# CHAPTER III

# EXPERIMENTAL

## 3.1 Materials

3.1.1 Four types of polymerized toner: Cyan, Magenta, Yellow and Black toner

3.1.2 Three types of carriers: TSV-200, F-150 and Z-250

3.1.3 Mesh screen: mesh screen diameter of 25 micrometers, 500 mesh/inch

## 3.2 Apparatus

3.2.1 Rolling type of toner charger: MS 1 Minishaker, IKA-WORK, Inc., Wilmington, USA

3.2.2 Horizontal rotator (home made)

3.2.3 Blow-off measurement unit comprising two important units:

3.2.3.1 A vacuum cleaner: VC-K50K 500, TOSHIBA, Chiba, Japan

3.2.3.2 Electrometer: R8240 Digital Electrometer, Advantest Corporation, Tokyo, Japan

3.3.3 E-SPART analyzer: EST-1, Hosokawa micron Corporation, Osaka, Japan

3.3.4 Electronic balance: Mettler PM 2500 Delta Range, Switzerland

3.3.5 Image analyzer: LUZEX F, PM 10-AD, Olympus, Nireco Corporation, Tokyo, Japan

3.3.6 Scanning Electron Microscope: JSM-5800LV, JEOL, Tokyo, Japan

3.3.7 Differential Scanning Calorimeter: DSC 822, Mettler Toledo, Switzerland

3.3.8 Fourier Transform Infrared Spectroscopy: Impact 410, Nicolet Instrument Corporation, USA

3.3.9 X-ray fluorescence: PW 2400, Philips, Netherlands

3.3.10 Densitometer: RD 915, Macbeth Corporation, Newburg, USA

3.3.11 Spectrophotometer: X-Rite SP 62, X-Rite Incorporated (Color Shop program), Granville, USA

3.3.12 Printer: Canon Laser Shot LBP-2710, Canon, Japan

3.3.13 Printer: Fuji Xerox Color Laser Wind 3310, Fuji Xerox, Japan

3.3.14 Spatula

3.3.15 Forceps

3.3.16 Glass bottle: diameter of 32 millimeters



## 3.3 Procedure

### 3.3.1 Developer preparation

The developers (toners and carriers) are different in component and concentration (wt%). They are shown in Table 3-1.

Table 3-1: Developers with different compositions and contents

| Toner | Carrier | Concentration (wt%) |
|---|---|---|
| Cyan | TSV-200, F-150, Z-250 | 1, 3, 5, 7, 10 |
| Magenta | TSV-200, F-150, Z-250 | 1, 3, 5, 7, 10 |
| Yellow | TSV-200, F-150, Z-250 | 1, 3, 5, 7, 10 |
| Black | TSV-200, F-150, Z-250 | 1, 3, 5, 7, 10 |

### 3.3.2 Measurement of toner charging properties

3.3.2.1 Toner charge dependence on toner concentration (wt%)

The toners (cyan toner) and carrier (TSV-200) were mixed together with 1, 3, 5, 7, and 10 wt% of toner by Minishaker at the rotating speed of 800 rpm. The Minishaker was stopped after 15, 30, 60, 90, 120, 240 and 360 seconds of mixing in order to measure the q/m by a blow-off measurement unit that was connected to an electrometer and a vacuum cleaner.

The measurement of q/m by blow-off method was carried out as follows:

- The developer was poured into a cage of the blow off measurement unit, which had a metal mesh size of 25 micrometers on the bottom.
- The cage with the developer was weighed by an analytical balance.
- The toner was blown off through the mesh screen for 15 seconds.

- The q value was read from the electrometer.
- The cage with the developer was weighed again to determine the weight of the toner only.
- The toner charge-to-mass ratio (q/m) was calculated.
- The developer was experimented again 5 times.
- The developer was changed by mixing the carrier and magenta, yellow and black, respectively. They are shown in Table 3-2. The charge-to-mass ratio was determined using the same conditions and the same measurements as mentioned above.

Table 3-2: Developers for toner charge dependence on toner concentration

| Toner | Carrier | Concentration (wt%) | Rotating speed (rpm) |
|---|---|---|---|
| Cyan | TSV-200 | 1, 3, 5, 7, 10 | 800 |
| Magenta | TSV-200 | 1, 3, 5, 7, 10 | 800 |
| Yellow | TSV-200 | 1, 3, 5, 7, 10 | 800 |
| Black | TSV-200 | 1, 3, 5, 7, 10 | 800 |



3.3.2.2 Toner charge dependence on carrier

The developer in Table 3-3 was experimented in the same measurements as mentioned in Section 3.3.2.1.

Table 3-3: Developers for toner charge dependence on carrier

| Toner | Carrier | Concentration (wt%) | Rotating speed (rpm) |
|---|---|---|---|
| Black | TSV-200 | 5 | 800 |
| Black | F-150 | 5 | 800 |
| Black | Z-250 | 5 | 800 |

3.3.2.3 Toner charge dependence on mixing method

The toner (black toner) and carrier (TSV-200) were prepared with various toner concentrations (1, 3, 5, 7 and 10%). Their charge was generated by the different charging methods.

3.3.2.3.1 By rotating charging (vertical mixing)

The developer was rotated vertically by a rotator (MS 1 Minishaker). The rotating speed was 800 rpm. The rotator was stopped after 15, 30, 60, 90, 120, 240, and 360 seconds of mixing in order to measure the q/m by a blow-off method as mentioned in Section 3.3.2.1

3.3.2.3.2 By rotating charging (horizontal mixing)

The developer was rotated horizontally by a horizontal rotator. The rotating speed was 120 rpm. The rotator was stopped after 30 seconds, 1, 2, 5, 10, 15, 20 and 30 minutes of mixing in order to measure the q/m by a blow-off method as mentioned in Section 3.3.2.1

### 3.3.3 Measurement of charge properties by the E-SPART analyzer

The toner and carrier in Table 3-4 were selected to measure their charges by E-SPART analyzer. The developer was mixed together by vertical mixing and horizontal mixing.

Table 3-4: Developers for toner charge measurement (E-SPART analyzer)

| Toner | Carrier | Concentration (wt%) | Rotating mixing |
|---|---|---|---|
| Black | TSV-200 | 1, 3, 5, 7, 10 | Vertical mixing, horizontal mixing |
| Black | F-150 | 1, 3, 5, 7, 10 | Vertical mixing, horizontal mixing |
| Black | Z-250 | 1, 3, 5, 7, 10 | Vertical mixing, horizontal mixing |

### 3.3.4 Toner and carrier characterization

3.3.4.1 Morphology of toners and carriers

The toners and carriers were analyzed for the morphology in terms of particle size and particle shape by SEM technique. This technique coated the toners and carriers with a thin layer of gold in vacuum and the toner/carrier mixture was photographed using scanning electron microscopy.

3.3.4.2 Thermal properties of toners

Glass transition temperature and melting temperature of the toners were measured calorimetrically using a differential scanning calorimeter. The measurement was programmed at a heating rate 20°C/min without liquid nitrogen, over a temperature range of 25-125°C.

3.3.4.3 Characterization of toners

The four types of toners (black, cyan, magenta and yellow toners) were analyzed for functional groups using Fourier Transform Infrared Spectroscopy.

3.3.4.4 Particular elements of carriers

The three types of carriers (TSV-200, F-150 and Z-250) were analyzed for the elements using x-ray fluorescence.

**3.3.5 Printing experiment**

The plain paper sheets were printed with a test form, which was produced by the Adobe Illustrator program, by the Canon printer (Canon Laser Shot LBP-2710) using polymerized toner and compared with another set of print-outs, printed by a Fuji printer (Fuji Xerox Color Laser Wind 3310) using pulverized toner.

The test form was consisted of A-Z alphabets, the seven color types of different percentage halftones of 0-100 (cyan, magenta, yellow, red, green, blue and black), the color chart and the lines with the size of 87.75, 175.5, 263.25, 351, 702

and 1053 micrometers in order to analyze the sharpness. Twenty plain paper sheets were printed. (1 point = 0.351 millimeters)

### 3.3.6 Evaluation of print quality

The print-outs were measured for solid density, tone reproduction, the sharpness of the alphabets and lines, color gamut and gamut volume.

3.3.6.1 The density was measured at solid density by a reflection densitometer (Macbeth, RD 915), which was calibrated with black and white tiles.

3.3.6.2 Tone reproduction was measured at percentage halftone of 0-100 by the reflection densitometer (Macbeth, RD 915).

3.3.6.3 The sharpness of the alphabets and lines was measured by image analyzer (LUZEX F, PM 10-AD).

3.3.6.4 Color gamut and gamut volume were measured at color charts by the spectrophotometer (X-Rite SP 62), measurement geometry d/8°, Illuminants D65, CIE 1931 10° observer. For gamut volume, the method of calculation for objective evaluation of SHIPP Image, evaluation for three kinds of items was done as follows. (see the detail in Appendix A)

1) Statistics value of image data (minimum and maximum values, mean value, variance and covariance, one dimensional histogram)

2) Principal component analysis by analysis of amount of information on color

3) Characteristics of spatial frequency of image (auto-correlation function)