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Dyeing Properties and Color of Silk Fabrics Dyed with Safflower Yellow Dye

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홍화 황색소의 견섬유에 대한 염색성과 색상

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Abstract

The objective of this study is to investigate the dyeing properties of safflower yellow dye on silk for the standardization of dyeing process and color reproducibility. Yellow colorants were water-extracted from safflower petals, concentrated, and freeze-dried to obtain colorants powder. The effects of dye concentration, dyeing temperature, and pH of dye bath were studied in terms of dye uptake and shade. Fastness to dry cleaning and light was evaluated. Dye uptake increased with raising temperature and brighter and more vivid yellow shade was obtained when dyed at 30°C. As colorants concentration increased, dye uptake increased progressively and the shade got darker and deeper. Maximum color strength was obtained at pH 3.5. It was speculated that the adsorption of colorants seemed to occur mainly by hydrogen bonding and physical force at pH 5.5 and by ionic bonding as well as hydrogen bonding below isoelectric point(pH 3.8-4.0). The results of reproducibility test showed acceptable color difference in the range of 1.11~2.01. Washing fastness was fairly good as 4/5 rating, while light fastness was 2/3 rating.

Key words: Safflower yellow colorants, Silk, Dyeing properties, Reproducibility, Color fastness; 홍화 황색소, 견, 염색성, 재현성, 염색견뢰도

I. Introduction

Safflower(*Carthamus Tinctorius* L.) has long been used as a natural colorant in Korea. The flower petals contain carthamin(red) and safflower yellow B(orange yellow) colorants, and mainly the red colorant has been used for cosmetics and textiles. The water-soluble yellow components of safflower petals have been hardly used as a natural dye for textiles and are currently using as a natural food colorants(Saito et al., 2005; Yoon et al., 2003). Comparing with carthamin, large quantities of yellow colorants are present in safflower and safflower yellow is more stable than carthamin under the UV light(Kanehira & Saito, 2001). We became to notice that it has great possibility as a yellow natural dye for textiles. There have been few studies(An & Kim, 2001; Cho, 1997; Nam et al., 1995) to investigate possibility to utilize yellow colorants as a natural dye for textiles. Recently,

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studies on the improvement of dyeability and light-fastness on cellulose fibers have been reported(Jung & Jang, 2004; Shin et al., 2007; Shin & Choi, 2007).

Traditionally, aqueous colorants extract has been used in natural dyeing. So, it is difficult to reproduce the color of a dyed fabric. The introduction of natural dyes into modern textile dyehouse requires standardized quality with regard to color depth and shade of dyed fabrics(Bechtold et al., 2007). The objective of this study is to investigate the dyeing properties of safflower yellow dye on silk for the standardization of dyeing process and color reproducibility. For better color reproducibility and the standardization of dyeing process, colorants powder was prepared and concentration could be controlled more consistently. Yellow colorants were water-extracted from safflower petals, concentrated, and freeze-dried to obtain colorants powder. The prepared yellow colorant was characterized in terms of dyeability and color on silk. Dyeing was carried out varying dye concentration, dyeing temperature, and pH of dye bath. Color of the dyed silk fabrics was characterized by CIELab coordinates, HV/C, and K/S values. Reproducibility of dyeing process was checked. Fastness to dry cleaning and light was also evaluated.

II. Experimental

1. Materials

The fabric used was a scoured and bleached 100% silk(plain weave, 160×98/cm^2, 42g/m^2, 0.11mm thickness). Dried safflower petals were purchased commercially in oriental medicine market. Chemicals used were reagent grade.

2. Preparation of Colorants

Safflower yellow colorants was extracted from dried safflower petals in a liquor ratio of 1:100 in distilled water at 40°C for 2 hrs two times using a constant temperature shaking bath. The first and second extracts were mixed together, filtrated, concentrated with a vacuum evaporator, and freeze-dried at -40°C to obtain colorants powder. The prepared colorants powder was used for dyeing without further purification. The absorption property of the colorant solution showed the yellow shades in the visible range of 400~420nm. The pH value of aqueous colorant extract was 5.5. Yield was about 34%.

3. Dyeing

Dyeing was done in material to liquor ratio at 1:100 varying dye concentration, temperature, pH of bath with an automatic laboratory dyeing machine (Ahiba Nuance, Datacolor International, USA). The dyed fabrics were then washed, rinsed, and dried. The pH of dyeing solution was varied from 3 to 11 with acetic acid and sodium hydroxide.

4. Color Measurement

Color was evaluated in terms of K/S values, ΔE, and CIELab coordinates(Illuminant D_{65}/10° Observer) with a Macbeth Coloreye 3100 spectrophotometer at 400nm(λmax). H V/C values were obtained from L*a*b* data using CIE Munsell conversion program.

5. Fastness Testing

Fastness to dry cleaning of the pre-mordanted samples was evaluated by AATCC method 61-1989. Light fastness was assessed in terms of color difference (ΔE) and color change against the appropriate grey scale according to AATCC method 16-1998 with Fade-Ometer(Atlas Electric Devices Co, USA). Color differences were measured with a Macbeth Coloreye after irradiating for 5, 10, 20, and 40 hours.

III. Results and Discussion

1. Effect of Dyeing Temperature on Dye Uptake and Color

<Fig. 1> shows the effect of temperature on the dye uptake of silk fabrics. Dye uptake, expressed as K/S at the maximum absorption wavelength, increased with the increase of temperature. The conglomeration of colorant molecules reduced with the rise in

temperature, leading to easy and rapid diffusion of colorants into the fibers and more dye uptake at higher temperature. This attributed to the change in lightness(L*) and saturation(C) and produced color differences, as shown in <Table 1>. The L* value indicates perceived lightness in CIELab color space. The L* scale runs from 0(black) to 100(white); the higher the L reading, the lighter the color. The a* value indicates redness(+a*) and greenness(-a*) while the b* value indicates yellowness(+b*) and blueness(-b*)(Billmeyer & Saltzman, 1981). As temperature increased, L* value decreased with the increase of dye uptake, and thus color got darker. The minimum value of a* was seen at 30°C and maximum at 50°C, and thereafter a* value decreased by little. This indicates that the shade of fabrics dyed at 50°C got more red character than those dyed at lower temperature. We speculated the inclusion of red pigment in safflower yellow extract. The red colorant has high affinity to cotton and thus it was removed by processing the extract with cellulose powder(Saito, 1990) and then dyeing was carried out at 50°C. The results are presented in parentheses in <Table 1>. The redness of dyed fabric(a*) is lowered from 5.27 to 2.73. Even so, a* value was higher with the silk fabric dyed at 50°C than that dyed at 30°C. Comparing with the fabric color dyed at higher temperature above 50°C, the fabric color dyed at 30°C was brighter and more vivid yellow with less redness. Therefore, optimum dyeing temperature was set at 30°C.

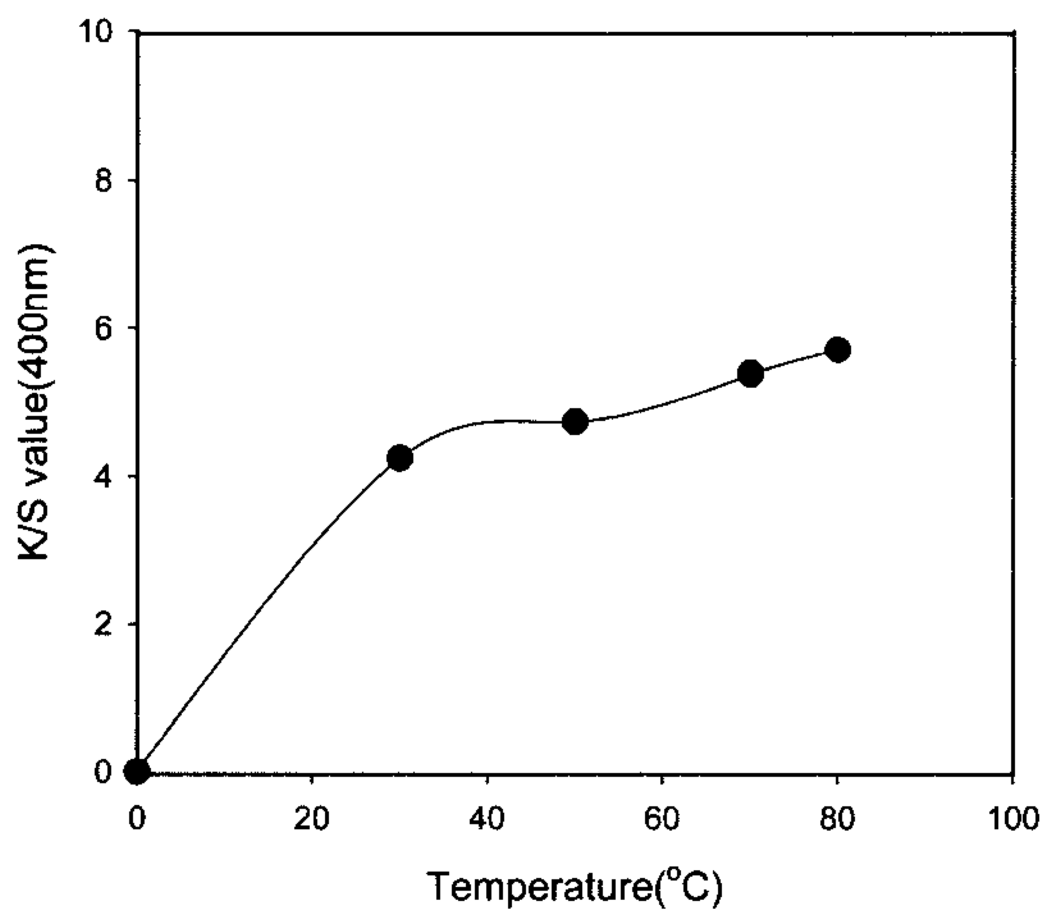

Fig. 1. Effect of dyeing temperature on dye uptake (0.6% owb, 60min).

Table 1. CIELab coordinates at different dyeing temperature

Temp. (°C)	L*	a*	b*	H V/C
30	80.20	2.24	47.77	4.0Y 8.0/6.8
50	77.26 (78.66)	5.27 (2.73)	46.50 (47.30)	2.8Y 7.7/6.8 (3.9Y 7.8/6.7)
70	73.15	4.60	43.05	3.0Y 7.3/6.2
80	71.46	3.19	42.16	3.6Y 7.1/6.0

( ) dyed after removing red pigments.

2. Effect of Dye Concentration on Dye Uptake and Color

<Fig. 2> shows the effect of dye concentration on dye uptake. Dyeing was carried out at original pH 5.5 without adjusting. Dye uptake increased progressively as colorant concentration increased. With increasing concentration, more dye transferred to fabric and the depth of color became stronger. The adsorption isotherm in <Fig. 2> seems similar to Freundlich type curve, indicating that adsorption of the colorants occurs mainly by hydrogen bonding and physical force(Trotman, 1975). The attachment of colorants is not at specific sites so that there is no stoichiometric limiting factor. The limitation is the available surface within the pores and affinity to silk fiber. Therefore,

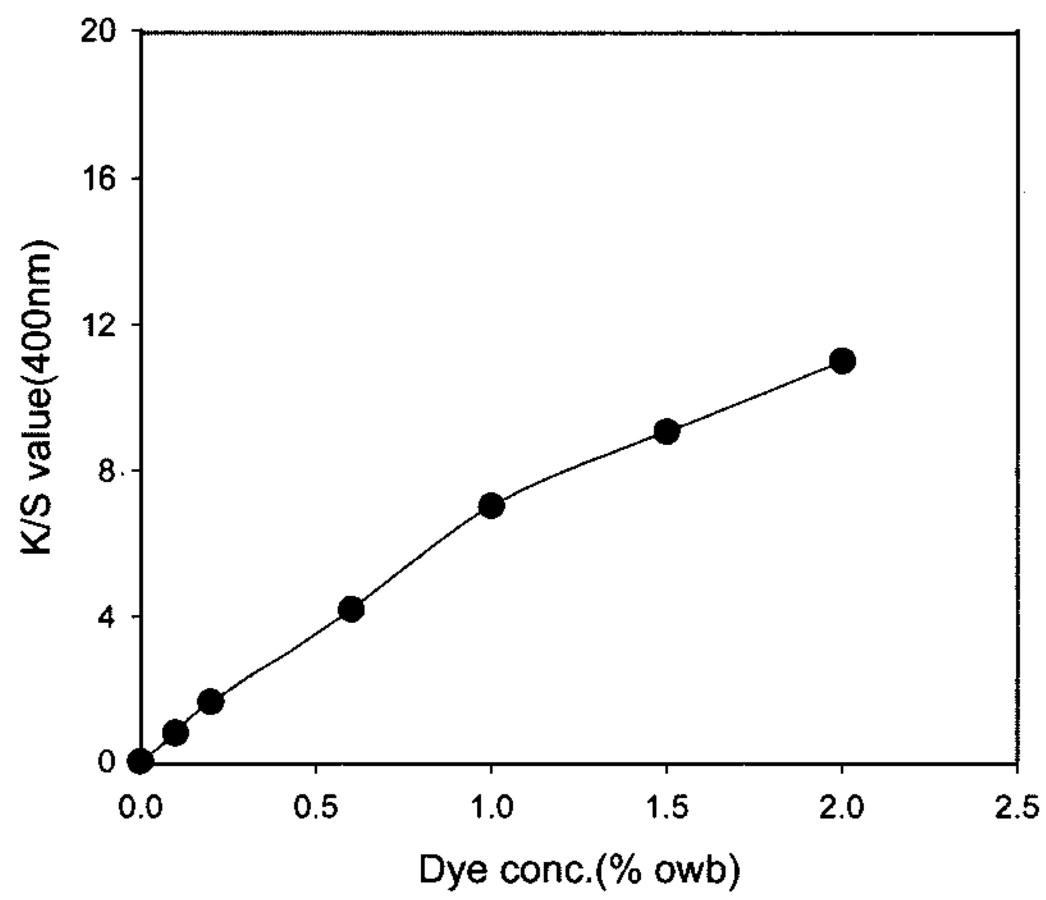

Fig. 2. Effect of dye concentration on dye uptake (30°C, 40min).

adsorption is rapid at first because the sites are easily accessible. It becomes slower as the dye molecules have to find the more remote sites of attachment. <Fig. 3> shows the dyeing rate. It was confirmed that adsorption occurred rapidly at first 10min and then it

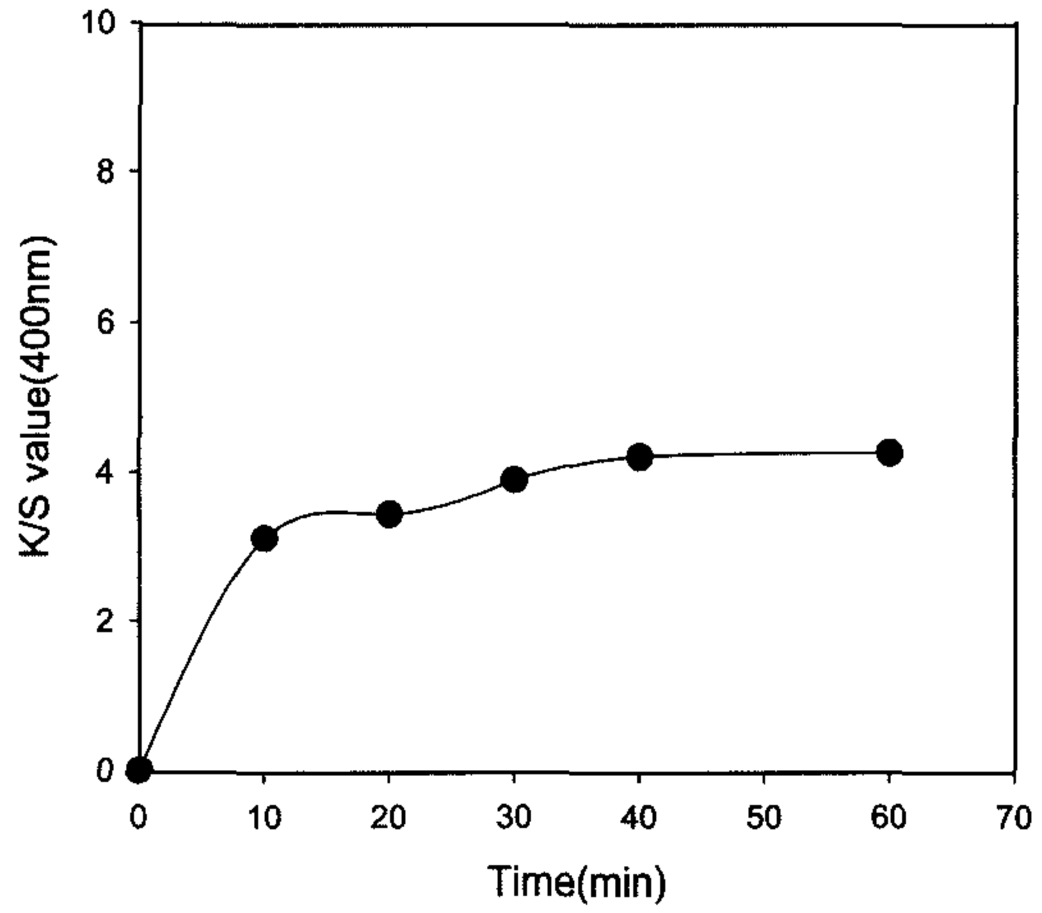

Fig. 3. Effect of dyeing time on dye uptake(0.6% owb, 30°C).

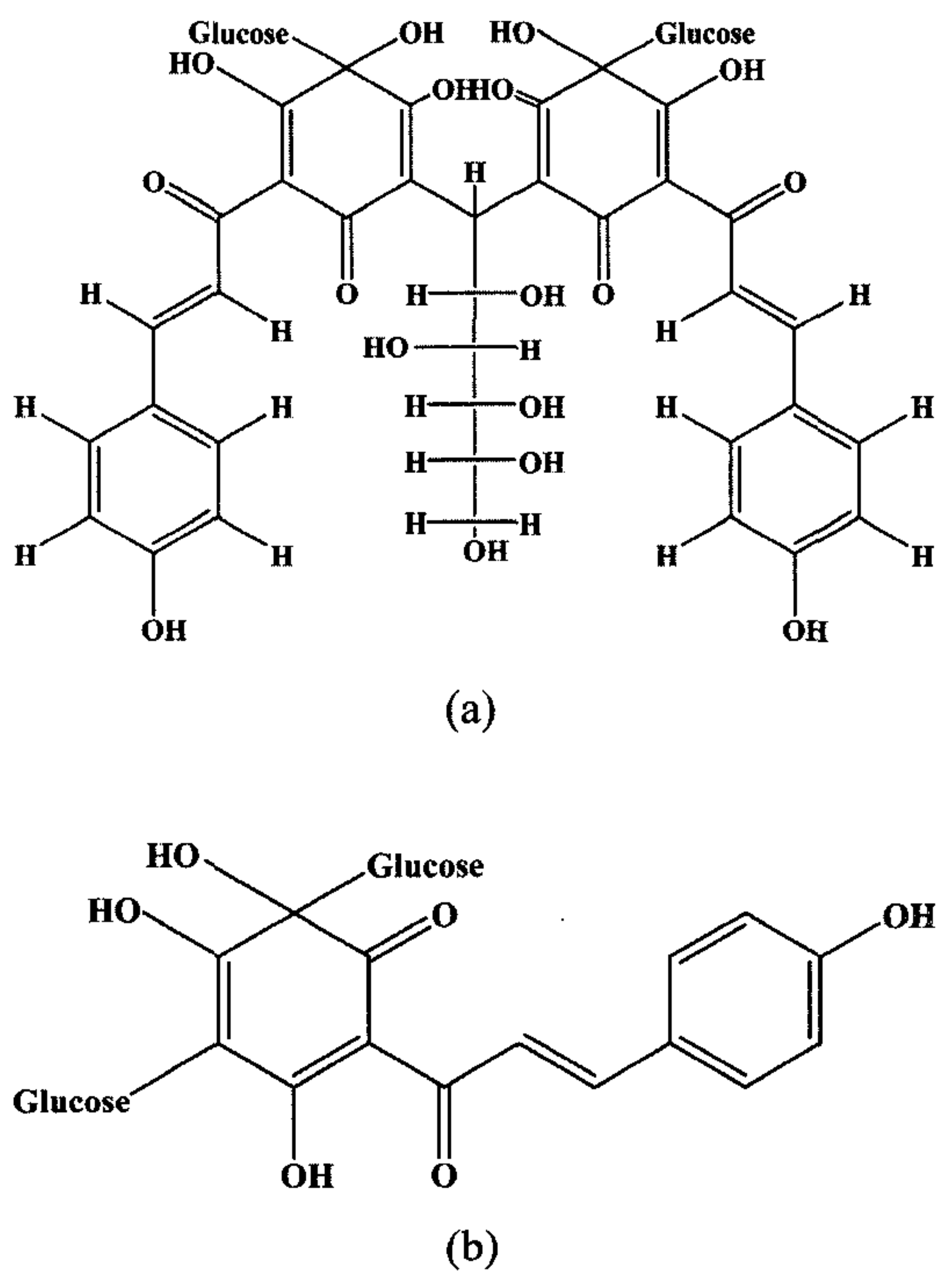

Fig. 4. Chemical structures of safflower yellow B (a) and safflomin (b).

slowed down, and reached to almost maximum dye uptake at 40min, as seen in <Fig. 3>. Optimum dyeing time was set at 40min. The major yellow components of safflower petals are safflower yellow B and safflomin(Saito et al., 2005). They have several hydroxyl groups(-OH) in their structures shown in <Fig. 4>. So, they are capable of forming hydrogen bonding with amide groups(-CONH-) in silk fibers.

As shown in <Table 2>, a* increased slightly with the increase of concentration, while the increase of b* value is much larger compared with a* value. The yellow shade of fabric got stronger and more saturated as V decreased and C increased with increasing dye concentration.

Table 2. CIELab coordinates at different dyeing concentration(0.6% owb, 60min)

Conc. (%)	L*	a*	b*	H V/C
0.1	87.10	-3.18	24.64	7.5Y 8.6/3.1
0.2	83.61	-0.61	33.63	4.7Y 8.3/4.5
0.6	80.23	1.59	46.69	4.2Y 8.0/6.6
1.0	77.08	3.08	52.84	4.0Y 7.7/7.5
1.5	75.37	4.34	56.19	3.8Y 7.5/8.1
2.0	74.63	5.22	59.56	3.7Y 7.4/8.6

3. Effect of pH on Dye Uptake and Color

<Fig. 5> shows the effect of pH on dye uptake. Maximum adsorption wavelength was shifted from 400nm to 420nm below pH 3.5. Dye uptake in alkaline condition was very low. It increased sharply from pH 4.5 to 3.5 and reached to maximum color depth at pH 3.5 in the range of pH of this experiment. This result attributed to the structural features of colorants and silk fiber. In alkaline condition, (-) charges are formed on both of colorants and fibers and repulsion between colorants and fibers are occurred, resulting in low dye uptake. Below the isoelectric point(pH 3.8-4.0) of silk fiber(Kim & Lee, 1996), more (+) charges are formed on the silk fibers. It has been known that yellow colorants exist as aglycons in acidic condition and (-) charges are formed on the surface of colorants(Cho, 1997). The colorants can form ionic bondings with the amino groups(NH_3^+) of silk fibers. Considering that silk has a few basic

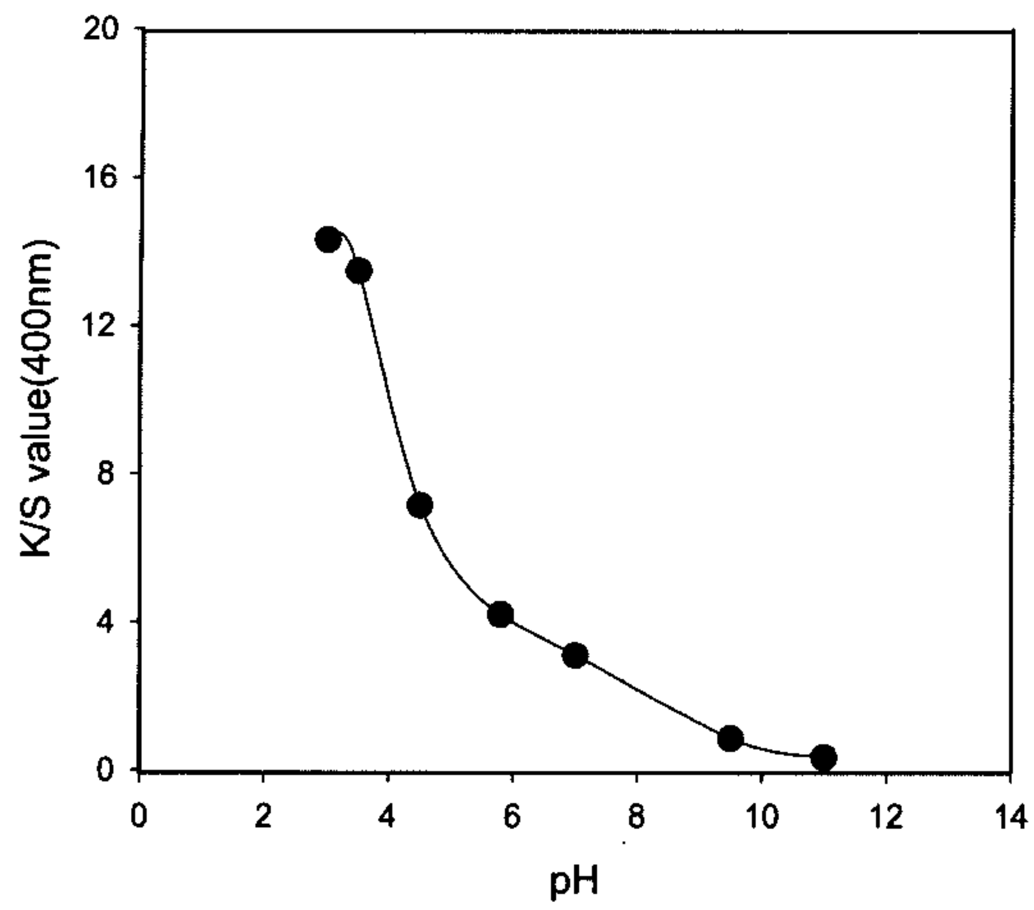

Fig. 5. Effect of pH on dye uptake(30°C, 40min, 0.6% owb).

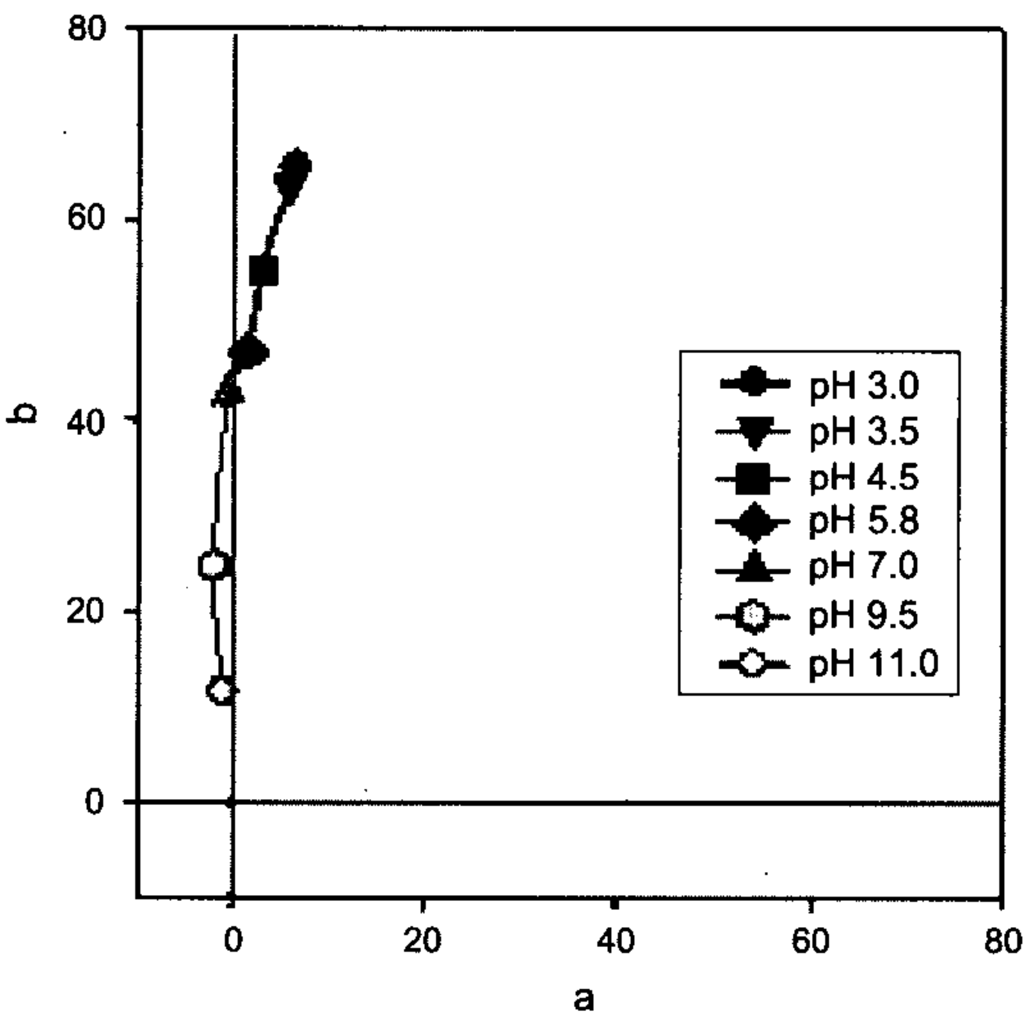

Fig. 6. a* vs. b* value of dyed fabrics with different pH(30°C, 40min, 0.6% owb).

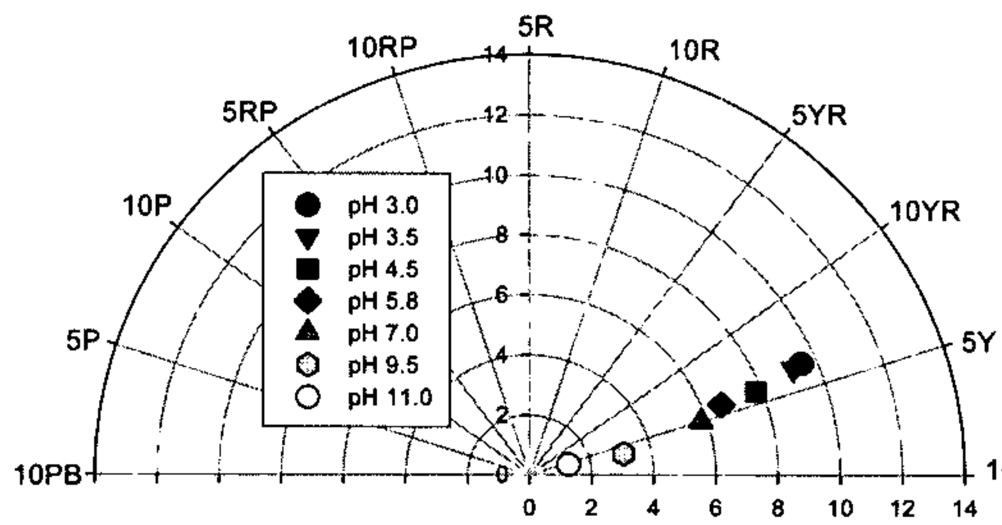

Fig. 7. Hue vs. chroma of dyed fabrics with different pH(30°C, 40min, 0.6% owb).

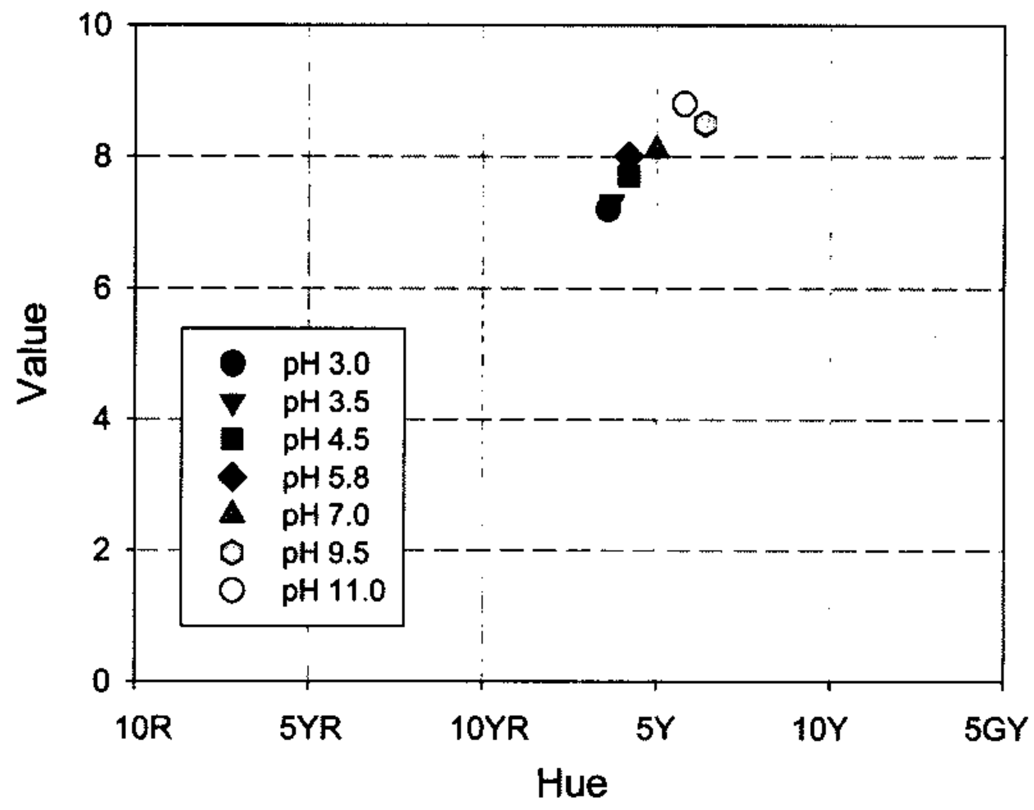

Fig. 8. Hue vs. value of dyed fabrics with different pH(30°C, 40min, 0.6% owb).

groups, it is speculated that the adsorption of yellow colorants on silk might be involved by ionic bonding as well as hydrogen bonding.

<Fig. 6> shows a* vs. b* coordinates of dyed fabrics with different pH of dye bath. As pH increased, b* value increased sharply while a* showed (-) value and increased slightly above pH 7.0. It indicates that yellow shade got stronger and deeper in acidic dyeing condition. In <Fig. 7 and 8>, coordinates of hue vs. chroma and hue vs. value are presented depending on the pH of dye bath. Hue of the dyed fabrics was in the range of 3.6Y-6.4Y, value in the range of 7.2-8.8, and chroma in the range of 9.5-1.3. The color values of silk fabrics followed a general trend for more saturated and stronger yellow with decreasing the pH of dye bath.

4. Color Reproducibility

Reproducibility of laboratory dyeing process was checked for different batch and the results are presented in <Table 3>. Dyeing conditions were 0.6% (owb) dye concentration, pH 3.5, 30°C, 40min. Color difference(ΔE) between dyeing experiments varied in the range of 1.11~2.01. In dyeing with synthetic dyes a color difference of ±1 is tolerable technically. For natural dyeing a somewhat more color difference of $\Delta E = \pm 2$ could be argued because the use of natural resources makes possibly wider variations due to non-synthetic raw material(Bechtold et al., 2007).

Table 3. Variation in CIELab color coordinates and K/S values between dyeings

Batch no.	K/S values (420nm)	L*	a*	b*	H V/C	ΔE*
1	13.95	73.52	5.97	63.76	3.7Y 7.3/9.2	-
2	13.92	74.54	5.79	64.16	3.7Y 7.4/9.3	1.11
3	13.71	73.94	6.11	65.81	3.8Y 7.4/9.5	2.01

5. Colorfastness to Dry Cleaning and Light

<Table 4> shows colorfastness to dry cleaning and light. Samples for fastness evaluation were dyed in 0.6%(owb) dye concentration, pH 3.5, at 30°C for 40min. Fastness to washing was relatively good as 4/5 rating for color change and 5 for stain. Lightfastness of the dyed fabrics was 2/3 rating on the face side and 4/5 rating on the back side after 20 hrs of light exposure. The lightfastness is influenced by several factors: the chemical and physical state of dye, the dye concentration, the nature of fibers, the mordant type, etc. It has been known that the physical state of dye is more important than the chemical structure of dye. Fibers with large aggregates of dye show better lightfastness.

Table 4. Fastness of the silk fabrics dyed with safflower yellow

Dry cleaning fastness		Light fastness
Color change	Stain	Color change
4/5	5	2/3(face), 4/5(back)

IV. Conclusions

Dye uptake, expressed as K/S, increased with increase in temperature. The shade of dyed silk fabric was brighter and more vivid yellow when dyed at 30°C. Dye uptake increased progressively and the color of the dyed silk fabrics got darker and deeper as colorant concentration increased. The adsorption isotherm is considered as Freundlich type, indicating that adsorption of the colorants occurs mainly by hydrogen bonding and physical force at pH 5.5. On the other hand, it was considered that ionic bonding as well as hydrogen bonding involved below isoelectric point(pH 3.8-4.0). Dye uptake increased sharply from pH 4.5 to 3.5 and reached to maximum color depth. In reproducibility test for dyeing, color difference(ΔE) between dyeings was in the range of 1.11~2.01. Colorfastness to dry cleaning was very satisfactory as 4/5 rating. Lightfastness of the dyed fabric with 20 hrs exposure was 2/3 rating on front side and 4/5 on back side of fabric.

References

An, K. C. & Kim, J. H. (2001). A study of the dyeability and physical properties of mordanted and finished fabrics dyed with natural dye of safflower. *J. of Korean Society of Dyers and Finishers, 13*(1), 23–31.

Bechtold, T., Mahmud-Ali, A., & Mussak, R. (2007). Natural dyes for textile dyeing: A comparison of methods to assess the quality of Canadian golden rod plant material. *Dyes and Pigments, 75*(2), 287–293.

Billmeyer, Jr. F. W. & Saltzman, M. (1981). *Principles of color technology* (2nd ed.). New York: Wiley Pub. Co..

Cho, K. R. (1997). Study on the natural dyes. *J. of Korean Society of Dyers and Finishers, 9*(5), 10–18.

Cristea, D. & Vilarem, G. (2006). Improving light fastness of natural dyes on cotton yarn. *Dyes and Pigments, 70*(3), 238–245.

Jung, S. & Jang, J. (2004). Light fastness of silk fabric dyed with safflower and amur cork tree extract for combining dyeing. *J. of Korean Society of Dyers and Finishers, 16*(5), 8–18.

Kanehira, T. & Saito, K. (2001). Stability of carthamin and safflower yellow B on silk powders under continous irradiation of fluorescent or UV-C light. *Lebensmmittel-Wissenschaft und-Technologie, 34*, 55–56.

Kim, G. J. & Lee, J. M. (1996). *Chemistry of dyeing*. Seoul: Hyungsul Pub. Co..

Nam, S. W., Chung, I. M., & Kim, I. H. (1995). Dyeing cotton by natural dyes (I). *J. of Korean Society of Dyers and Finishers, 7*(2), 47–54.

Saito, K. (1990). Possible locality of affinity site(s) on cellulose for preservation of stable red colour of carthamin. *Food Chemistry, 36*, 243–251

Saito, S., Kusakari, T., Suda, T., Kasai, T., Kumazawa, T., Onodera, J., & Obara, H. (2005). Efficient synthesis of analogs of safflower yellow B, carthamin, and its precursor: Two yellow and one red dimeric pigments in safflower petals. *Tetrahedron, 61*, 9630–9636.

Shin, Y., Cho, A., & Yoo, D. I. (2007). Improving dyeability of safflower yellow colorants on cellulose fibers. *J. Korean Society of Clothing and Textiles, 31*(12) 1754–1760.

Shin, Y. & Choi, S. Y. (2007). Imroving UV-cut ability of natural dyed fabrics. *J. Korean Home Economics Association, 45*(10), 73–81.

Trotman, E. R. (1975). *Dyeing and chemical technology of textile fibers* (5th ed.). London: Charles Griffin & Co. Ltd.

Yoon, J. M., Cho, M. H., Park, J. E., Kim, Y. H., Hahn, T. R., & Paik, Y. S. (2003). Thermal stability of the pigments hydroxysafflor yellow A, safflor yellow B, and precarthamin from safflower. *J. of Food Science, 68*(3), 839–845.

요 약

본 연구는 염색공정의 표준화와 재현성을 확립하기 위해 홍화 황색소의 견섬유에 대한 염색성을 조사하는데 목적이 있다. 홍화 황색소는 물로 추출한 후 농축, 동결건조하여 분말상태로 만들어 사용하였다. 염색온도 및 시간, 염료농도, 염액의 pH 등에 따른 염착성과 색상 변화에 대해 조사하였으며, 세탁 및 광견뢰도를 평가하였다. 염색온도가 증가함에 따라 염착량은 증가하였으며 30°C에서 염색할 때 가장 선명한 노랑색을 얻을 수 있었다. 염료농도가 증가함에 따라 염착량이 계속 증가하여 점점 진하고 어두운 노랑색이 되었다. pH 3.5에서 최대염착량을 보였으며, 최적조건에서 색차는 1.11-2.01로서 재현성은 양호하였다. 색소와 견섬유간의 결합은 pH 5.5 부근에서는 주로 수소결합에 의해, 등전점(pH 3.8-4.0) 이하에서는 수소결합과 함께 이온결합이 관여하는 것으로 판단되었다. 세탁(드라이크리닝)견뢰도는 4/5등급으로 좋은 편이었으나 일광견뢰도는 2/3등급이었다.