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A Study on the dyeing of wool felt using cochineal and mordants

- change of color and image analysis of dyed felt -

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Abstract

Felt fabric is one of the nonwovens characterized by the entanglement of the constituent fibers, resulting from the application of heat, moisture, and mechanical action to a fibrous web. This method has been applied to the wool fibers for long. As a natural dyestuff, cochineal dyestuff was employed for dyeing felt specimens. The color of the dyed felt was measured using a colorimeter. Along with this, an attempt was made in order to evaluate the variance of the color of the felted specimens having irregular orientation of wool fibers.

Key Words : felt, mordant, cochineal, color measurement, image analysis

I. Introduction

Product development in textile-related industry has focused on the environment-friendliness in recent years. Therefore, natural dyeing has become one of the interesting topics. Red color may be obtained from various plant sources such as madder root and related alizarin-based dyestuffs. The other main source of red came from insects. The best of these insect sources was American cochineal, which provided the best intensity of color and was most readily available.[1,2)]

Cochineal dyestuff is obtained from the bodies of the female *Dactylopius coccus*. This animal produces a deep maroon pigment and stores the pigment in body fluids and tissues.[3)] Cochineal forms a very fine crimson on an aluminum mordant, and scarlet on tin oxide.[4)] Main component of the cochineal is carminic acid($C_{22}H_{20}O_{13}$), and a natural anthraquinone dyestuff. It has a melting point of 135℃, and is easily soluble in water, ethanol, ether, propylene glycol, and NaOH solution, while insoluble in benzene, and chloroform. Since it has carboxylic end groups in the molecular structures, it behaves similar to acid dyestuffs.[1)3)]

Felting of wool is commonly used to improve the properties of woolen garments or materials.[1)] One of the definitions of felt is "a textile fabric characterized by the entangled condition of many or all of its component fibers." As one form of the felts, pressed felt is usually consolidated by the application of moisture, mechanical action and heat.[4)]

The CIE(International Commission on Illumination) tristimulus value system, using X, Y and Z that represent the spectrum of primary colors red, green, and blue, respectively, was formulated in 1931. In the $L^*a^*b^*$ color system, L^* is the lightness variable; a^* and b^* are chromaticity coordinates.

RGB values of the acquired image region of specimen may represent the distribution or variance of the colors of the specimen. Therefore, using a color CCD camera, one can measure the variance or evenness of the dyed specimen, in this case, dyed wool felt specimen. The evenness measurement of the dyed fibers, especially wool felt specimens, may be affected by the fiber configurations including the orientation of the fibers in the felt, and the reflection and transmission characteristics of the fibers, and the optical density of the dyed fibers. Using conversion equations, the RGB values under specified measuring conditions may be converted to the corresponding $L^*a^*b^*$ values.

$$[X\ Y\ Z] = [r\ g\ b]\ [M] \qquad \text{<Eqn. 1>}$$

where [M] is a transformation matrix.
Equations for converting XYZ into $L^*a^*b^*$ may be found in the literature.[5)]

The wool fiber is structurally complex and composed of the cuticle, the cortex and the medulla. The cuticle is composed of thin scales, comprising epicuticle, exocuticle, and endocuticle. The epicuticle and the exocuticle contain high amount of sulphur with cystine cross-links having high resistance to biological and chemical degradation along with water repellency. The cuticles also endow the wool fiber subtle luster, which is one of the important apparel textile fiber properties.

Leeder[6)] reported that the dyes have been found to enter thefiber at the junctions between the cuticle cells and to diffuse through the wool via the relatively lightly crosslinked nonkeratinous regions, and in the later stages of the dyeing cycle, the dyes migrate into the sulfur-rich keratinous regions.

The previously reported data in the study of dyeing differences of the cotton using cochineal[7)] was also compared with the results from this study.

In this study, wool fiber rovings were formed into felt specimens. The felt specimens were then pre-mordanted and subsequently dyed using cochineal dyestuff. Color of the dyed specimens were measured using a colorimeter. Wool fibers often exhibit natural variation in color yield fiber by fiber. Therefore, an attempt was made to quantify the variation of color in the cochineal dyed wool felt specimens, using a color CCD camera coupled with an objective lens.

II. Samples and Experiments

1. Sample and Mordants

1) Sample

Commercially available rovings of wool fibers of 70's were used for felting.

2) Mordants

Three types of mordants, $CuSO_4 \cdot 5H_2O$, $AlK(SO_4)_2 \cdot 12H_2O$, and $SnCl_2 \cdot 2H_2O$(Dukasan Pure Chemical Co., Ltd.)were used for mordanting.

2. Experimental Procedures

1) Felting

Wool fibers were pulled from the wool fiber rovings and placed on a plate. Three layers were needed for the felt. The first layer has rows of fibers placed horizontally on the plate. The second layer was placed perpendicular to the first layer, and the third perpendicular to the second layer. A screen was laid flat on top of the layers. Water was sprayed over the screen until the wool fibers underneath were thoroughly wetted. A detergent solution (Beat, CJ Lion Co., Korea) was evenly sprayed on the wet sample. Pressure and manual rubbing was applied on the fiber layers to disperse the detergent solution and to further the felting procedure.

2) Mordanting

Each 2% o.w.f. mordanting agent was added to the bath of deionized water of liquor ratio 1:75. Wool felt specimen was dipped in the bath at 40℃ bath temperature. The bath temperature was gradually raised from 40 to 60℃ within 20minutes. The specimens was further mordanted for 20 minutes at 60℃, and the bath was cooled down to room temperature.

3) Dyeing of specimen

Cochineal was used to prepare dyeing solutions of L.R. 1:75 at 30℃. After bath was heated to reach 40℃, cochineal was introduced to the warm bath and solubilized. Specimens were dipped in the bath and the initial dyeing temperature of 50℃ was maintained for 10 minutes. The temperature was again raised to 60℃ and it was maintained for 20 minutes. When the dyeing procedure was finished, the dye liquor was cooled down to room temperature. The dyed specimens were rinsed with deionized water and room-dried.

4) Color measurement

The color of dyed specimens were measured using Chroma-meter(CR-200b, Minolta, Japan). Using the L^*, a^*, b^* values obtained, color difference(ΔE) was calculated as follow :

$$\Delta E = \sqrt{(L_1^* - L_2^*)^2 + (a_1^* - a_2^*)^2 + (b_1^* - b_2^*)^2}$$

<Eqn. 2>

where L_2^*, a_2^*, and b_2^* were the corresponding values of the control wool felt specimen.

5) Image acquisition using a CCD camera and image analysis

A CCD color camera, ProgRes®C10plus (Jenoptik JENA, Germany), was used for acquiring images of the dyed specimens under illumination.

The CCD used in the camera wasa diagonal 8.93mm interline CCD solid-state image sensor with 3.24 million pixels and unit cell size of 3.45x3.45㎛. RGB primary color mosaic filters are used for color filtering. The CCD also employs a peltier cooling device to lower the electrical noise generated during the acquisition.

A magnifying lens(type 350N) was attached to the C-mount of the CCD camera.

As an image analysis tool, ImageJ(NIH, USA) was employed for histogram analysis and others.

III. Results and Discussion

<Table 1> shows the color measurement results of pre-mordanted and cochineal dyed wool felt specimens. The ΔE value of unmordanted and cochineal dyed wool felt specimen was 44.25, which is apparently high. By introducing mordanting procedure, it was clear that dye uptake was promoted based on the E values of the Al and Cu mordanted wool felt specimens, while that of Sn mordanted specimen was relatively lower than that of unmordanted specimen. The cochineal color of the dyed fabric changed appreciably according to the characteristics of mordants. Sn mordanted specimen gave the dyed wool felt specimen bright color and the highest L^* value among the cochineal dyed specimens. Cu mordanted specimen gave the lowest b^* value, leaning toward bluish and purplish color.

From the previous study,[7] the ΔE value of the unmordanted and cochineal dyed cotton fabric specimen was 17.48, which is visually discernible, though pale. By introducing mordanting procedure, it was clear that dye uptake was promoted based on the ΔE values of the Sn mordanted fabric, while that of Cu mordanted fabric was almostsimilar to that of unmordanted. In the case of cotton fabric specimen, the mordanting effect was not appreciable except for the case of Sn mordanting. Sn mordanting gave the fabric specimen reddish (a^*=42.1) and deep color development. Cu mordanting gave the fabric more bluish color compared to the other mordants.

<Table 1> Color measurement results of cochineal dyed wool felt (pre-mordanted)

Mordanting method	L^*	a^*	b^*	ΔE
Control(undyed)	87.0	-0.9	8.4	-
unmordanted	52.2	25.3	0.3	44.25
Sn mordant	63.9	29.1	7.9	37.83
Al mordant	46.3	34.3	2.7	54.11
Cu mordant	50.0	22.4	-4.1	45.43

This indicates that there were mordanting effects according to the types of metallic ions bonded with the cochineal dyestuffs.

Compared to these cases of cotton, dyeing of unmordanted wool felt gave relatively deep shade, and Al mordant gave the highest ΔE value. However, the differences of ΔE values among the mordanted specimens were not large when compared to those of the cotton cases.

Dyeing of protein fibers with acid dyes is primarily governed by the salt formation with amino groups. Wool fibers may be regarded as a matrix dispersed with $-NH_3$ end groups attracting the dye anionic molecules.

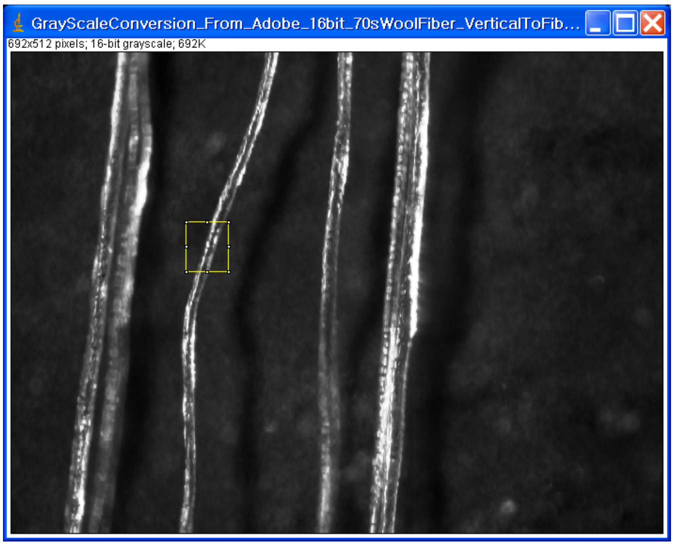
In <Table 2>, converted values of cochineal dyed wool felt specimens are given based on the L*C*h system(CIE1976). Al mordant gave the wool felt the highest value of C*(chroma), while Cu mordant gave the felt the lowest value of C*.

Reflections from the wool fibers may affect the measured color values. As shown in <Fig. 1>, the intermittent intensity patterns of reflections due to the wool scales may affect the measurement under specific conditions. Since the wool fibers have natural crimps with undetermined orientation in the felt specimen, the influence from the gloss might not be precisely predictable, leading to some uncertainty in the color measurement values.

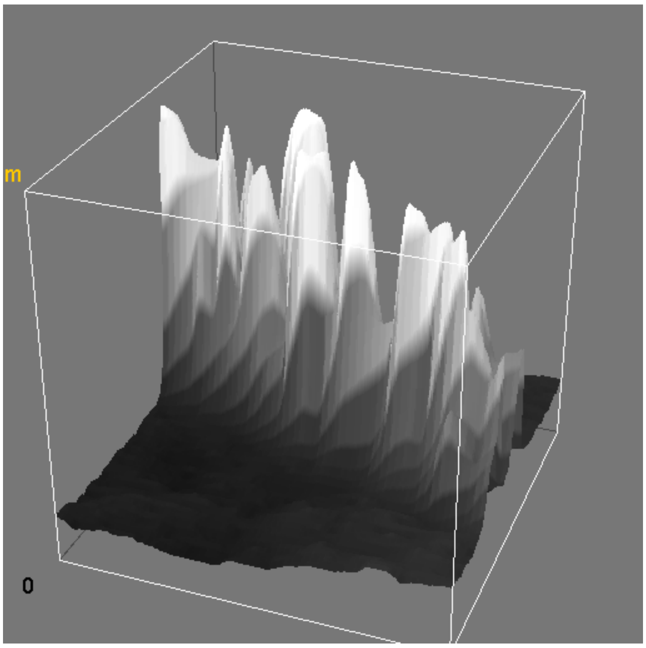
The sharp vertical peak on the rightmost part of <Fig. 2> may originate from the gloss of the fibers. This pattern may affect the measurement of wool felt color. In relatively flat specimens such as plastic film or painted surface, there are designs of measuring instruments allowing to remove the specular component of the reflection. In the textile materials, it is difficult to remove the gloss or specular component due to the complexities of the specimen configurations. However, it might be possible to remove the specular component contributions by applying

<Table 2> Converted values of cochineal dyed wool felt specimens (L*C*h system, CIE1976)

Mordanting methods	L*	C*	h
Control(undyed)	87	8.45	96.1
unmordanted	52.2	25.30	0.7
Sn mordant	63.9	30.15	15.2
Al mordant	46.3	34.41	4.5
Cu mordant	50.0	22.77	349.6



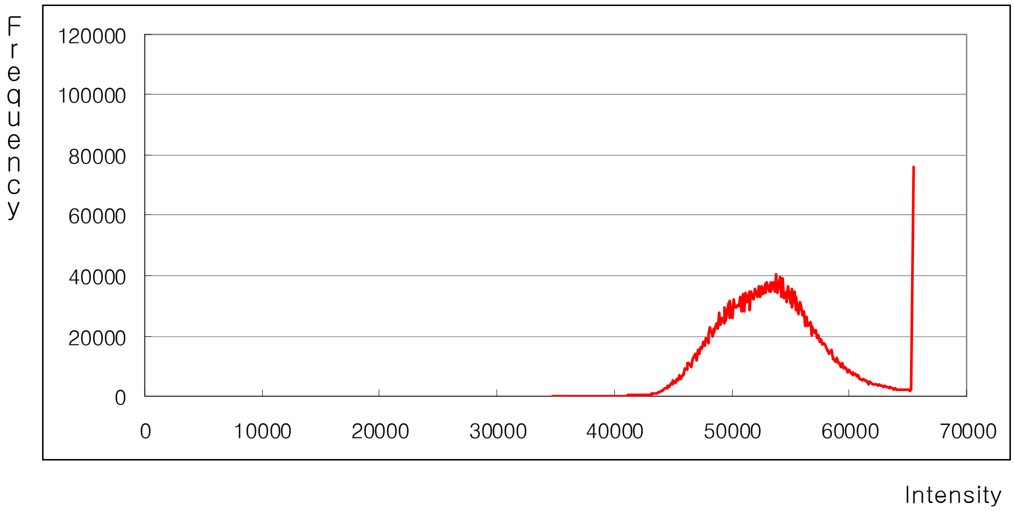
(a)



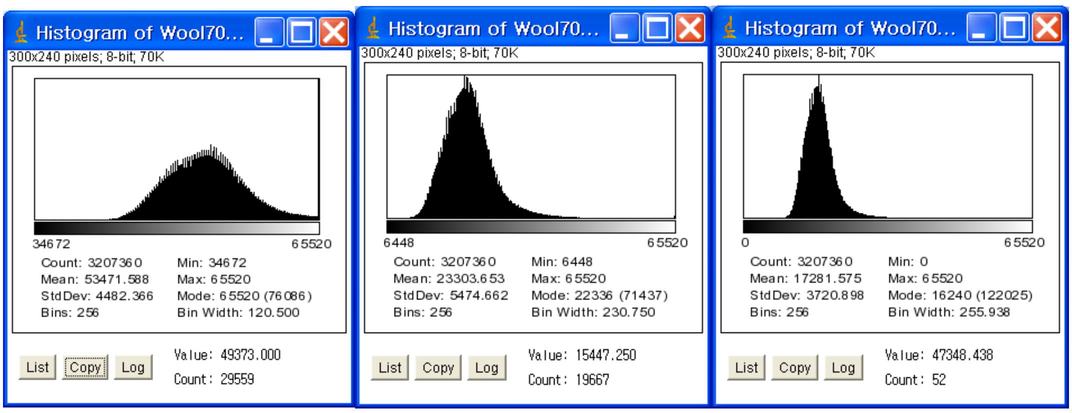
(b)

<Fig. 1> (a) Gray scale image of the wool fibers under illumination. (b)Small square in the image (a) is 3-dimensionally represented with the vertical axis of intensity, showing the intermittent reflection patterns from the scales of wool fibers

image processing and analysis techniques. Overall histograms of the red, green, and blue (RGB) components of the acquired image are presented in <Fig. 3>, and from the peak shape one can figure out the distribution or uniformity of the specimen's color. A simple method to compare distributions is to calculate the standard deviation or coefficient of variation of the readings.



<Fig. 2> Histogram of the R component(Red) intensity from the color image of the Sn pre-mordanted and cochineal dyed wool felt



Red Green Blue

<Fig. 3> Histogram of the acquired RGB color image of the Sn pre-mordanted and cochineal dyed wool felt: Red, Green, and Blue

<Table 3> Average and standard deviation values calculated from the acquired images of the dyed wool felt specimens

Mordanting Method	Average			Std.dev.		
	R	G	B	R	G	B
unmordanted	36,382	19,172	15,293	6,635	5,737	4,113
Sn mordant	53,472	23,304	17,282	4,482	5,474	3,721
Al mordant	34,232	14,415	12,666	6,698	5,627	3,948
Cu mordant	33,586	18,969	16,938	7,061	6,004	4,174

In <Table 3>, the standard deviation values are given in order to compare the variation from the acquired image of felt specimens. Standard deviations of R, G, and B components of the Cu mordant were relatively high compared to those of other mordant including unmordanted specimen, which may be an indication of the color variation across the dyed specimen. Standard deviations of Sn mordanted specimen were relatively low compared to the others. This may indicate that the Sn mordant gave more uniform shade across the specimen.

IV. Conclusions

From wool fiber rovings, felt specimens were prepared and dyed using a natural dyestuff, cochineal. The effect of pre-mordanting of wool felt was compared. L^*, a^*, and b^* values were measured using a colorimeter. In order to evaluate the variation of the colors of dyed specimens, color images were acquired using a color CCD camera.

1. The ΔE value of unmordanted and cochineal dyed wool felt specimen was 44.25, which is apparently high.
2. By introducing mordanting procedure, it was clear that dye uptake was promoted based on the ΔE values of the Al and Cu mordanted wool felt specimens, while that of Sn mordanted specimen was relatively lower than that of unmordanted specimen.
3. The cochineal color of the dyed fabric changed appreciably according to the characteristics of the mordants. Sn mordanted specimen gave the dyed wool felt specimen bright color and the highest L^* value among the cochineal dyed specimens. Cu mordanted specimen gave the lowest b^* value, leaning toward bluish and purplish color.
4. Standard deviations of R, G, and B components of the Cu mordant were relatively high compared to those of other mordant including unmordanted specimen, which may be an indication of the color variation across the dyed specimen.

References

1) E.R. Trotman, *"Dyeing and Chemical Technology of Textile Fibres",* Charles Griffin & Co. Ltd., London 1975.

2) *R. Hage,* http://www.bell.lib.umn.edu/Products/cochinea.html

3) *"Aztec Dye cochineal: A blend of culture and Science",* http://scienckit.com

4) *M. J. Denton and P. N. Daniels, "Textile Terms and Definitions",* The Textile Institute, Manchester, 2002.

5) *"Chroma Meter CR-200b Instruction Manual",* Minolta Co., Osaka, Japan, 1987.

6) J. D. Leeder, J. A. Rippon, F. E. Rothery, and I. W. Stapleton, "Use of the Transmission Electron Microscope to study Dyeing and Diffusion Processes," *Proceedings of the 7th International Wool Textile Research Conference,* pp99-108, Tokyo, 1985.

7) D. W. Jeon, J. J. Kim and H. J. Kwon, "A Study on the Dyeing Characteristics in Cochineal Dyeing of Chitosan-treated Fabrics according to the Sequence of Mordanting Procedure", *Fashion Business,* Vol.7, No.5, pp. 83-91 (2003).

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