

## Natural Dyeing Using Tea Extract II

– The Effect of Dyeing Condition on Dyeing Characteristics of  
Coffee Extract Dyed Silk Fabric–

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### Abstract

Coffee extract dyeing on silk fabric using various mordanting agents at different mordanting condition was studied in our previous research. Coffee extract dyeing can exert beautiful colors and have several advantages over the conventional natural dyeing. Since most of natural dyes are expensive for mass production, strongly season and source dependent, and hard to store, and therefore, coffee waste was used as natural dye to create beautiful colors in this study. Coffee waste is inexpensive and easy to store, and provide a comparatively regular quality regardless of season and source. The effects of dyeing condition such as dyeing temperature, dyeing time, and liquor ratio on dyeing characteristics of silk fabrics were investigated. Coffee extract dyed silk showed brilliant colors, and the colors were different with the kind of mordant. Cu, Sn, and Al mordanted silks showed golden yellow with subtle change of hue and value, while Fe mordanted silk showed a brownish green color upon coffee extract dyeing. As the dyeing temperature increased, the dyeability generally increased in all mordanted silk fabrics except Fe mordanted silk until 45°C. The dyeing temperature did not affect the dyeability of Fe mordanted silk fabric. As the liquor ratio increased, the dyeability showed a gradual increase in all mordanted silk except Cu mordanted silk. Cu mordanted silk fabric showed an abrupt increase in dyeability at liquor ratio 1:150. The dyeing time effect can be separated into 2 stages. During the initial half an hour, the dyeability did not change significantly, and then, increased after 45 min. dyeing time duration. A further increase in dyeing time after 45 min. did not affect the dyeing efficiency in all mordanted silk fabrics except the Cu mordanted.

**Key Words :** Natural dyes, Coffee waste, Dyeing temperature, Dyeing time, Liquor ratio

### I. Introduction

Natural dyeing technology has been vigorously studied nowadays because of its environmental

advantages over synthetic dyes and its beautiful color.<sup>1)-5)</sup> Compared to the conventional natural dyeing in the past, current studies about natural dyeing became more scientific and systematic.

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The term "natural dyes" refer to the dyes obtained from insects, plants, and mineral substances, and used for dyeing textile material.<sup>4)</sup> The study about natural dyes were mostly focused on the property of antibacterial activity.<sup>6)-14)</sup> Most natural dyes studied so far are traditional medicines and strongly season-dependent. In other words, conventional natural dyes are expensive to use as dye for mass production and show irregular qualities upon season, source, storage methods, and etc. In this study, tea waste was used as natural dye source since tea waste is inexpensive and provides a comparatively regular quality compared to conventional natural dyes.

Among tea waste, coffee waste is easy to obtain and easy to store, and coffee extract dyed fabrics showed a brilliant color in our previous study.<sup>15)</sup> Coffee is one of the most widely and frequently consumed beverage in the world because of its pleasant aroma, taste, and mental stimulating effect.<sup>16)-21)</sup> Coffee also has some beneficial aspects such as antioxidant and antibacterial agent. Daglia et al. isolated the active antibacterial components from coffee brew, and proved these components have antibacterial activities over *Staphylococcus aureus* ATCC 25923 and *Streptococcus mutans* 9102.<sup>17)</sup> And it is well-known that coffee contains antioxidants.<sup>19),20)</sup> Coffee brew have over 100 different active chemicals depending brew method.<sup>20),22)</sup> The organic constituents of the coffee bean include representatives of carbohydrates, proteins, fats, oils, waxes, and a considerable number of other groups of organic compounds, including caffeine and trimethyl xanthin.<sup>22)</sup> Coffee color is generated from roasting green bean by caramelizing sugars and starches.<sup>22)</sup> However, there is no study related to coffee colorants and coffee extract dyeing so far.

As mentioned earlier, the quantity of coffee

consumption is very large, and therefore, the coffee waste is also coming out in large quantity.

Coffee waste has no usage so far, although coffee waste would still have many biologically or chemically active components.

Therefore, we want to use coffee waste as the source of dyeing material and study the effect of dyeing condition such as dyeing temperature, dyeing time, and liquor ratio on dyeing characteristics of silk fabric.

## II. Experimental

### 1. Materials

Ungummed silk fabric was kindly supported by KSRI (Korea Silk Research Institute). Used coffee powder (Jamaica Blue mountain) was also kindly supplied by Krema Coffee Company (Seoul, Korea). Ferrous sulfate heptahydrate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ), cupric sulfate pentahydrate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ), aluminum ammonium sulfate dodecahydrate ( $\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ ), and stannous chloride dihydrate ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ) were purchased from DaeJung Chemical Co. (Gyeonggido, Korea).

### 2. Preparation of coffee extract solution

Used coffee powder was completely dried at the oven at 110°C for 6 hours before use. Coffee extract solution prepared by boiling used coffee powder in water (5% o.w.b.) for 10 min. The solution was cooled down to room temperature and subsequently used as dyeing stock.

### 3. Mordanting silk with various metal compounds

Silk fabrics were mordanted with Al, Sn, Cu, and

Fe, respectively. Silk fabric was immersed with tap water, mordanted with metal mordant (1% o.w.f.) at room temperature for 5 min., and subsequently at 40°C for 30 min. The mordanted silk fabrics were washed several times with running tap water, and dried under shade.

#### 4. Dyeing silk with coffee extract

Mordanted silk fabrics with various mordanting agents and concentrations were immersed with tap water sufficiently, and dyed with prepared coffee extract solution. Dyeing started at room temperature, and subsequently conducted at the different temperatures, 20°C, 40°C, and 60°C. Dyeing time also varied from 15min. to 60min. by 15 min. interval. The liquor ratio also changed 1:50, 1:75, 1:100, 1:125, and 1:150. Dyeing batch was cooled down to room temperature, and dyed silk was washed several times with running tap water until no colorant come out. Coffee extract dyed silk fabrics were dried under shade and pressed.

#### 5. Color measurement

The  $L^*a^*b^*$  and  $\Delta Lab$  values were measured using colorimeter (Pantone color cue TX, Accuracy Microsensors, Inc., NY, USA). Color value of each sample was measured five times and averaged.  $\Delta E$  value was calculated from the following equation:

$$E = (\Delta L^2 + \Delta a^2 + \Delta b^2)^{1/2}$$

$$\Delta L = L^1 - L^2$$

$$\Delta a = a^1 - a^2$$

$$\Delta b = b^1 - b^2$$

Surface reflectance values were also measured using spectrophotometer (Gretag Macbeth Color-Eye 3100, USA). All measured sample showed the

greatest  $\lambda_{max}$  value at 400nm. The K/S was calculated by the following equation (Kubelka-Munk equation)<sup>23)</sup>

$$K/S = \frac{(1 - R)^2}{2R}$$

R: Surface reflectance

K: Light absorption

S: Light scattering

### III. Result and Discussion

#### 1. Dyeing color using various mordanting agents

Silk fabrics were very well dyed by coffee extract showing various colors upon different mordanting agents. Copper mordanted silk showed a luxurious gold tan color (pantone number 16-1334 TC), tin mordanted silk showed a bright gold taffy color (pantone number, 16-0940 TC), aluminium mordanted silk showed a beautiful curry color (pantone color, 16-0928 TC), a similar color with Tumeric (WoolGeum) dyed silk fabrics, and iron mordanted silk showed a elegant Elmwood color (pantone number, 17-0919 TC). Copper, tin, and aluminium mordanted silk all showed a similar hue, yellow gold, while iron mordanted silk showed a totally different color, dark shade brownish green.

#### 2. The effect of dyeing temperature

As the dyeing temperature increased, the  $\Delta E$  values of coffee extract dyed silk fabrics increased in case of Al, Cu, and Sn mordants (Fig. 1, Table 2.). However, dyeing temperature increase did not affect  $\Delta E$  value of Fe mordanted silk fabric. This means that the dyeing temperature increase resulted in the increase in the dyeing efficiency in all mordanted fabrics except Fe mordanted fabrics.

<Table 1> The pantone number and RGB of coffee extract dyed silk fabrics with various mordants.\*

Mordants	Pantone number	R	G	B
$\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	14-1116 TC	82	72	58
$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	16-0928 TC	73	61	42
$\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$	14-1038 TC	85	71	50
$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	17-1312 TC	61	55	47

\* Pre-mordanted silk fabrics with various mordants were dyed with coffee extract at 40°C for 30min., liquor ratio 1:150.

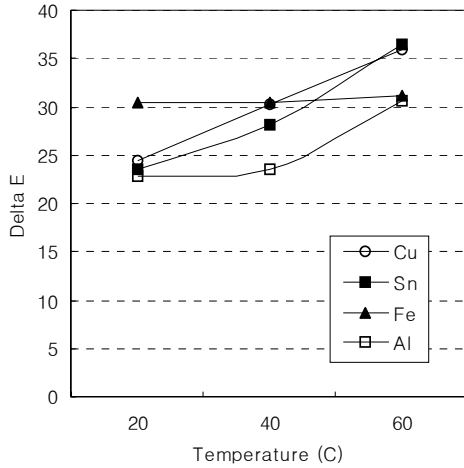
<Table 2> The effect of dyeing temperature on the L\*a\*b\* and delta Lab values of coffee extract dyed silk fabrics.

Mordants	Temperature	L*	a*	b*	delta L	delta a	delta b	delta E
	Control	90.9	-2.04	2.7	-	-	-	-
Cu	20°C	76.44	3.78	21.48	-14.46	5.82	18.78	24.40
	40°C	70.70	-1.08	25.13	-20.20	0.96	22.43	30.20
	60°C	71.31	5.18	31.89	-19.59	7.22	29.19	35.89
Sn	20°C	80.08	2.24	23.27	-10.82	4.28	20.57	23.64
	40°C	76.40	2.35	26.51	-14.50	4.39	23.81	28.22
	60°C	58.36	1.74	18.68	-32.54	3.78	15.98	36.45
Al	20°C	80.54	2.28	22.52	-10.36	4.32	19.82	22.78
	40°C	77.20	2.65	21.21	-13.70	4.69	18.51	23.50
	60°C	69.94	3.97	24.26	-20.96	6.01	21.56	30.66
Fe	20°C	62.27	1.45	12.73	-28.63	3.49	10.03	30.54
	40°C	62.30	1.45	12.73	-28.60	3.49	10.03	30.51
	60°C	72.32	3.86	26.91	-18.58	5.90	24.21	31.08

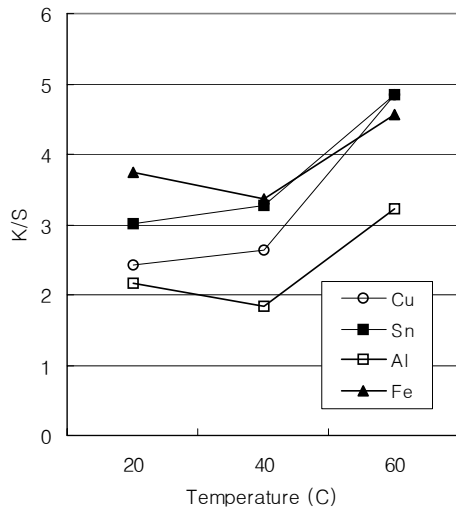
Therefore, an increase in dyeing temperature in case of Fe mordanted dyeing would be unnecessary. The highest  $\Delta E$  was achieved in both Cu and Sn mordanted silk fabrics at 60°C condition.

K/S value did not increase as the temperature increase from 20°C to 40°C in all mordanted silk fabrics as shown in Fig. 2. However, K/S increase

significantly as the temperature increase from 40°C to 60°C in all mordanted silk fabrics. Therefore, to get a better dyeing efficiency, it would be required to increase dyeing temperature up to 60°C. Al mordanted silk showed the lowest K/S compared to the other mordanted fabrics at all temperature.



<Fig. 1> The effect of dyeing temperature on the  $\Delta E$  values of coffee extract dyed silk fabrics with various mordants.



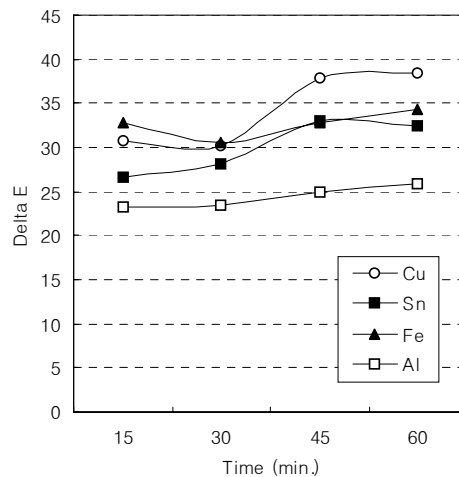
<Fig. 2> K/S values of coffee extract dyed silk fabrics with various mordants in the change of dyeing temperature.

### 3. The effect of dyeing time

<Table 3> and <Fig. 3> shows the effect of dyeing time on the  $\Delta E$  values of coffee extract

dyed silk fabrics with various mordants. During the initial half an hour,  $\Delta E$  values of coffee extract dyed silk fabrics did not change significantly in all mordants.  $\Delta E$  values of coffee extract dyed silk fabrics increased after 45 min. dyeing time, and a further change was not observed upon the dyeing time duration after 45 min. in all mordants. Especially, Fe mordanted fabrics showed any dramatic changes in  $\Delta E$  values compared with other mordanted fabrics. Al mordanted fabrics did not show any significant changes over dyeing time duration and showed the lowest  $\Delta E$  of all mordanted fabrics. Therefore, in case of Al and Fe mordanted fabrics, dyeing time duration was not necessary to increase the dyeing efficiency. Only Cu mordanted silk fabric was required to increase the dyeing time duration to get a darker shade.

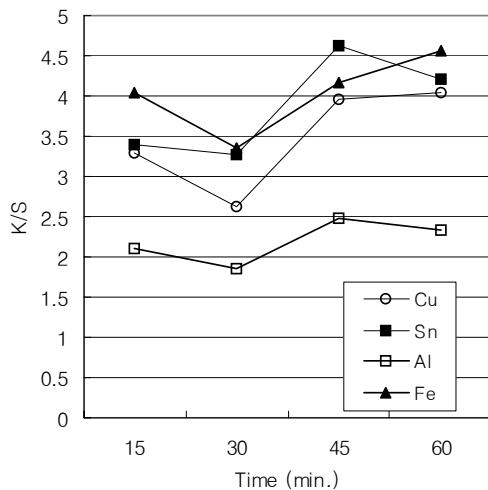
K/S value and  $\Delta E$  of Al mordanted silk showed the lowest among all mordanted coffee extract dyed silk fabrics as shown in <Fig. 4> K/S did not increase after 45 min. in all mordants except the Fe mordanted. Only Fe mordanted silk showed the further increase in K/S upon time duration.



<Fig. 3> The effect of dyeing time on the  $\Delta E$  values of coffee extract dyed silk fabrics with various mordants.

<Table 3> The effect of dyeing time on L\*a\*b\* and delta Lab values of coffee extract dyed silk fabrics using various mordant agents.

Mordants	Time (min.)	L*	a*	b*	delta L	delta a	delta b	delta E
Control	-				-	-	-	-
Cu	15	70.28	4.67	24.57	-20.62	6.71	21.87	30.79
	30	70.70	-1.08	25.13	-20.20	0.96	22.43	30.20
	45	65.87	5.34	30.11	-25.03	7.38	27.41	37.85
	60	64.39	8.27	28.42	-26.51	10.31	25.72	38.35
Sn	15	78.54	4.37	25.30	-12.36	6.41	22.60	26.55
	30	76.40	2.35	26.51	-14.50	4.39	23.81	28.22
	45	76.13	8.31	30.36	-14.77	10.35	27.66	33.02
	60	73.29	6.70	28.56	-17.61	8.74	25.86	32.48
Al	15	79.94	3.57	22.33	-10.96	5.61	19.63	32.93
	30	77.20	2.65	21.21	-13.70	4.69	18.51	30.51
	45	76.06	4.34	21.61	-14.84	6.38	18.91	32.92
	60	76.61	5.73	22.69	-14.29	7.77	19.99	34.39
Fe	15	59.90	2.36	12.89	-31.00	4.40	10.19	23.17
	30	62.30	1.45	12.73	-28.60	3.49	10.03	23.50
	45	60.90	4.26	14.72	-30.00	6.30	12.02	24.87
	60	58.57	3.50	13.04	-32.33	5.54	10.34	25.77



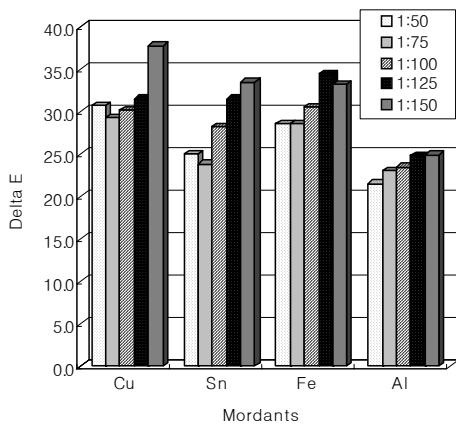
<Fig. 4> K/S values of coffee extract dyed silk fabrics with various mordants in the change of dyeing time.

#### 4. The effect of liquor ratio

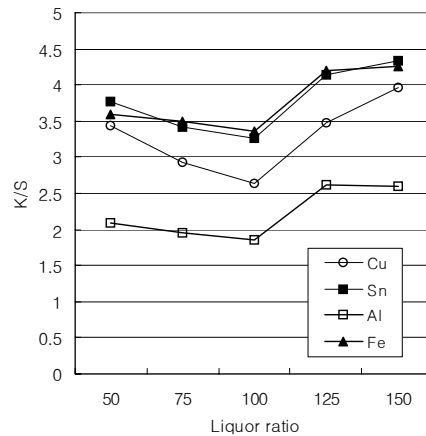
<Table 4> and <Fig. 5> shows the effect of liquor ratio of coffee extract dyed silk fabrics with various mordants. As the liquor ratio increased, the  $\Delta E$  values of coffee extract dyed silk fabrics generally increased. The gradual increases in  $\Delta E$  values of coffee extract dyed silk fabrics with mordants, Al, Sn, and Fe, were observed. In case of Cu mordanted silk, same  $\Delta E$  values at liquor ratio at both 1:50 and 1:100 were shown, and an abrupt increase in  $\Delta E$  value at liquor ratio 1:150 was observed. The highest  $\Delta E$  values of coffee extract dyed silk fabrics was observed at Cu mordanted silk fabric at liquor ratio 1:150. Therefore, the increase in liquor ratio resulted in the increase in dyeing efficiency.

<Table 4> The effect of liquor ratio on L\*a\*b\* and delta Lab values of coffee extract dyeing with various metal mordants.

Mordants	Liquor ratio	L*	a*	b*	delta L	delta a	delta b	delta E
	Control	90.9	-2.04	2.7	-	-	-	-
Cu	1:50	69.94	3.97	24.26	-20.96	6.01	21.56	30.66
	1:75	71.22	5.69	23.04	-19.68	7.73	20.34	29.34
	1:100	70.70	-1.08	25.13	-20.20	0.96	22.43	30.20
	1:125	71.65	7.46	25.80	-19.25	9.50	23.10	31.53
	1:150	65.87	5.34	30.11	-25.03	7.38	27.41	37.85
Sn	1:50	80.03	1.28	24.98	-10.87	3.32	22.28	25.01
	1:75	79.51	5.84	22.10	-11.39	7.88	19.40	23.84
	1:100	76.40	2.35	26.51	-14.50	4.39	23.81	28.22
	1:125	71.65	7.46	25.80	-19.25	9.50	23.10	31.53
	1:150	76.01	7.40	31.14	-14.89	9.44	28.44	33.46
Al	1:50	80.23	3.04	20.64	-10.67	5.08	17.94	21.48
	1:75	80.05	3.00	22.39	-10.85	5.04	19.69	23.04
	1:100	77.20	2.65	21.21	-13.70	4.69	18.51	23.50
	1:125	76.06	4.34	21.61	-14.84	6.38	18.91	24.87
	1:150	76.06	4.34	21.61	-14.84	6.38	18.91	24.87
Fe	1:50	63.90	1.19	11.47	-27.00	3.23	8.77	28.57
	1:75	63.90	1.19	11.47	-27.00	3.23	8.77	28.57
	1:100	62.30	1.45	12.73	-28.60	3.49	10.03	30.51
	1:125	59.74	7.11	14.22	-31.16	9.15	11.52	34.46
	1:150	59.87	0.86	14.11	-31.03	2.90	11.41	33.19



<Fig. 5> The effect of liquor ratio on the ΔE values of coffee extract dyed silk fabrics with various mordants.



<Fig. 6> K/S values of coffee extract dyed silk fabrics with various mordants in the change of liquor ratio.

K/S did not increase until the liquor ratio 1:100, then increased in all mordanted silks. K/S did not increase further in all mordanted silks except the Cu mordanted after the liquor ratio 1:125. Only Cu mordanted silk showed a constant increase from 1:100 to 1:150.

#### IV. Conclusion

From this study, the effect of dyeing conditions on the dyeing efficiency of silk fabrics pre-treated with various mordants was successfully investigated, and the following conclusions were made:

1. Coffee extract from coffee waste can effectively dye silk fabrics exerting brilliant colors.
2. The color was varied with the change of mordanting agents under the same dyeing conditions. Al, Cu, and Sn mordanted silk fabrics showed luxurious gold colors with subtle change of tone and shade, while Fe mordanted silk fabric showed a beautiful khaki color.
3. As the dyeing temperature increased, the dyeability generally increased in all mordanted silk fabrics except Fe mordanted silk until 45°C. The dyeing temperature did not affect the dyeability of Fe mordanted silk fabric.
4. As the liquor ratio increased, the dyeability showed a gradual increase in all mordanted silk except Cu mordanted silk. Cu mordanted silk fabric showed an abrupt increase in dyeability at liquor ratio 1:150.
5. The dyeing time effect can be separated into 2 stages. During the initial half an hour, the dyeability did not change significantly, and then, increased after 45 min. dyeing time duration. A further increase in dyeing time after 45 min. did not affect the dyeing efficiency in all mordanted silk fabrics except the Cu mordanted.

6. The desired color can be reproduced in the coffee extract dyeing with this dyeing condition kinetic study.

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