CHAPTER III

EXPERIMENTAL

3.1 Chemicals

3.1.1 Pigment Based Inks

FUJI SP YELLOW 4254, Fuji Pigment Co., Tokyo, Japan,

percent volatile by weight: 84%,

C.I. Pigment yellow 138, specific gravity 1.80 - 1.87

FUJI SP MAGENTA 9345, Fuji Pigment Co., Tokyo, Japan,

percent volatile by weight: 83%,

C.I. Pigment red 122, specific gravity 1.4 - 1.5,

melting point 440 °C

FUJI SP BLUE 6447, Fuji Pigment Co., Tokyo, Japan,

percent volatile by weight: 78%,

C.I. Pigment blue 15:3, specific gravity 1.4 - 1.7,

melting point 480 °C

FUJI SP BLACK 8796, Fuji Pigment Co., Tokyo, Japan,

percent volatile by weight: 80%,

C.I. Pigment 7 (carbon Black), specific gravity 1.80 - 1.85

3.1.2 Binders

PERMALIN UA150, Sanyo Chemical Industries, Kyoto, Japan,

polyurethane emulsion, pH 9.3, non-volatile 29.7%, viscosity 79 mPa s

NK BINDER A12, Shin-Nakamura Chemical Co., Tokyo, Japan,
acrylate emulsion, pH 6.5, non-volatile 45%, viscosity >100 mPa s

NK BINDER M302, Shin-Nakamura Chemical Co., Tokyo, Japan,
acrylate emulsion, pH 5.5-7.5, non-volatile 47%, viscosity 300 mPa s

NK VANATEX S711, Shin-Nakamura Chemical Co., Tokyo, Japan,
acrylate emulsion, pH 5, non-volatile 48.5%, viscosity >1000 mPa s

3.1.3 Surfactants

BC-TX Series, Nikkoh Chemical, Osaka, Japan,
poly(ethylene oxide) cetyl ether

3.1.4 Other Chemicals

Ethylene glycol ($C_2H_6O_2$), Merck, Darmstadt, Germany,
analytical grade, M = 62.07 g mol^{-1}

Glycerol ($C_3H_8O_3$), Merck, Darmstadt, Germany,
analytical grade, M = 92.10 g mol^{-1}

Aluminum oxide (Al_2O_3), Degussa A.G., Frankfurt, Germany,
product name : aluminum oxide C®

PVA205, Thai Mitsui Specialty Chemicals Co.,
Poly(vinyl alcohol), 86-89% hydrolysis, degree of polymerization 1000

Denacol EX-313, Nagase Chemical Co., Osaka, Japan,
Glycerol poly(glycidyl ether)

3.2 Materials

1. Bleached and unfinished cotton fabric: plain weave, construction 140x75, weight 136 g m^{-2}

2. Bleached and unfinished polyester fabric: plain weave, construction 136x90, weight 127 g m^{-2}
3. Bleached and unfinished cotton/polyester blend fabric: plain weave, construction 54x60, weight 152 g m^{-2}
4. Bleached and unfinished silk fabric: plain weave, construction 82x85, weight 102 g m^{-2}

3.3 Equipment

1. Inkjet Printer: Epson Stylus 3000, Seiko Epson Corporation, Tokyo, Japan
2. Cantilever Stiffness Tester: Shirley Developmented Limited, England
3. Crockmeter: AATCC Crockmeter, ATLAS Electric Device Corporation, USA
4. Spectrophotometer: Digital Swatchbook, X-Rite Inc., Granville, USA
5. Image analyzer: LUZEX F, PM 10-AD, Olympus, Nireco Corporation, Tokyo, Japan
6. Viscometer: Brookfield DVIII Programmable Rheometer, MA, USA
7. Scanning Electron Microscope (SEM): JSM 6400, Joel, Japan
8. Surface tensiometer : K8, Kruss, Germany
9. Drying oven: Rapid, Labortex Corporation, Taiwan
10. pH meter: SA 720, Orion Research Incorporated, USA
11. Homogenizer: Robo Mics, Tokushu Kika, Japan
12. Padding machine: Tsuji Dyeing Machine Mfg, Osaka, Japan
13. Mechanical stirrer: RE16, IKA-Labortechnix, Germany
14. Dynamic permeability tester: Toyo Seiki Seisaku-Sho, Ltd, Tokyo, Japan
15. Dynamic Mechanical Analyzer: DMA 7e, Perkin Elmer, USA

3.4 Procedures

3.4.1 Preparation of Aqueous-Based Pigmented Inkjet Inks

The aqueous pigmented inkjet inks were prepared by varying the binder types of the inks, which are soluble binder and emulsion binder. The components of these inks were kept constant and the binder-to-pigment ratio was 1 to 1. An appropriate ink formula was investigated.

3.4.1.1 Mixing Step

The ink components were mixed proportionally in which the deionized water was used as a main solvent, ethylene glycol as a cosolvent or humectant, BC30 as a nonionic surfactant, pigment dispersion as a colorant, and emulsion binder. These components were gradually added and agitated. The concentration of the ink components were kept constant as follows:

- Binder : 4%wt based on total weight of the ink
- Pigment-based inks: 4%wt based on total weight of the ink
- Ethylene glycol : 10%wt based on total weight of the ink
- Surfactant : 0.5%wt based on total weight of the ink
- Deionized water : residual amount

NaOH could be added for controlling the ink to a range of pH around 7-9.

3.4.1.2 Filtering Step

The prepared inks were filtered directly with 400 mesh nylon screen. The coarse particles were therefore removed to prevent plugging of the printing head.

3.4.2 Preparation of Fabrics

3.4.2.1 Preparation of Nontreated Fabrics

Four kinds of unfinished fabrics, which are cotton, silk, polyester, and

cotton/polyester blend fabrics, were washed with soap, then rinsed with clean water and dried at ambient atmosphere. The dry fabrics were ironed to obtain a flat and smooth surface.

3.4.2.2 Preparation of Pretreated Cotton Fabrics

Alumina pigments were dispersed in water by a homogenizer. The alumina dispersion was then homogenized with 10% poly(vinyl alcohol) solution. Glycerol poly(glycidyl ether) was added as a crosslinking agent to improve waterfastness of the pretreatment. The speed of rotation was 8000 rpm and the time of rotation was 30 minutes. The formulation of this pretreatment agent is shown as follows:

- Alumina dispersion concentration : 10%wt.
- Poly(vinyl alcohol) concentration : 10%wt
- Alumina dispersion: PVA : 4:1

The clean and smooth cotton fabrics were padded with the pretreatment agent to have 100% pick up ratio using a padding machine. The padded fabrics were dried in an oven at 80 °C for 10 minutes.

3.4.2.3 Backing of Fabrics

The nontreated and pretreated fabrics in Sections 3.4.2.1 and 3.4.2.2 were cut into A4 size or otherwise specified. It was taped with two-sided sticky tapes onto a smooth, flat surface, such as a plastic film as a backing material. The backing material should have a uniform thickness.

3.4.3 Inkjet Printing Step

Cyan, magenta, yellow, and black inks were printed on cotton, silk, polyester and cotton/polyester blend fabrics using a modified Epson printer. The printer was calibrated according to the manufacture instruction. The test forms were

created and printed with Adobe Photoshop program without any color matching function with a resolution of 1440 dpi.

3.4.4 Characteristics of Binder and Ink

3.4.4.1 Viscosity

The pigmented inks were investigated for their viscosity using a Brookfield viscometer DVIII. The inks were also brought into the viscometer and measured at the temperature of 25°C by which many shear rates were varied. The variation of the shear rates were between 100 to 250 rpm.

3.4.4.2 Surface Tension

The pigmented inks were measured for their surface tensions by a ring method in K8 surface tensiometer.

3.4.4.3 Mechanical Stability of Emulsions

The filtered emulsion was poured into a mechanical blender, and mixed for 10 min. It was then refiltered to separate any solid residue which was left on the screen.

3.4.4.4 Water Uptake of Binder Films

A dried binder film was prepared and weighted. The dried film was placed into distilled water for 24 hours. It was then reweighted and the percent water uptake was then calculated.

3.4.4.5 Hardness of Ink Films

Ink films of the pigmented inks were prepared and their hardness was measured by a pencil test method (ASTM D 3363).

3.4.4.6 Young's Modulus of Binder Films

The Young's modulus of the film of the binders was measured to obtain a correlation of film property and stiffness of the printed fabrics. They were then loaded

to a tensile tester. Strain of the film was measured and Young's modulus was calculated, which from a stress-strain curve was plotted to get a slope for Young's modulus.

3.4.5 Characterization of Fabrics

3.4.5.1 Wicking Test

The fabric samples 25 mm. wide and 100 mm. long were prepared. During the test, each fabric strip was positioned vertically over the glass beaker containing the inkjet ink and the end of the fabric strips was immersed in the ink for 3 sec. The ink was absorbed along the thread of the fabrics. The wicking test was measured in terms of the height of absorbed ink.

3.4.5.2 Absorption by Dynamic Permeability Tester

The fabrics were cut into the strips, and then the strip was mounted on the dynamic permeability tester. The ink was then drawn on the mounted fabric using a headbox of the tester at a speed of drawing, then the length of the drawn ink was measured. The ink absorption in the term of the volume per unit area of the fabric was calculated.

$$\text{Ink absorption} = \frac{\text{volume of the tested ink}}{W \times L} \qquad (3.1)$$

where

W is the width of headbox

L is the length of the drawn ink

3.4.5.3 Absorption of Inks on Fabrics by Optical Microscope

Cross-sectioned printed fabrics were observed for depth of ink penetration

using an optical microscope. The depth of ink penetration were photographed, and investigated to characterize absorption of the inks into the fabrics.

3.4.5.4 Crockfastness of the printed fabrics

A crockmeter was used for testing the printed colors of cyan, magenta, yellow, and black. The crockfastness of the printed fabrics was evaluated based on the AATCC 8-1969 test method.

3.4.5.5 Stiffness

The stiffness of the non-printed fabrics was measured in terms of the bending length. After printing, the printed colors on the fabrics: cyan, magenta, yellow, green, blue, red, and black were measured for the bending length using a stiffness tester (a cantilever type). The stiffness test method was based on ASTM D 1388 test method.

3.4.5.6 Surface Morphology of Fabrics

Surface morphology of the fabrics was viewed and photographed using the scanning electron microscopic technique (Joel JSM-6400).

3.4.5.7 Whiteness (CIE/ASTM E313-96)

The raw fabrics were measured for the xyY values using a spectro-photometer (X-Rite Digital Swatchbook), measurement geometry $45^{\circ}/0^{\circ}$, Illuminants D65, CIE 1931 2° observer. The whiteness, W, was calculated from the following equation:

$$W = Y+800(x_n-x)+1700(y_n-y) \quad (3.2)$$

where

Y is the Y tristimulus value of the sample

x and y are the x,y chromaticity coordinates of the sample

x_n and y_n are the chromaticity coordinates of the perfect reflecting diffuser

3.4.5.8 Color Measurement

Colors of the printed fabrics: cyan, magenta, yellow, red, green, and blue were measured using a spectrophotometer (X-Rite Digital Swatchbook), measurement geometry 45°/0°, Illuminants D65, CIE 1931 2° observer. The u′ and v′ color values were calculated as color saturation, S_{uv}, from the following equation:

$$S_{uv} = 13\,((u' - u'_n)^2 + (v' - v'_n)^2)^{1/2} \quad (3.3)$$

where

u'_n, v'_n are the values of u′,v′ for the reference white

u′, v′ are the u′, v′ diagram, which are calculated from x,y color values as follows:

$$u' = 4x/(-2x+12y+3) \quad (3.4)$$

$$v' = 9y/(-2x+12y+3) \quad (3.5)$$