Hopper Flow Electrostatics of Tableting Material I

Instrumentation and Acetaminophen Formulations

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Instrumentation is described for measuring the sign and magnitude of static charges generated by particles flowing through a tablet hopper. Acetaminophen in the crystalline form had a higher negative hopper flow static charge than granulations prepared from the powder. Tablet excipients, such as diluents (dicalcium phosphate dihydrate, mannitol, and spray-dried lactose) and lubricants (magnesium stearate and talc), lowered the hopper flow static charge of acetaminophen. Particle size and water concentration were shown to influence the magnitude of the hopper flow static charge.

Static electricity in the strict sense means electricity that is standing still. The term is used primarily to distinguish effects which are observed between electrically charged bodies from effects associated with electricity in motion. It is referred to frequently as frictional or contact electricity because a convenient method of producing such a charge is by rubbing one body upon another. It seems highly probable, however, that in all such cases the friction between the two bodies is merely incidental, and the charge results from the fact that in the process of rubbing, the materials are first brought into intimate contact, then separated (1). When two surfaces are brought into contact, it is possible for some of the electrons of one surface to be attracted to the adjoining surface and to remain there when the two surfaces are separated abruptly. The surface which now carries an excess of electrons is negatively charged, while the surface with a deficiency of electrons has an equal positive charge. The resulting forces of attraction and of repulsion between charges have been recognized to be a problem in the tableting process (2).

Static charges on particle surfaces make many powdered materials difficult, if not impossible, to handle in blending and feeding operations. Methods suggested to overcome flow problems include modification of the crystalline habit, the addition of glidants, use of antistatic agents, and humidity control (3). Although humidification is an accepted method of mitigating static charges, its detrimental effect on the stability of many materials restricts its use. The beneficial effect of high atmospheric humidity is mainly the result of films adsorbed on the surface of the solid insulating materials (4). There is also some indication that adsorbed moisture films on the contacting surfaces reduce the tendency for the separation of electric charges, quite apart from the leakage effect. Other methods used to eliminate static electricity include grounding and neutralization. Particle size, shape, distribution, density, surface characteristics, and the relative size and geometry of the hopper affect the flow rate (5) and, therefore, may influence hopper flow static charges.

This study was initiated to determine the factors influencing the accumulation of static charges caused by the flow of materials through a hopper during the tableting operation. This phase describes instrumentation for measuring hopper flow static charges and defines several tablet formulation variables which influence the hopper flow static charge of acetaminophen. The variables considered include particle form and size, tablet excipients, and added moisture.

EXPERIMENTAL

Instrumentation.—Figure 1 presents a diagram of the instrumentation which consists of tablet hopper A, glass receptacle B, Brookfield[1] helipath stand C, induction transfer system D, E, and F, and ionostat[2] G. The ionostat is an ionization chamber device containing a 10-μc. radium foil source which ionizes the air within the chamber. The ions are accelerated by an electric field, producing a current proportional to the field intensity, which is amplified by an electrometer tube circuit and transferred to an indicating meter. The ionostat contains an iris diaphragm which permits a selection of measuring ranges. The scale of the meter is calibrated in volts per centimeter, where the centimeter refers to the

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[1] Model C, Brookfield Engineering Laboratory, Stoughton, Mass.
[2] Model H1407, Herfurth G.m.b.H., Hamburgh-Altona. Distributed by U. S. Radium Corp., Morristown, N. J.

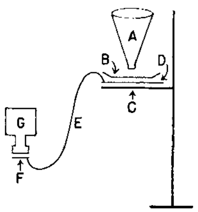

Fig. 1.—Instrumentation for measuring hopper flow static charges. Key: A, stainless steel tablet hopper with 2.5-cm. orifice; B, Pyrex 20-cm. diameter glass receptacle; C, platform of Brookfield helipath stand; D, 20-cm. diameter copper disk positioned 1 cm. directly beneath B; E, 20-gauge copper wire; F, 5-cm. diameter copper disk; G, ionostat, positioned 1 cm. above F. A, C, and G were grounded and entire apparatus confined in glass fronted enclosure.

distance between the front of the chamber and the object to be measured.

Measurements were made as follows. The receptacle was positioned on the platform of the helipath stand 0.6 cm. below the tablet hopper orifice, and a premeasured sample was transferred to the hopper. Two minutes were allowed for the dissipation of any charge developed during the introduction of sample before the platform was lowered. As the platform descended at a rate of 2.0 cm./min., granules flowed through the hopper and generated a static charge. The resulting charge was transferred by induction from the glass receptacle to the ionostat for measurements. The charge was recorded when the platform had descended 5.7 cm., irrespective of the amount of granules which flowed through the hopper. Experiments were conducted in a glass fronted enclosure in which the temperature varied from 22° to 28° and the relative humidity from 20 to 50%. The following arbitrarily selected ranges of values were chosen to express the data: 0–50, 51–175, 176–400, 401–650, and 651–1050 v./cm. The range reported for each material represents in volts per centimeter the high limit and low limit for three consecutive readings, each made on a different day.

Materials.—Commercially available materials of either a U.S.P., N.F., or pharmaceutical grade were used. The forms of acetaminophen studied were crystalline,[3] fine crystalline,[4] and fine powder.[5] Acetaminophen fine powder, dicalcium phosphate dihydrate powder, and mannitol powder were granulated to flow through the tablet hopper. Granulations were prepared using a kitchen aid mixer[6] with one of the following granulating solutions: 5% w/w ethylcellulose,[7] 10% w/w starch paste, and 85% w/w sucrose. Starch paste and syrup were used to granulate acetaminophen, and ethylcellulose was used to granulate acetaminophen, dicalcium phosphate dihydrate, and mannitol. Wet granulations were passed through a No. 6 stainless steel hand screen and dried overnight at 120° F. Dried granules were sized through a No. 12 stainless steel hand screen. Magnesium stearate, talc, cornstarch, Avicel,[8] and Solka Floc BW100 special[9] were passed through a No. 60 stainless steel hand screen prior to their addition in 2% concentrations. Materials were placed in polyethylene bags and stored in cardboard drums.

Moisture Experiments.—Water was sprayed from laboratory prepared aerosols onto granules tumbling in a 6-in. coating pan. Measurements for static charges were made immediately after the addition of water.

Loss on Drying.—Samples were subjected to 70° for 16 hr. Less than 0.1% weight loss was obtained for each material or formulation, except for the following: starch granulated acetaminophen (0.20%); 50% dicalcium phosphate dihydrate and 50% fine crystalline acetaminophen (0.18%); 2% cornstarch and 98% fine crystalline acetaminophen (0.18%); and 48% dicalcium phosphate dihydrate, 2% magnesium stearate, 2% cornstarch, and 48% fine crystalline acetaminophen (0.33%).

RESULTS AND DISCUSSION

Figure 2 compares the static charge of various forms of acetaminophen. Higher negative static charges were obtained for acetaminophen in the fine crystalline and crystalline forms with range values of 401–650 and 176–400 v./cm., respectively. Fine powder acetaminophen granulated with either ethylcellulose, sucrose, or starch had lower negative static charge ranges of 51–175, 51–175, and 0–50 v./cm., respectively. The form of acetaminophen, therefore, influences the magnitude of the hopper flow static charge. Flow difficulties are visualized for the crystalline forms because of their high hopper flow static charge. This would be a problem in direct compression formulations where the crystal-

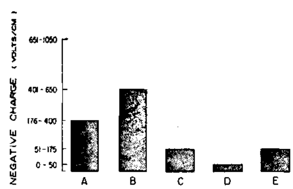

Fig. 2.—Static charges of selected forms of acetaminophen. Key: A, crystalline; B, fine crystalline; C, fine powder granulated with ethylcellulose; D, fine powder granulated with starch paste; E, fine powder granulated with syrup.

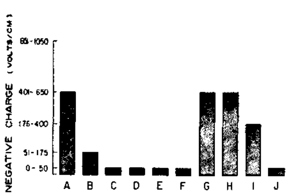

Fig. 3.—Effects of added tablet excipients on static charges of fine crystalline acetaminophen. Key: A, fine crystalline acetaminophen; the following materials were added to fine crystalline acetaminophen in the concentrations indicated: B, dicalcium phosphate dihydrate granules (50%); C, mannitol granules (50%); D, spray-dried lactose (50%); E, magnesium stearate (2%); F, talc (2%); G, cornstarch (2%); H, Avicel (2%); I, Solka Floc BW100 special (2%); J, dicalcium phosphate dihydrate granules (48%), cornstarch (2%), and magnesium stearate (2%).

[3] Granular (16–30 mesh), Miles Chemical Co., Elkhart, Ind.
[4] Fine granular (25–40 mesh), Miles Chemical Co., Elkhart, Ind.
[5] Fine powder (90% through 100 mesh), Miles Chemical Co., Elkhart, Ind.
[6] Precision Scientific Co., Chicago, Ill.
[7] Ethocel, 7 cps., the Dow Chemical Co., Midland, Mich.
[8] American Viscose Corp., Philadelphia, Pa.
[9] Brown Co., New York, N. Y.

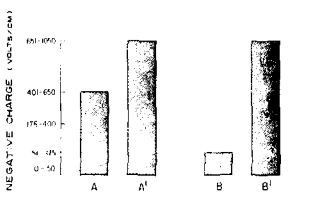

Fig. 4.—Effects of particle sizes on static charges of acetaminophen. Key: A, fine crystalline acetaminophen; A′, 30–50 mesh separated from A; B, fine powder acetaminophen granulated with ethylcellulose; B′, 20–30 mesh separated from B.

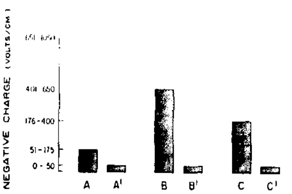

Fig. 5.—Effects of added water in 0.5% concentrations on the static charges of acetaminophen. Key: A, fine powder acetaminophen granulated with syrup and A′ after addition of water; B, fine crystalline acetaminophen and B′ after addition of water; C, crystalline acetaminophen and C′ after addition of water.

line raw material would be required. Consequently, attention was concentrated on efforts to determine the effect the formulation has on the static charge of the fine crystalline form.

Figure 3 illustrates the effect of selected tablet excipients on the static charge of fine crystalline acetaminophen. The negative hopper flow static charge of fine crystalline acetaminophen was 401–650 v./cm. The addition of the tablet fillers, ethylcellulose granulated dicalcium phosphate dihydrate, ethylcellulose granulated mannitol, or spray-dried lactose in 50% concentrations, lowered the negative static charge to 51–175, 0–50, and 0–50 v./cm., respectively. In 2% concentrations, lubricants, magnesium stearate or talc, lowered the negative static charge to 0–50 v./cm. Disintegrants, cornstarch or Avicel, when added in 2% concentrations, did not alter the static charge, whereas Solka Floc BW100 special in 2% concentration lowered the negative static charge to 176–400 v./cm. A formulation containing 48% fine crystalline acetaminophen, 48% ethylcellulose granulated dicalcium phosphate dihydrate, 2% magnesium stearate, and 2% cornstarch had a negative static charge of 0–50 v./cm. Tablet excipients are, therefore, capable of altering the hopper flow static charge. The significant lowering of the static charge by the addition of lubricants in relatively low concentrations suggests their possible use as antistatic agents. Experiments are currently in progress to study the effect of lubricants on the hopper flow static charge of a variety of materials.

Figure 4 illustrates the variation of static charge with particle size. Fine crystalline acetaminophen had a negative static charge in the range of 401–650 v./cm. A 30–50 mesh size separated from these crystals had a negative charge in the higher range of 651–1050 v./cm. Ethylcellulose granulated acetaminophen fine powder had a negative static charge of 51–175 v./cm. and a higher negative static charge of 651–1050 v./cm. for a separated 20–30-mesh size. Particle size, particle size distribution, or both influence the magnitude of the static charge and should be considered in formulations in which static is a problem.

The effect of moisture on the static charge of crystalline, fine crystalline, and syrup granulated acetaminophen is illustrated in Fig. 5. Prior to the addition of water, crystalline, fine crystalline, and syrup granulated acetaminophen had negative static charges of 176–400, 401–650, and 51–175 v./cm., respectively. The addition of 0.5% water reduced the negative static charge of each of the above to 0–50 v./cm. In formulations where static is a problem, the moisture of the tableting material becomes an important consideration.

SUMMARY AND CONCLUSIONS

This study was initiated to determine the factors influencing the accumulation of static charges caused by the flow of materials through a hopper during the tableting operation. A method was developed to measure hopper flow static charges, and a study was made of several tablet formulation variables which could influence the static charge of acetaminophen. From the results obtained, the following conclusions can be drawn.

The instrumentation described provided a satisfactory method for measuring the sign and magnitude of hopper flow static charges.

Acetaminophen had a negative hopper flow static charge, and the form of acetaminophen influences the magnitude of the hopper flow static charge. Fine crystalline and crystalline acetaminophen had higher negative hopper flow static charges than granulations of the fine powder prepared with either ethylcellulose, starch paste, or syrup.

Tablet excipients are capable of altering the hopper flow static charge of fine crystalline acetaminophen. The addition of diluents, such as dicalcium phosphate dihydrate, mannitol, or spray-dried lactose in 50% concentrations, and lubricants, such as magnesium stearate or talc in 2% concentrations, lowered the negative static charge. The significant lowering of the static charge by the addition of lubricants in relatively low concentrations suggests their possible importance as antistatic agents.

Particle size influences the hopper flow static charge. A 30–50-mesh size and a 20–30-mesh size separated from fine crystalline acetaminophen and ethylcellulose granulated fine powder acetaminophen, respectively, had higher negative hopper flow static charges than the granules from which they were separated.

The addition of water modifies the magnitude of the hopper flow static charge. Water, when added to crystalline, fine crystalline, and syrup granulated fine powder acetaminophen in 0.5% concentration, lowered the negative hopper flow static charge.

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