Dyeing, Fastness, and Deodorizing Properties of Cotton, Silk, and Wool Fabrics Dyed with Coffee Sludge (*Coffea arabica L.*) Extract

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ABSTRACT: A natural colorant was extracted from *Coffea arabica L.*, using water as extractant at 90°C for 90 min. Studies have been made on the dyeing, color fastness, and deodorization properties of cotton, silk, and wool fabrics dyed with *Coffea arabica L.* extract solutions. The best mordants were found to be $FeSO_4$, $CuSO_4$, and $SnSO_4$ for improving the color strength (K/S) of cotton, silk, and wool fabrics. Mordants $MnSO_4$, $ZnSO_4$, and $NiSO_4$ for cotton (Rating 3), and all mordants except $MnSO_4$ for silk (Rating 3), mordants $CuSO_4$, $FeSO_4$, $CoSO_4$, $Al_2(SO_4)_3$, and $MnSO_4$ for wool (Rating 4) were the best mordants to improve the light fastness. It was found that $FeSO_4$ and $CuSO_4$ were the best mordants for the improvements of color strength (K/S) and light fastness for silk and wool fabrics. In addition, it was found that cotton, silk, and wool fabrics dyed with the *Coffea arabica L.* extract showed good deodorization performance. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 103: 251–257, 2007

Key words: natural dye; coffee sludge (*Coffea arabica L.*); mordant; dyeing; color fastness; deodorization performance

INTRODUCTION

The coloring of textiles, wood, leather, and other natural commodities with dyes from plants and other natural product is receiving increasing attention. What attracts people to textiles colored with natural dyes may be one or a combination of factors, including a preference for naturalness, environmental friendliness, lower toxicity, antibacterial/antiallergic/deodorizing/anticancer properties, harmonizing natural shades or just the novelty.[1,2]

However, there are a small number of companies that are known to produce natural dyes commercially. For example, de la Robbia, which began in 1992 in Milan, produces water extracts of natural dyes such as weld, chlorophyll, logwood, and cochineal under the Eco-Tex certifying system and supplies the textile industry. In the USA another company, Allegro Natural Dyes, produces natural dyes under the Ecolor label for the textile industry.[3] Aware of the Toxic Substance Act and the Environmental Protection Agency, they claim to have developed a mordant using a nontoxic aluminum formulation and biodegradable auxiliary substances. In Germany, Livos Pflanzenchemie Forschungs-und Entwicklungs marketed numerous natural products. In France, Bleu de Pastel sold an extract of woad leaves. There are several small textile companies using natural dyes. India is still a major producer of most naturally dyed textiles.

In the last decade, investigations about the possible use of natural dyes in textile dyeing processes have been performed by various research groups. In general, to improve its fastness, most of the dyeing processes were conducted using metal salts (e.g., potassium dichromate, stannous chloride, ferrous sulfate, and copper sulfate) as mordants.[4] The metal ions can act as electron acceptors to electron donor to form coordinate bonds with the dye molecules, which are insoluble in water.[5]

The dyeing of cotton and jute with tea as a natural dye using alum, copper sulfate, or ferrous sulfate mordants has been studied by Deo and Dosai.[6] Bhattacharya et al. investigated the properties of selected natural dyes on jute.[7] Nishida and Kobayashi reported properties of natural dyes on silk, cotton, and cashimilon using alum or ferrous sulfate mordants.[8] Brucker et al. investigated the color depth and fastness properties of selected natural dyes on wool and synthetic fiber, e.g., polyester, polyamide, and polyacrylonitrile.[3] Lokhande and Dorugade presented results with selected natural dyes on polyamide using various mordants, e.g., alum, ferrous sulfate, stannous chloride, and tannic acid.[9] Cho et al. reported results with natural dyes (Gardenia and Turmeric) on silk and cotton using various mordants.[10,11] Lim et al. investigated the dyeing characteristics with selected natural dyes (amur cork tree, palmatin, gomwell, red wood,

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and cochineal) on cotton and wool using Al mordant.[12] Cho presented results with natural dye (cochineal) on wool fabric using various mordants.[13] Nam et al. reported the dyeing of cotton fabric with natural dye (safflower) using aqueous acid.[14] We investigated the dyeing properties and fastness of cotton and silk fabrics dyed with cherry extract and *Cassia tora L.* extract using various mordants.[15,16] However, research in the natural dyes obtained from *Coffea arabica L.* extract can hardly be found.

The *Coffea Arabica L.* is described under the class of *Rubiaceae Juss*, its common name is coffee, arabian coffee, and arabica coffee. The major chemical constituents of *Coffea Arabica L.* beans are caffeine 1–2%, coffee oil 10–15%, sucrose, and other sugars about 8%, protein about 11%, ash about 5%, chlorogenic and caffeic acids about 6%. Other constituents include cellulose, hemicellulose, trigonelline, tannic acid, and volatile oils.[17]

Generally, problems in dyeing with natural dyes are related to low exhaustion of dyes and to the poor fastness of dyed fabrics. Attempts to overcome these problems have been mainly focused on the use of metallic salts as mordants, which are traditionally used to improve exhaustion or fastness properties and develop different shades with same dye.[18–21]

In this study, the influence of the kind of mordants ($MnSO_4$, $CuSO_4$, $ZnSO_4$, $Al_2(SO_4)_3$, $FeSO_4$, $NiSO_4$, $CoSO_4$, and $SnSO_4$) on the dyeing property and color fastness (light, water, and perspiration fastness) of cotton, silk, and wool fabrics was investigated. In addition, the values of K/S and apparent color, and the color fastness of premordanted and unmordanted cotton, silk, and wool fabrics were compared. Later on, the influence of deodorization performance on the various fabrics, mordanted fabrics, and dyed fabrics with *Coffea arabica L.* extract solution was studied in detail.

EXPERIMENTAL

Materials

Cotton, silk, and wool fabrics (Standard Adjacent Fabrics for Staining of Fastness: KS K 0905) were used without further purification. The colorimetric data (L^*, a^*, b^*, brightness, yellowness, and whiteness, respectively) for the reference samples are given in Table I. Aluminum sulfate hydrate ($Al_2(SO_4)_3 \cdot 13–14H_2O$, M_W 585; Junsei Chemical), manganese (II) sulfate hydrate ($MnSO_4 \cdot 5H_2O$, M_W 241; Junsei Chemical), zinc sulfate hydrate ($ZnSO_4 \cdot 7H_2O$, M_W 287.56; Junsei Chemical), nickel sulfate hydrate ($NiSO_4 \cdot 6H_2O$, M_W 262.87; Junsei Chemical), copper sulfate hydrate ($CuSO_4 \cdot 5H_2O$, M_W 249.68; Junsei Chemical), iron(II) sulfate hydrate ($FeSO_4 \cdot 7H_2O$, M_W 278.03; Junsei Chemical), stannous sulfate ($SnSO_4$, 95%, M_W 214.75; Hayashi Pure Chemical Industries), and cobalt (II) sulfate hydrate ($CoSO_4 \cdot 7H_2O$, MW : 281.10, Shinyo Pure Chemicals) were used without further purification.

Extracting/Mordanting/Dyeing

Extraction: Coffee sludges were dried and extracted using water as extractant at 90°C for 90 min in material–liquor ratio 1 : 10. The coffee sludge extract solution (natural dye solution) obtained in this study was a yellowish brown solution.

Mordanting: Cotton, silk, and wool fabrics were premordanted at 40°C for 60 min using 3% owf mordant solutions (except aluminum sulfate hydrate 10% owf) with liquor ratio 1 : 50.

Dyeing: Cotton, silk, and wool fabrics were dyed by exhaustion method with a fixed bath ratio (1 : 100) at 80°C for 60 min.

Characterization

An FTIR (computerized Nicolet Impact 400D Fourier Transform Infrared spectrometer, USA) was used to identify the structure of *Coffea arabica L.* extract. For each sample, 32 scans at 4 cm^{-1} resolution were collected in the transmittance mode. The content of mordanted metal ion of premordanted fabrics was determined using an Induced Coupled Plasma Atomic Emission Spectrometer (Optima 4300 DV, Perkin–Elmer, USA). The reflectance values and the corresponding CIE L^*, a^*, b^*, H (hue) V (value)/C (chroma), and K/S values for the dyed samples were measured using computer color matching (Gretag Macbath Color-Eye 7000A, USA) interfaced to a digital PC under illuminant D_{65}, with a 10^0 standard observer. CIE (Commission Internationale de l'Eclairage or International Commision on Illumination) L^*, a^*, b^* are CIE 1976 psychromic (or metric) lightness, redness

TABLE I
Color Characteristics of Cotton, Silk and Wool Fabrics

Fabric	L^*	a^*	b^*	Brightness (T452)	Yellowness (ASTMD1925)	Whiteness (CIEGanz82)
Cotton	87.752	−0.395	0.457	71.133	0.742	69.321
Silk	78.960	−0.169	0.251	54.670	0.578	53.525
Wool	82.088	−1.545	8.294	52.947	15.469	17.664

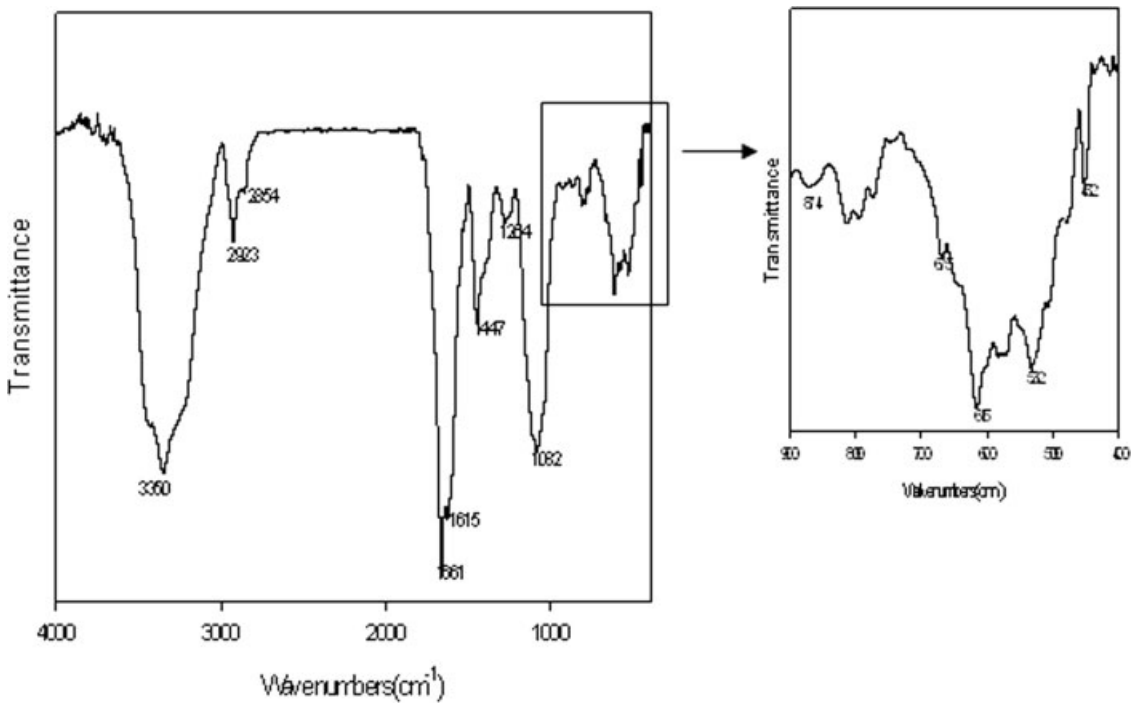

Figure 1 IR spectrum of *Coffea arabica L.* extract solution.

TABLE II
The Dyeing Results, the Colorimetric Data (*L, *a**, *b** and HV/C) and Color Strength (*K/S*) for the Dyed Samples with or without Metallic Salts**

Fabric	Mordant	*L**	*a**	*b**	H	V/C	*K/S*
Cotton	None	88.113	1.668	10.876	9.39YR	8.71/1.65	0.510
	Mn	80.589	0.253	6.484	1.49Y	7.93/0.92	2.613
	Cu	72.252	3.329	10.074	7.89YR	7.07/1.77	5.601
	Zn	81.625	0.218	6.378	1.51Y	8.03/0.90	2.348
	Al	80.655	0.165	6.652	1.70Y	7.94/0.94	2.613
	Fe	75.427	0.008	6.907	0.18Y	7.39/1.06	4.266
	Ni	80.838	0.397	6.717	1.18Y	7.95/0.97	2.550
	Co	80.790	0.375	6.706	1.22Y	7.95/0.97	2.594
	Sn	75.430	1.416	12.308	0.67Y	7.39/1.85	6.533
Silk	None	74.477	5.036	21.337	9.13YR	6.42/4.35	5.100
	Mn	63.342	2.028	12.970	0.65Y	6.15/1.99	17.950
	Cu	52.856	4.439	13.404	8.75YR	5.11/2.32	27.853
	Zn	64.026	1.945	12.988	0.71Y	6.22/1.98	13.664
	Al	62.393	2.254	13.876	0.64Y	6.06/2.14	15.971
	Fe	44.130	1.776	8.727	0.81Y	4.25/1.34	45.594
	Ni	63.058	24.54	12.854	0.68Y	6.13/1.97	14.375
	Co	62.633	2.313	12.901	0.37Y	6.08/2.00	14.906
	Sn	62.226	1.493	16.638	1.81Y	6.04/2.48	31.437
Wool	None	74.477	5.036	21.337	9.35YR	7.29/3.53	9.070
	Mn	60.255	5.003	17.029	9.04YR	5.85/2.89	14.234
	Cu	52.576	4.855	15.981	9.23YR	5.08/2.71	35.577
	Zn	61.746	4.794	17.294	9.23YR	5.99/2.90	21.596
	Al	55.401	6.618	18.194	8.35YR	5.36/3.22	34.519
	Fe	42.257	1.686	8.953	1.09Y	4.08/1.35	54.925
	Ni	60.307	4.783	30.836	9.21YR	5.85/2.84	23.239
	Co	59.061	5.137	17.370	9.07YR	5.73/2.95	25.693
	Sn	62.825	3.905	20.627	0.51Y	6.10/3.28	36.441

TABLE III
The Content of Metal Ion (ppm) of Cotton, Silk, and Wool Fabrics Premordanted with Various Metal Salts

	Mordant							
Fabric	Mn	Cu	Zn	Al	Fe	Ni	Co	Sn
Cotton	998	1515	1546	596	1365	1125	1254	4341
Silk	1699	2802	2058	1155	2371	2414	1665	10470
Wool	825.2	1135	996	888.1	1144	761.7	783.5	3433

(or greenness), and yellowness (or blueness), respectively. L^*, a^*, b^* was calculated as follows:

$$L^* = 116(Y/Y_n)^{1/3}$$

$$a^* = 500[(X/X_n)1/3 - (Y/Y_n)^{1/3}]$$

$$b^* = 200[Y/Y_n]1/3 - (Z/Z_n)^{1/3}]$$

where X, Y, and Z are CIE tristimulus values, and X_n, Y_n, and Z_n are CIE tristimulus values to the reference white under the source used. Color strength, K/S, was calculated from the reflectance values using the Kubelka–Munk equation as follows:

$$K/S = (1 - R)^2/2R - (1 - R_0)^2/2R_0$$

where R is the reflectance of the colored fabric, R_0 is the reflectance of the uncoloured fabric, and K/S is the ratio of the absorption coefficient (K) to scattering coefficient (S); the higher the value, the greater the color strength. TAPPI brightness, yellowness-C, and whiteness were determined according to T452, ASTM D1925, and CIE Ganz 82, respectively. The light fastness, the water fastness, and the perspiration fastness were determined according to KS K 0700, KS K 0645, and KS K 0715, respectively. Fade and stain in color were assessed using gray scales. The deodorizing performance was calculated as follows:

$$\text{Deodorization performance (\%)} = (C_b - C_s)/C_b$$

where C_b is the gas concentration of blank and C_s the gas concentration under specimen existence. Ammonia aqueous solution (2 μL) was taken in a 1000-mL flask, the cloth was suspended as sheets (10 × 10 cm^2) at 30°C and RH 40%, and the concentration of ammonia was determined at each time.

RESULTS AND DISCUSSION

Identification of *Coffea arabica L.* extract

Figure 1 shows the IR spectrum of *Coffea arabica L.* extract solution. Generally, it is known that the extract of *Coffea arabica L.* beans contains caffeine 1–2%, coffee oil 10–15%, sucrose and other sugars about 8%, protein about 11%, ash about 5%, and chlorogenic and caffeic acids about 6%. Other constituents include cellulose, hemicellulose, trigonelline, tannic acid, and volatile oils.

The characteristic peaks corresponding to the OH stretch and NH stretch at 3372 cm^{-1}, CH antisymmetric and symmetric stretch at 2923 cm^{-1} and 2854 cm^{-1}, C=O stretch at 1661 cm^{-1}, OH deformation at 1615 cm^{-1}, in-plane OH bending at 1417 cm^{-1}, CH_2 scissors vibration at 1350 cm^{-1}, C—O—C stretch at 1264 cm^{-1}, C—N stretch at 1082 cm^{-1}, CH out of plane deformation and OH out of plane deformation at 716 cm^{-1}, C—O—H bending at 675 cm^{-1}, C=O out-of-plane bend at 615 cm^{-1} ,C—C=O bend at 532 cm^{-1}, and C—O—C bend at 452 cm^{-1} were observed indicating the presence of coffee's component.

Dyeing properties

From the preliminary experiment, it was found that the optimum dyeing time and temperature were 60 min

TABLE IV
Color Fastness of Cotton Fabrics Dyed with *Coffea arabica L.* Extract

Mordant				None	Mn	Cu	Zn	Al	Fe	Ni	Co	Sn
	Light			2	3	2	3	2	2	3	2	2
Water		Fade		4	4–5	4–5	4–5	4	4–5	4	4–5	4
		Stain	Silk	4	4	4–5	4–5	4	4	4–5	4–5	4–5
			Cotton	4	4–5	5	4–5	5	4	4–5	5	4–5
Perspiration	Acidic	Fade		3	4–5	2	4	3–4	4–5	4–5	4	3
		Stain	Silk	4	4–5	3	3–4	4	4–5	4–5	4–5	4
			Cotton	4	4–5	4	3–4	4	4–5	4–5	4–5	4
	Alkaline	Fade		4	4	2–8	3	4	3–4	3–4	4	3–4
		Stain	Silk	3	4	4	3	4	4	4	4	4
			Cotton	3–4	4	4	3	4	4	4	4	4

TABLE V
Color Fastness of Silk Fabrics Dyed with *Coffea arabica L.* Extract

	Mordant			None	Mn	Cu	Zn	Al	Fe	Ni	Co	Sn
	Light			2	2	3	3	3	3	3	3	3
Water		Fade		3	3	5	4	3	5	4	4	5
		Stain	Silk	4	4	5	3	4	5	5	4–5	5
			Cotton	4	4	4–5	4	3	5	5	4–5	5
Perspiration	Acidic	Fade		4	4	4	4	4	4	4	4	5
		Stain	Silk	3	4	5	5	5	5	5	5	5
			Cotton	4	4	5	5	4–5	5	5	5	5
	Alkaline	Fade		4	4	3	3	4	4	4	4	5
		Stain	Silk	3–4	5	3–4	3	4	4–5	4–5	5	5
			Cotton	3	5	3	3	3	4	4	5	4

and 80°C, respectively, to give the highest K/S values for cotton, silk, and wool fabrics. Therefore, the pretreated cotton, silk, and wool fabrics, with metallic salts (mordants), were dyed with *Coffea arabica L.* extract solution at 80°C for 60 min. The dyeing results, the colorimetric data (L^*, a^*, b^*, and HV/C), and color strength (K/S) for the dyed samples with and without metallic salts pretreatment are given in Table II. It was found that the K/S values of pretreated cotton, silk, and wool fabrics, with mordants, were higher than those of unmordanted fabrics. The K/S values of silk fabrics were higher than those of cotton fabrics. This might be due to the higher mordanted metal ion content (see Table III). This may be due to the easier formation of complex between metal ions and ligands of silk compared with cotton. The K/S values of wool were higher than those of silk fabrics. This may due to the higher yellowness and the lower whiteness (see Table I). The best mordants were found to be $FeSO_4$, $CuSO_4$, and $SnSO_4$ for improving the color strength (K/S) of cotton, silk, and wool fabrics.

The variation in color due to the use of different mordant is a well-known phenomenon. The apparent colors of cotton and silk fabrics pretreated using $CuSO_4$ were 7.89 YR and 8.75 YR. And the apparent colors of cotton and silk fabrics pretreated using $MnSO_4$, $ZnSO_4$, $Al_2(SO_4)_3$, FeSO4, $NiSO_4$, $CoSO_4$, $SnSO_4$ were 1.49–1.81 Y. However, the apparent colors of wool fabrics pretreated using $FeSO_4$ and $SnSO_4$ were 1.09 and 0.51 Y. And the apparent colors of wool fabrics pretreated using $MnSO_4$, $CuSO_4$, $ZnSO_4$, $Al_2(SO_4)_3$, $FeSO_4$, $NiSO_4$, $CoSO_4$ were 9.04–9.07 YR. With varying the kind of mordant, the color of dyed cotton, silk, and wool fabrics was changed from yellow-red to yellow or yellow-red. The value of Munsell coordinates (value and chroma) were lower for dyed samples pretreated with metallic salts than for those without pretreatment. It related to darkness and dullness of colors. From the above findings, it was concluded that the apparent colors depended on the kind of mordant used in this study.

Color fastness

The rating of fastness (light, water, and perspiration fastness) of premordanted and unmordanted cotton, silk, and wool fabrics dyed with *Coffea arabica L.* extract are shown in Tables IV–VI, respectively. It was found that water and perspiration fastness of unmordanted cotton, silk, and wool fabrics was considerably good. However, the light fastness of unmordanted cotton, silk, and wool fabrics was bad. Generally, it is well known that poor light fastness is a problem for natural dyes. The light fastness of fabrics were found to increase from Rating 2 to Rating 3, from Rating 2

TABLE VI
Color Fastness of Wool Fabrics Dyed with *Coffea arabica L.* Extract

	Mordant			None	Mn	Cu	Zn	Al	Fe	Ni	Co	Sn
	Light			1	4	4	2	4	4	3	4	2
Water		Fade		4	4	5	5	5	5	5	5	5
		Stain	Silk	4	4	5	5	5	5	5	5	5
			Cotton	4	5	4	5	5	5	5	5	5
Perspiration	Acidic	Fade		4	5	5	4	5	5	5	5	5
		Stain	Silk	3–4	4	5	5	5	5	5	5	5
			Cotton	4	5	5	5	5	5	5	5	5
	Alkaline	Fade		4	5	3	4	5	4	5	5	5
		Stain	Silk	3	4	3	5	4	4	5	3	5
			Cotton	3	5	4	5	3	3	4	4	4

TABLE VII
Deodorizing Rate (%) of Various Fabrics

Time (min)	Fabric					
	Cotton	Silk	Nylon	Polyester	Acryl	Wool
30	26	47	7	11	11	78
60	27	51	7	12	12	80
90	28	55	8	13	13	83
120	28	61	11	14	14	86

to Rating 3, and from Rating 1 to Rating 4 by using mordants $MnSO_4$, $ZnSO_4$, and $NiSO_4$ for cotton, all mordants except $MnSO_4$ for silk, and $FeSO_4$, $CoSO_4$, $Al_2(SO_4)_3$, $CuSO_4$, and $MnSO_4$ for wool, respectively. However, the other mordants among the mordants used in this study did not significantly affect the lightfastness of premordanted cotton, silk, and wool fabrics.

From these results, it was concluded that mordants $MnSO_4$, $ZnSO_4$, and $NiSO_4$ for cotton, and all mordants except $MnSO_4$ for silk, mordants $CuSO_4$, $FeSO_4$, $CoSO_4$, $Al_2(SO_4)_3$, and $MnSO_4$ for wool were the best mordants to improve the light fastness.

Deodorizing performance

The rate of deodorization of various fabrics are given in Table VII. As can be seen, the deodorization performance increased in the order of Nylon < polyester = acryl < cotton < silk < wool. Table VIII shows the deodorization performance of cotton, silk, and wool fabrics pretreated with metallic salts. It was found that the deodorization performance was good with pretreatment with only metal salts except $MnSO_4$. The deodorizing mechanism of metallic salts employs chemical adsorption. Foul smelling molecules (ammonia) form coordinate bonds with transition metal ions, and thus are fixed, so that the foul odor is never rereleased. Table IX shows the deodorization rate of cotton, silk, and wool fabrics dyed with *Coffea arabica L.* extract solution and unmordanted metal salts. In the case of cotton, the deodorization performance is worse than that of the one pretreated with metal salts;

TABLE VIII
Deodorizing Performance (%) of Cotton, Silk, and Wool Fabrics Mordanted with Metallic Salts

Fabric	Mordant								
	None	Mn	Cu	Zn	Al	Fe	Ni	Co	Sn
Cotton	28	67	99	97	99	89	99	86	99
Silk	61	62	89	87	88	79	87	84	92
Wool	86	96	98	98	99	99	98	96	99

Time, 120 min.

TABLE IX
Deodorization Rate (%) of Cotton, Silk, and Wool Fabrics Dyed with *Coffea arabica L.* Extract

Time (min)	Fabric		
	Cotton	Silk	Wool
30	63	84	93
60	64	86	94
90	65	88	95
120	65	90	96

however, in the case of silk and wool fabrics, the deodorization performance is similar to that of fabrics treated with premordanted metal salts. Cotton, silk, and wool fabrics dyed with the *Coffea arabica L.* extract showed good deodorization performance.

Therefore, it was found that cotton, silk, and wool fabrics dyed with the *Coffea arabica L.* extract is not disadvantageous, when compared with pretreatment with metallic salts, with regard to the deodorization performance.

CONCLUSIONS

The purpose of this study is to investigate dyeing properties, color fastness (light, water, and perspiration fastness), and the deodorization performance of cotton, silk, and wool fabrics dyed with *Coffea arabica L.* extract solutions. The best mordants were found to be $FeSO_4$, $CuSO_4$, and $SnSO_4$ for improving the color strength (K/S) of cotton, silk, and wool fabrics. Mordants $MnSO_4$, $ZnSO_4$, and $NiSO_4$ for cotton (Rating 3), and all mordants except $MnSO_4$ for silk (Rating 3), mordants $CuSO_4$, $FeSO_4$, $CoSO_4$, $Al_2(SO_4)_3$, and $MnSO_4$ for wool (Rating 4) were the best mordants used to improve the light fastness. It was found that $FeSO_4$ and $CuSO_4$ were the best mordants for improving color strength (K/S) and light fastness for silk and wool fabrics. In addition, it was found that cotton, silk, and wool fabrics dyed with the *Coffea arabica L.* extract showed good deodorization performance.

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