CHAPTER 3

EXPERIMENTAL

3.1 Materials

3.1.1 Toner, two types of toner were used in this work.

Red toner is the mixture of a red color pigment and some styrene/acrylate resin with a mean diameter measured by the manufacturer of 14 μm, which is the commercially used one in the two-color copier such as Canon NP-4080.

Cyan toner is the mixture of a cyan color pigment and polyester resin with a mean diameter measured by the manufacturer of 8 μm, which is the commercially used one in the full-color copier such as Canon CLC 500.

3.1.2 Carrier, eight types of ferrite spherical carrier samples and one type of iron irregular carrier sample were used in this work.

Carrier A : fluorine/acrylate coated ferrite particles with a mean diameter measured by the manufacturer of 100 μm.

Carrier B : fluorine/acrylate coated ferrite particles with a mean diameter measured by the manufacturer of 70 μm.

Carrier C : fluorine/acrylate coated ferrite particles with a mean diameter measured by the manufacturer of 60 μm.

Carrier D : fluorine/acrylate coated ferrite particles with a mean diameter measured by the manufacturer of 50 μm.

Carrier E : acrylate coated ferrite particles with a mean diameter measured by the manufacturer of 70 μm.

Carrier F : silicone coated ferrite particles with a mean diameter measured by the manufacturer of 70 μm.

Carrier G : un-coated ferrite particles with a mean diameter measured by the manufacturer of 70 μm.

Carrier H : fluorine/acrylate coated iron particles with a mean diameter measured by the manufacturer of 70 μm.

Carrier K : fluorine/silicone coated ferrite particles with a mean diameter measured by the manufacturer of 70 μm.

3.2 Apparatus

E-SPART analyzer, model EST-II of Hosokawa Micron Corp, Japan.

Scanning Electron Microscope (SEM), S-4500 of Hitashi and JSM-6400 of JEOL, Japan.

Blow-off method, a home-made machine, comprising a Faraday cage, metal tube, screen mesh no. 635, digital electrometer of KEITHLEY model 6011 and air pump of a vacuum cleaner, TC-L 350 of MITSUBISHI, Japan.

Copier, a two-color Canon NP-4080 copier and a full-color copier of Canon CLC 500 copier, Japan.

Reflection densitometer, RD 914, 915 of Macbeth and Densitometer of TC-6MC, USA.

Image analyzer of LUZEX FS with a NIKON CCD video camera module and an OLYMPUS camera, Japan.

Differential Scanning Colorimetry, DSC 7 of PERKIN ELMER, USA.

Electronic balance of ER - 180 A and METTLER AE 240, USA.

Thermometer/Hygrometer and Humidity Control, HONEYWELL, USA.

3.3 Procedure

3.3.1 Determination of toner and carrier particle morphology

3.3.1.1 The toner and the carrier particles were analyzed for the morphology in term of the particle shape, the particle surface, and the average particle sizes from the micrographs taken by the SEM. For the size measurement, the micrographs were 3 time enlarged, and the particle diameters were measured by a digitized pen of the Size Distribution System in a home-made program of a conventional computer. The cyan toner particle size distribution were also measured with an E-SPART analyzer.

3.3.1.2 The color optical micrographs of the developers, the red and the cyan toners mixed with the carriers A - D at various toner concentrations, were taken by an optical microscope coupled with the CCD camera. These pictures were considered on the covering ratio of toners on carriers.

3.3.2 Determination of developer charge properties and the effective parameters

3.3.2.1 The red and the cyan toners were measured for the charge-to-mass ratios by the blow-off method with a specially constructed Faraday cage at 20 - 30°C and 50 - 60% RH as follows:

a) Twenty grams of the two-component developers were prepared by adding the toner concentrations each of 1, 3, 5, 8, 10 and 15% with a corresponding weight to make 100%.

b) The toner charging of each developers was measured by varying the hand-shaking time of 10, 20, 30, 60 and 120 seconds with a constant rate of 3 times per second before a charge measurement.

c) The blow-off method was prepared by mounting the metal mesh screen no. 635 (38 μm of the hole diameter) in the bottom of the cage.

d) The developers were weight about 0.2 g and put into the cage, and then the exact weight was recorded (W_d).

e) The cage was covered with the cap, placed on the suction table and connected with the electrometer.

f) The indication of the electrometer was adjusted to zero, the coulomb meter mode was selected .

g) The toner was blown off through the screen mesh with a pressure of about 250 mm.H_2O for 30 seconds which the charge was saturated.

h) The remained charge in carrier, opposite polarity from the toner charge, was measured and recorded in micro-coulomb unit.

i) The cage was disconnected and weight of the toner mass lost (W_t).

j) The toner charge-to-mass ratio (q/m) was calculated and recorded.

k) The measured T/C of developer was confirmed after blow-off measurement as Equation 3-1. The measured T/C value divided by the mixing T/C value should be ≤ 1. If this value > 1, it means that some carrier particles pass through the screen mesh, and the toner q/m value are not correct.

$$\text{Measured T/C} = W_t / W_d \times 100 \qquad (3\text{-}1)$$

l) The triplet experiments were carried out and the result was repeated as the average value.

3.3.2.2 The charge distribution (q/d) of the cyan toner was measured in some cases with an E-SPART analyzer which was prepared by the following steps:

a) Calibration of the analyzer: monodispersed polystyrene standard with a particle size of 3.09 μm was used as a size calibration of the E-SPARTs, with 3.9×10^{4} N/m^{2} pressure under a 5×10^{-5} m^{3}/s gas flow and a zero volt field voltage.

b) The developer was put in the glass tube and was flown to adhere on the rotating magnetic wheel.

c) The wheel containing the magnetic brush of the developer was slowly rotated to pass a nitrogen blow nozzle.

d) The toner particles were separated from the carrier bead and fallen down passing a vibration electrode.

e) The toners with the different charges were fallen down with different deflected distances by the field voltage of the vibration electrode, and were detected by the laser beam.

f) The field voltage value was adjusted to 50 volts for the high charge toner, but was adjusted to 100 volts for the low charge toner.

g) The maximum count of 3,000 toner particles was measured for the toner charge per diameter (q/d), the particle size distribution, and the charge distribution, by calculating the deflected angles, and then the q/m values were also obtained. The data is shown in Appendix A.

3.3.2.3 Determination of the dependence of toner charge on the toner concentration (T/C), the shaking time, the carrier size, the carrier surface coating, and the carrier core particle.

3.3.3 Analysis of copy print quality

3.3.3.1 A Canon NP 4080 copier, the currently commercial available machine in Japan, was set up and calibrated for a standard tester of red color developer. The color developer (CD) unit was adjusted to a maximum level of VR851 and VR852 in order to be printed with too low toner concentration.

3.3.3.2 The single-color red copies were produced from a test form on the uncoated papers, PB paper QKD A4, and the special transparencies by the NP 4080 copier, as following steps:

a) One hundred and fifty grams of the red developer were prepared for the carriers A - G, with toner concentrations each of 1, 3, 5, 8, 10, and 15%. Carrier H, the high conductive carrier, is unable to be tested with the NP-4080 copier.

b) The developer was mixed and hand-shaken for the optimum shaking time, which was determined by the charge measurement.

c) The red color developer was then poured into the CD unit.

d) The magnetic sleeve was rotated to be totally covered by the developer.

e) The developer mass per area on the magnetic sleeve was controlled by adjusting the gap between the blade and the sleeve.

f) The CD unit was installed in the copy machine before turning on the power switch.

g) The control panel of the copier was set at the medium exposure keys, and one copy of red color was printed.

h) After one copy had made, the power switch was turned off and turned on again to control the same condition of the copier.

i) The remainder of developer in the CD unit was poured out and the CD unit was cleaned before changing the developer.

3.3.3.3 A Canon CLC 500 full-color copier, the currently commercial available machine, was set up and calibrated for a standard tester of cyan color developer. The single-color cyan copies were produced by the CLC 500 copier with some different parameters from the red copies, as follows:

a) Two hundred grams of the red developer were prepared for the carriers A - D, with toner concentrations each of 2, 5, 8, 12, 15 and 18%.

b) The developer was machine-shaken with 250 rpm.

c) The developer mass per area of about 45 - 55 mg/cm^2 on the sleeve surface was controlled by adjusting the gap distance between the blade and the magnetic roller of about 0.6 - 0.8 mm.

3.3.3.4 Densities of copy prints were measured with a reflection densitometer at the solid area and the background. The solid density was measured by using the green filter for red copy and the red filter for cyan copy. The background density was measured by calibration on the reference white paper. The optimum quality of the copy was determined for producing the maximum density in

the solid area higher than 1.35 for red copy and higher than 1.8 for cyan copy, with a background density lower than 0.03.

3.3.3.5 Fourteen continuous gradation levels was observed for the tone reproduction and the print contrast by measuring the copy densities and plotting versus the original densities.

3.3.3.6 The print resolution was determined by measuring the lines width at 5.0 lines per millimeter in the copy with an analyzed system of an image analyzer as the following steps:

a) The copy sheet was placed below the optical lens of the videomicroscope with 80 magnification.

b) The distance of a micrometer was calibrated by a standard ruler.

c) The lines of 5 l/mm were captured and displayed.

d) The function of fiber length was used and the length of line width was measured about 50 points to find a mean length.

3.3.3.7 The 14 halftone gradation levels of the copy was measured for the dot gain with the reflection densitometer by calibration of 0% dot on the white paper and 100% dot on the solid area.

3.3.4 Analysis of thermal behavior of the toners

Thermal behavior of the toner resins was examined by DSC to obtain the glass transition temperature or the melting temperature, which affect on the fixing properties of the toner, as the following procedures:

a) The toner sample was put into the aluminum pan with 5.63 mg for the red toner and 5.5 mg for the cyan toner.

b) The DSC was run from 30°C to 250°C, with the heating rate of 20°C /min.

c) Thermal energy and Tg values were evaluated by the Thermal Analysis System.