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## Electrostatic Microencapsulation of Composite Particulate Materials for Manufacturing and Environmental Applications

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# Electrostatic Microencapsulation of Composite Particulate Materials for Manufacturing and Environmental Applications

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## Abstract

Electrostatic microencapsulation is a dry coating process where two powders, one containing the fines and the other relatively larger particles, are separately dispersed in air and pre-charged with opposite polarity, using corona charging for electrostatic coagulation. These oppositely charged core and guest particles experience attractive electrostatic forces and generate composite particles. Preliminary experiments of electrostatic microencapsulation were performed using Anionic Exchange Resin (AG 1-X8) as the host particle and Red Toner (Omega 4000) as the guest particles. An electrostatic microencapsulation tower has been designed for generation of composite particles using particles of different particle size distribution.

## Introduction

Microencapsulation of particles with different sizes and compositions is widely used in many industrial, pharmaceutical, agricultural, and consumer product applications. Examples include controlled-release drugs, sustained-release pesticides, slow-release fertilizers, and triggered-release cosmetics. In most cases, the outer shell protects the encapsulated ingredients until the material is needed. The release mechanisms include mechanical, thermal, chemical, dissolution, and other processes. Most of the products are made from liquid phase dispersion commonly termed wet particle coating. In some applications, it is not desirable to have a protective outer shell completely covering the core material. In those applications, the encapsulating material must be highly porous, but stable. The porosity of the outer shell allows the inner core material to come in contact with the surrounding fluid. The outer shell and the inner core materials have two different functions. A cluster of particles from two different materials, serving as a composite material, can also be used to perform different functions.

In many cases, the encapsulation of particles must be made using a dry process. Electrostatic microencapsulation is a dry process in which core particles can be coated with fine particles of different materials. The method employs a mixing process (Fig.1) with an oppositely charged relatively large size particle (called the host) and a fine particle (called the guest). The host forms the core and is coated with the guests, which form the outer layer. The electrostatic microencapsulation provides dry dispersion of powder and avoids mechanical mixing of the particles and generates composite particles by polydisperse coagulation of the

precharged host and guest particles. Research in the field of dry particle coating shows that simultaneous fluidization and mixing improve the efficiency of the coating (Pfeffer, 2001) in comparison to many other mechanical devices, such as Mechanofusion™ or Hybridizer™, which primarily work by the application of shear force on the host and guest particles to make them coalesce. The electrostatic microencapsulation technique demands an effective precharging of the host and guest particles. Corona charging is an effective precharging method, which combines both field and diffusion charging methods. A corona discharge is

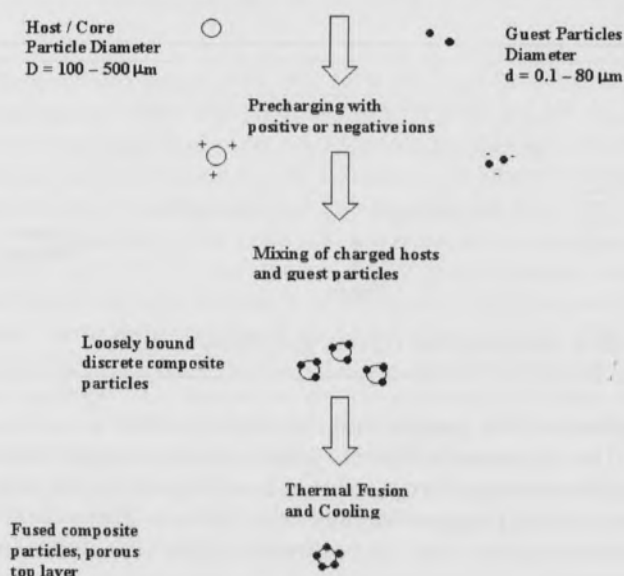


Fig. 1. Schematic of electrostatic microencapsulation technique to generate composite.

generated by dielectric breakdown of air or other gases due to the non-uniform electrostatic field generated between a needle and a grounded plate. The corona discharge generates electrons, which promote field and diffusion charging of particles present in the corona region. Corona charging also generates a stream of ions, called an "ion wind", from the point to the grounded plane. The corona gun (Fig. 2) is an effective device to disperse particles and generate highly charged particles. Powder is fed through a vibrating feeder into a diffuser. A regulated airflow through the diffuser conveys powder through a region rich in ions from a corona electrode, which is maintained at a high voltage and generates highly charged particles. The interparticle attachment force that holds the host and the guest particles is a summation of the London-van der Waals force, the electrostatic force, and the liquid bridge force (Hinds, 1999a). The above forces are dependent upon several factors including material, shape, surface roughness, relative humidity, and temperature. The London-van der Waals forces, which are short-range forces, are effective when two particles are very close to each other. These forces arise due to the random motion of electrons on the surface of the particle forming dipoles, which in turn induce an opposite polarity on the surface of another particle in close proximity. The resultant adhesive force between the particle and a plane surface can be estimated by the following equation:

$$F_{adh} = (AD) / (12X^2) \quad (1)$$

where A is the Hamaker constant, which depends on the material involved and ranges from  $6 \times 10^{-20}$  to  $150 \times 10^{-20}$  J for common materials, D is the diameter of the particle, and X is the separation distance, that depends on the surface

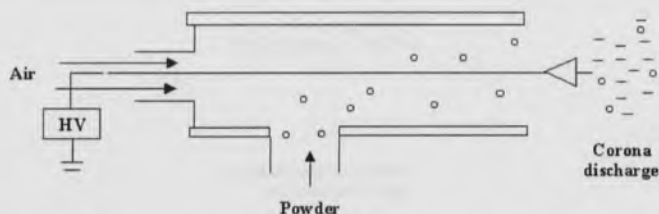


Fig. 2. A conventional corona gun (Bailey, 1998).

roughness of the particle and the contact surface.

The electrostatic forces, which are long-range forces, arise due to image forces induced by any particle carrying a charge when it approaches any other surface. The attractive coulombic force can be estimated from the following equation:

$$F_E = (K_E Q^2) / X^2 \quad (2)$$

where  $K_E$  is a constant of proportionality ( $9 \times 10^9 \text{ N}\cdot\text{C}^2/\text{m}^2$ ), Q is the charge of the particle, and X is the separation

distance of opposite charges.

The capillary force between a surface and a particle is created by the surface tension of the liquid drawn into the capillary space at the point of contact. The force between a particle and a plane surface at relative humidities higher than 90 % can be estimated from the following equation:

$$F_{bridge} = 2 \pi \gamma d \quad (3)$$

where  $\gamma$  is the surface tension of the liquid, and d is the diameter of the particle. For low humidity, the liquid bridge force is dependent on the curvature of the asperities at the point of contact and not the particle diameter.

In addition to long-range forces, the interaction of oppositely charged host and guest particles is dependent on polydisperse coagulation (Hinds, 1999b). The rate of change of number concentration of host and guest particles or of the rate of composite formation can be estimated from the following equation:

$$dN/dt = - K_{12} \beta N^2 \quad (4)$$

where N is the number concentration of particles,  $K_{12}$  is the coagulation coefficient (which is dependent upon particle diameter), and  $\beta$  is a correction factor dependent on particle charge and diameter. The coagulation coefficient for interaction of particles of two different sizes,  $d_1$  and  $d_2$ , can also be estimated from the following equation:

$$K_{12} = \pi (d_1 D_1 + d_1 D_2 + d_2 D_1 + d_2 D_2) \quad (5)$$

where  $D_1$  and  $D_2$  are the diffusion coefficients of the two particles respectively.

The correction factor  $\beta$  for thermal coagulation of aerosols having charged particles  $+q_1$  and  $-q_2$  was estimated by Fuchs (1964) to be:

$$\beta = \lambda_{12} / \{ \exp(\lambda_{12}) - 1 \} \text{ and } \lambda_{12} = q_1 q_2 / (2rkT) \quad (6)$$

where r is the radius of the particle, k is the coagulation constant, and T is the absolute temperature. It can be concluded that a high charge-to-mass ratio and lower number concentration of host and guest particles would improve the efficiency of electrostatic microencapsulation technique. Works performed by Borra et al. (1999) show that electrostatically induced bipolar coagulation is an efficient process to generate "tailored" particles by dispersing oppositely charged droplets and micro-mixing of particles inside the droplets.

## Materials and Methods

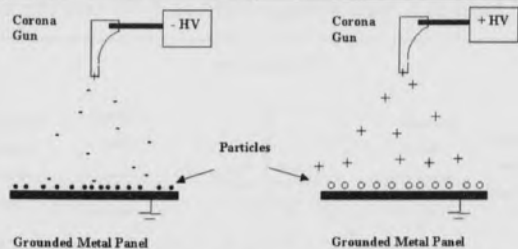
Composite particles were generated using AG 1-X8 Resin (Catalogue No. 140-1441), manufactured by Bio-Rad Laboratories (Richmond, CA), as the host particles and Omega 4000 Red Toner, manufactured by AEG Olympia (Somerville, NJ), as the guest particles. These host and guest particles were chosen because of their ready availability and low cost. Corona charging of particles was performed to generate charged host and guest particles of opposite polarity. A corona-charging device (Nordson® Versa Spray®) was used, where the particles were exposed to

positively or negatively charged ions for a few seconds (Fig. 3). The ions collide with the particles, sticks to them, and cause the particles to attain the charge. The charged host and guest particles were then transferred into a steel tumbler for mixing. The charge decay characteristics of the particles were studied using an electrostatic voltmeter held close to the surface to monitor the surface potential. When the particles were sprayed with positive ions, the electrostatic voltmeter measured a positive surface potential and vice versa when sprayed with negative ions. The charge-to-mass ratio of the particles was measured.

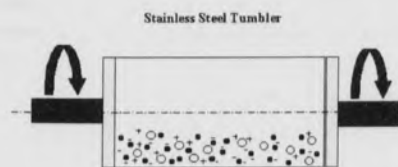
Mixing of host and guest particles in different mass ratios were investigated. The mixing was performed in a stainless steel tumbler of 8 cm. diameter and 11 cm. long rotated at approximately 50 – 60 rev / min (Fig. 3). Experiments were performed using neutral host and guest particles, only charged guest particles and uncharged host particle, and oppositely charged host and guest particles. The host and the guest particles were tumbled for 10 to 20 minutes to form a loosely bonded composite powder. The mixture of loosely bonded composite particles were transferred to a metal panel and placed in an oven maintained at 100°C. The particles were then removed from the oven after 1, 3, and 5 minutes and observed under an optical microscope.

## Results and Discussion

The particle size range of the as received resin and red toner powders (Table 1) was measured using MICROTRAC®. Both resin and red toner particles were widely spread with  $d_{50}$  of around 133  $\mu\text{m}$  and 15  $\mu\text{m}$  respectively. The MICROTRAC® was calibrated using



Step 1 - Corona Charging of Host and Guest Particles placed on a grounded metal



Step 2 Mixing of Pre-charged Host and Guest Particles

Fig. 3. Preliminary experiments of electrostatic micro-encapsulation.

fluorescent particles of 10  $\mu\text{m}$  size (Duke Scientific), which showed a  $d_{50}$  of 10.13  $\mu\text{m}$  ( $\sigma^2 = 2.28$ ). As received uncharged resin (AG 1-X8) and red toner (OMEGA) particles were mixed at different mass ratios (95%-5%, 96%-4%, and 97%-3%) and cured in an oven.

The objective was to find the mixing ratio that would yield the lowest  $d_{50}$  of the cured composite to exclude the effect of tribocharging with the stainless steel container. The cured mixture of uncharged resin and toner powders of

Table 1. Particle size distribution of resin and toner particles.

Parameters	AG 1-X8 ( $\mu\text{m}$ )	Red Toner (Omega) ( $\mu\text{m}$ )
$d_{10}$	55.43	9.94
$d_{50}$	133.80	15.60
$d_{90}$	180.05	21.17

Table 2. Particle size distribution of mixtures of uncharged resin and toner particles.

Conditions of Mixing (No Corona Charging)	$d_{10}$ ( $\mu\text{m}$ )	$d_{50}$ ( $\mu\text{m}$ )	$d_{90}$ ( $\mu\text{m}$ )
95% Resin, 5% Red Toner	29.74	104.77	162.24
96% Resin, 4% Red Toner	42.50	138.07	176.95
97% Resin, 3% Red Toner	124.12	147.18	173.17

different mass ratios (95%-5%, 96%-4%, and 97%-3%) were dispersed in water and analyzed in MICROTRAC® (Table 2). It was found that a resin-red toner mass mixture of 95%-5% yielded the lowest  $d_{50}$  (104.77  $\mu\text{m}$ ) and hence this ratio was chosen to be the ratio for the charging studies.

Precharging of the resin and toner particles was done at +70kV or -70 kV and charge decay measurements were performed for approximately 360 seconds. The host and the guest particles were found to retain their charge (Fig. 4) while they are being transferred to the stainless steel tumbler for mixing. The charge-to-mass of resin and red toner was found to be + 0.5  $\mu\text{C}/\text{gm}$  (corona charging at + 70 kV) and - 1.3  $\mu\text{C}/\text{gm}$  (corona charging at - 70 kV) respectively. The mixing of precharged particles generated composites (Fig. 5) with  $d_{50}$  of close to 145  $\mu\text{m}$ . The precharged particles were analyzed before (Table 3) and after (Table 4) being cured. No significant variation in  $d_{50}$  was observed due to curing, but significant change in  $d_{50}$  was observed between the uncharged (104.77  $\mu\text{m}$ ) and precharged (145  $\mu\text{m}$ )

formulations at 95% - 5% ratios. The resin particles were found to be stable up to 150°C and did not melt or deform. The toner particles melted at a few degrees above 100°C resulting in coverage of the entire surface area of the resin particle (Fig. 5).

### Conclusions

The preliminary experiments conducted with particles of  $d_{50}$  15 $\mu$ m and 133  $\mu$ m respectively did generate composite particles with a  $d_{50}$  close to 145 $\mu$ m. The composite particles had a single resin particle as the core and a cluster of red toner particles encapsulating the core.

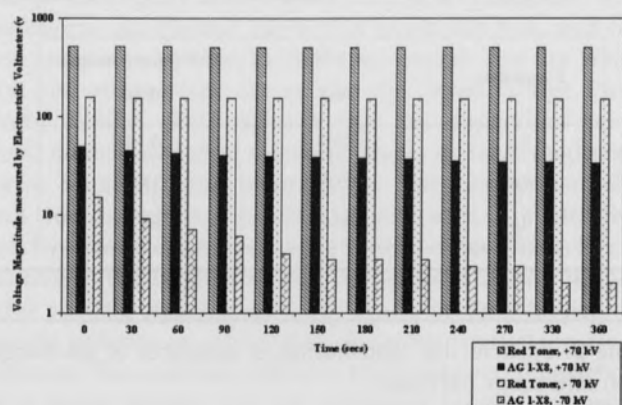


Fig. 4. Charge decay characteristics of red toner (OMEGA) and Resin (AG 1-X8) particles corona charged at +70 kV or -70 kV.

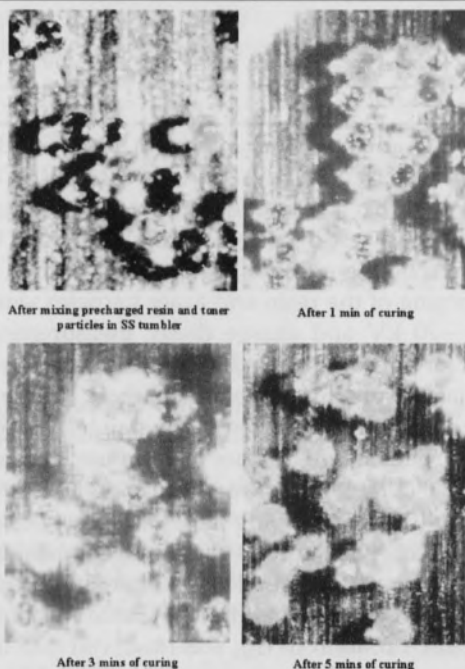


Fig. 5. Optical microscope pictures of AG 1-X8 resin particles and red toner particles.

Table 3. Particle size distribution of mixtures of corona charged resin and toner particles before curing.

Conditions of Mixing	$d_{10}$ ( $\mu$ m)	$d_{50}$ ( $\mu$ m)	$d_{90}$ ( $\mu$ m)
95% Resin, 5% Red Toner, Toner charged at -70kV	118.42	145.81	173.04
95% Resin, 5% Red Toner, Resin charged at +70kV, Toner charged at -70kV	104.40	142.57	178.27

Table 4. Particle size distribution of mixtures of corona charged resin and toner particles after curing in oven at 100°C for 5 minutes.

Conditions of Mixing	$d_{10}$ ( $\mu$ m)	$d_{50}$ ( $\mu$ m)	$d_{90}$ ( $\mu$ m)
95% Resin, 5% Red Toner, Toner charged at -70kV	125.82	147.61	171.75
95% Resin, 5% Red Toner, Resin charged at +70kV, Toner charged at -70kV	123.28	146.69	173.78

These composite particles were found to be stable when dispersed in water. Based on these preliminary findings, an electrostatic microencapsulation tower (Fig. 6) has been constructed, where the host (100 to 500  $\mu$ m) and guest (0.1 to 80 $\mu$ m) particles can be used to generate composite particles.

The electrostatic microencapsulation tower was designed to simultaneously disperse and charge host and guest particles using two corona guns (Nordson® Versa Spray®) held at a high voltage (one at + 70 and the other at

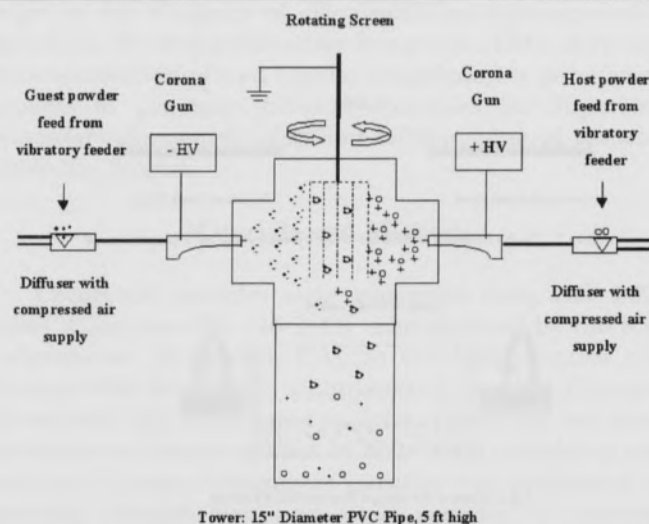


Fig. 6. Proposed electrostatic microencapsulation tower.

- 70 kV). Negative and positive corona guns are used to generate oppositely charged host and guest particles. A grounded rotating screen was installed inside the tower at the middle of two charging corona guns. A screen was used to collect the composite particles at the bottom of the tower. A jet of air was used to dislodge the particles from the screen and the composite particles were cured to promote bonding.

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