Removal of Fine Powders from Film Surface. I. Effect of Electrostatic Force on the Removal Efficiency

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A novel fine particle removal system composed of a corona-discharge neutralizer, a pulse-jet air unit and an image processing system has been developed. First of all, adhesion force between particle and film was directly measured and effect of electrostatic force on the adhesion force was calculated experimentally and theoretically. The electrostatic force was found to be significant, leading to the suggestion that the countermeasure for the electrostatic force was required to effectively remove fine particles. This system was then applied to the removal of fine particles from surface of a gelatin film used for conventional capsule material. The number of particles removed by the system was calculated by an image processing system and number base removal efficiency was computed with and without the elimination of electrostatic charge by the neutralizer. It was found that the difference between the removal efficiency of particles with elimination of electrostatic charge and that of without the elimination showed linear relationship with the electrostatic adhesion force. The data confirmed the necessity of electrostatic charge elimination for the effective removal of fine particles.

Key words removal; fine particle; electrostatic force; charge neutralizer; corona discharge

Electrostatic charge causes many troubles even in the manufacturing processes of pharmaceutical oral dosage forms. Especially in the capsule filling, film packaging and tabletting processes, sticking of fine particles onto the product surfaces occurs, leading to the stain on lens of video automatic inspection machines, unpredictable movement of electronic devices or machines, and deteriorates of product quality. In order to prevent these problems, removal of fine particles from the product surface is required.

So far, several devices have been used to remove fine particles from material surface. Otani *et al.*[1] used pulse air jet to remove fine particles from wafer surface. Shimada *et al.*[2] used pulsating air stream to remove pharmaceutical fine particles from film or capsule surface and reported that the particles smaller than 10 μm were difficult to be removed. Also, they reported that the countermeasure for electrostatic charge would be very important to reduce fine powder adhesion.[3] Generally, van der Waals and electrostatic forces mainly cause adhesion of fine particles, and the latter is remarkable if the particle is easy to be electrically charged. In this case, the removal of fine particles is difficult, because of the re-adhesion due to the electrostatic force. Therefore, elimination of electrostatic charge is necessary to completely remove fine particles from material surface.

In this study, a fine particle removal system composed of a corona-discharge neutralizer, a pulse-jet air unit and an image processing system has been developed and applied to the removal of fine particles from film surface. The effect of electrostatic force on the adhesion force between particle and film was investigated theoretically and experimentally. The relationship between electrostatic adhesion force and particle removal efficiency was also investigated.

Experimental

Powder Samples A gelatin film widely used for a conventional gelatin capsule was adopted as film material. For powder samples, cornstarch, talc and gelatin particles were used (Table 1). The gelatin particles were prepared by grinding the gelatin lump (2 to 5 mm), which was used for the gelatin film. The size ranges of these particles were almost the same. Prior to the experiments, powder samples and gelatin film were dried in a shelf drier under the conditions of 323 K and 24 h.

Equipment Figure 1 shows a schematic diagram of experimental setup. The experimental system composes of a corona-discharge neutralizer, an x–y stage and an image processing system.[4] Figure 2 illustrates a schematic diagram of a novel corona-discharge neutralizer. This neutralizer ionizes air molecules (supplied by a pneumatic air source) by using a corona discharge generated between a needle electrode and a nozzle cap. The nozzle launches positive and negative ionized air repeatedly at a cycle of 1/120 s as a commercial AC 100 V (60 Hz) is used for the electric supply. The launched ionized air molecules stick to electrical charged object and neutralize the charge. It is noteworthy that an electric resistance of $1.0\times10^{8}\,\Omega$ is installed to suppress the corona discharge not to be a cause of ignition or fire.

Table 1. Powder Samples

Powder samples	Size range (mass median size)
Cornstarch[a)]	10—30 μm (15 μm)
Talc[b)]	15—25 μm (18 μm)
Ground gelatin[c)]	15—25 μm (17 μm)

a) Nippon Shokuhin Kako Co. Ltd. (Cornstarch W). *b*) Nippon Talc Co. Ltd. *c*) Shionogi Qualicaps, Co. Ltd.

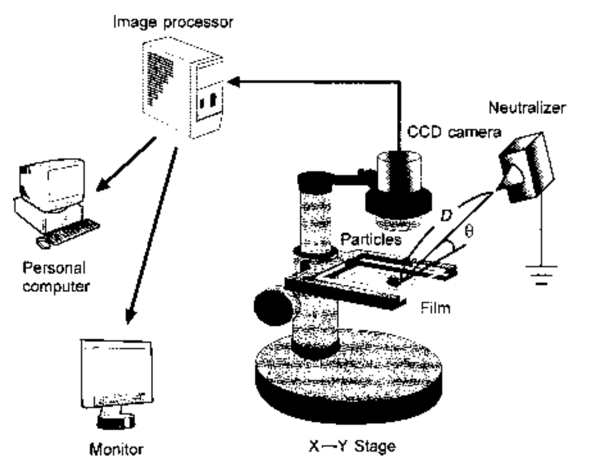

Fig. 1. Experimental Set-up

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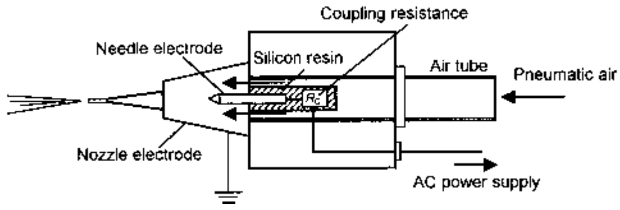

Fig. 2. Schematic Diagram of a Novel Corona-Discharge Neutralizer

Table 2. Operating Conditions

Number of particles	300
Distance between nozzle and film, *D*	30 mm
Number of pulses	4
Air (jet) pressure	0.15 MPa
Duration of air jet	0.5 s
Jet interval	0.5 s
Jet spray angle, θ	30 deg.

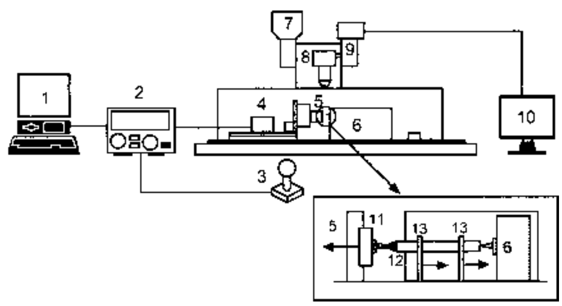

Fig. 3. Coherent Strength Measuring System

1, personal computer; 2, controller; 3, joystick; 4, motor; 5, stage; 6, laser displacement unit; 7, still camera; 8, microscope lens; 9, CCD camera; 10, video monitor; 11, sample; 12, contact needle; 13, flat spring.

The fine particle removal experiment was conducted as follows. A gelatin film (25×25 mm) was placed flat onto the x–y stage, and then approximately 300 particles were dropped inside a circle of 10 mm diameter on the film through a vibrated wire mesh having 53 μm (280 mesh) opening. The use of the vibrated wire mesh was to uniformly disperse the fine particles. The fine particles were removed by the neutralizer and then the number of fine particles remaining inside the circle was measured by using the image processing system.[4] The size distribution of the remaining particles and the number base removal efficiency was computed *via* a personal computer connected to the image processing system. The operating conditions for the fine particle removal experiments are listed in Table 2.

Figure 3 illustrates an apparatus for measuring adhesion force between single particle and a gelatin film directly (PAF-300, Okada Seikou).[5] This system composes of an accurate flat spring and a laser displacement sensor. A computer calculates the force required to peer off the particle from the film by directly measuring the displacement of the flat spring connected to a contact needle when the particle is about to leave from the film.

Figure 4 describes a measurement apparatus for contact potential difference (Contechter CPD-1000, Sankyo Pio-Tech).[6] The electrical force arisen when two materials contact each other can be calculated based on the electrical potential. This value is independent to material and can be measured as a contact potential.

As shown in Fig. 4, the equipment composes of movable upper/lower electrodes, electric shield box with temperature and humidity controllers, electrometer and computer. The upper electrode is made of gold and contact potential difference between gold and sample ($V_{\mathrm{Sample/Au}}$) can be measured. Based on the following equation, the apparent contact potential difference V_0 of the measuring sample can be calculated

$$V_0 = V_{\mathrm{Sample/Au}} + \frac{\rho_a}{2\varepsilon_a} d^2 \tag{1}$$

where, ρ_a, ε_a and d indicate electric volume density,[7] dielectric constant, and

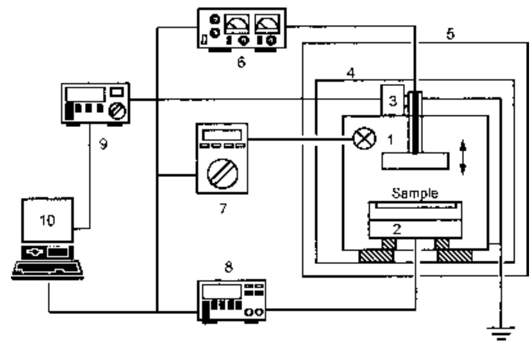

Fig. 4. Experimental Apparatus for Measuring Contact Potential Difference

1, upper electrode; 2, lower electrode; 3, motor; 4, electric shield box; 5, desiccators; 6, DC-power supply; 7, temp./humid. sensor; 8, electrometer; 9, upper electrode control unit; 10, PC.

thickness of the sample bed, respectively. The apparent contact potential difference V_0 changes with temporal change in the electric volume density (ρ_a) of the sample due to the electric relaxation, and finally becomes constant when the ρ_a reaches 0. The apparent contact potential difference V_0 at the constant value ($\rho_a=0$) is equivalent to the sample's contact potential difference ($V_{\mathrm{P/Au}}$) against gold upper electrode. Thus the sample's contact potential against gold can be measured by measuring the apparent contact potential difference.

A work function of the sample can be calculated by the following equations,[7]

$$V_{\mathrm{Sample/Au}} = \frac{\phi_{\mathrm{Au}} - \phi_{\mathrm{sample}}}{e} \tag{2}$$

thus,

$$\phi_{\mathrm{sample}} = \phi_{\mathrm{Au}} - eV_{\mathrm{Sample/Au}} \tag{3}$$

where, ϕ_{Au} and ϕ_{sample} show work functions of gold (4.78 eV) and sample respectively, and e indicate charge of electron (1.6×10^{-19} C).

Results and Discussion

Measurement of Coherent Strength When two dry particles contact each other, coherent strengths such van der Waals force and electrostatic force arise. Both of the forces can be described as follows.

van der Waals force:

$$F_{\mathrm{vdw}} = \frac{H}{6Z_0^2} r \tag{4}$$

Electrostatic force:

$$F_{\mathrm{el}} = \pi\varepsilon\varepsilon_0 \frac{\Delta V^2}{Z_0} r \tag{5}$$

Here, H, Z_0, r, ε_0 and V indicate Hammker constant (1.0×10^{-19} J), distance between two atoms (4.0×10^{-9} m), radius of particle, dielectric constant of air and contact potential difference between two materials, respectively. The ΔV between materials 1 and 2 is calculated based on Eq. (6).

$$\Delta V = \frac{|\phi_1 - \phi_2|}{e} \tag{6}$$

where ϕ_1 and ϕ_2 show work functions of materials 1 and 2, respectively.

By using the equipment described in Fig. 4, the work func-

Table 3. Measurement Results of Work Function

Material	Cornstarch	Gelatin	Talc
Work function (eV)	4.75	3.23	5.24

Table 4. Coherent Strength between Cornstarch and Gelatin Film

	Coherent strength
Theoretical (N)	8.56×10^{-8}
Experimental (N)	8.36×10^{-8}

Radius of cornstarch, r: 5 μm.

tions for cornstarch particle, talk and gelatin film can be calculated. The results are shown in Table 3. Based on the data, the electrostatic force can be actually calculated by substituting ΔV into Eq. (5).

Figure 5 indicates electrostatic and van der Waals forces of talc and cornstarch particles against the gelatin film as a function of particle radius. Here, the effect of gravity on the coherent strength was ignored. In addition, comparison between the measured and calculated values of total coherent strength of cornstarch particle against gelatin film is shown in Table 4. Here, the radius of cornstarch was determined as 5 μm (diameter was 10 μm) for both experiment and calculation; for experiment, cornstarch particles having approximately 10 μm diameter were selected.

Seen from Table 4, the measured and calculated coherent strengths indicated a farley good agreement, which confirmed the accuracy of the theoretical calculation as well. As seen in Fig. 5, ratio of the electrostatic force among the total coherent strength was enormously large. In order words, when the electrostatic force is awfully large, elimination of electrostatic charge is necessary to increase the removal efficiency.

Figure 6 shows the removal efficiency of three kinds of particles against the gelatin film with and without the elimination of electrostatic charge by the novel neutralizer. In order to estimate the neutralization effect, the same amount of air was supplied to the neutralizer even if the electric supply was off (no ion generation).

Seen from the figure, the removal efficiency differed depending on the materials. It was because the surface conditions such as roughness and shape differed. In addition, the difference between the removal efficiency of neutralizer and that of air only increased awfully with an increase in the electrostatic force (see Fig. 5). To quantitatively investigate the effect of electrostatic force on the removal efficiency, the difference between the removal efficiency was calculated, and then plotted against the calculated electrostatic force (Fig. 7).

Seen from Fig. 7, the deference in the removal efficiency has a liner correlation with the electrostatic force. In the case that the electrostatic force between the particles and film was large, the removal efficiency increased remarkable when the neutralizer removed the electrostatic charge. Based on this concept, the deference in the removal efficiency should be zero when there was no electrostatic charge; in the case of removal of gelatin particles from the gelatin film surface, the electrostatic charge was theoretically zero because the work functions of gelatin particle and film were the same.

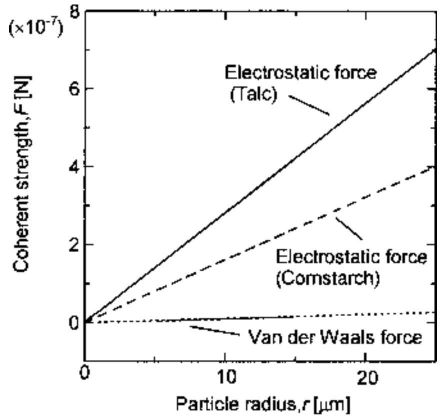

Fig. 5. Theoretical Calculations for Electrostatic and van der Waals Forces

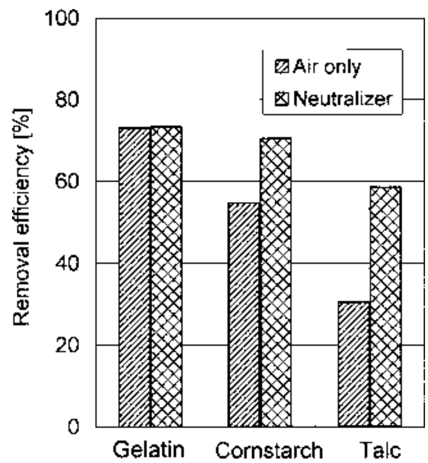

Fig. 6. Removal Efficiency

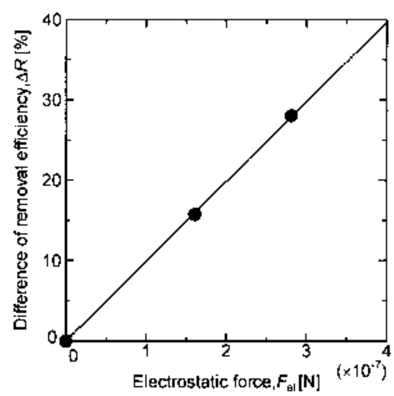

Fig. 7. Relationship between Difference of Removal Efficiency and Electrostatic Force

As the results, it can be pointed out that the elimination of electrostatic force is necessary in order to increase the removal efficiency of particles, which are easy to be charged. In the next paper, we will investigate the effects of the operating parameters of the neutralizer on the removal efficiency of fine particles against film.

Conclusions

A novel fine particle removal system composed of a corona-discharge neutralizer, a pulse-jet air unit and an image processing system has been developed and applied to the removal of fine particles from film. Adhesion force between particle and film was calculated theoretically, which agreed well with the measured value. The removal efficiency was measured with and without the elimination of electrostatic charge by the neutralizer. The difference between the removal efficiency of particles with the elimination of electric charge by the neutralizer and that of without the elimination was calculated, which showed a linear correlation with the electrostatic force. This confirmed the electrostatic force should be eliminated to increase the removal efficiency of particles, which were easy to be charged.

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