

Charging Characteristics of Spherical Mono-dispersed Particles

*Takahide Mizawa**, *Yoosuke Nakata**, *Yusuke Mizuno** and *Toshihiko Oguchi***
*: *Soken Chemical & Engineering Co., Ltd.*, **: *Morimura Chemicals Ltd.*
Tokyo and Kanagawa***, *Japan*

Abstract

Six types of spherical colored methylmethacrylate particles that have a particle diameter of 2, 3, 4, 5, 6, and 7 μm were prepared by seed polymerization. Obtained particles were completely separated mono-dispersed particles having a uniform diameter. The amount of tribo-charge q/m generated by mixing with reference particles having an irregular or a spherical shape was measured. The amount of q/m increased in proportion to the increase of the specific area of the mono-dispersed particles at a restricted particle concentration. With the combination of the spherical mono-dispersed particles and spherical reference particles, the tribo-charging characteristics can be measured reproducibly even when small mono-dispersed particles having a diameter of 2 μm are used. The combination can be used as an ideal standard mixture for the tribo-charge measurements.

Introduction

In most cases, toner particles that were prepared by a pulverizing process have an irregular particle shape and a broad particle size distribution. The surface of each toner particle were not uniform and the fine particle having a particle size less than 5 μm sometimes cause trouble in obtaining a reproducible amount of tribo-charge. Usually, the amount of tribo-charge q/m is measured for the mixture of toner and carrier particles by the blow-off method. In the mixture, the smaller toner particles preferentially cover the carrier particle surface and serves as a barrier for the contact of the larger toner particles to the surface.

In this report, six types of spherical mono-disperse methylmethacrylate particles having a particle diameter of 2, 3, 4, 5, 6 and 7 μm were used as model particles to investigate tribo-charging characteristics. The amount of q/m of the mixture of mono-dispersed particles and reference particles showed high reproducibility even when particles having such a small diameters as 2 μm are used. The effect of the size and concentration of the mono-dispersed particles to the amount of q/m are discussed.

Experimental

Preparation of the Mono-Dispersed Particle

The mono-disperse particles were prepared by seed polymerization using methylmethacrylate (MMA) seed particles and a black metal-azo-dye solution of a MMA monomer.¹ As an initiator, azobisisobutyronitril was used. Impurities such as remained dye and emulsifying agents deposited on the mono-disperse particle surface were washed out with ethyl alcohol and water. Obtained press cakes were dried and pulverized to acquire the mono-disperse particles. Six mono-disperse particle samples from CM-2 to CM-7 having a target particle diameter of 2, 3, 4, 5, 6, and 7 μm , respectively, were prepared and used for the experiment.

Reference Particles

As the reference particles for tribo-charge (q/m) measurements, two types of carrier particles usually employed for electrophotography were selected. One of them was an irregularly shaped iron particle (IF) the surface of which was treated with oxygen and nitrogen at high temperatures. The other was a spherically shaped ferrite particle (SF) the surface of which was coated with a fluoro-silicon compound. The diameters of both particles were ranging from 44 to 140 μm , and the average diameter was about 80 μm .

Evaluation of the Particles and Tribo-Charge Measurements

The shape and surface structure of the mono-dispersed particles and the reference particles were observed with an electron microscope (SEM: Keyence VE-7800) operated at low acceleration voltages. The particle distribution was measured with an image analyzer attached to the microscope VE-7800.

The amount of q/m was measured by the blow-off method according to the standard measurement procedure issued by ISJ.² Each combination of mono-dispersed particles and reference particles were mixed with each other at prescribed ratios. Twenty grams of mixed particles in a 100 ml uncapped polyethylene bottle were kept at a standard atmosphere of 25°C, 60%RH for 24 hours to adjust the moisture content. Then 200 times of hand shocks were given to obtain measurement samples. A blow-off apparatus equipped with a suction-blow type Faraday-cage (Toshiba

Chemical, Type TB-201) was used for the q/m measurement.

Result and Discussion

Observation of the Mono-disperse Particles and Reference Particles

Figure 1 shows the SEM photograph of the CM-5 mono-disperse particles (target particle size: 5µm). All particles have a perfect spherical shape and smooth surface.

The variation of the particle diameter for each mono-dispersed sample was less than 5%.



Figure 1. SEM photograph of mono-dispersed particles

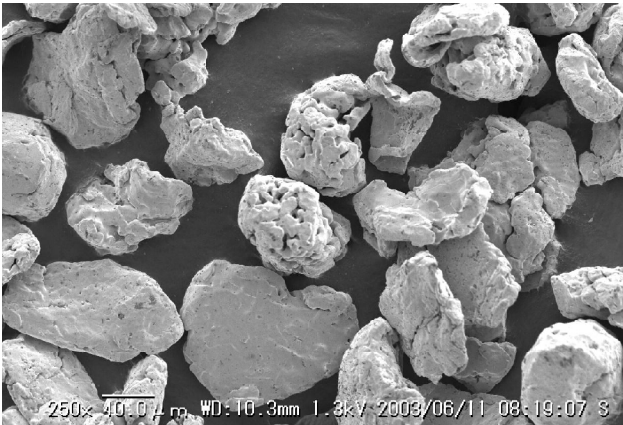


Figure 2. SEM photograph of irregularly shaped reference particle

The average diameter of CM-2 to CM7 particles measured by the image analyzer mentioned above is listed in table 1. The variation between the target diameter and the average diameter for each type of mono-disperse particles was 10%.

The SEM photographs of the irregular type reference particles (IF) and spherical type reference particles (SF) are shown in Figs. 2 and 3, respectively. The IF particle surface is rough and many cracks, dents and holes are present. The SF particle surface, on the other hand, is rather smooth and

small structures created by the sintering of ferrite powder in its manufacturing process are seen.

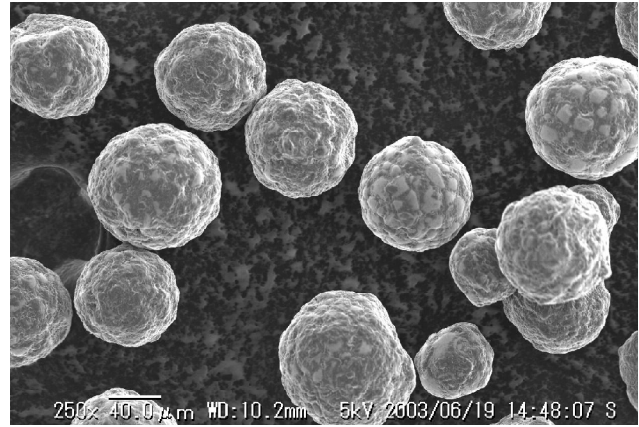


Figure 3. SEM photograph of spherically shaped particle

Table 1. Characteristics of mono-dispersed particles

	CM-2	CM-3	CM-4	CM-5	CM-6	CM-7
Target Diameter (µm)	2.0	3.0	4.0	5.0	6.0	7.0
Average Diameter (µm)	2.0	3.1	4.2	4.8	5.7	6.6
RSA	3.30	2.13	1.57	1.38	1.16	1.00
T _{c1} (Wt %)	5	5	5	5	5	5
T _{c2} (Wt %)	1.52	2.35	3.18	3.62	4.31	5.00

Amount of q/m on Each Mono-Dispersed Particle

Figure 4 shows the amount of tribo-charge (q/m) obtained for the mixture of the mono-dispersed particles and the reference IF particles. The q/m values were plotted against the relative specific surface area (RSA) of the mono-dispersed particle samples. The RSA value of each type of mono-dispersed particles is listed in Table 1. The values were calculated from equation (1) using the specific surface area of CM-7 as a unit.

$$RSA_{CM-X} = SA_{CM-X} / SA_{CM-7} = D_{CM-7} / D_{CM-X} \quad (1)$$

Here, RSA_{CM-X} is the RSA value of each type of mono-dispersed particles, SA_{CM-7} is the specific surface area of CM-7, SA_{CM-X} is the specific surface area of each type of mono-dispersed particles, D_{CM-7} is the average diameter of CM-7, and D_{CM-X} is the average diameter of each type of mono-dispersed particles.

In Fig. 4, the q/m plots in curve (a) were obtained for the mixtures containing the mono-disperse particles at a constant concentration of 5wt%. In this case, the amount of q/m remains nearly constant although the relative surface area (RSA) of the mono-disperse particles increases. The result suggests that, in the mixture containing larger RSA

(or smaller diameter) mono-disperse particles, the number of excess particles that cannot come in contact with the reference particle surface increases to reduce the q/m value. In other words, the particle concentration of 5wt% represents an excess concentration for the smaller size particles with a diameter less than 7 μm .

The q/m plots in curve (b) were obtained with the particle concentration of T_{c2} listed in Table 1. The values of T_{c2} were calculated using the equation (2) to obtain the same surface area as CM-7 in the mixture at the concentration of 5wt%.

$$T_{c2} = T_{c1} \cdot SA_{CM-7} / SA_{CM-X} = T_{c1} / RSA_{CM-X} \quad (2)$$

In this formula, $T_{c1} \cdot SA_{CM-7}$ is the surface area of CM-7 in the mixture at the concentration of 5wt%.

The q/m in curve (b) increases almost linearly with the increase of RSA. The result shows that, at the restricted particle concentration T_{c2} at which the surface area of the mono-dispersed particles is maintained constant, the amount of q/m increases proportionally with the increase of the specific surface area of the mono-disperse particles.

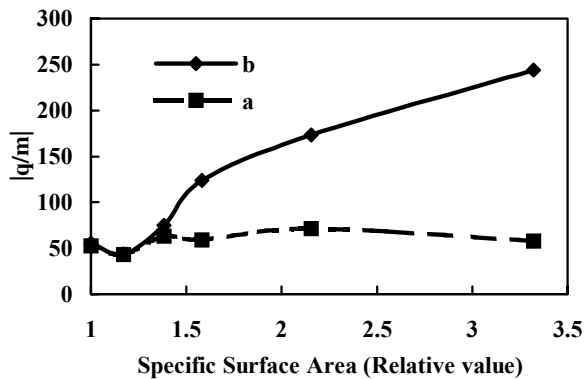


Figure 4. Relation between q/m and specific area of mono-dispersed particles (Reference particles:IF) Curve a: particle concentration is T_{c1} (5wt% const.) Curve b: particle concentration is T_{c2} in Table 1.

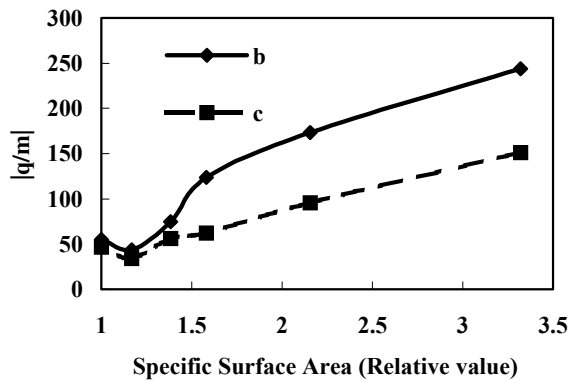


Figure 5. Relation between q/m and specific surface area of mono-dispersed particles. Curve c: Particle concentration is T_{c2} (Reference:IF); Curve b: Particle concentration is T_{c2} (Reference:SF)

Curve (c) in Fig. 5 shows the relation between T_{c2} and q/m obtained for the mixture of mono-disperse particles and the spherical reference particle (SF). Curve (b) of Fig.4 is also shown for comparison. The q/m increases linearly with the increase of RSA. In comparison with curve (b) that was obtained by using the irregular shape reference particles (IF), the linearity of curve (c) is improved.

The mixture of the mono-disperse particles and the spherical reference particles is regarded as the ideal combination for the investigation of tribo-charging characteristics between the two kinds of particles.

Amount of q/m and Mono-Disperse Particle Concentration (T_c)

The relation between mono-disperse particle concentration (T_c) and q/m was investigated by using the mixture of CM-5 particles and the two types of reference particles (IF and SF). The results are shown in Fig. 6. The q/m value remains constant in the low T_c region and then gradually decreases with increasing T_c . In the case of the mixture using the IF reference particles, the T_c region where a constant q/m value (q/m constant T_c region) was obtained lies between 0 and 3wt%. In the case of the mixture using SF reference particles, on the other hand, the T_c region where the q/m value stays constant ranges from 0 to 2wt%.

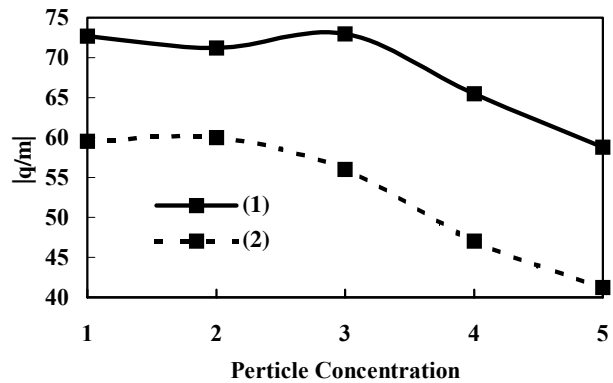


Figure 6. Relation between q/m and particle concentration. Curve 1: CM-5/IF mixture, Curve 2: CM-5/SF mixture

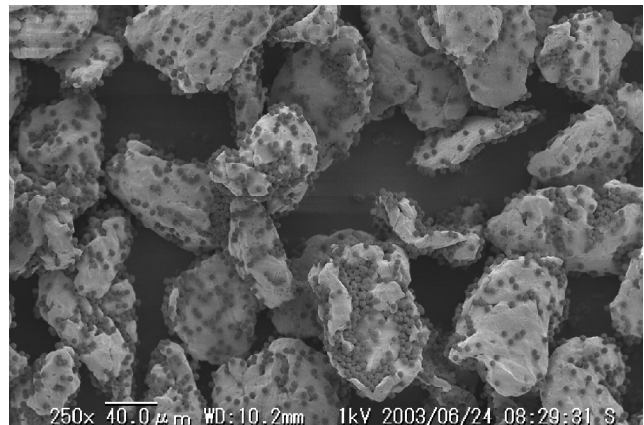


Figure 7. Mono-dispersed particle adhesion on IF particle surface

The difference of the q/m constant T_c region yielding a constant q/m value, in the two types of mixtures can be explained by the difference of surface space that can accommodate the adhering mono-disperse particles. The photographs of the CM-5 particles adhering on the IF and SF particle surface at 2wt% particle concentration are compared in Fig. 7 and Fig. 8.

In the case of the CM-5/IF mixture in Fig. 7, the CM-5 particles are adhering along the cracks or dents on the surface and there seems to exist more accommodation space for the CM-5 particles to adhere. In the case of the CM-5/SF mixture in Fig. 8, on the other hand, the accommodation space remaining on the SF surface seems to be smaller than that of the IF surface in Fig. 7.

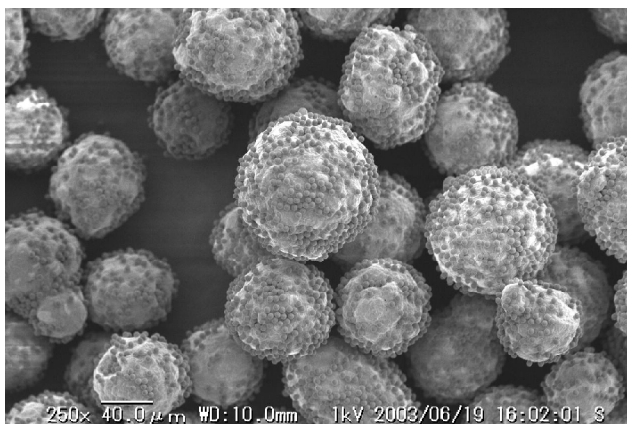


Figure 8. Mono-dispersed particle adhesion on SF particle surface

The accommodation space was also observed at 5wt% T_c . Actually no space was found on both reference particle surfaces and excess CM-5 particles were stacking on the surface or spilling over from the surface. More particles were spilling over from the SF particle surface than from the IF particle surface.

It was confirmed that within the T_c region where the q/m stays constant, all mono-disperse particles adhere on the reference particle surface and acquire a uniform amount of tribo-charge. In the T_c region showing decrease of q/m , some excess particles start to stack onto the reference surface while other excess particles start to spill over from the reference surface to make the q/m value gradually decrease.

Change of q/m by Agitation

The mixtures of CM-5/IF and CM-5/SF were prepared at 5wt% T_c and the amount of q/m was measured while the mixing time is increased. For the mixing apparatus, a paint conditioner was used. The shaking was very rigorous compared to that of shaking by hand.

The results were shown in Fig. 9. The q/m change for CM-5/SF mixture was very small during six hour shaking. The q/m change in the initial 1 hour shaking for CM-5/IF mixture was very large and the q/m amount (negative charge) was reduced to a half. As shown in Fig 6, in the case of CM-5/IF mixture, the mono-disperse particles adhering along the cracks or dents on the IF particle surface

easily stick to provide a spent particle layer and reduce the amount of q/m .

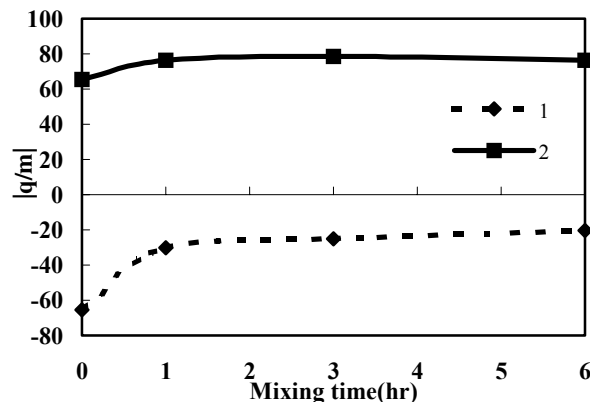


Figure 9. q/m change by mixing curve 1: CM-5/IF mixture, curve 2: CM-5/SF mixture

Conclusion

Six species of colored mono-disperse methylmethacrylate particles that have a target particle diameter of 2, 3, 4, 5, 6 and 7 μm were obtained. The particles are in spherical shape and have a smooth surface. The particles have a uniform diameter and the variation from the target particle diameter is less than 5%. The amount of tribo-charge q/m generated by mixing with reference particles having an irregular or spherical shape was measured. A large amount of q/m on the mono-disperse particle was obtained. At a restricted particle concentration at which the surface area of the mono-dispersed particle is maintained constant, the amount of q/m increases proportionally to the increase of specific area of the mono-disperse particles. With the mixture of the spherical mono-dispersed particles and spherical reference particles, the tribo-charging characteristics can be measured reproducibly even small sized particles having a diameter of 2 μm is used. The mixture will be an ideal standard material for the tribo-charge measurement

References

1. T. Mizawa, Y. Nakata, J. Powder Tech. Soc. Japan, 40, 16 (2003)
2. T. Oguchi, M. Kimura, N. Sawayama, C. Suzuki, Y. Takahashi, M. Takeuchi, T. Tada, and K. Hoshino, International Conf. on Digital Printing, 369 (2001)

Biography

Takahide Mizawa received his B. Sc. in 1995, and the Ph.D. Eng, in 2000 in polymer Eng, from Nagaoka University of technology. He has worked as the researcher at Soken chemical & Engineering Co., Ltd. since 2000. He is now developing the functional polymer materials and their application. He is especially interested in mono-disperse and color of the polymer particles.