# CHAPTER IV

# RESULTS AND DISCUSSION

In this research, two types of pigment dispersion technique understudy were related to the inkjet ink properties and print quality onto the silk fabrics. Two conditions were applied to the silk fabric. One silk fabric was not treated with any pretreatment reagent, as a control, while the other was treated with a cationic acrylate polymer. The printed fabrics were then characterized and evaluated for their print qualities.

## 4.1 The Characteristics of Pigmented Inkjet Inks

Two types of pigment dispersion for four color pigments are shown in Table 4-1. Some physical properties of the pigments used here are given as follows: PB 15:4 is phthalocyanine: copper complex pigment, which has bright blue shade. Its particular importance is partly due to its resistance to solvents, heat, light and chemicals. This is one of most important pigments used in printing ink. PR 122 is quinacridone pigment, which is bright bluish red, high performance and high degree of intermolecular bonding. PY 74 is a bright yellow monoazo pigment, which shows superior brightness and tinting strength. PY 128 is diazo pigment, which has bright greenish yellow shade. Its high molecular weight, high performance pigments with excellent tinting strength. Pigment

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black 7 is impervious to chemicals, light, weather and heat, excellent hiding power and UV absorption.[20]

**Table 4-1** Dispersion for four color pigments

| Dispersion technique | Color | Color index | Chemical type | Chemical class |
|---|---|---|---|---|
| Surface modification | Cyan | PB 15:4 | Organic | Phthalocyanine |
| | Magenta | PR 122 | Organic | Quinacridone |
| | Yellow | PY 74 | Organic | Monoazo |
| | Black | PBk 7 | Inorganic | Carbon black |
| Microencapsulation | Cyan | PB 15:4 | Organic | Phthalocyanine |
| | Magenta | PR 122 | Organic | Quinacridone |
| | Yellow | PY 128 | Organic | Diazo |
| | Black | PBk 7 | Inorganic | Carbon black |

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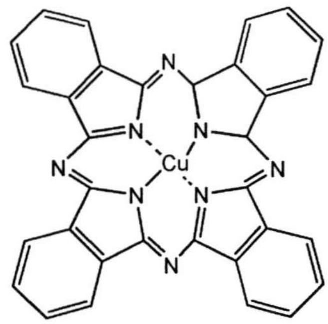
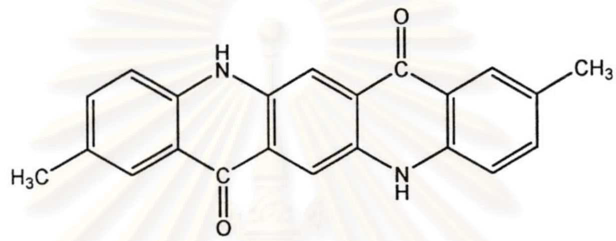
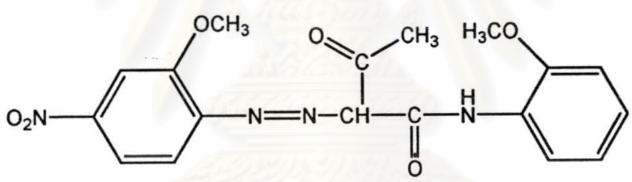


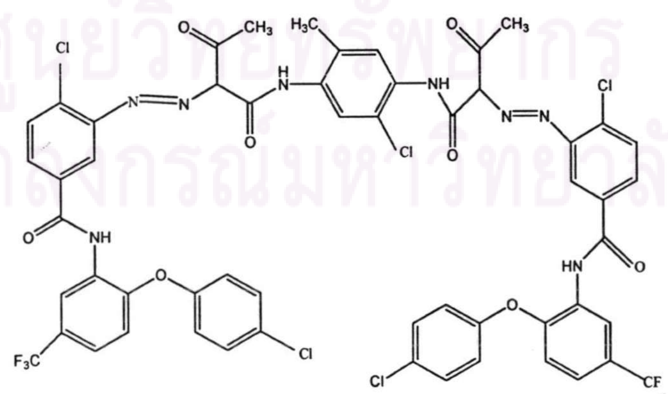
Pigment Blue 15:4



Pigment Red 122



Pigment Yellow 74



Pigment Yellow 128

**Figure 4-1** Chemical structure of pigments

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The physical properties of pigment dispersions used in this research for preparation of the inkjet inks are shown in Table 4-2 .

Table 4-2 The physical properties of four color pigments

| Dispersion technique | Color | Properties | | | |
|---|---|---|---|---|---|
| | | Pigment concentration (%) | pH | Particle size (μm) | Viscosity (mPa s) |
| Surface modification | Cyan | 10.4 | 8.09 | 0.24 | 1.55 |
| | Magenta | 10.2 | 7.94 | 0.25 | 1.86 |
| | Yellow | 9.8 | 8.15 | 0.30 | 1.83 |
| | Black | 19.6 | 7.60 | 0.24 | 3.23 |
| Microencapsulation | Cyan | 14.3 | 8.64 | 0.23 | 3.74 |
| | Magenta | 14.4 | 8.76 | 0.23 | 4.84 |
| | Yellow | 16.7 | 9.43 | 0.23 | 8.46 |
| | Black | 14.3 | 8.48 | 0.22 | 3.12 |

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The two types of pigment dispersion have a different functional group. The surface modification technique using sulfonic group to modify the surface of pigment particle, whereas the microencapsulation technique used the encapsulated polymer embracing pigment particles.[6,11] The ratios the pigment to polymer (by weight) for pigment blue, pigment red, pigment yellow and pigment black are shown in Table 4-3.

Table 4-3 The ratio of pigment to polymer of microencapsulated pigment

| Pigment | Pigment : Polymer ratio |
|---|---|
| PB 15:4 | 1 : 0.54 |
| PR 122 | 1 : 0.52 |
| PY 128 | 1 : 3.24 |
| PBk 7 | 1 : 0.41 |

The polymer binder, which is used in this research, is polyacrylate emulsion. The mean diameter of the particles of the emulsion is of approximately 0.24 μm. The properties of the binder S-711 are shown in Table 4-4.

Table 4-4 Characteristics of S-711 binder

| Properties | Amount |
|---|---|
| Nonvolatile (%) | 48.5 |
| Viscosity (mPa s) | 1000-1500 |
| pH of dispersion | 5 |
| Dispersion type | nonionic |
| Tg ($^{o}$C) | 1.2 |

The required properties of inkjet inks are excellent stability, high brilliance, low viscosity, high surface tension, long shelf life, and rapid drying time. When examining the rigors of the process, droplets are heated only for microseconds to reach 300-500°C in a print head and the droplets are then ejected through the nozzles of 20-100 μm diameter.[21] The inkjet ink properties used in this research conform to the above mentioned characteristics.

### 4.1.1 Viscosity of Pigmented Inkjet Inks

Print performance comes from how the inks generate images on the substrate with the specific properties within a designed range. To eject droplets correctly and to get enough density, inkjet ink must have the proper physical and optical properties (low viscosity and high surface tension). The ink formulation was kept constant at a pigment-to-binder ratio of 1:2 by weight based on total weight of the ink. Table 4-5 shows the viscosity of cyan, magenta, yellow and black inks for two types of pigment dispersion technique. The viscosity of the inkjet inks was measured at 25°C with a spindle #18 and shear rate at 250 $s^{-1}$.

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**Table 4-5** The viscosity of cyan, magenta, yellow and black pigmented inkjet inks

| Type of ink dispersion | Viscosity (mPa s) | | | |
|---|---|---|---|---|
| | Cyan | Magenta | Yellow | Black |
| Surface modified pigmented ink | 3.58 | 3.85 | 3.58 | 3.55 |
| Microencapsulated pigmented ink | 4.45 | 4.71 | 4.81 | 4.27 |

From Table 4-5, the viscosity of the inks made from surface modified pigments of cyan, magenta, yellow and black inks were found 3.58, 3.85, 3.58 and 3.55 mPa s, respectively. The inks made from microencapsulated pigments have the viscosity of 4.45, 4.71, 4.81 and 4.27 mPa s. The ink made from microencapsulated pigments, therefore gave the higher viscosity than those from the surface modified pigments because of the extra concentration of the polyacrylate emulsion used for encapsulating the former pigment dispersion.

Basically, the inkjet inks are colloidal suspension, they should exhibit a significant shear thinning behavior, which this is expected to impact upon the particle distribution across the nozzle capillary leading to a jet. The shear thinning behavior disturbs the flow of inkjet ink because the inkjet ink system should be of the Newtonian fluid, which maintains a constant viscosity regardless of shear rate.

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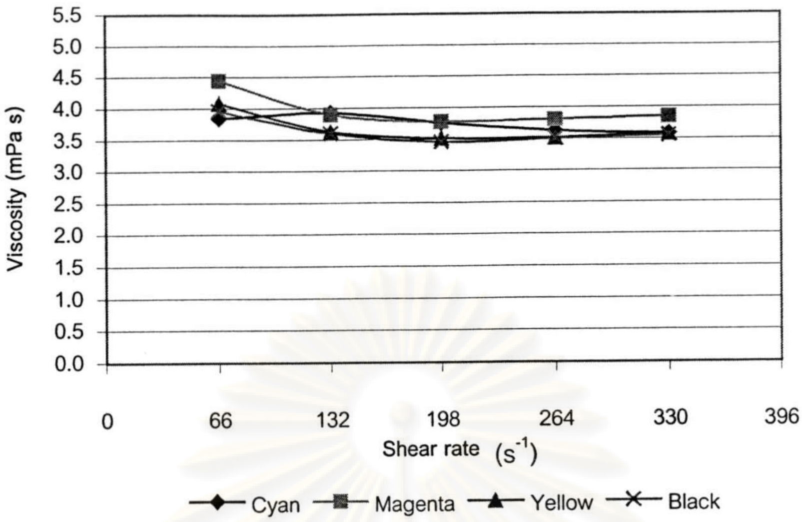


Figure 4-2 The viscosity of surface modified pigmented inkjet inks

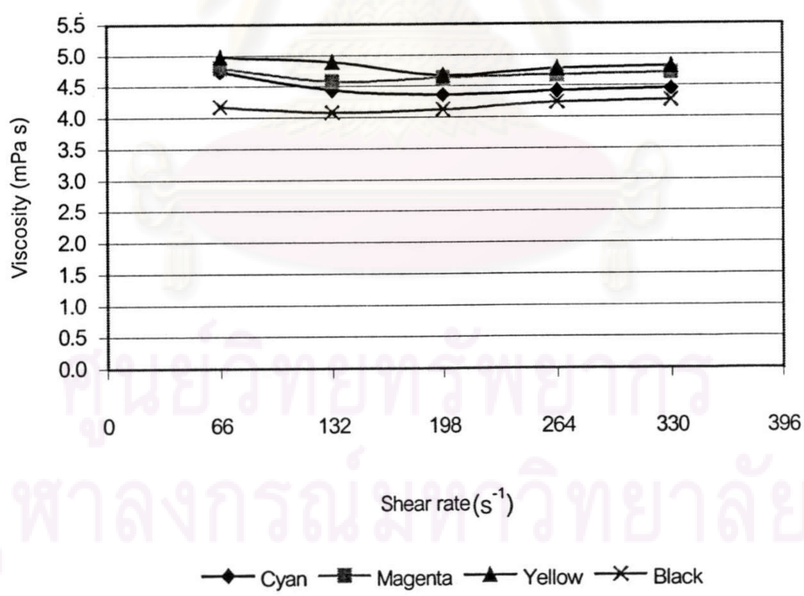


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Figure 4-3 The viscosity of microencapsulated pigmented inkjet inks

The two pigmented inkjet inks in this research perform somewhat like a non-Newtonian fluid, which might be caused by the other components in the ink ingredient. However, when we ignore the low shear region (the 66 $s^{-1}$), the other shear rates seem to give relatively constant viscosity since the differences between each shear rate is not significant. The high viscosity at low shear region could possibly be resulted from the buildup of structure between pigment and polymer binder.

### 4.1.2 Surface Tension of Pigmented Inkjet Inks

Surface tension helps to control the meniscus at the nozzle. A high surface tension is desirable for pigmented inkjet ink on uniform and stable drop formation on the nozzles. The surface tension of the ink has to be lower than the surface energy of the fiber of the fabrics. Considering the pigmented inkjet inks in Table 4-6, the surface tension of cyan, magenta, yellow and black inks is similar. The surface tension of surface modified pigmented inks is higher than microencapsulated pigmented inks. As a result of surface tension, regularity of droplet formation and pigment loading are critical to textile inkjet print quality.

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Table 4-6 The surface tension of cyan, magenta, yellow and black pigmented inkjet inks

| Type of ink dispersion | Surface Tension (mN $m^{-1}$) | | | |
|---|---|---|---|---|
| | Cyan | Magenta | Yellow | Black |
| Surface modified pigmented ink | 45 | 45 | 45 | 41 |
| iMicroencapsulated pigmented ink | 38 | 39 | 41 | 40 |

### 4.1.3 Particle Size

The particle size was measured using dynamic light scattering method from Malvern laser scattering analyzer. The sizes of dispersed pigments in the four pigmented inkjet inks are shown in Table 4-7.

Table 4-7 The particle sizes of dispersed pigments in the four pigmented inkjet inks

| Type of ink dispersion | Particle Size (μm) | | | |
|---|---|---|---|---|
| | Cyan | Magenta | Yellow | Black |
| Surface Modified pigmented Ink | 0.23 | 0.22 | 0.23 | 0.23 |
| Microencapsulated pigmented Ink | 0.23 | 0.22 | 0.22 | 0.23 |

The mean particle size of the cyan, magenta, yellow, and black inks were very similar at around 0.22 μm to 0.23 μm for both inks made from surface modified pigments and microencapsulated pigments. The clogging problem of the ink was not found during printing. The nozzles provide very good jetting, because the opening of the nozzles may vary from about 40 μm to about 100 μm.[4]

## 4.2 Stability of Pigmented Inkjet Inks

The pigmented inkjet inks were stored under room condition for 12 weeks in a stability study of the inks. The changes of viscosity after 12 weeks storage are shown in Figures 4-4 and 4-5 for surface modified and microencapsulated pigment inks, respectively.

The required properties of inkjet inks are excellent stability, high brilliance, low viscosity, high surface tension, long shelf life, and rapid drying time. Stabilization of the dissociated state can be achieved by controlling the added salts, ions, impurities in and from outside. These factors affect the size of the hydrostatic and electrostatic radii of the particles in the vehicle. The microencapsulated pigment contains the carboxylate group ($COO^-$) while the surface modified pigment has the sulfonate group ($SO_3^-$) on the modified surface. A better stability was observed in the surface modified pigmented inks than those of microencapsulated pigmented inks. Figures 4-4 and 4-5 show the increasing viscosity during storage. The viscosity of the inks made from microencapsulated pigments increase somewhat greater than those from surface modified pigments.

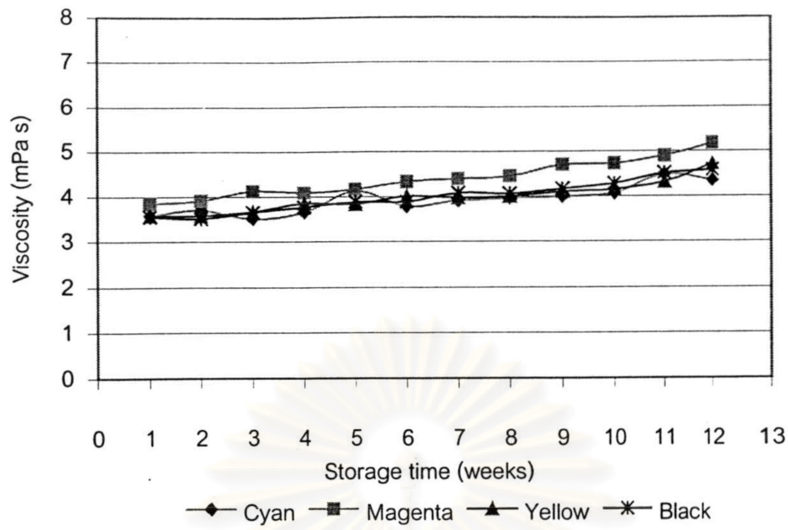


Figure 4-4 The change in viscosity of the surface modified pigmented inkjet inks in 12 weeks

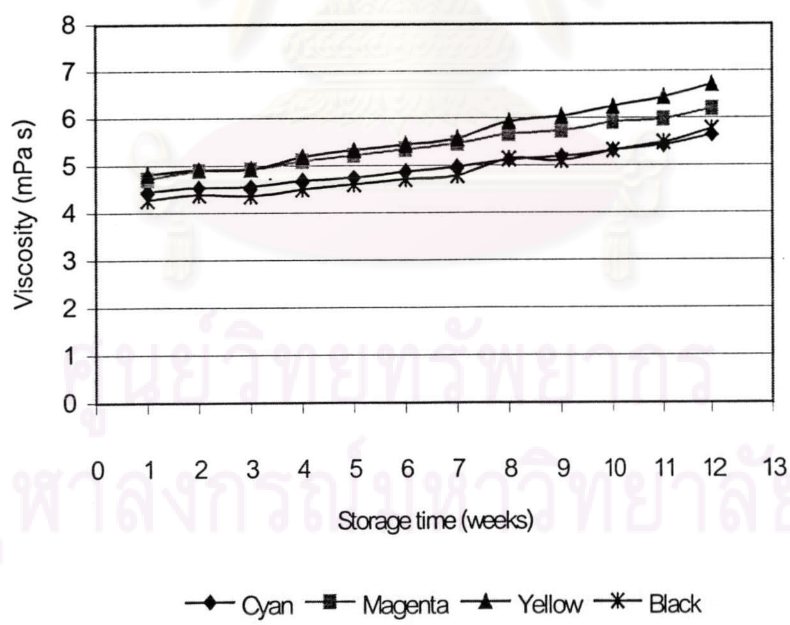


Figure 4.5 The change in viscosity of the microencapsulated pigmented inkjet inks in 12 weeks

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Runability of pigmented inkjet ink relies on various ink properties such as anti-kogation, heat stability, and anti-clogging performances. Especially anti-clogging property is thought to be the most serious problem of emulsion-based ink. Fortunately, by introducing several kinds of hydrogen bonding functional groups to polymer molecules, which can have mutual interaction with humectants, the ink could then be stabilized well under the dry condition. In this research, urea was added in the ink formulation for reducing the clogging problem.

The particle size distributions of the initial inks were compared with the particle size distribution after storage for 12 weeks. Figure 4-6 shows that the inks made from surface modified dispersion technique of cyan, magenta and black inks show a little change in size distribution except that of yellow ink. The particle size distribution of the inks made from microencapsulate pigment dispersion as shown in Figure 4-7 was almost constant for all colors of ink for 12 weeks. It is possible to state that the microencapsulated pigments are protected individually from coalescence by the encapsulating polymer. Therefore, the polymer shell behaves like a protective repulsion layer to keep each individual encapsulated pigment apart. Therefore, the particle size distribution before and after storage was the same, no changes were observed.

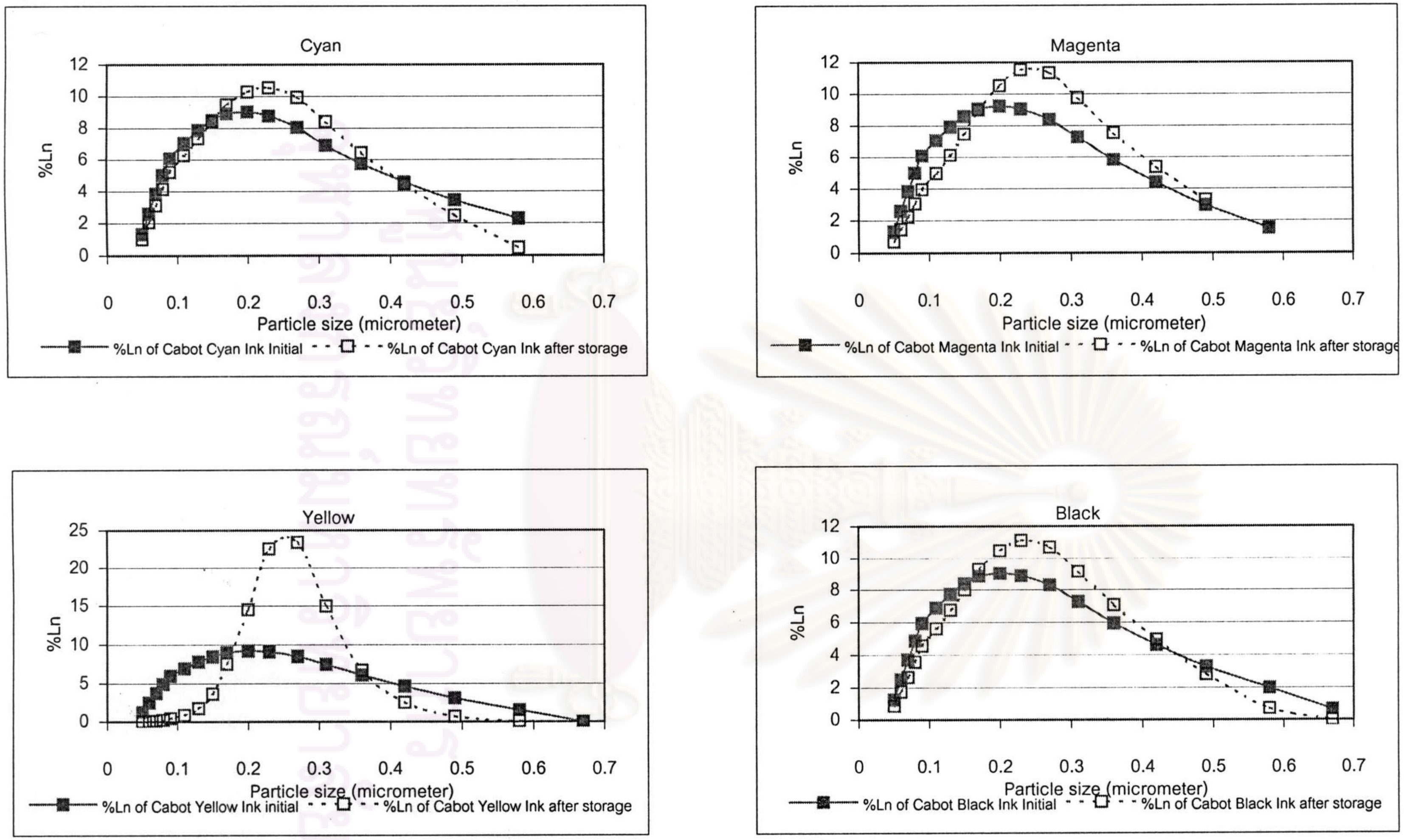



Figure 4.6 Dependence of storage on particle size distribution of surface modified pigmented inkjet inks

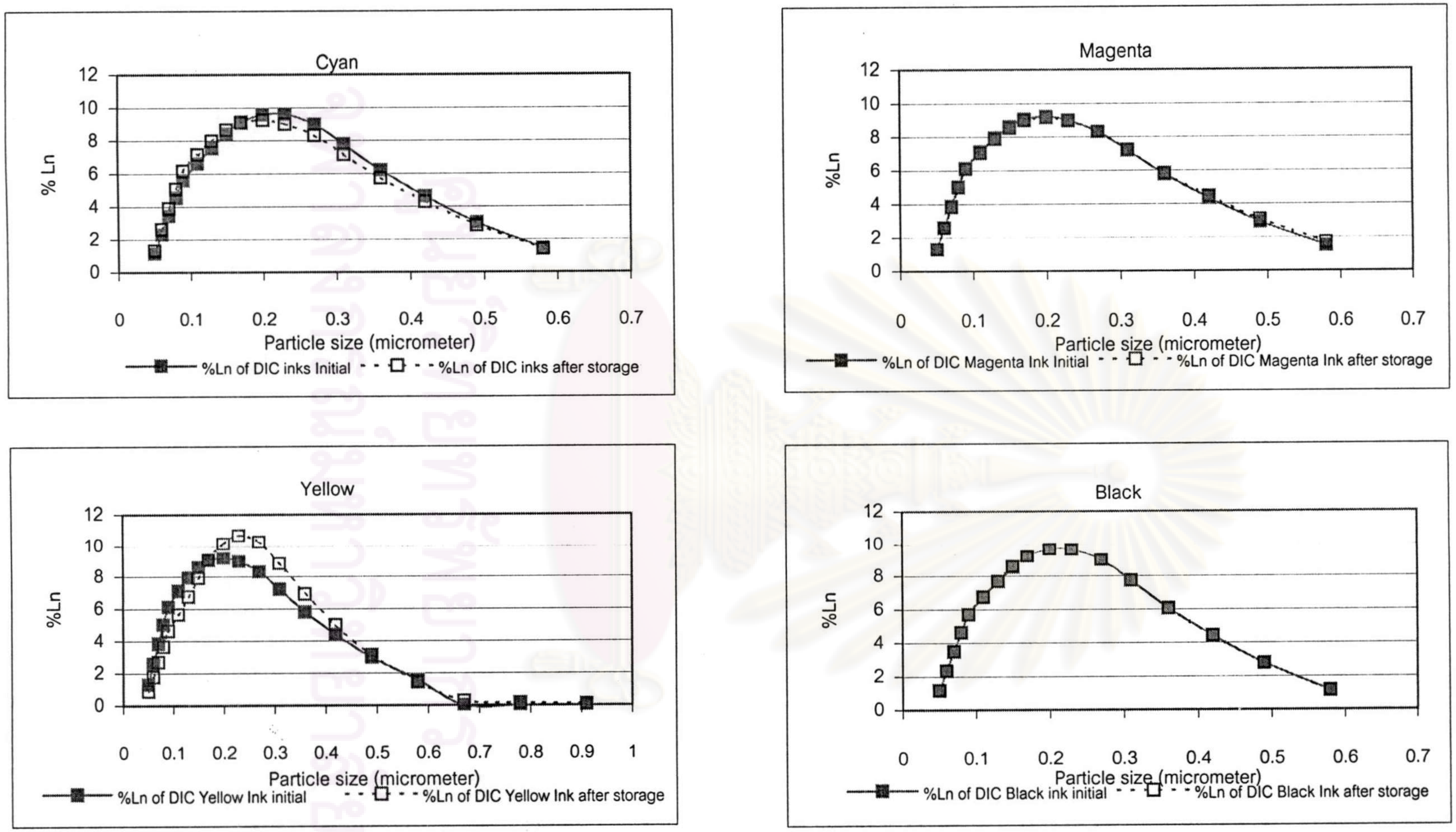



Figure 4.7 Dependence of storage on particle size distribution of microencapsulated pigmented inkjet inks

## 4.3 The Qualities of Printed Silk Fabrics

The pigment-resin printing forms a film when dried, which encloses the pigments and adheres to the fabric. Textile apparel has strict requirements on hand and color fastness. The silk fabrics were printed by a commercial desktop inkjet printer. The qualities of printed fabrics were analyzed in four categories: 1) appearance-related issues including optical density and tone reproduction; 2) color-related issues including color gamut, chroma, and color gamut volume; 3) permanence issues including crockfastness, washfastness and lightfastness; 4) usability issues including the presence of defects and hand, air permeability, and bending stiffness.

### 4.3.1 Effect of Pigment Dispersion on Optical Density

Optical density and tone reproduction were evaluated for the visual qualities of the printed fabrics. The original digital pattern consisting of a solid tone pattern and gray levels were printed onto the silk fabric. The optical density of the non-treated silk fabric is shown in Table 4-8.

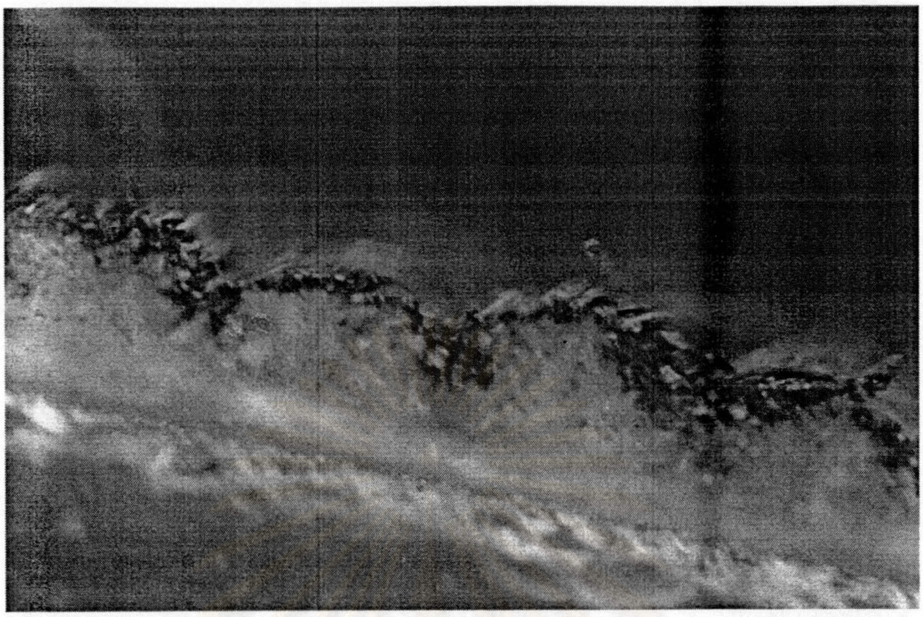
The cyan, magenta and black color inks made from the two different pigment dispersions contain the same chemical class of pigment (see Table 4-1). The optical density of the inks made from the surface modified pigments is higher than the inks made from the microencapsulated pigments.

Table 4-8 Surface tension - optical density relation on the non-treated silk fabric[a]

| Color | Surface modified pigmented ink | | Microencapsulated pigmented ink | |
|---|---|---|---|---|
| | Surface tension (mN $m^{-1}$) | Optical density | Surface tension (mN $m^{-1}$) | Optical density |
| Cyan | 45 | 1.29 | 38 | 1.20 |
| Magenta | 45 | 1.30 | 39 | 1.10 |
| Yellow | 45 | 1.43 | 41 | 1.14 |
| Black | 41 | 1.40 | 40 | 1.27 |

[a] The number of one sample measurement is 5 in each direction.

The difference in optical density between the two sets of ink depends on the illuminant and reflectance through the pigment particles in the ink layer. The microencapsulated pigment and surface modified pigment have different weight of polymer concentration, of course they are the same weight of dispersion while making inks but the microencapsulated pigment dispersion has a lot of polymer in the non-volatile part so they are not in the same quantity. The ratio of pigment to polymer of the microencapsulated pigment is shown in Table 4-3. The different weight % of pigment does not give the same tint strength.



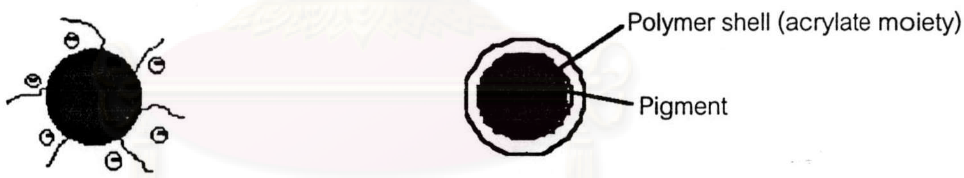
(a)

(b)

Figure 4-8 The cross section of photomicrographs showing the depth of ink penetrationon the non-treated silk fabric, (a) surface modified pigmented inkjet ink, (b) microencapsulated pigmented inkjet ink

Moreover, the structure of fiber imparts a strong influence on the scattering of light, when measuring the optical density of the silk fabric, the dependence of angle of reflection light is affected by the surface texture of fixed ink layer. The difference in surface tension affects the depth of penetration in fabrics, because the low surface tension causes the deeper penetration and the lower optical density.

Figure 4-8 (b) shows the ink penetration of microencapsulated pigmented ink with a low surface tension than the surface modified pigmented ink, the ink former can penetrate deeper than the surface modified pigment ink in Figure 4-8 (a).




(a) (b)

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**Figure 4-9** The dispersed pigment (a) surface modified pigment, (b) microencapsulated pigment

From Figure 4-9, the pigment in (b) has a thick layer of polymer shell enclosing pigment particles as a core, while the pigment in (a) is modified by chains of surfactant. On optical density measurement, the illuminating light from a spectrophotometer strikes on the encapsulated pigments in (b), the light is scattered and internal reflected. The reflected light collected at the spectrophotometer becomes less to give a lower ink density. On the other hand, the light reflected from the pigment surface in (a) is all collected by the spectrophotometer to give a higher ink density.

Macroscopically, the microencapsulated pigments reduce scattering of light in a fixed ink layer and gives better ink densities. In this context, the advantageous property can be observed in comparison to the ink layers of surface modified pigments, therefore, the densities of the microencapsulated pigmented ink are unfortunately lower. As mentioned previously in Chapter 3, the microdensitometer was used for evaluation of densities of the small printed areas including fiber. The optical microdensities are shown in Table 4-9. The optical microdensities of the microencapsulated pigmented inks are higher than those the surface modified pigmented inks.

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Table 4-9 The optical microdensity of the non-treated, printed silk fabric[a]

| Color | Surface modified pigmented ink | Microencapsulated pigmented ink |
|---|---|---|
| Cyan | 0.87 | 1.18 |
| Magenta | 0.70 | 0.85 |
| Yellow | 0.24 | 0.27 |

[a] One thousand points on the printed silk fabric were measured, with a sampling pitch of 1 μm.

#### 4.3.2 Effect of Pigment Dispersion on Tone Reproduction

The halftone pattern consisting of percent dot areas from 7 percent (highlight) to 100 percent or solid tone is printed onto the silk fabric. The densities of printed halftone dots on the printed fabric were measured for optical density and expressed in terms of tone reproduction.

In Figure 4-10, the tone reproduction of inkjet ink printed silk fabric is illustrated in terms of optical density versus percentage dot area. One can see that the tone reproduction of the inks made from the surface modified pigments is relatively higher than that of the microencapsulated pigments.

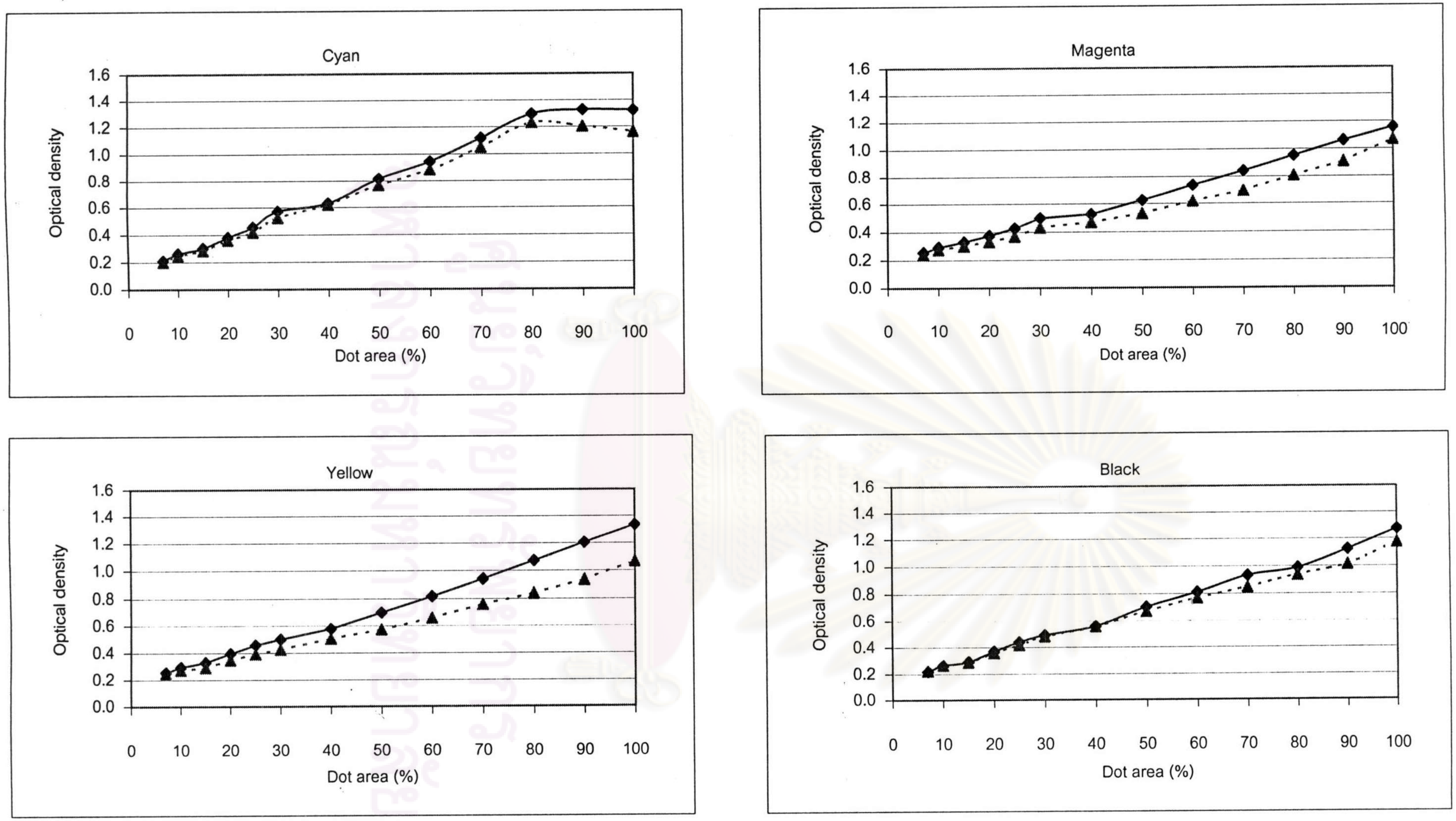


Figure 4.10 Tone reproduction on the non-treated silk fabric ( ——— Surface modified pigmented ink, - - - - Microencapsulated pigmented ink)

### 4.3.3 Effect of Pigment Dispersion on Color and Color Gamut

The gamut performance of different inkjet ink sets on the printed silk fabrics was investigated. The influence of different dispersion technique of the inks is compared. Gamut variation is caused by the different colorants in the inks. The colors on the printed silk fabrics were measured by CIELAB and CIEXYZ systems for evaluating the color strength of different dispersion technologies.

It is well known that the water-soluble dye-based ink has a large color space, high brightness, and high transparency, while the pigmented ink inherents inferior properties in color gamut and strength. As shown in Figure 4-11, comparison between different pigment dispersion techniques, one can see that cyan, magenta, and black pigments by both techniques have the same shade, but the surface modified yellow pigment is yellowish and the microencapsulated yellow pigment is greenish. The surface modified pigmented ink shows a larger color space in the x,y chromaticity diagram on the non-treated silk fabric with a larger gamut volume than those of the microencapsulated pigmented ink.

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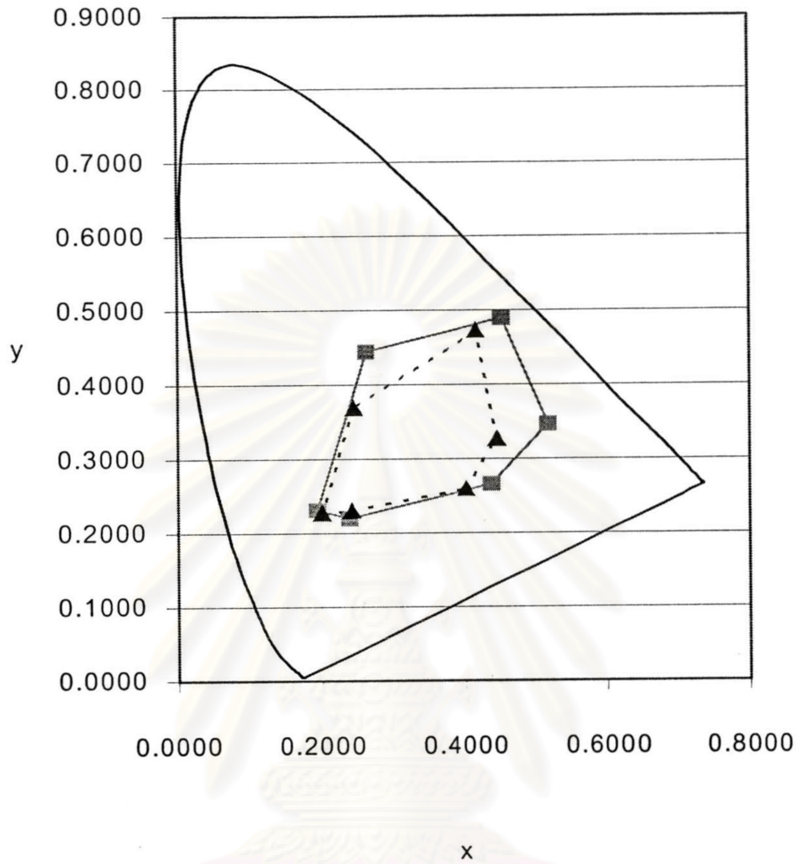


**Figure 4-11** The chomaticities of colors printed using surface modified pigmented ink and microencapsulated pigmented ink on the non-treated silk fabric (——— Surface modified pigmented ink, – – – Microencapsulated pigmented ink)

Penetration of a liquid flowing under its own capillary pressure in a horizontal capillary is described by the Lucas-Washburn equation.[23]

$$\ell = \left( \frac{r\gamma \cos\theta}{2\eta} \right)^{\frac{1}{2}} t^{1/2} \qquad (4.1)$$

where

$\ell$ = penetration distance after time, t

r = pore radius

$\gamma$ = surface tension

$\theta$ = contact angle

$\eta$ = liquid viscosity

This equation shows that the high surface tension ink is infiltrated in the fabric slowly, so the ink is held on the top of the fabric. The more ink holdout, the higher the ink color gamut is found. As shown in Figure 4-11, the higher surface tension of the surface modified pigmented inks produces the larger color gamut than the microencapsulated pigmented ink.

The gamut volumes of the two ink sets on the non-treated silk fabric are listed in Table 4-10. The larger gamut volume is presented by the surface modified pigment ink set than the other. It is common to consider the three relevant attributes of perception of color as hue and chroma, (or saturation) which are the colorfulness or richness of the color, and lightness, which refers to the amount of reflected light. These three attributes are described using the concept of color space, which shows a relation ship of colors to one another and which illustrates the three dimensional nature of color.

Table 4-10 Effect of pigment dispersion on color gamut volume

| Type of ink dispersion | Gamut volume |
|---|---|
| Surface modified pigmented ink | 7043 |
| Microencapsulated pigmented ink | 5337 |

4.3.4 Effect of Pigment Dispersion on Color Fastness

The requirement of no wet post processing to achieve the suitable color and durability is a common practice in pigmented inkjet inks. The color fastness of printed silk fabric was evaluated in several ways.

4.3.4.1 Crockfastness

Comparing the print durability of the inkjet ink made from different pigment dispersions, crockfastness or rub resistance is also needed to know along with other service properties. Table 4-11 indicates that the printed silk fabrics by both pigmented inkjet inks suffer from both dry crockfastness and wet crockfastness.

As expected, the microencapsulated pigmented ink would show the better rub resistance than the surface modified pigmented ink on the printed silk fabrics. Because the microencapsulated pigments are encompassed by a polymer shell, the higher amount of the carboxylate group can adhere better on the silk fiber to withstand rubbing.

Table 4-11 Effect of crockfastness on the non-treated silk fabric

| Type of ink dispersion | Color | Color staining | | | |
|---|---|---|---|---|---|
| | | Dry | | Wet | |
| | | Gray scale rating | Color change ($\Delta E^*_{ab}$) | Gray scale rating | Color change ($\Delta E^*_{ab}$) |
| Surface modified pigmented ink | Cyan | 3-4 | 8.8 | 1-2 | 36.1 |
| | Magenta | 3-4 | 7.3 | 2 | 21.3 |
| | Yellow | 3 | 12.7 | 2-3 | 20.4 |
| | Black | 3-4 | 6.8 | 1 | 47.0 |
| Microencapsulated pigmented ink | Cyan | 3 | 11.0 | 3 | 13.2 |
| | Magenta | 2 | 22.0 | 2-3 | 15.2 |
| | Yellow | 3 | 13.7 | 3 | 14.4 |
| | Black | 2-3 | 11.7 | 2 | 15.8 |

5 stands for excellent, 4 for good, 3 for fair, 2 for poor, 1 for very poor

Table 4-11 shows the crockfastness of the pigmented inkjet inks prepared by the different dispersion techniques printed on the silk fabric. The inks made from surface modification technique show the better dry crockfastness than the ink made from microencapsulation technique. On the contrary, the inks made from microencapsulation technique present the better wet crockfastness than the surface modified pigmented inks.

The cyan, magenta and yellow inkjet inks from both pigment dispersion exhibit almost the same dry crockfastness. On the contrary, the wet crockfastness of the microencapsulation pigmented inks is better than the other. Figure 4-8 shows the depth of ink penetration of the silk fiber. Figure 4-8 (a) shows the higher amount of the surface modified pigmented ink deposited on the surface of the fabric, which could be the attribute of the lower level of crockfastness. That is the ink holdout is higher, which is prone to be removed when it is crocked. For the microencapsulated pigmented inks, the ink can penetrate deeper, i.e. more ink is absorbed into the silk fibers as shown in Figure 4-8 (b). When it is wet crocked, less pigment is removed. Beside the influence of the ink pick-up, the damage of the fiber of fabric after crocking also affects the crockfastness level.

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4.3.4.2 Washfastness

The cyan, magenta, and yellow inks used in the pigment dispersion technique are PB 15:4, PR 122, PY 74, and PY 128 from which their chemical class and structure are shown in Table 4-1 and Figure 4-1. The corresponding color pigments are resistant to alkaline ratings of 5, 5, 4, and 5. [20] Commonly, the absence of acid group is essential to alkaline resistance. In this research the pigments were modified using the sulfonate group for the surface modified pigmented dispersion and the carboxylate group for the microencapsulated pigmented dispersion. The sulfonate group is a strong acid and renders the lower soap resistance than the carboxylate group, which is a weak acid. The mutual compatibility of S-711 polymer binder and encapsulation polymer is good, the soap resistance should also be good. The result of washfastness of the silk printed by the two sets of pigment dispersion is shown in Table 4-12.

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Table 4-12 Effect of pigment dispersion on washfastness

| Color | Color change | | | |
|---|---|---|---|---|
| | Surface modified pigmented ink | | Microencapsulated pigmented ink | |
| | Gray scale rating | Color change ($\Delta E^*_{ab}$) | Gray scale rating | Color change ($\Delta E^*_{ab}$) |
| Cyan | 1-2 | 8.5 | 2-3 | 5.3 |
| Magenta | 1-2 | 8.3 | 2-3 | 5.1 |
| Yellow | 1-2 | 9.4 | 1 | 11.6 |
| Black | 1-2 | 9.1 | 1-2 | 8.6 |

5 stands for excellent, 4 for good, 3 for fair, 2 for poor, 1 for very poor

One can see that the washfastness of surface modified pigmented inks printed on the silk is much lower than those of microencapsulated pigmented inks. The greater and loose ink holdout on the printed silk fabric could be easily washed off. This result is important in the pigmented ink system. The pigments are not absorbed in the fiber structure and fix on the surface of fiber. Therefore the fixing by the polymer is necessary.

4.3.4.3 Lightfastness

Lightfastness of pigmented inks was compared using a Xenon weather-o-meter. The printed fabrics were kept under ISO 105-B02 condition for 300 h. The change of color was calculated using Equation 3-5, which is listed in Table 4-13. It is ordinary known that the pigments exhibit the high lightfastness. Lightfastness depends mainly on the molecular formula of pigments. The pigments, which are used in the two types of inks, made from different dispersion techniques are excellent in lightfastness. [20] When the inkjet inks were printed on the silk fabric, the pigments still exhibit the excellent lightfastness. The color change expressed in the terms of $\Delta E^*_{ab}$ is shown in Table 4-13. The $\Delta E^*_{ab}$ values of the corresponding color before and after exposure are significantly different.

Table 4-13 Effect of pigment dispersion on lightfastness

| Color | Surface modified pigmented ink | | Microencapsulated pigmented ink | |
|---|---|---|---|---|
| | Color Index | Color change ($\Delta E^*_{ab}$) | Color Index | Color change ($\Delta E^*_{ab}$) |
| Cyan | PB 15:4 | 6.8 | PB 15:4 | 4.5 |
| Magenta | PR 122 | 4.4 | PR 122 | 5.6 |
| Yellow | PY 74 | 7.0 | PY 128 | 2.8 |
| Black | PBk 7 | 2.2 | PBk 7 | 2.0 |

Considering in the structure of pigment (see Table 4-1 and Figure 4-1), the pigment blue and pigment red, PB 15:4 and PR 122 are the phthalocyanine and quinacridone, respectively. The symmetrical polycyclic pigments tend to possess high lightfastness. The yellow pigment, PY 74, containing the diazo component, alkyl and alkoxy groups in the coupling component tends to improve lightfastness. The chlorine group, as well as the high molecular weight of the azo component contributes to much better lightfastness.[20]

### 4.3.5 Effect of Pigmented Inks on Wearing Comfort

#### 4.3.5.1 Air Permeability

The amount of air passing through the printed fabric with the surface modified pigment inks and microencapsulated pigment inks is reported in Figure 4-12 which show the change of the air permeability of printed silk fabric when compared with the non-printed silk fabric.

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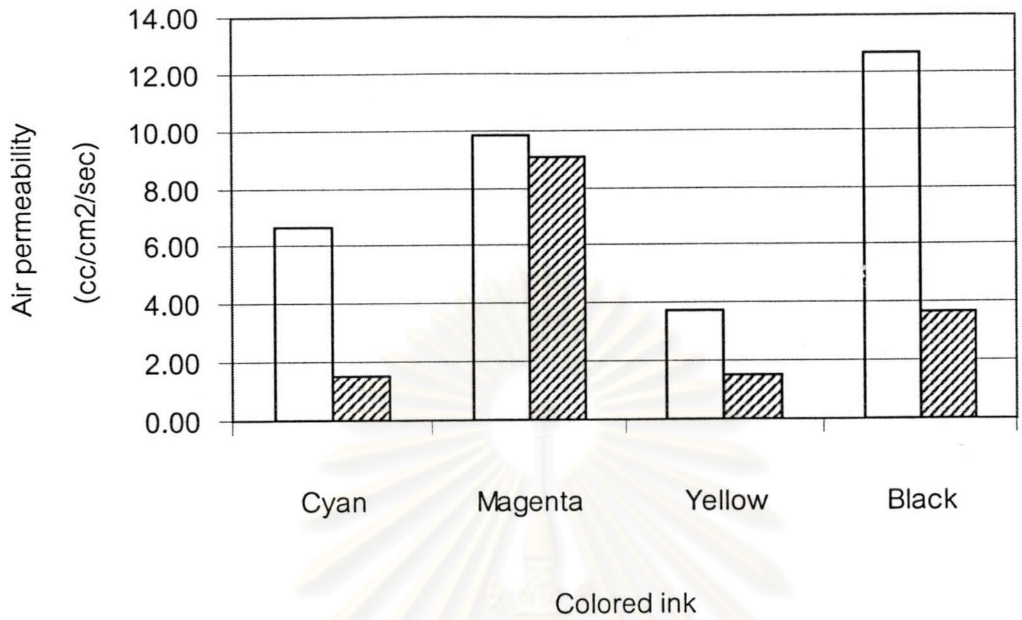
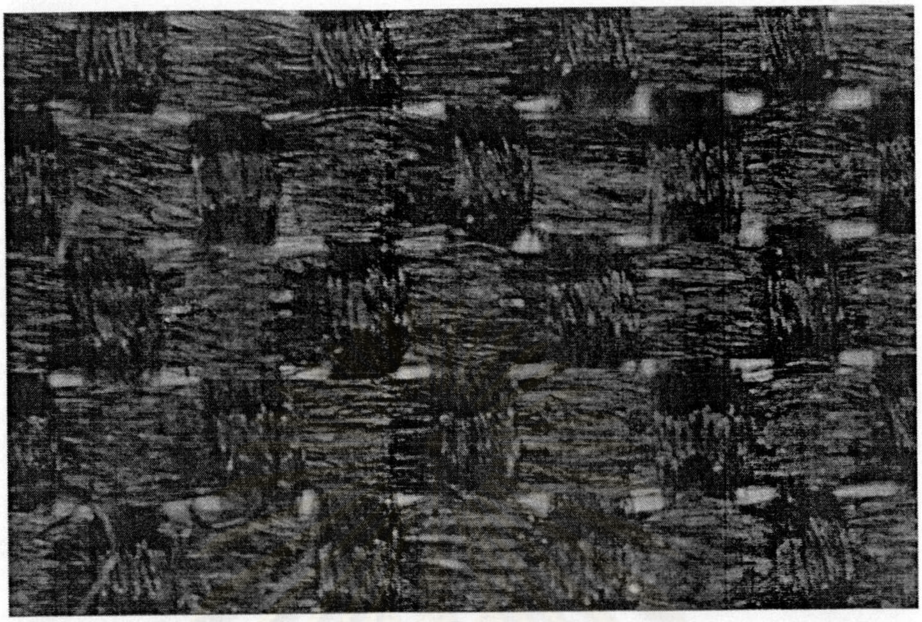
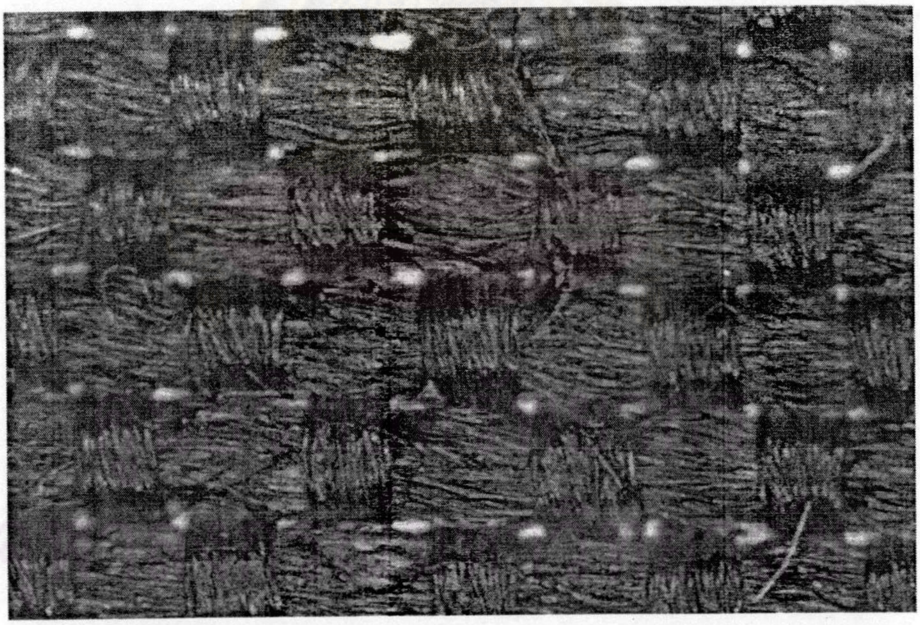



**Figure 4-12** The change of air volume after printing on silk fabric ( □ Surface modified pigmented inks, ▨ Microencapsulated pigmented inks)

Air permeability is the ability of air to pass through a fabric. The most common permeability of fabrics to gases ever measured is that to air or to wind. Obviously, wherever openings between the fibers are large, a good deal of air will pass through the fabric.



(a)



(b)

Figure 4-13 Photomicrographs of the non-treated printed silk fabric : (a) the surface modified pigmented ink, (b) the microencapsulated pigmented ink

As shown in Figures 4-13 (a) and (b), we can see that there are voids or spaces between fibers, regardless of the fibers being treated or not. When the silk fiber was printed by either inkjet inks, the ink coverage and penetration on the fabric surface govern the extent of air permeability. As illustrated in Figure 4-12, the ink penetration of microencapsulated pigmented inkjet ink is higher, therefore, the ink coverage on the fiber surface is also higher, which fills the voids, interstitial cavities to decrease the air volume through the fabric, as shown in the results of Figure 4-12.

4.3.5.2 Bending Stiffness

Stiffness is the ability of a material to resist deformation. The bending length of fabric reflects its stiffness when bent in one plane under the force of gravity. The higher bending length value means the higher stiffness of the fabric. Table 4-14 shows the bending length of printed silk fabric.

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Table 4-14 Bending length of the non-treated, printed silk fabric

| Color | Bending length (cm) | | | |
|---|---|---|---|---|
| | Surface modified pigmented ink | | Microencapsulate pigmented ink | |
| | Warp direction | Weft direction | Warp direction | Weft direction |
| Non-printed | 1.98 | 4.89 | 1.98 | 4.89 |
| Cyan | 2.70 | 5.96 | 2.94 | 5.99 |
| Magenta | 2.73 | 6.03 | 2.94 | 6.13 |
| Yellow | 2.53 | 5.28 | 2.11 | 4.68 |
| Black | 2.61 | 5.86 | 2.83 | 5.91 |
| Red | 2.56 | 5.79 | 2.65 | 5.41 |
| Green | 2.60 | 5.71 | 2.78 | 5.26 |
| Blue | 2.58 | 5.98 | 2.60 | 5.04 |

Then, the relative bending length was calculated from Equation 4-2, which results are shown in Figure 4 –14.

$$\text{Relative bending length} = \frac{\text{bending length of printed fabric}}{\text{bending length of non-printed fabric}} \qquad (4\text{-}2)$$

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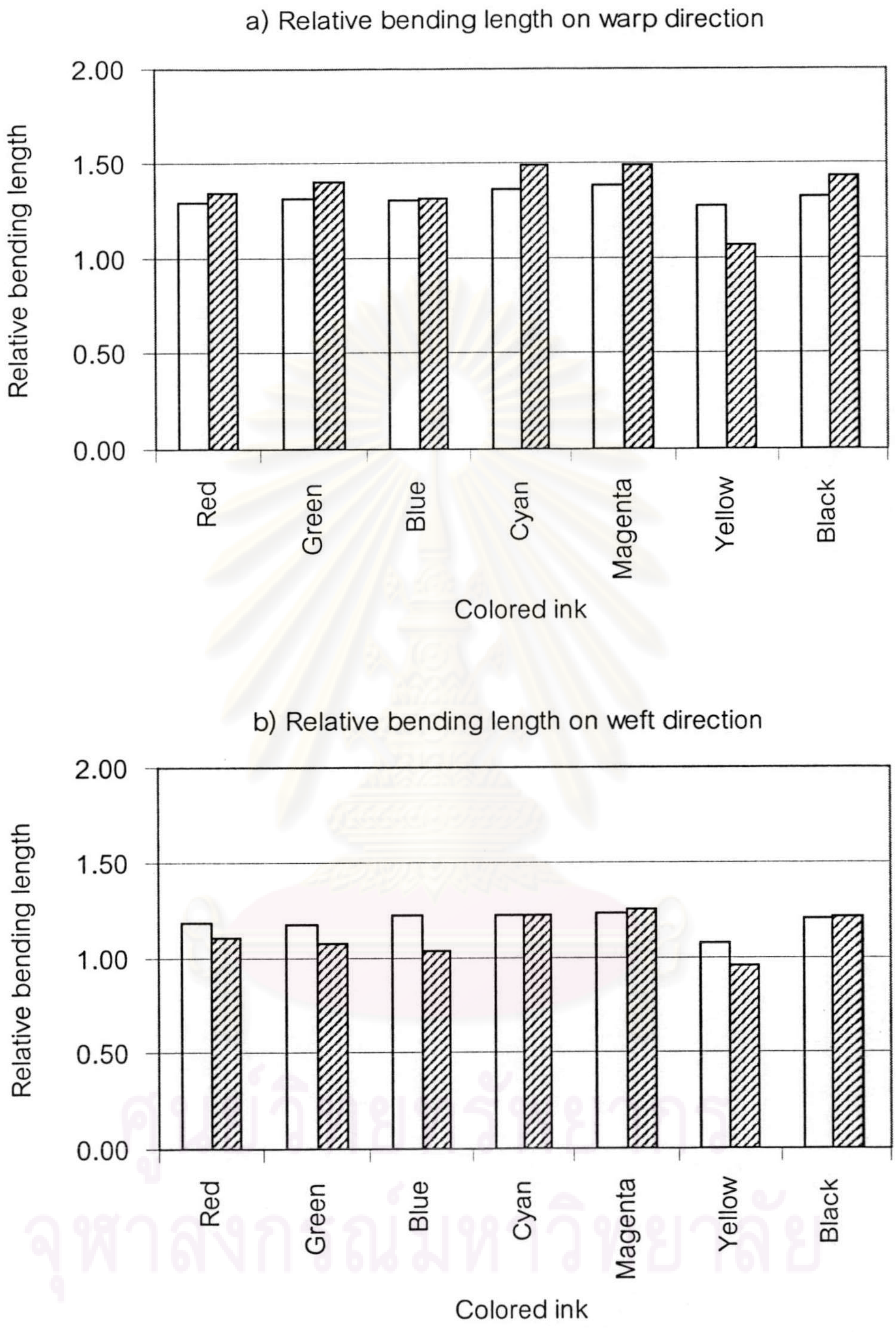


**Figure 4.14** Relative bending length of the non-treated printed silk fabric, (a) warp direction, (b) weft direction (☐ Surface modified pigmented inks, ▨ Microencapsulated pigmented inks)

The ink made from the surface modification technique produced the minimum increase in bending length in both fiber directions. The printed silk fabric by the microencapsulated pigmented inks showed the minimum relative bending length on cyan, magenta, yellow and black color printed silk fabric. The photomicrographs in Figure 4-8, after the ink has been dried, some pigment particles and solid binder particles densely deposited on the fibers on the surface of the fabric. Because the silk fiber is hydrophilic, therefore, the water-based inks were absorbed and swelled the fibers by both the ink vehicle and water. Of course, both fiber directions could absorb the aqueous vehicle of the ink. The term weft direction is used to indicate the direction analogous to coursewise or filling direction in knitted or woven fabrics. The stiffness of the weft direction is lower than the warp direction, because the density of thread of the warp direction is higher than the weft direction. The stiffness of both directions was calculated by Equation 4-3, which results are shown in Table 4-15.

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$$G = W \times c^3 \qquad (4\text{-}3)$$

where

$G$ = flexural rigidity, mg cm,

$W$ = fabric mass per unit area, mg cm$^{-1}$,

$c$ = bending length, cm.

Table 4-15 The stiffness of the non-treated printed silk fabric

| Color | Stiffness (mg cm) | | | |
|---|---|---|---|---|
| | Surface modified pigmented ink | | Microencapsulated pigmented ink | |
| | Warp direction | Weft direction | Warp direction | Weft direction |
| Non-printed | 85.59 | 1289.32 | 85.59 | 1289.32 |
| Cyan | 218.64 | 2354.60 | 285.57 | 2418.32 |
| Magenta | 234.02 | 2529.44 | 262.87 | 2382.98 |
| Yellow | 182.45 | 1663.51 | 96.46 | 1045.40 |
| Black | 203.60 | 2300.73 | 251.51 | 2305.74 |
| Red | 186.53 | 2149.00 | 212.11 | 1807.22 |
| Green | 189.52 | 2010.12 | 243.01 | 1657.36 |
| Blue | 181.51 | 2267.71 | 204.57 | 1487.86 |

## 4.4 Effect of Pretreatment on Print Quality

A good quality inkjet print is characterized by high dot intensity and low print-through. The printing of textiles has shown that the ink has been jetted on the fabric with a pick up sufficiently high to achieve adequate penetration, there is a pronounced tendency for the ink to wick laterally along the individual yarns of the fabric.[22] The pretreatment process is necessary for the inkjet textile printing. The fabric is porous, soft and pliable. In order to achieve well-registered, clear and sharp printing results, the fabric need to be pretreated. The silk fabric was treated by 10% Sanfix 555 (cationic acrylate polymer) and 10% urea solution by 100 % pick up ratio of padding. The cationic acrylate polymer can interact with the anionic dye and pigment rapidly, fix and bond to the surface of fiber of textile. At the same time this type of polymer limits the excess spreading of inks, the effect of which is a well-defined shape of dots, color and print quality.

### 4.4.1 Effect of Pretreatment on Optical Density

The printed colors onto the pretreatment silk fabric were evaluated for print density in comparison with the non-treated silk fabric. Table 4-16 shows the higher print density on the pretreated fabric.

Table 4-16 Optical density on the pretreated fabric

| Color | Surface modified pigmented ink | | Microencapsulated pigmented ink | |
|---|---|---|---|---|
| | Non-treated silk fabric | Pretreated silk fabric | Non-treated silk fabric | Pretreated silk fabric |
| Cyan | 1.29 | 1.45 | 1.20 | 1.36 |
| Magenta | 1.30 | 1.46 | 1.10 | 1.35 |
| Yellow | 1.43 | 1.52 | 1.14 | 1.34 |
| Black | 1.40 | 1.45 | 1.27 | 1.31 |

Figures 4.15 and 4.16 show the SEM photographs of the non-treated and pretreated silk fabric. Surface enlargement was found in both the non-treated and pretreated silk fabrics. The pretreatment reagent was absorbed on the surface of fiber. Consequently, the yarn size increased, which retarded the absorption of inks. Upon printing, pigments were deposited on the layer of pretreatment while the ink vehicle and solvent gradually penetrated the fabric. Due to the pigment deposition, the ink penetration becomes less than that on the non-treated fabric, as shown in Figures 4-17 (a) and 4-17 (b) in comparison with Figures 4-8 (a) and 4-8 (b).

Figure 4.15 SEM photomicrograph of the non-treated silk fabric

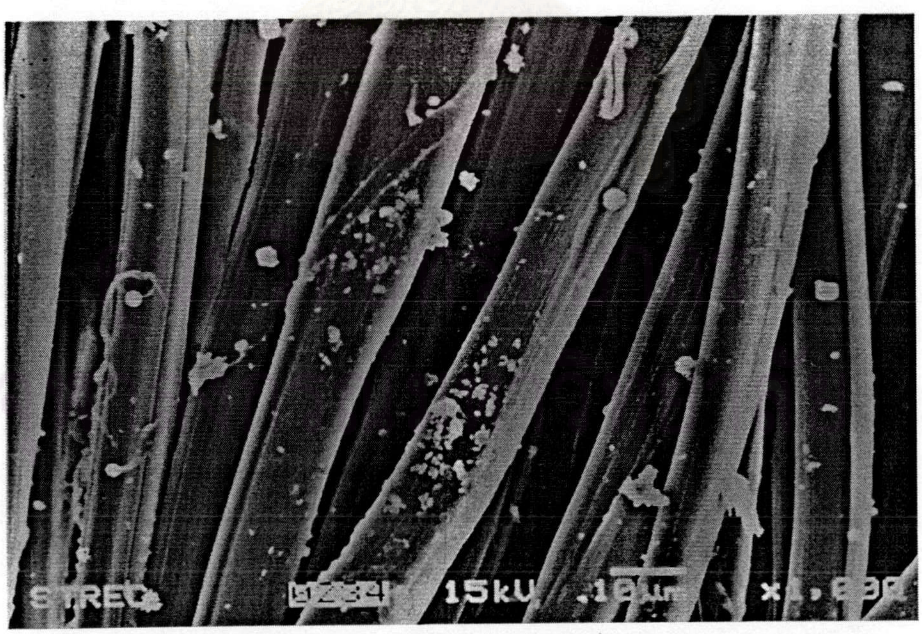
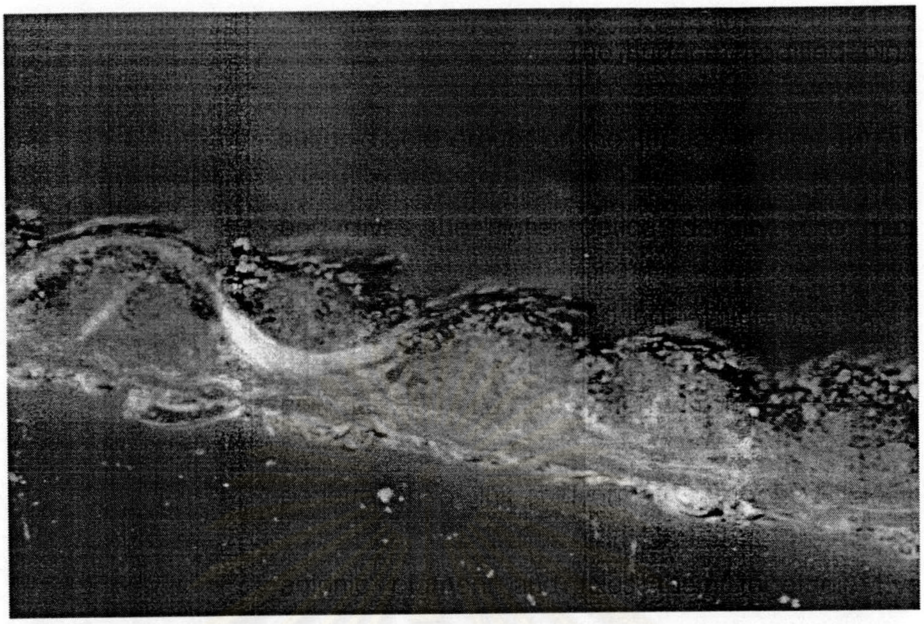
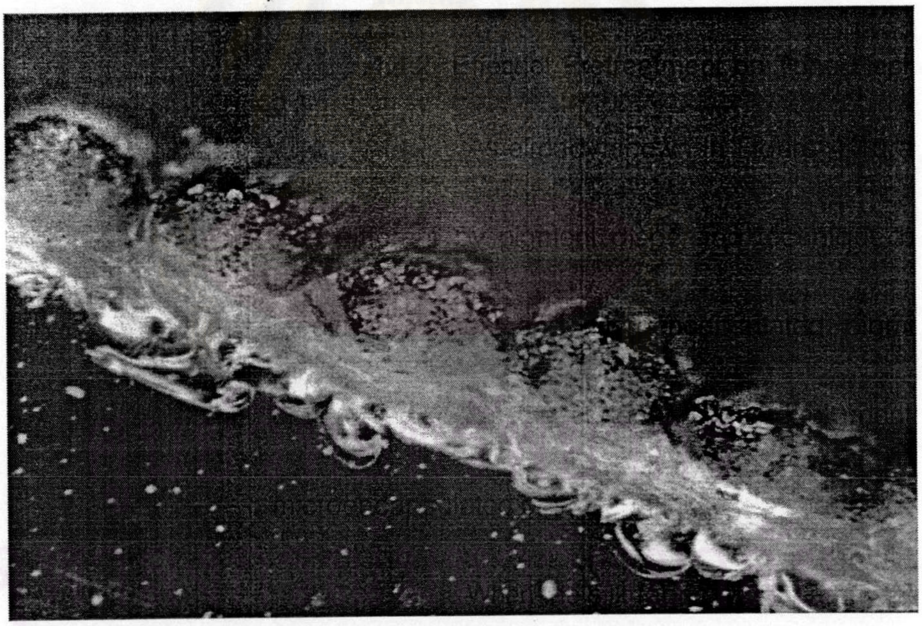


Figure 4.16 SEM photomicrograph of the pretreated silk fabric



(a)



(b)

Figure 4-17 The cross section of photographs showing the depth of ink penetration on the pretreated silk fabric, (a) the surface modified pigmented ink, (b) the micro encapsulated pigmented ink.

The surface modified pigmented ink has increased number of sulfonic acid groups on the surface of pigment, so the penetration speed becomes slow and gives the higher optical density. The microencapsulated pigmented ink gives increased penetration speed, which reduces the optical density. Sanfix 555, a commercial cationic polymer, which was used to treat the silk fabric prevents excess penetration of ink solution by a dipole-dipole interaction with anionic moiety in the anionic pigment and fixes them together. Even thought the original property of pigmented solution still shows, some difference on penetration depth, the depth of ink penetration on the treated silk fabric is shown in Figure 4-17.

### 4.4.2 Effect of Pretreatment on Tone Reproduction

As already shown in Figure 4-10, the tone reproduction of the cyan ink was different by pigment dispersion technique between the inks made from surface modified pigment and microencapsulated pigment. For the non-treated silk fabric, optical density of the surface modified pigmented ink was higher than that of the microencapsulated pigmented ink at the shadow area.

When the silk fabric was treated, the optical density of printed fabric from the both inks microencapsulated pigmented ink and the surface modified pigmented ink was similar. The tone reproduction of the surface modified pigmented ink is insignificantly higher that that of microencapsulated pigmented ink for all colors (Figures 4-18 and 4-19).

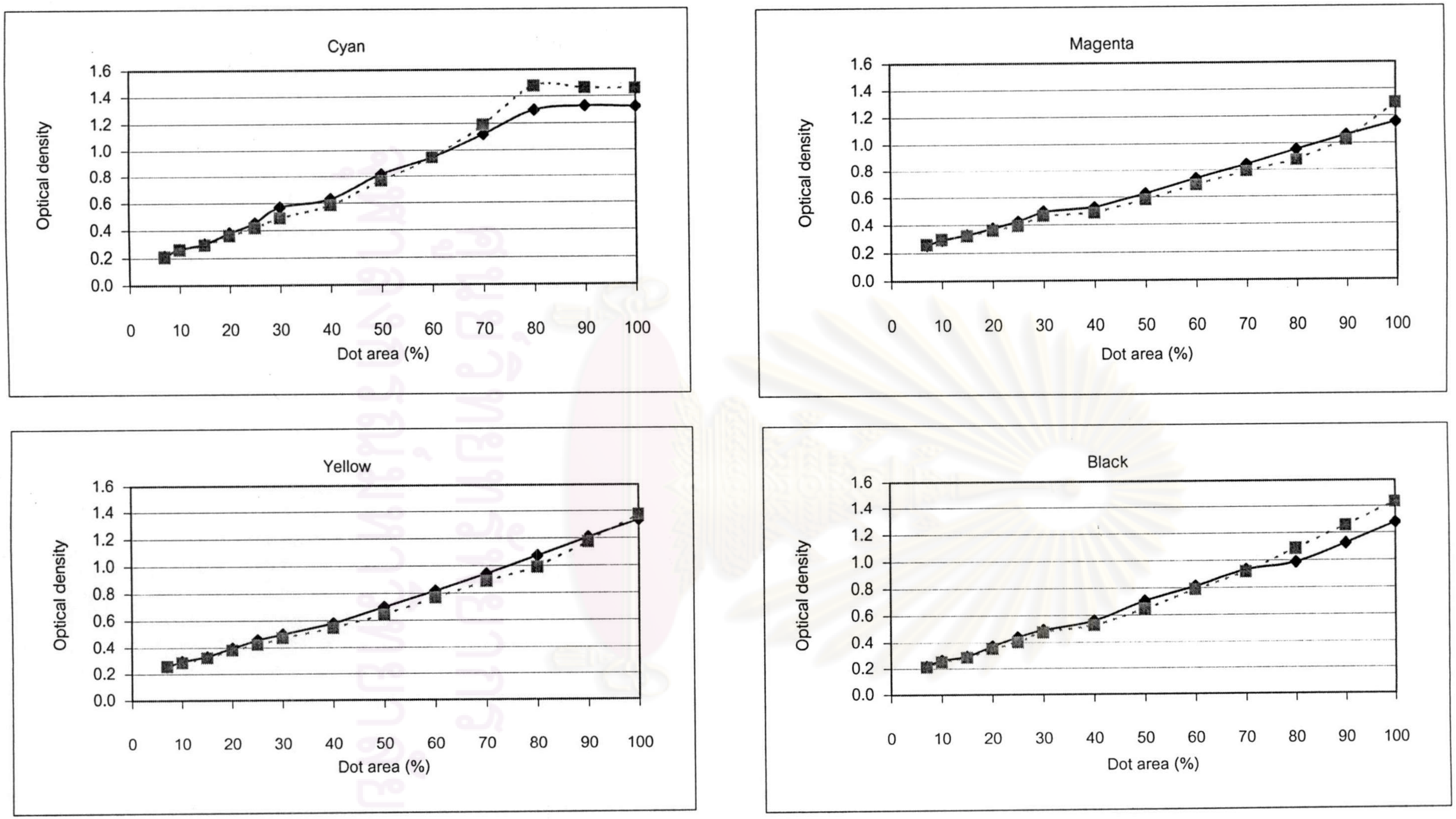



**Figure 4.18** Effect of pretreatment on tone reproduction of the surface modified pigmented inks ( ——— non treated fabric, ........ treated fabric)

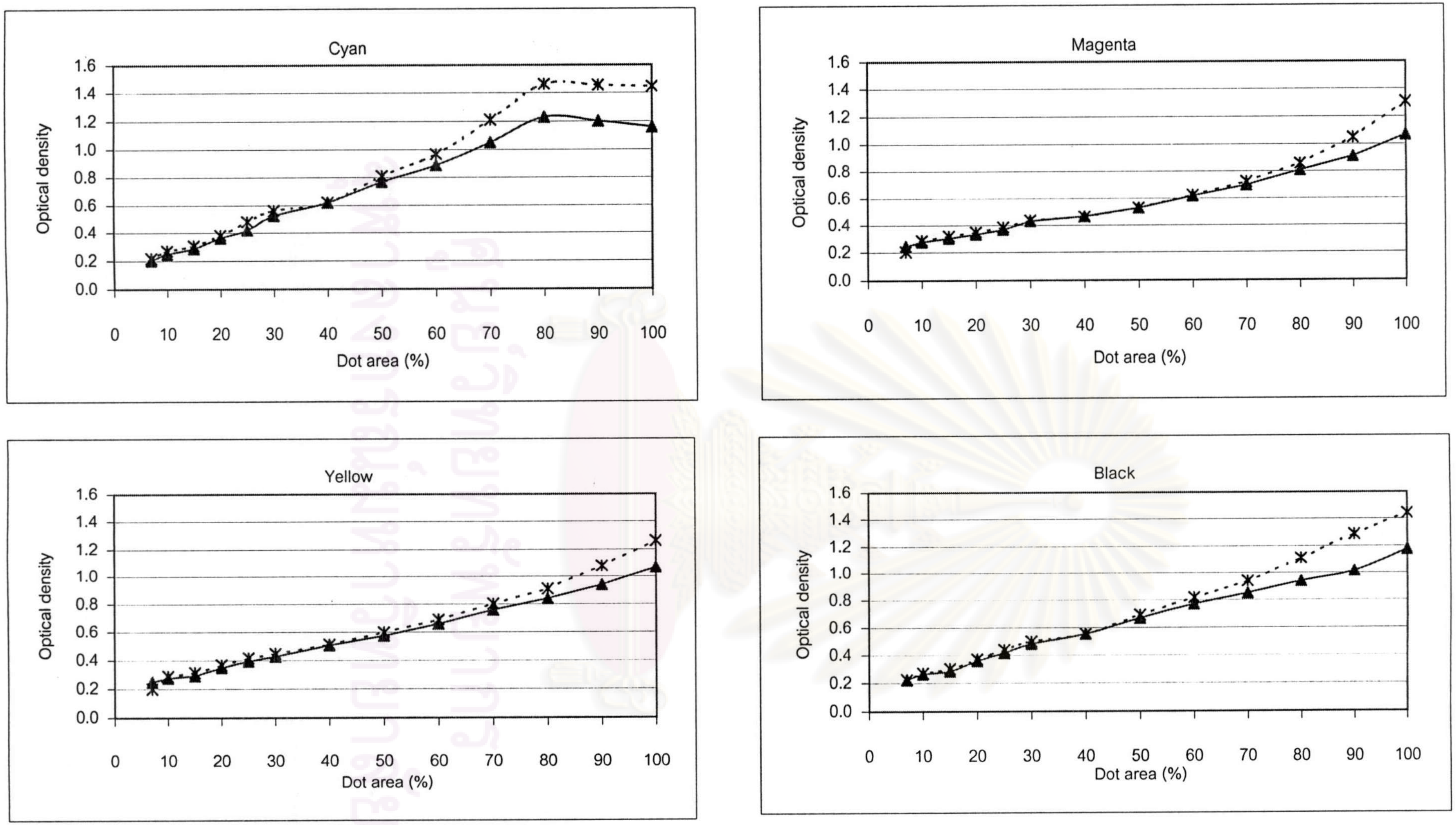



Figure 4.19 Effect of pretreatment on tone reproduction of the microencapsulated pigmented inks ( ——— non treated fabric, ▪▪▪▪▪▪▪ treated fabric)

4.4.3 Effect of Pretreatment on Color and Color Gamut

The depth of the ink penetration could indicate the amount of the ink localized on the surface of fabric. Since the greater amount of pigment was deposited on the surface of pretreated silk fabric than that on the surface of the non-treated silk fabric, consequently, the color of the pretreated silk fabric was higher than that of the non-treated silk fabric. As shown in Figures 4-20 and 4-21, the pretreated silk fabric surface is flatten, the color value is thus higher.

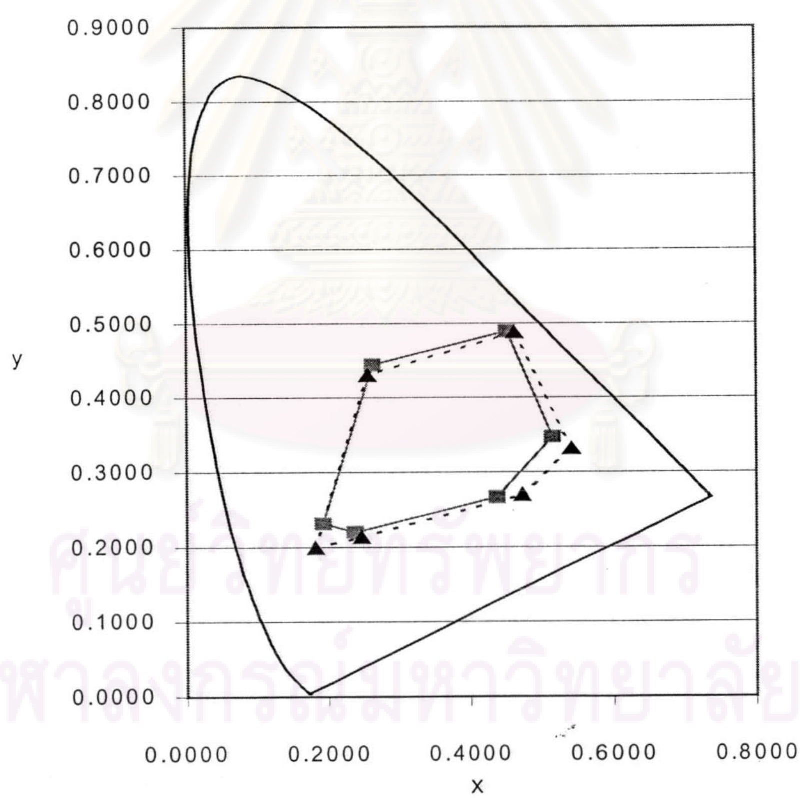



Figure 4-20 Effect of fabric pretreatment on color gamut printed by the surface modified pigmented ink (—— Non-treated printed silk fabric, - - - Pretreated printed silk fabric)

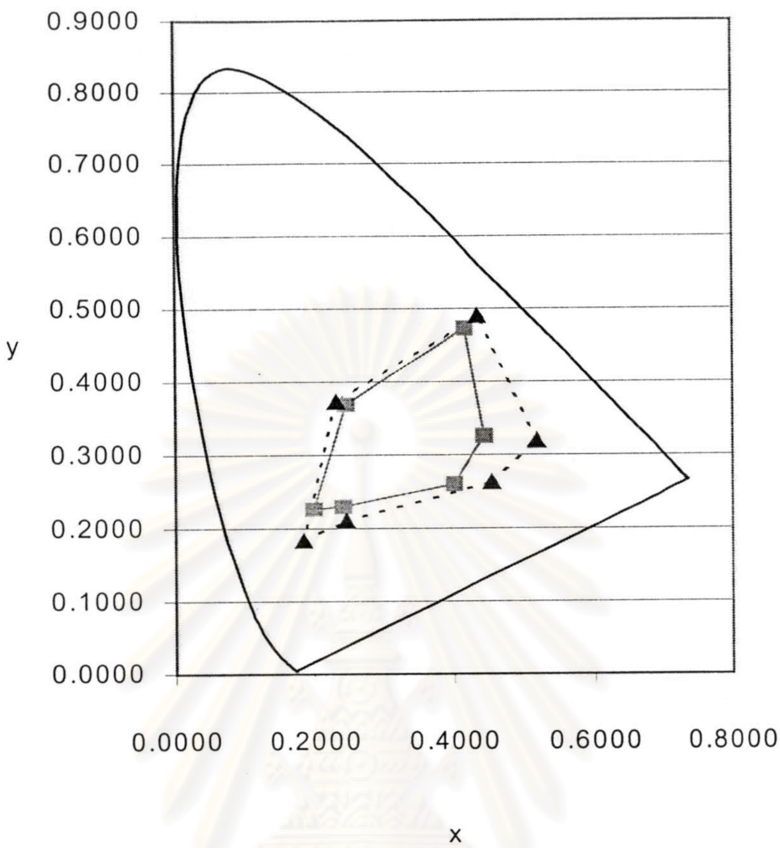



Figure 4-21 Effect of fabric pretreatment on color gamut printed by the micro-encapsulated pigmented ink ( —— Non-treated printed silk fabric, - - - Pretreated printed silk fabric)

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With the same printed fabrics as shown in Figure 4-17 and Table 4-18, the color gamut and gamut volume of the pretreated fabrics were greater than the printed colors on the non-treated fabrics, especially with the ink made from the microencapsulated pigment. As shown in Figure 4-16, the fabric pretreated with the particular padding solution has a smooth surface and increased surface area for absorbing the ink vehicle. Regardless of a padding or pretreatment, the printed fabric showed that the ink had been jetted nicely on the fabric with a sufficiently high pick-up to achieve adequate penetration, there is a pronounced tendency for the ink to wick laterally along the individual yarns of the fabric, the ink will then be spread.

To confirm the flatten surface of pretreated non-printed silk fabric, the standard deviation of optical density on the pretreated silk fabric was analyzed using the optical density data from the microdensitometric measurement. In Table 4-17, the pretreated non-printed silk fabric gave the lower standard deviation, that means, the smoothness of the printed surface increased. In addition, the pretreated printed silk fabric produced significantly the larger color gamut and gamut volume than the non-treated printed silk fabric for the microencapsulated pigmented ink; and to a lesser extent for the surface modified pigmented ink.

Moreover, variation of the reflection light by microdensitometry, might reflect the homogeneity of the ink layer on the fabric after drying. The microencapsulated pigmented ink improved or reduced light scattering because the polymer shell of the microcapsule along with the ink vehicle contributes to the better film forming and the higher smoothness of the ink film surface.

Table 4-17 The standard deviation of optical density data from the microdensitometer

| Type of ink dispersion | Color | Non-treated silk fabric | Pretreated silk fabric |
|---|---|---|---|
| Surface modified pigmented ink | Non-printed | 0.0792 | 0.0552 |
| | Cyan | 0.1192 | 0.1284 |
| | Magenta | 0.1059 | 0.0704 |
| | Yellow | 0.0491 | 0.0469 |
| | Black | 0.1355 | 0.1150 |
| Microencapsulated pigmented ink | Cyan | 0.1373 | 0.1281 |
| | Magenta | 0.0753 | 0.0751 |
| | Yellow | 0.0595 | 0.0529 |
| | Black | 0.1787 | 0.1365 |

As for a good quality inkjet print, print through is not only a function of droplet penetration, but also of the light scattering properties of the substrate. The pretreatment exhibited higher color values because the pretreatment reagent had been accepted on the fabric before excessive spreading of the inkjet inks and high swelling capacity took place, comparable with the fibrous material in retention of the ink. The pre-treatment on fabrics before printing is still necessary to prevent the entry of ink liquid to the capillary spaces and increase the availability of surface area for the drops rapidly permeating into the fabric. The amount of the pigment on the surface of pretreated fabric was more than the amount of the pigment on the surface of the non-treated fabric, therefore the color performance of the pretreated fabric was better than the non-treated fabric.

Table 4-18 Effect of pretreatment on color gamut volume

| Type of ink dispersion | Gamut volume | |
|---|---|---|
| | Non-treated fabric | Pretreated fabric |
| Surface modified pigmented ink | 7043 | 8475 |
| Microencapsulated pigmented ink | 5337 | 7933 |

Considering the non-treated and the pretreated silk fabrics at the solid pattern, which the color chroma is shown in Table 4-19. The pretreated silk fabric with Sanfix 555 gave the increasing chroma with the high color values ($a*$ and $b*$). The reason might be that the pretreatment reagent, cationic acrylate polymer, has a large amount of cations in the molecular structure, the interaction between the cations from the pretreatment reagent and the anions from the pigment dispersion gave a good fixing and a higher color value. The correlation between lightness and chroma is shown in Figures 4-22 and 4-23. The lightness and chroma of the color of the prints increased when the ink is each printed onto the pretreated fabric and purity of the color also increased.

Table 4-19 Effect of the pretreatment on chroma

| Type of ink dispersion | Color | Chroma | | |
|---|---|---|---|---|
| | | Non-treated silk fabric | Pretreated silk fabric | % Increase |
| Surface modified pigmented ink | Cyan | 31.68 | 33.44 | 5.57 |
| | Magenta | 48.05 | 51.06 | 6.28 |
| | Yellow | 84.26 | 87.65 | 4.02 |
| | Red | 50.92 | 55.20 | 8.40 |
| | Green | 36.37 | 30.53 | -16.05 |
| | Blue | 25.93 | 24.51 | -5.46 |
| Microencapsulated pigmented ink | Cyan | 32.90 | 38.94 | 18.39 |
| | Magenta | 46.26 | 51.92 | 12.24 |
| | Yellow | 66.91 | 81.47 | 21.77 |
| | Red | 38.52 | 52.95 | 37.48 |
| | Green | 28.88 | 28.55 | -1.13 |
| | Blue | 26.04 | 27.37 | 5.12 |

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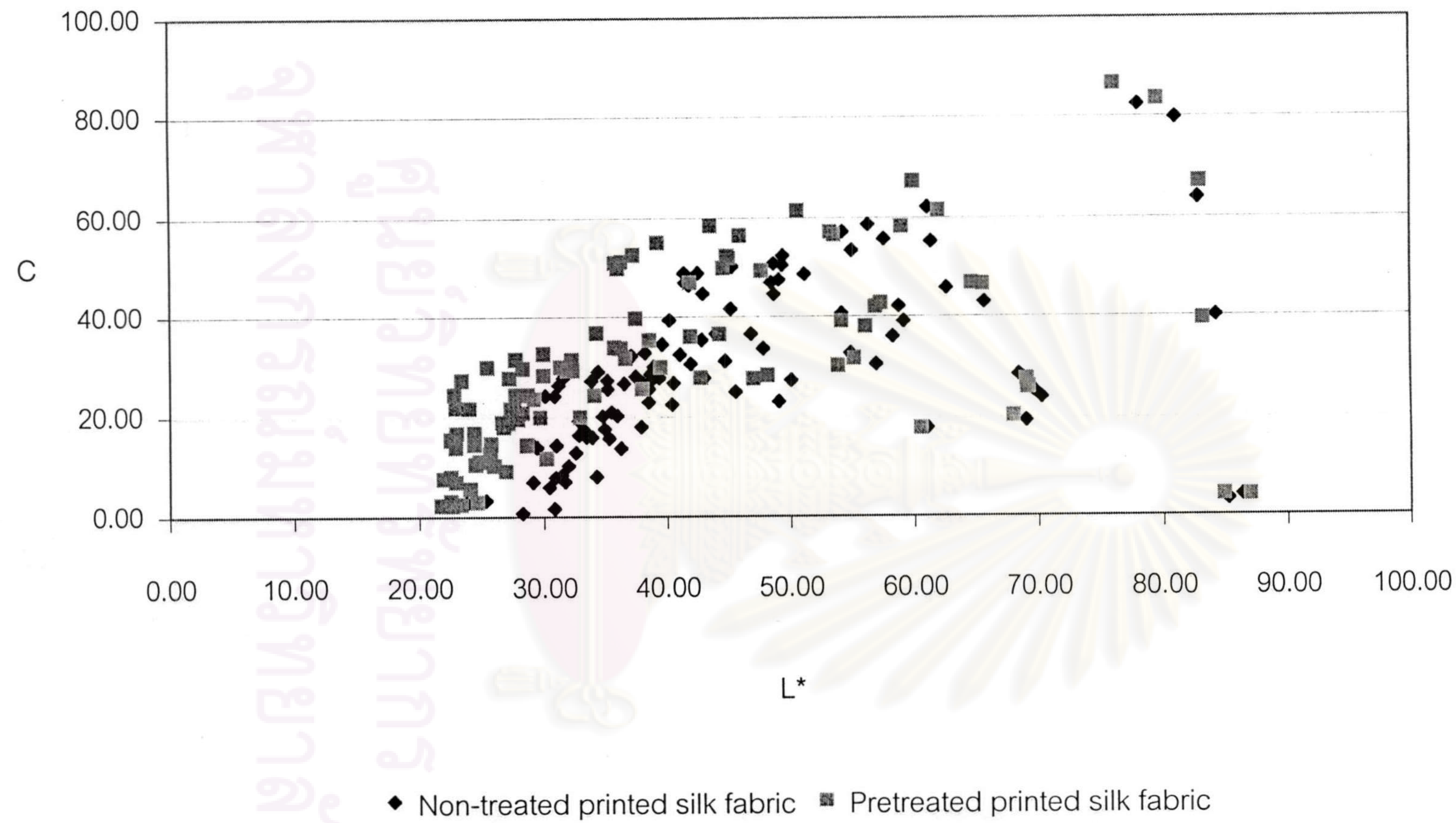


Figure 4.22 Correlation of lightness and chroma of the surface modified pigmented ink

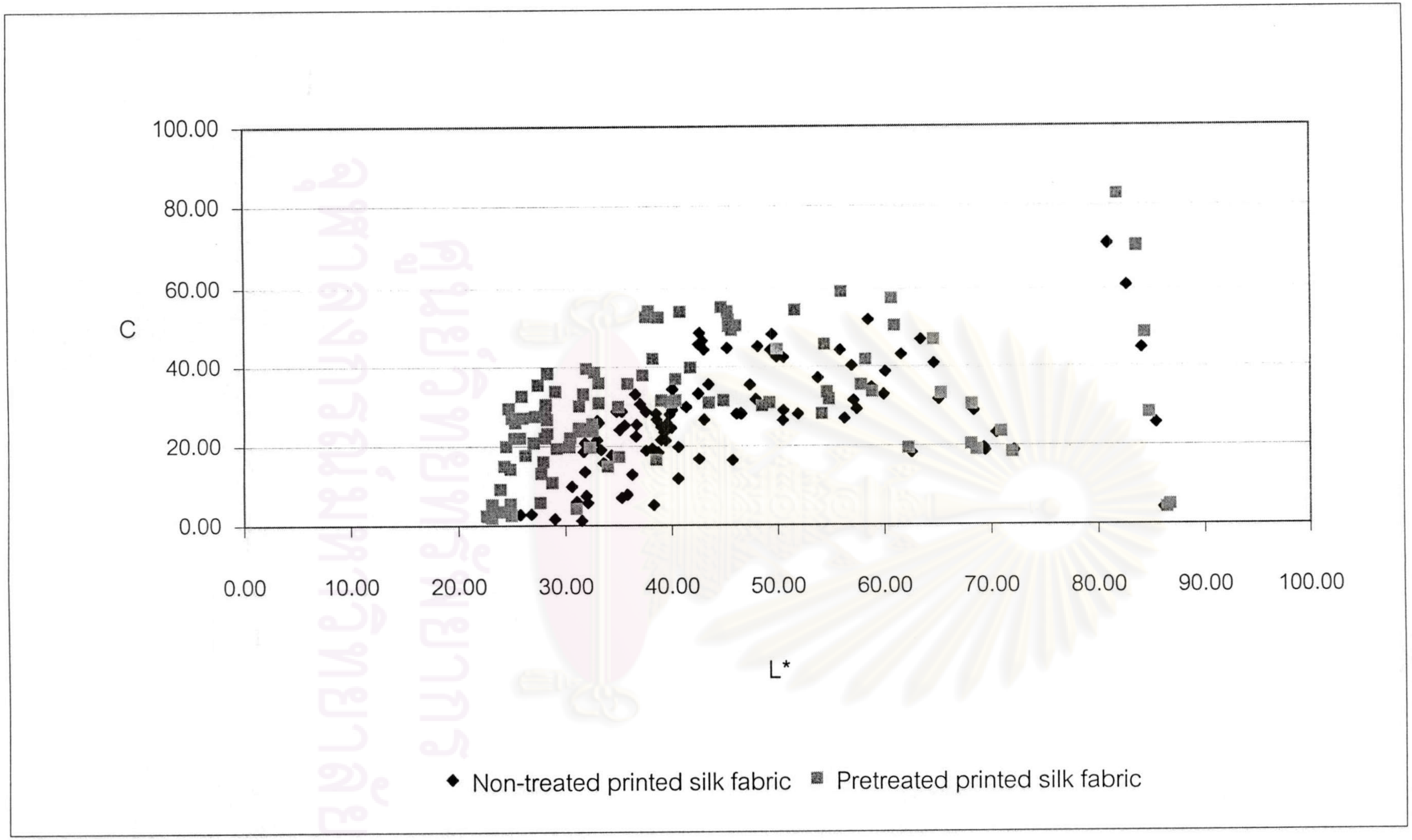


Figure 4.23 Correlation of lightness and chroma of the microencapsulated pigmented ink

#### 4.4.4 Effect of the Pretreatment on Color Fastness

##### 4.4.4.1 Crockfastness

Table 4-20 Effect of crockfastness on the treated fabrics

| Type of ink dispersion | Color | Color staining | | | |
|---|---|---|---|---|---|
| | | Dry | | Wet | |
| | | Gray scale rating | Color change ($\Delta E^*_{ab}$) | Gray scale rating | Color change ($\Delta E^*_{ab}$) |
| Surface modified pigmented ink | Cyan | 3 | 11.3 | 1 | 49.8 |
| | Magenta | 2 | 20.1 | 1 | 42.3 |
| | Yellow | 2-3 | 21.9 | 1 | 50.2 |
| | Black | 3 | 10.9 | 1 | 54.8 |
| Microencapsulated pigmented ink | Cyan | 2-3 | 18.0 | 1-2 | 29.5 |
| | Magenta | 2 | 25.1 | 1-2 | 32.1 |
| | Yellow | 3 | 13.2 | 2-3 | 22.9 |
| | Black | 2 | 17.4 | 1 | 49.3 |

5 stands for excellent, 4 for good, 3 for fair, 2 for poor, 1 for very poor

Table 4-20 shows crockfastness of the printed fabrics on the treated printed silk fabric. Sanfix 555 contributes a good fixing characteristic by an interaction between the cationic acrylate polymer and anionic pigment. Good adhesion

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of the printed fabric, pretreated with Sanfix 555, showed the high level of dry crockfastness. The inks made from the surface modified pigments gave a different crockfastness of 0.5 to 1 unit in comparison with the inks made from the microencapsulated pigment. The printed silk fabrics yielded a lower wet crockfastness as a direct attribute from their pretreatment reagent of a hydrophilic nature, which is relatively water-soluble. The microencapsulated pigmented ink shows the better wet crockfastness.

#### 4.4.4.2 Washfastness

Table 4-21 Effect of the pretreatment on washfastness

| Color | Color change | | | |
|---|---|---|---|---|
| | Surface modified pigmented ink | | Microencapsulated pigmented ink | |
| | Gray scale rating | Color change ($\Delta E^*_{ab}$) | Gray scale rating | Color change ($\Delta E^*_{ab}$) |
| Cyan | 4 | 1.7 | 4 | 1.4 |
| Magenta | 4 | 1.6 | 5 | 0.4 |
| Yellow | 2 | 5.7 | 4 | 1.7 |
| Black | 4-5 | 1.0 | 4-5 | 1.1 |

5 stands for excellent, 4 for good, 3 for fair, 2 for poor, 1 for very poor

On the non-treated fabric, the pigments in the microcapsules are protected by the acrylate polymer shell, which is one of the advantages of this technology. The pigments in the microcapsules are not directly attached by the washing solution. Physico-chemical interaction of the padding solution behavior with the pigments of ink could be one solution to enhance better adhesion of the inkjet ink film on the padding chemicals of silk fabric. Therefore, the stronger interface between the padding chemicals and polyacrylate contributes to a better washfastness. The cationic acrylate polymer can be attached with the surface modified or polyacrylate shell anionic pigment, fix and bond to the surface of fiber of textile. From the structure of pretreatment agent in Figure 3-1, the electron-with-drawing group adjacent to the pigment can improve alkaline resistance.

### 4.4.5 Effect of the Pretreatment on Wearing Comfort

#### 4.4.5.1 Air Permeability

The air permeability of the pretreated and printed silk fabrics was observed and compared between the treated printed silk fabric and the treated non-printed silk fabric. Figure 4-24 shows the changes of air permeability of the pretreated silk fabric before and after printing. The air permeability of the printed fabrics was decreased because more coating film was deposited onto the treated layer. As mentioned previously, the pretreatment reagent may swell the silk fibers, which enlarges them and results in interfiber voids. The ink components cover these voids and thus reduce the air permeability.

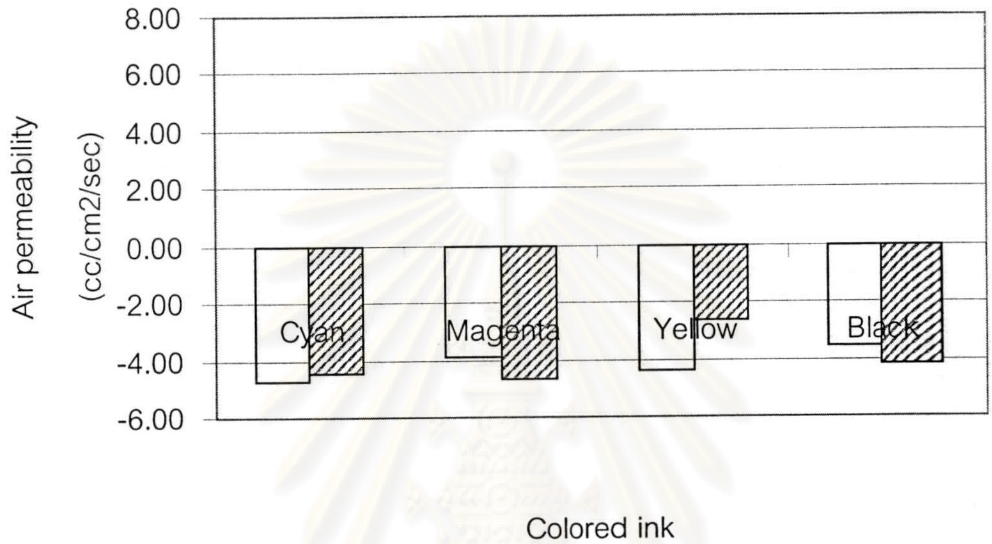


Figure 4-24 Changes in air permeability caused by inkjet printing of ☐ surface modified pigmented inks, and ▨ microencapsulated pigmented inks

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4.4.5.2 Bending Stiffness

The bending length of the pretreated printed fabric was evaluated and presented in Table 4-22. The relative bending length of printed fabric was compared with the pretreated non-printed fabric using Equation (4-1) and the result is shown in Figure 4-25.

Table 4-22 Bending length of the pretreated printed silk fabric

| Color | Bending length (cm) | | | |
|---|---|---|---|---|
| | Surface modified pigmented ink | | Microencapsulated pigmented ink | |
| | Warp direction | Weft direction | Warp direction | Weft direction |
| Non-printed | 2.35 | 5.25 | 2.35 | 5.25 |
| Cyan | 2.44 | 5.30 | 2.78 | 5.56 |
| Magenta | 2.39 | 5.30 | 2.59 | 5.36 |
| Yellow | 2.53 | 5.10 | 2.38 | 4.90 |
| Black | 2.42 | 5.44 | 2.56 | 5.30 |
| Red | 2.71 | 4.99 | 2.38 | 4.03 |
| Green | 2.70 | 5.08 | 2.29 | 4.03 |
| Blue | 2.59 | 4.95 | 2.23 | 3.68 |

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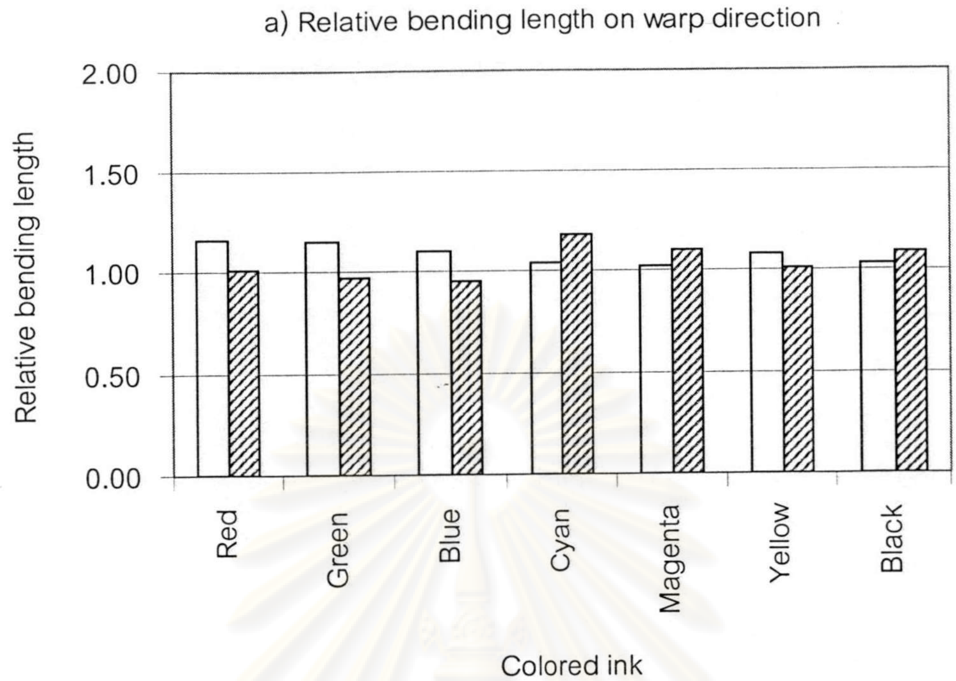


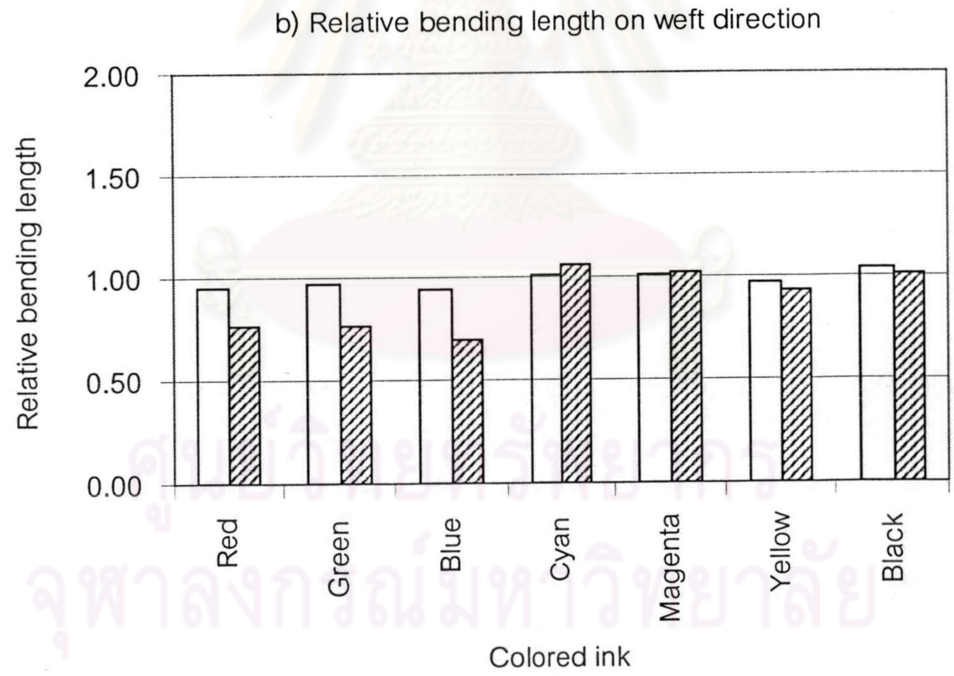


**Figure 4.25** Relative bending length of the treated, printed silk fabric ☐ for surface modified pigmented inks and ▨ for microencapsulated pigmented inks, (a) warp direction, (b) for weft direction

The stiffness of the pretreated printed silk fabrics was calculated using Equation 4-3, which results are shown in Table 4-23.

**Table 4-23** The stiffness of the pretreated printed silk fabric

| Color | Stiffness (mg cm) | | | |
|---|---|---|---|---|
| | Surface modified pigmented ink | | Microencapsulated pigmented ink | |
| | Warp direction | Weft direction | Warp direction | Weft direction |
| Non-printed | 177.34 | 1977.39 | 177.34 | 1977.39 |
| Cyan | 169.97 | 1747.26 | 258.55 | 2082.37 |
| Magenta | 164.16 | 1795.88 | 220.74 | 1964.91 |
| Yellow | 203.85 | 1679.74 | 158.20 | 1389.36 |
| Black | 190.48 | 2160.79 | 201.56 | 1783.37 |
| Red | 261.91 | 1628.11 | 176.43 | 858.78 |
| Green | 256.78 | 1705.19 | 153.51 | 836.27 |
| Blue | 225.51 | 1578.82 | 149.15 | 672.07 |

On average, ink color does not affect significantly the bending length of the pretreated silk fabric for both inks and both fiber directions (warp and weft direction). However, the fibers in the warp direction are stiffer than in the weft direction. The bending stiffness of the printed fabric is somewhat stronger for microencapulated pigmented inks than that from surface modified pigmented inks. The stronger the stiffness, the less the comfort the fabric is. Likewise, for the pretreated, and non-printed silk fabric, the bending length of the weft direction is higher than that of the warp direction as shown in Table 4-22. The effect of inkjet ink film on bending length of the pretreated, and printed silk fabric is not significantly pronounced. The main attribute is caused by the fiber itself.

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