Explaining Electrostatic Charging and Flow of Surface-Modified Acetaminophen Powders as a Function of Relative Humidity Through Surface Energetics

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ABSTRACT: Powder flow involves particle–particle and particle–vessel contacts and separation resulting in electrostatic charging. This important phenomenon was studied for uncoated and dry-coated micronized acetaminophen (MAPAP) as a function of relative humidity. The main hypothesis is that by modifying powder surface energy via dry coating of MAPAP performed using magnetically assisted impaction coating, its charging tendency, flow can be controlled. The examination of the relationship between electrostatic charging, powder flow, and the surface energies of the powders revealed that an improvement in flow because of dry coating corresponded to a decrease in the charging of the particles. A general trend of reduction in both electrostatic charging and dispersive surface energy with dry coating and relative humidity were also observed, except that a divergent behavior was observed at higher relative humidities (≥55% RH). The uncoated powder was found to have strong electron acceptor characteristic as compared with the dry coated. The adhesion energy between the particles and the tubes used for the electrostatic charging qualitatively predicted the decreasing trend in electrostatic charging from plastic tubes to stainless steel. In summary, the surface energies of the powders and the vessel could explain the electrostatic charging behavior and charge reduction because of dry coating. © 2015 Wiley Periodicals, Inc. and the American Pharmacists Association J Pharm Sci 104:2225–2232, 2015

Keywords: surface energy; adhesion energy; cohesion energy; electrostatic charge; coating; moisture sorption; interaction; nanoparticles; structure property relationship; flowability

INTRODUCTION

Industrial powder handling operations involve particle–particle and particle–vessel contacts that can result in electrostatic charging of the particles. The charging can be classified as contact charging (particles brought into contact and separated) or tribo-charging (particles rubbing or sliding against each other or a surface). Electrostatic charging could be harmful or useful in industrial processes, for example, in the pharmaceutical industry, it can lead to powder flow problems, segregation,[1–3] and explosions,[4] whereas in xerography[5] and coatings,[6–8] particle charge can be used to control the movement and coating of the particles.

Electrostatic charging is a surface phenomenon, which depends upon various factors including humidity,[9] surface chemistry,[10] surface contamination,[11] and particle size.[12] Usually, Electrostatic charging is associated with different materials in contact; however, it has been shown that identical materials can charge one another as a result of the nature of contact between them[13] and as a result of different particles sizes.[14] Also, coating the surface of the equipment[15] or the particles[16] changes the charging behavior of some materials. Some of these coating processes however do not produce uniform surface coating and involve the use of solvents, which will have to be completely evaporated, thus complicating the process and are not cost-effective.

Dry coating with nanoparticles leads to the dispersion of nanoparticles on the surface of micron-sized particles, and has been shown to create uniform surface coating.[17–20] Therefore, it is potentially a very cost-effective alternative to wet methods while achieving better performance. The magnetically assisted impaction coating (MAIC) method is one of several dry coating techniques,[18] and was considered in the present study for its applicability to pharmaceutical powders, although other methods would be equally applicable. The presence of these nanoparticles on the surface of the particles is expected to change their electrostatic behavior.[21,22]

Because of the complex nature of electrostatic charging process, it is impossible to quantify the charge acquired by the particles unless the contact area is accurately obtained. However, it is possible to determine the differences between the dynamic behaviors of charged/uncharged particles of the same powder or coated/uncoated particles. For instance, surface properties such as the surface energy of the powders can be correlated to the electrostatic charge as both are surface properties that are affected by surface roughness,[23] as well as electron donating and accepting capacity. As an example, the electrostatic charge of pharmaceutical powders has been correlated with their acid–base characteristics[24] obtained from surface energy measurements using inverse gas chromatography (IGC). Such measurements would also allow for examining the stability of solid-state pharmaceutical products, which is highly affected by moisture. Unfortunately, there is very limited information on this topic in the literature to assess whether surface modification can indeed make a positive difference. Therefore, one of the goals of the current work is on evaluating the effect of moisture (relative humidity) on the material properties, such as electrostatic charge and relating that with surface energy

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for the purpose of providing an understanding of the sorption behavior of these materials.

The use of IGC has been established in nonpharmaceutical as well as pharmaceutical industries. For example, it has been used to analyze the surface energy of inorganic compounds, such as alumina,[25] silica,[26–28] carbon,[29] and aluminum,[30] and in organic pharmaceutical powders, it has been used to detect batch to batch variability[31] and solubility parameters.[32] IGC measurements are based on the physical adsorption of gaseous probes on the solid surface. From the elution peak, the retention time is obtained and is used to calculate the retention volume, which, in turn, allows the surface energy of the material to be calculated. The surface energy is considered to be consisting of two components: a Lifshitz–van der Waals (LW) dispersion component and an acid–base component. The IGC approach utilized in this paper employs methods proposed by Dorris and Gray[33] and Good et al.,[34] discussed in more detail later in the paper, for the determination of the dispersive and acid–base components, respectively.

The overall objective of this study is to alter the electrostatic behavior and surface energy of uncoated micronized acetaminophen (MAPAP) through dry coating, and thus to improve its flow and handling. The specific objectives are: (1) to relate the flow and electrostatic behavior of the uncoated and dry coated powders; (2) to correlate electrostatic charging data with the surface energetics of the uncoated and dry-coated powders; (3) to understand the effect of relative humidity on both surface energy and electrostatic charging of the powders; (4) to propose a new approach of using the adhesion energy between particles and the processing vessel to explain tribocharging. Towards these objectives, first, surface modification of MAPAP powders via MAIC was performed using hydrophobic silica, hydrophilic silica, or titania as guest nanoparticles. The electrostatic charge was measured using a simple apparatus employing powder flow through a tube discharged in to a faraday cup. Powder flow with and without dry coating was assessed through measuring the angle of repose (AOR). The surface energy of the uncoated and dry-coated MAPAP was characterized under different relative humidity conditions using IGC.

EXPERIMENTAL

Materials

Listed in Table 1 are the properties of the raw materials used in this study. The MAPAP was purchased from Mallinckrodt Inc., St. Louis, MO, hydrophobic silica (R972) was obtained from Evonik, Parsippany, NJ, and hydrophilic silica (M5) as well as titania were obtained from Cabot Inc., Boston, MA. The nanosilicas and titania particles were used as surface modifiers to coat the surface of MAPAP.

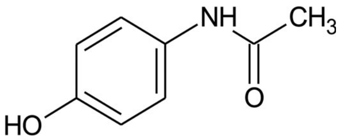

Figure 1. Molecular structure of acetaminophen.

The molecular structure of MAPAP as shown in Figure 1 consists of a core benzene ring, a hydroxyl group, and an amide group in para position to each other on the ring. It is highly conjugated and electrophilic because of charge delocalization, leading to decrease in basicity of the amide group and an increase in the acidity of the hydroxyl group.

Dry Coating Method

The surface modification was achieved through dry coating with the MAIC. The details of the dry coating method have been described elsewhere.[17,19] Briefly, the method employs the use of permanent magnets that spin and move rapidly under the influence of an oscillating magnetic field to disperse the nanoparticles on the surface of the MAPAP through fluidization and collisions between the magnets and the powders.

Electrostatic Charge Measurements

The set-up presented in Figure 2 was used to measure the electrostatics charge that is generated because of contact and frictional sliding of powders through foot long (or 30.48 cm) stainless steel, polycarbonate, polypropylene, or acrylic pipes. The same experiment was also performed without using any tube and is termed “direct” measurement. Prior to the start of the experiment, the powders and the set-up were stored in a temperature–humidity-controlled chamber at the required

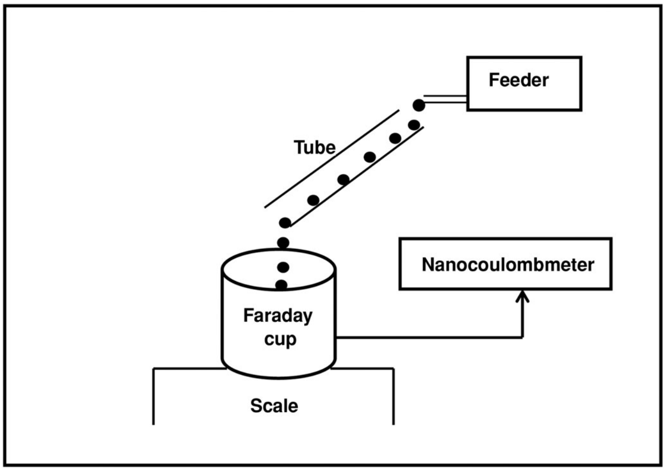

Figure 2. Set-up for measuring electrostatic charge of powders.

Table 1. Properties of the Raw Materials

Powder	D10	D50	D90	Particle Density (g/mL)	Dispersive Surface Energy (mJ/m^2)	Function
MAPAP	2.8 μm	11.2 μm	38 μm	1.29	–	Host
M5 (hydrophilic silica)	–	16 nm	–	2.20	38.26	Guest
R972 (hydrophobic silica)	–	16 nm	–	2.65	34.38	Guest
Titania	–	21 nm	–	4.23	34.60	Guest

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