

## Effect of Extraction Method of Loess in Cotton Fabric Dyeing

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In this research, cotton samples dyeing with ball-milled loess showed larger increase in dye uptake and the change to more reddish yellow colour than that with the elutriated one. With the increase of ball-milling time, it was decreased to smaller size and changed from multi-layer to ragged spherical or rod-like form. For narrower particle size distribution and smaller particle size, K/S value of cotton fabric was increased and the colour was changed to more reddish yellow. SEM analysis confirmed that the loess particle did not penetrate inside the fiber but adhered the surface of cotton fiber.

**Key Words:** Loess, Elutriation, Ball-milling, Particle size distribution.

### INTRODUCTION

Loess has been used in Korea as construction materials, cosmetics, ceramic industry, heavy metal elimination from aqueous solutions, control of algal blooms and natural dyestuff<sup>1,2</sup>. Especially, due to far-infrared radiation, loess-dyed fabric is widely used for health products such as underwear, beddings and other home furnishings. Commercial loess powder is prepared by extraction, that is, elutriation or ball-milling. The particle size of loess commonly used for dyeing is smaller than 0.005 mm in diameter. The particle, linear and flat form, is combined with cotton fabric by hydrogen bonding and vander Waals force<sup>3-6</sup>. However, the effect of extraction and particle size distribution onto dyeing was not reported until now.

This study is to compare the extraction methods in terms of particle shape, size and distribution and to investigate the dye uptake and colour change for loess-dyed cotton fabrics<sup>7</sup>.

### EXPERIMENTAL

Loess soil for elutriation was collected in Bayer Co., Germany. Loess powder for ball-milling was purchased commercially. A bleached and scoured cotton fabric (plain weave, 25 × 21 cm<sup>-2</sup>, 132 g/m<sup>2</sup>) was used.

#### Extraction method

**Soil:** 480 g of loess soil and 180 mL of water were blended in the mixer. After removing the impurities and large particles by 190 μm mesh, fine particles were collected. They were dispersed in water and left for 30 d. To compare with elutriation soil, it was ball-milled at 210 rpm for 50 min and 3 h.

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**Powder:** 40 g of loess powder and 200 mL of water were mixed in 480 mL stainless steel tank with 50 g of ceramic balls (0.3 cm in diameter) at 230 rpm from 50 min to 9 h. The ceramic balls were removed and then, the powder was classified and dried *in vacuo*<sup>8-10</sup>. Here, it was classified by using the meshes of 26, 45 and 75  $\mu\text{m}$ , subsequently.

**Analyses of loess:** The chemical components of loess powder were analyzed by X-ray fluorescence spectrometer (RIX 2000, Rigaku, Japan). The particle size and the surface contour were assessed using a Particle Size Analyzer (Malvern PSA, UK) and scanning electron microscope (JSM 5400, Jeol Inc., Japan), respectively. Colour was measured by the method of JS-777 (Japan C.T.S.).

**Dyeing process:** Dyeing was carried out at a liquid ratio of 1:50 with dye concentration of 4 % owb (on the weight of bath). The temperature was raised to 65 °C over 10 min and maintained at this level for 40 min, using infrared automatic dyeing machine (Ahiba, Daerim Eng., Switzerland). The samples were dried at room temperature for over night, rinsed with water and then dried at 40 °C for 1 h.

Dye uptake was assessed by measuring reflectance on a Macbeth colorimeter (Color-Eye 3100, USA). The K/S value was calculated by using Kubelka-Munk equation

$$K/S = (1-R)^2/2R$$

where K/S is the absorption coefficient, S is the scattering coefficient and R is the reflectance at wavelength of 440 nm. Colour was measured by the Hunter Lab system and Munsell system at light source D<sub>65</sub>, 2 degree.

## RESULTS AND DISCUSSION

**Analysis of loess components:** Table-1 show two types of loess components used in this study. Both samples contain SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> components. But the soil has more Fe<sub>2</sub>O<sub>3</sub> than the powder. So the soil colour is more reddish yellow than the powder one.

TABLE-1  
ELEMENTAL ANALYSIS OF LOESS (wt. %)

Element	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	MnO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	ZrO <sub>2</sub>	CaO
Soil	49.83	23.41	11.99	0.98	0.26	1.05	0.06	1.13	0.10	0.01	0.11
Powder	44.96	37.01	4.13	0.30	0.03	0.39	0.12	0.61	0.02	0.01	0.21

**Elutriation and ball-milling:** Table-2 shows that the particle size of loess was decreased with the increase of elutriation time. It can be controlled by weathering, such as, freezing, contraction and swelling by temperature, abrasion by running water. Because of this, some traditional dyers elutriate the loess in autumn and kept the container under the ground until spring<sup>10-14</sup>.

The average particle size per volume of the elutriated sample D (V, 0.5)) decreased from 12  $\mu\text{m}$  to *ca.* 9  $\mu\text{m}$  when it was maintained for 30 d. But by ball-milling for 3 h, it was decreased up to 8  $\mu\text{m}$ . K/S value of ball-milling for 3 h (1.60) is higher than that of elutriation (1.44) in dyeing cotton fabric with loess.

TABLE-2  
PARTICLE SIZE AND LOESS COLOUR BY EXTRACTION METHOD IN SOIL

Extraction method	Time	D (V, 0.5) ( $\mu\text{m}$ )	L	a	b	H	V/C
Elutriation	1 d	11.91					
	10 d	10.02					
	20 d	8.91					
	30 d	8.19	66.81	11.09	26.02	6.91YR	7.71/5.94
Ball-milling	1 h	8.37					
	3 h	8.05	65.97	13.01	24.93	6.18YR	6.94/6.03

Dyed cotton fabric with ball-milling loess shows darker and more reddish yellow than that of elutriation at the colour difference of Hunter Lab (Fig. 1). The particle obtained by elutriation takes multi-layer sheet form but ball-milled particle takes rather rugged form (Fig. 2).

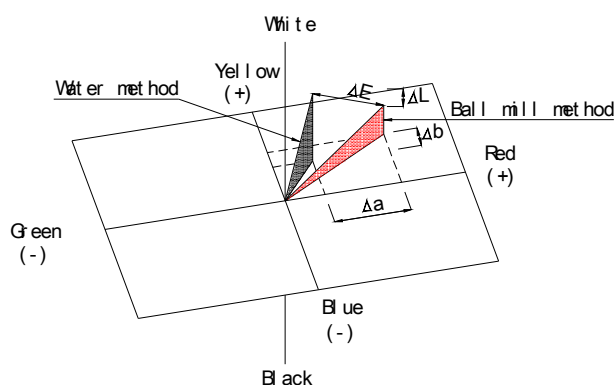


Fig. 1. Colour difference of Hunter Lab dyed cotton by elutriation and ball-milling

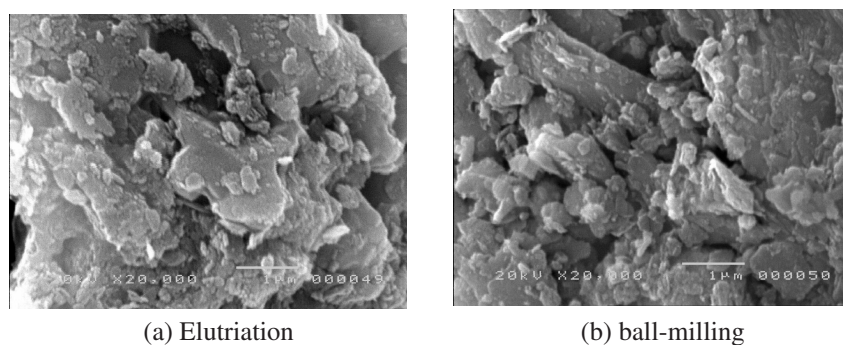


Fig. 2. SEM pictures of the elutriated and the ball-milled loess ( $\times 20,000$ )

**Ball-milling time:** Particle size of loess was decreased with the increase of ball mill time. As shown in Table-3, the average particle size of  $15.12 \mu\text{m}$  (0 h) is changed to  $13.41 \mu\text{m}$  (1 h) and  $11.01 \mu\text{m}$  (9 h). With the increase of ball-mill time,

the colour has changed darker, more reddish and yellowish. The dyed cotton fabric also shows the increased K/S value at 440 nm (Fig. 3). For the smaller particle size, the higher K/S value was obtained. SEM pictures show that the shape predominantly of multi-layer (0 h) is changed to more spherical or rod-like form (3 and 9 h) (Fig. 4).

TABLE-3  
PARTICLE SIZE DISTRIBUTION AND COLOUR BY  
BALL MILL TIME OF LOESS POWDER

Ball-mill time (h)	D (V, 0.5) ( $\mu\text{m}$ )	L	a	b	H	V/C
0	16.10	81.08	9.85	25.49	7.72YR	7.91/5.51
1	14.31	79.17	10.31	25.89	7.60YR	7.24/5.89
3	13.61	79.13	10.63	25.99	7.53YR	7.01/6.11
5	13.49	77.58	10.91	25.36	7.43YR	6.98/5.79
7	13.28	76.31	11.33	26.39	7.44YR	6.81/6.11
9	12.11	74.21	12.21	26.59	7.31YR	6.75/6.23

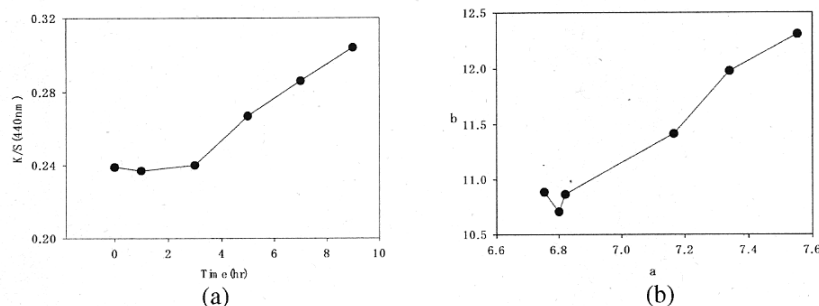


Fig. 3. Dye uptake (a) and colour change of Hunter Lab (b) with ball-mill time (60/40 min)

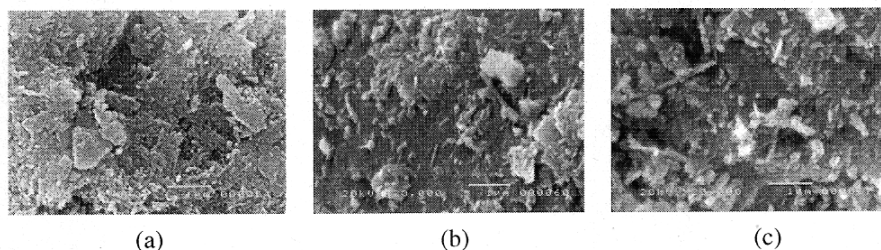


Fig. 4. SEM pictures of loess particle by ball-milling ( $\times 20,000$ ): (a) 0 h, (b) 3 h and (c) 9 h

**Particle size distribution:** The loess powder was classified with the size of 0-26, 26-45 and 45-75  $\mu\text{m}$  (Table-4). The average particle size was decreased from 14.7  $\mu\text{m}$  before classification to 8.13, 11.5 and 19.8  $\mu\text{m}$ . With the decrease of average particle size, it is changed to darker and more reddish colour. For the sample of smaller average size and of broader particle size distribution, it is changed to brighter colour. K/S value of dyed cotton fabric is also increased with the increase of concentration

and for the narrower particle size distribution (Fig. 5). SEM pictures also indicate that the loess particles are distributed at the surface of cotton fiber not penetrated inside the fiber (Fig. 6). This confirms that loess, though commonly treated as natural dyestuff, is quite different from natural and manmade dyes but is a kind of pigment.

TABLE-4  
PARTICLE SIZE DISTRIBUTION AND COLOUR OF LOESS POWDER

Sample	D (V, 05) ( $\mu\text{m}$ )	L	a	b	H	V/C
~ 26 $\mu\text{m}$	8.13	72.68	11.92	26.06	7.12YR	7.63/6.09
26~45 $\mu\text{m}$	11.53	73.13	11.67	25.62	7.09YR	7.97/5.95
45~75 $\mu\text{m}$	19.86	75.54	11.66	26.97	7.34YR	7.88/6.14
Original	14.70	77.81	10.72	26.60	7.54YR	8.08/5.85

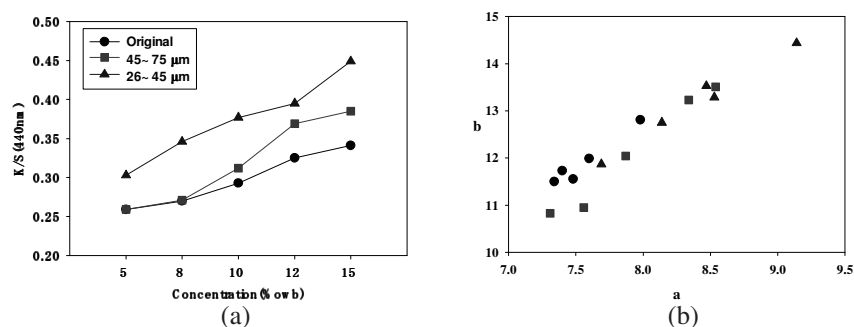


Fig. 5. Dye uptake (a) and colour change of Hunter Lab (b) with particle size distribution (60/40 min)

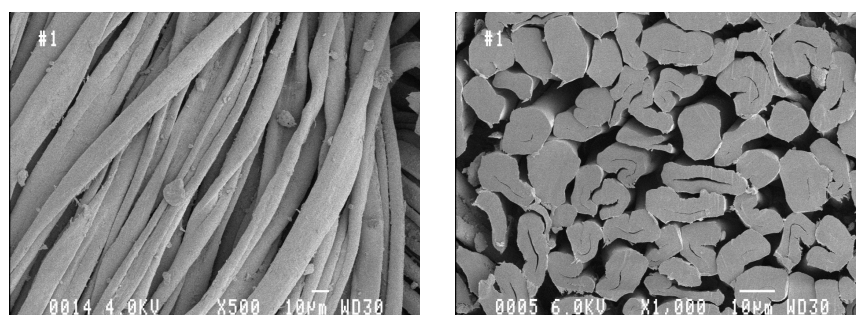


Fig. 6. SEM pictures of the fabrics dyed with loess: longitudinal ( $\times 500$ ) (a) and cross-sectional ( $\times 1,000$ ) views (b)

## Conclusion

The extraction of loess, *i.e.*, the elutriation and the ball-milling and its uptake and colour difference onto cotton fabric was investigated and summarized as follows:

The dyeing with ball-milled loess showed larger increase in dye uptake and the change to more reddish yellow colour than that with the elutriated one. With the increase of ball-milling time, it was decreased to smaller size and changed from multi-layer to ragged spherical or rod-like form. For narrower particle size distribution and smaller particle size, K/S value of cotton fabric was increased and the colour was changed to more reddish yellow. SEM analysis confirmed that the loess particle did not penetrate inside the fiber but adhered the surface of cotton fiber.

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(Received: 18 May 2009; Accepted: 24 August 2009)

AJC-7778