CHAPTER III

EXPERIMENTAL

3.1 Materials

3.1.1 Colorants

Two types of pigment dispersion technique were used:

3.1.1.1 Microencapsulation: microencapsulated pigments from Dainippon Ink and Chemicals, Inc. Tokyo, Japan. were used as follows:

Cyan (MCC-146-K60C) : C.I. Pigment Blue 15:4

Magenta (MCM-146-K59C) : C.I. Pigment Red 122

Yellow (MCY-146-K61C) : C.I. Pigment Yellow 128

Black (MCB-146-K62C) : C.I. Pigment Black 7

3.1.1.2 Surface modification: surface modified pigments from Cabot Corporation, Massachusetts, USA. were used as follows:

Cyan (IJX 253) : C.I. Pigment Blue 15:4

Magenta (IJX 266) : C.I. Pigment Red 122

Yellow (IJX 273) : C.I. Pigment Yellow 74

Black (CABOT-O-JET 200) : C.I. Pigment Black 7

3.1.2 Binder

The binder is NK Vanatex S-711 from Shin-Nakamura Chemical Co., Tokyo, Japan, acrylate emulsion, pH 5, non-volatile 48.5%, viscosity >1000 mPa s

3.1.3 Solvents

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

3.1.3.2 Glycerin ($C_3H_8O_3$) from Merck, Darmstagt, Germany, analytical grade, M = 92.10 g mol^{-1}

3.1.3.3 Urea (CH_4N_2O) from AJAX, New Southwales, Australia, analytical grade, M = 60.06 g mol^{-1}

3.1.4 Pretreatment agent

3.1.4.1 Cationic polymer: Sanfix 555 (cationic acrylate polymer) from Sanyo Chemical Industries, Ltd. Kyoto, Japan

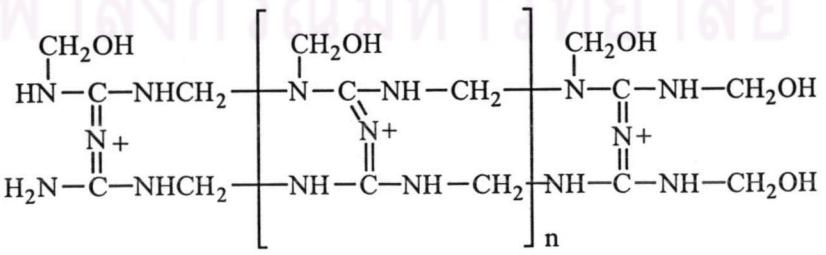

Figure 3-1 Chemical structure of cationic acrylate polymer (Sanfix 555)

3.1.5 Fabric

Degummed silk fabric: plain weave, construction 82x85, 104 g m^{-2}

3.2 Apparatus

3.2.1 Inkjet Printer : Epson Stylus Color 3000, Seiko Epson Corporation, Tokyo, Japan

3.2.2 Viscometer : Brookfield DV III Programmable Rheometer/TC500, Brookfield Engineering Laboratories, Inc., Stoughton, USA

3.2.3 Surface Tensiometer : K 810, Kruss, Hamberg, Germany

3.2.4 pH Meter : SA 720, Orion Research Incorporation, Boston, USA

3.2.5 Drying Oven : 6-2 FI, Rapid Labortex Corporation, Taiwan

3.2.6 Mechanical Stirrer : RE 16, IKA-Labortechnix, Staufen, Germany

3.2.7 Spectrophotometer : Gretag-Macbeth spectrolino AG, Rogensdorf, Switzerland

3.2.8 Crockmeter : AATCC Crockmeter, Atlas Electric Devices Corporation, Chicago, USA

3.2.9 Cantilever Stiffness Tester : Shirley Development Limited, Stockport, England

3.2.10 Air Permeability Tester : Shirley, England

3.2.11 Padding Machine : Laboratory, Tsujh Dyeing Machine Mfg., Osaka, Japan

3.2.12 Xenon Arc Lamp Weather-O-Meter : X75, Suga Test Instrument Co.,Ltd., Tokyo, Japan

3.2.13 Launder-O-Meter : AATCC Launder-o-meter Test, USA

3.2.14 Scanning Electron Microscope (SEM) : JSM 6400, Joel, Tokyo, Japan

3.2.15 Laser Scattering Analyzer : Malvern Instrument Ltd., Malvern, UK

3.2.16 Image Analyzer : LUZEX F, PM 10-AD, Olympus, Nireco Corporation, Tokyo, Japan

3.2.17 DSC : DSC 200, NETZSCH, Thermische Analyse, Bayern, Germany

3.2.18 Microdensitometer : PDM-7, KONICA, Tokyo, Japan

3.3 Procedure

3.3.1 Preparation of Fabrics

3.3.1.1 Preparation of Non-treated Fabric

Silk fabric was washed with soap then rinsed with clean water and dried at ambient atmosphere. The fabric was ironed for a smooth surface.

3.3.1.2 Preparation of Treated Fabric

Silk fabric was treated using the 10% Sanfix and 10% urea solution as a pretreatment agent. The silk fabric was padded by a padding machine with 100% pick up and then dried in an oven at 80^{o}C for 10 minutes.

3.3.1.3 Backing of Fabrics

In order to print textile substrates, it is necessary to assure fabric stability in the printer feed rollers. Deformation of the fabric was prevented by applying a transparency film or plastic film, which has a flat surface and should have a uniform-thickness strip of masking tape to the back of the fabric.

3.3.2 Preparation of Pigmented Inkjet Inks

The inkjet inks were prepared by varying the dispersed pigment, either by the microencapsulation technique or the surface modification technique. The ratio of pigment to binder was 1 to 2. The formulation of pigmented inkjet inks is shown in Table 3-1.

Table 3-1 Fomulation of Pigmented Inkjet Inks

Composition	Concentration (%wt)
Pigment dispersion	4.0
Diethylene glycol	10.0
Glycerine	10.0
Urea	5.0
Binder (S-711)	8.0
Deionized water	63.0
Total	**100.0**

The Ink components were mixed together, in which the dispersed pigment was used as a colorant, S-711 polymer resin as a vehicle or binder, the deionized water as a main solvent, diethylene glycol, glycerine and urea as a co-solvent. The mixture was then stirred to be a homogeneous solution. Then, sodium hydroxide was added to control the ink pH in a range of 7-9. The inks were later filtered through 5 μm pore filtering paper for preventing clogging problems. A set of dispersed pigmented inks contained four colors, which cyan, magenta, yellow, and black. Then the inks were stored in a dessicator, which was connected to a suction pump for eliminating any air bubbles. Then the four colors of inkjet ink were loaded into the inking unit of the printer.

3.3.3 Characterization of the Materials

The pigmented inkjet inks were evaluated for the ink properties in terms of viscosity, surface tension, particle size distribution, content of dispersing agents and glass transition temperature of the polymeric binder.

3.3.3.1 Viscosity

The pigmented inkjet inks were measured for their viscosity using a Brookfield viscometer model DVIII. The inks were filled into a sample holder of

the equipment and then measured at the temperature of 25°C by varied shear rates. The shear rates under study were between 50 to 250 rpm.

3.3.3.2 Surface Tension

The surface tensions of the inks were evaluated using a ring method in K8 surface tensiometer.

3.3.3.3 Particle Size Distribution of Dispersed Pigments and Pigmented Inkjet Inks

A small amount of dispersed pigments and pigmented inkjet inks were suspended and fed into the input channel of the equipment for measuring their particle sizes. The information of particle size distribution was taken from Malvern laser scattering analyzer model Mastersizer S long bed Ver. 2.11.

3.3.3.4 Concentration of the Dispersing Agent

The microencapsulated pigments were evaluated for the concentrations of the dispersing agent using DSC technique.

3.3.3.5 Glass Transition Temperature of the Polymer Binder

The glass transition temperature, Tg, of polymer binder S-711 was evaluated using NETZSCH DSC200.

3.3.4 Characterization of the Printed Fabrics

The printed fabrics were evaluated for the print qualities. The print qualities were measured in terms of color of the printed fabrics, dry/wet crockfastness, washfastness, lightfastness, air permeability, and bending stiffness.

3.3.4.1 Color Measurement

Colors of the printed fabrics: cyan, magenta, yellow, black, red, green and blue were measured in terms of density and color values in tristimulus value and CIELAB color space using a spectrophotometer model spectrolino, measurement geometry $45^{\circ}/0^{\circ}$, Illuminants D65, CIE 1931 2° observer.

The tristimulus values were transformed to chromaticity coordinates for creating a color gamut (2 dimension) from the following equations:

$$x = \frac{X}{X+Y+Z} \tag{3-1}$$

$$y = \frac{Y}{X+Y+Z} \tag{3-2}$$

$$z = \frac{Z}{X+Y+Z} \tag{3-3}$$

where

X, Y, Z = tristimulus values

x, y, z = chromaticity coordinates

The L^*, a^* and b^* color values were used to calculate a color volume. The color volume was calculated by using the color gamut volume

program provided by Canon Inc. and chroma, C^*_{ab}, was calculated from a^* and b^* from the following equation:

$$C^*_{ab} = \sqrt{a^{*2} + b^{*2}} \quad (3\text{-}4)$$

where

C^*_{ab} = chroma

a^* = redness-greenness

b^* = yellowness-blueness

3.3.4.2 Dry/Wet Crockfastness

The printed colors on the fabrics: cyan, magenta, yellow, black, red, green and blue were rubbed with white crock test cloth under controlled conditions, based on the AATCC 8-1995 test method. Then color transferred to the white test cloth is assessed by a comparison with the gray scale for staining and a grade as well as ΔE^*_{ab} are assigned.

3.3.4.3 Washfastness

The washfastness method is evaluated for the effect of washing only on the color fastness on textile. The printed colors on fabrics were evaluated for color fastness to washing using a Launder-o-meter based on ISO 105-C01 test method.

3.3.4.4 Lightfastness

The printed fabrics were composed of cyan, magenta, yellow, black, red, green and blue on the non-treated and treated fabrics. The specimens were placed into the Xenon arc lamp of a weather-o-meter for 300 h. The color values in CIELAB color space of each printed fabric specimens were measured using a spectrophotometer. The color difference was calculated using the color difference equation as follows:

$$\Delta E^*_{ab} = \sqrt{(L^*_1 - L^*_2)^2 + (a^*_1 - a^*_2)^2 (b^*_1 - b^*_2)^2} \qquad (3\text{-}5)$$

where

ΔE^*_{ab} = color difference

L_1^*, L_2^* = CIE-L* of color before and after exposure

a_1^*, a_2^* = CIE-a* of color before and after exposure

b_1^*, b_2^* = CIE-b* of color before and after exposure

3.3.4.5 Air Permeability

The printed color fabrics were measured for the air permeability using a Shirley air permeability tester. The rate of airflow passing perpendicularly through the area of fabric is adjusted to obtain a prescribed air pressure. From this rate

of airflow, the air permeability of the fabric is determined. The rate generally expressed in SI units $cm^3\ s^{-1}\ cm^{-2}$ and in English units as $ft^3\ s^{-1}\ ft^{-2}$ at operating conditions. The air permeability of the printed fabrics was evaluated based on the ASTM D 737-96 test method.

3.3.4.6 Bending 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, black, red, green and blue were measured for the bending length using a stiffness tester (a cantilever type). The stiffness test method was based on the ASTM D 1388 test method.