed.

When samples 1 through 5 were examined under the microscope, it was found that many of the particles were approximately 0.0895 mm in diameter and it therefore was quite easy to prepare samples of that particle size. There were very few particles as large as 0.1795 mm in diameter. In most cases the larger particles were made up of groups of smaller particles. The number of particles in a group appeared to vary from 3 to 7. The charge per particle as reported represents the charge per group when the larger

sizes were so constituted. The results of these tests are shown in Table II. The decay times shown are the times required for the charge to fall to 36.8% of the original value. This corresponds to the system time constant if one assumes that the charge would eventually decrease to zero. Although no measurements were made of charge acceptance, it was observed that powders which had slow decay had rapid acceptance and vice versa. Thus it appears that decay time might be used as a figure of merit for both acceptance and decay characteristics.
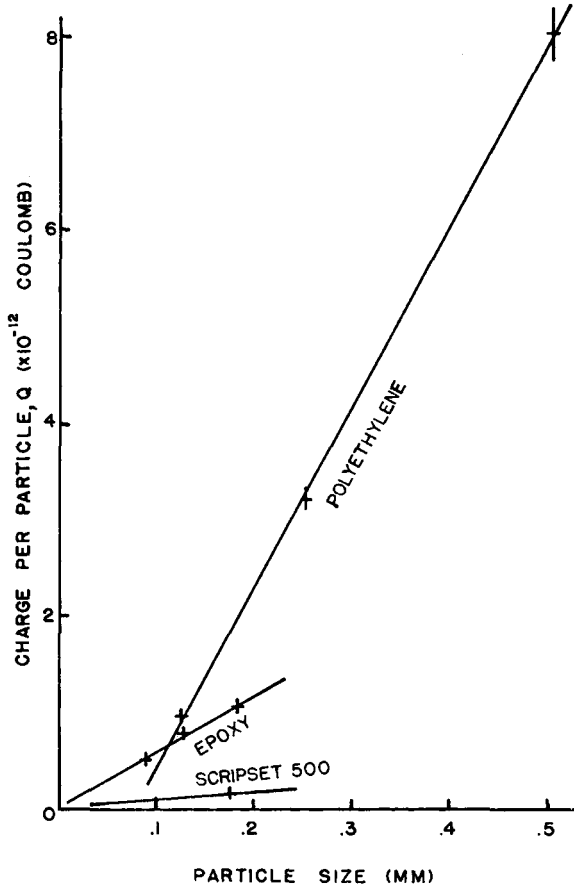

Fig. 4. Powder properties.

The results of the measurements on various powders produce some interesting data. Some conclusions can be drawn from the results although more information about particle geometry is probably necessary before some aspects of the results can be fully evaluated. The ratios of surface area and volume to apparent diameter of the particles would be particularly useful. It is claimed[3] that previous work showed that the limiting charge carried by a particle, of a definite material, is given by the equation

$$Q = pE_c \frac{d^2}{4}$$

where

p = (3 × dielectric constant)/(dielectric constant + 2),
E_c = field strength, and
d = particle diameter.

The results obtained here show that the range in limiting charge accepted by different powders is much greater than variation of the dielectric could account for if the above equation described the situation. Also, particularly in the case of the epoxy powder samples, the limiting charge accepted was proportional to the diameter d, not d^2 as in the equation cited. Figures
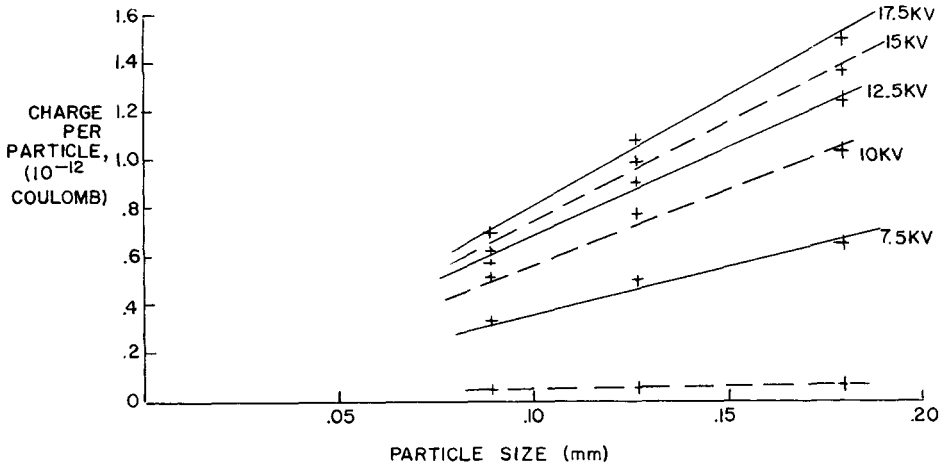

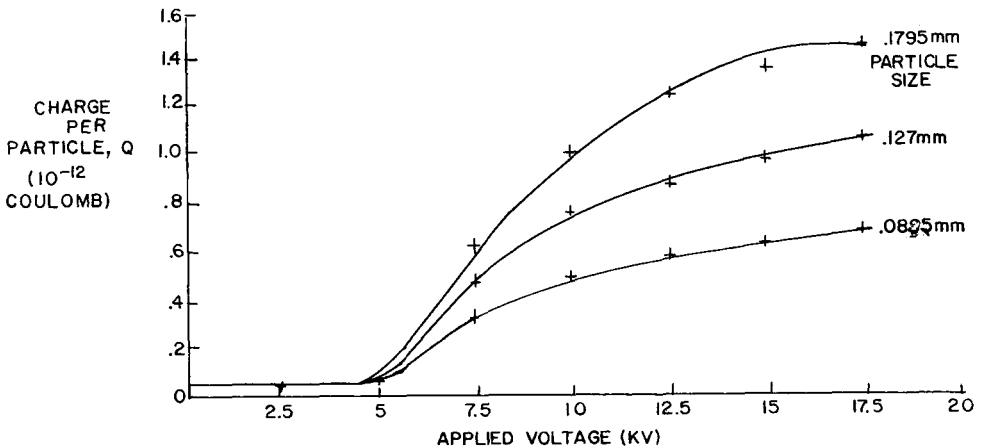Fig. 5. Effect of varying charging potential.

Fig. 6. Effect of varying charging potential.

4 and 5 illustrate this relationship. Further work with the method would allow such characteristics to be adequately investigated. Figure 6 shows the nonlinear characteristic of charge acceptance vs. applied charging voltage. This is almost certainly a property of the corona charging phenomenon rather than a powder property.

In conclusion, we can say that we can make measurements which are adequately precise to permit characterization and ranking of different powders. Absolute measurements can be made with an accuracy probably better than 50%.

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