



## 여러 가지 천연 염재를 이용한 면, 견, 모직물의 염색 및 소취 특성

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### Dyeing and Deodorizing Properties of Cotton, Silk, and Wool Fabrics Dyed with Various Natural Colorants

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**Abstract**— Four kinds of natural dyeing solutions (natural colorant extracts) were obtained by extraction from sappan wood, black tea, peony, and clove using water as extracting solvent at 90°C for 90 min with liquor ratio (solid natural colorant material/solvent water, weight ratio) of 1/10. The dyeing, colour fastness and deodorizing properties of fabrics (cotton, silk, and wool fabrics) dyed with natural colorant extracts were compared. It was found that these properties were significantly dependent on the concentration of extracts, the structure of colorant, and the kind of fabrics. The K/S value of dyed cotton fabric increased in the order of peony < sappan wood < clove < black tea, however, the values of dyed silk and wool fabrics were in the order of peony < sappan wood < black tea < clove. Colour fastness (light, water, and perspiration fastness) was in the range of 3 - 5 grade except for sappan wood. The deodorizing performance of fabrics dyed with various natural colorant extracts was in the range of 56 - 99%. The deodorizing performance increased in the order of peony < black tea < sappan wood < clove. Especially, the deodorizing performance of all fabrics dyed with clove was found to be the highest at 98-99%.

**Keywords:** natural colorants (Sappan wood, Black tea, Peony, Clove), natural colorant extraction, dyeing, fastness, deodorizing performance

## 1. Introduction

Since prehistoric times, natural dyes have been used for many purposes such as the coloring of natural fibers like wool, cotton and silk as well as fur and leather. The natural dyes have been also used for color cosmetics, watercolour painting and inks. The use of natural dyes to color textiles declined rapidly after the discovery of synthetic dyes in 1856<sup>1-2</sup>.

Recently the world has become increasingly aware

of environmental issue. Synthetic dyestuffs in particular have come under severe criticism on the grounds of being highly polluting in their manufacturing and application. A search for safer alternatives has created a widespread renewal of interest in natural dyes<sup>3</sup>.

This is a result of the stringent environmental standards imposed by many countries in response to the toxic and allergic reactions associated with synthetic dyes. Natural dyes are more friend to the environment than synthetic dyes

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and can exhibit better biodegradability. They have low toxicity and allergic reactions and also show beautiful and multi-hued shades<sup>4)</sup>.

Problems in dyeing using natural dyes are the low exhaustion of the dyes and bad 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 fastness properties or exhaustion and to develop different shades with same dye<sup>5)</sup>.

Metal ions can act as electron acceptors to electron donor to form co-ordinate bonds with the dye molecules, which are insoluble in water<sup>6)</sup>. There were many studies on the dyeing and fastness properties of various fabrics using various natural dyes with various mordants<sup>7-13)</sup>.

We also investigated the dyeing and fastness properties of cotton and silk fabrics dyed with cherry extract and *Cassia tora* L. extracts using various mordants<sup>14-16)</sup>.

However, the synthetic mordants are also pollutive components. Researches on dyeing property using natural colorants without mordants are not available. Especially studies on the functions such as deodorization performance, antimicrobial ability, and the emission of far-infrared radiation using natural colorants can be hardly found.

In recent years, concern for health and hygiene is constantly rising. People are extremely sensitive to smells, and deodorizing is becoming an entrenched social need. There are smells which almost everyone finds unpleasant. Deodorizing

performance aims to protect the health and living environment of citizens by regulating the discharge of foul smelling substances which are generated by factories and other commercial activity<sup>17)</sup>. Therefore, we investigated the improvement of deodorization performance of various fabrics (cotton, silk, and wool) dyed using various natural colorants without mordants.

In this study, the influences of natural colorant extracts (sappan wood, black tea, peony, and clove) on dyeing, fastness (light, water, and perspiration fastness), and deodorizing properties of fabrics (cotton, silk, and wool) were investigated.

## 2. Experimental

### 2.1 Materials

Cotton, silk and wool fabrics (Standard Adjacent Fabrics for Staining of Fastness Test: KS K 0905) were used. The characteristics of these fabrics are shown in Table 1.

Mordants [Aluminium sulfate hydrate ( $\text{Al}_2(\text{SO}_4)_3 \cdot 13 \sim 14\text{H}_2\text{O}$ , MW: 585, Junsei Chemical Co. Ltd), Manganese (II) sulfate hydrate ( $\text{MnSO}_4 \cdot 5\text{H}_2\text{O}$ , MW: 241, Junsei Chemical Co. Ltd), Zinc sulfate hydrate ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ , MW: 287.56, Junsei Chemical Co. Ltd), Nickel sulfate hydrate ( $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ , MW: 262.87, Junsei Chemical Co. Ltd), Copper sulfate hydrate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , MW: 249.68, Junsei Chemical Co. Ltd), Iron(II) sulfate hydrate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , MW: 278.03, Junsei Chemical Co. Ltd), Stannous sulphate ( $\text{SnSO}_4$ , 95%, MW: 214.75, Hayashi Pure Chemical Industries Ltd), and

**Table 1.** Characteristics of control fabrics

Fabric	Thickne ss <sup>a</sup> (mm)	Yarn number (Tex)		Fabric counts (thread/cm)		Weight (g/m <sup>2</sup> )	K/S	L*	a*	b*	WI	YI	BI
		Warp	Weft	Warp	Weft								
Cotton	0.27	14	16.5	31	35	115	0.08	88.97	-0.25	1.10	68.51	1.97	73.08
Silk	0.10	2.3/2	2.3	38	55	26	0.11	86.87	-0.21	1.03	64.34	1.93	68.88
Wool	0.33	15.6/2	15.6/2	18	21	125	0.27	86.38	-1.23	5.00	43.65	8.69	64.76

Weave: plain

<sup>a</sup>Thickness: under 1kpa pressure

WI: Whiteness Index, 10deg./D65/Ganz

YI: Yellowness Index, 2deg./C/ASTM D1925

BI: Brightness Index, 2deg./C/TAPP1452/ISO2470

Cobalt (II) sulfate hydrate ( $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ , MW: 281.10, Shinyo Pure Chemicals Co. Ltd)] were used without further purification.

Sappan wood, black tea, peony, and clove were used as natural colorants.

## 2.2 Extraction

Dyeing solutions were obtained by extraction from sappan wood, black tea, peony, and clove using water as extracting solvent at 90°C for 90 min. at a fixed liquor ratio (solid natural colorant /water weight ratio) of 1:10.

## 2.3 Mordanting

To compare the deodorizing properties of mordanted fabrics and dyed fabrics with natural colorant extracts, fabrics were pre-mordanted at 40°C for 60 min using 3% o.w.f. mordant solutions (except aluminium sulfate hydrate 10% o.w.f) with bath ratio 1:50.

## 2.4 Dyeing

Dyeing was carried out using a 1:100 bath ratio at 80°C for 60 min by exhaustion method.

## 2.5 IR spectrometry

To confirm the structure of natural colorants, FTIR spectrometer (Impact 400D, Nicolet, Madison, WI) was used to measure the infrared spectra of sappan wood, black tea, peony, and clove extract solution in the wavenumber of 400-4000  $\text{cm}^{-1}$  at room temperature. For each IR spectrometer samples 32 scans at 4  $\text{cm}^{-1}$  resolution was collected in the transmittance mode.

## 2.6 Colour measurement

The reflectance values and the corresponding CIE  $L^*$ ,  $a^*$ ,  $b^*$ ,  $C^*$ ,  $HV/C$ , and colour strength (K/S) values for the dyed samples were measured using a CCM (Gretag Macbeth Color-Eye 7000A, USA) interfaced to a digital PC under illuminant  $D_{65}$ , with a  $10^\circ$  standard observer. 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 coloured 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 colour strength.

## 2.7 Colour fastness test

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.

## 2.8 Deodorizing test

Gas detecting tube method was used to measure ammonia gas concentration. The change of concentration of ammonia gas in the tube with fabrics over the elapse of time was measured at 25°C. The concentration of ammonia gas in blank (reference) tube was about 500ppm. The deodorizing performance was calculated as follows:

$$\text{Deodorization performance (\%)} = (C_b - C_s) / C_b \times 100$$

Where,  $C_b$  is the gas concentration (ppm) of test tube without fabric (blank state), and  $C_s$  is the concentration of tube with fabrics.

## 3. Results and Discussion

### 3.1 Confirmation of sappan wood, black tea, peony, and clove extracts

Fig. 1 show the IR spectra of sappan wood (a), black tea (b), peony (c), and clove (d) extracts. Main components in these extracts were identified as follows:

Sappan wood extract (a) have the characteristic peaks corresponding to the OH stretch at 3354 $\text{cm}^{-1}$ , ring stretch (benzene ring in aromatic compounds) at 1613 $\text{cm}^{-1}$  and 1507 $\text{cm}^{-1}$ , C-O-C stretch in cyclic ethers at 1248 $\text{cm}^{-1}$ , C-OH stretch (secondary alcohol) at 1113 $\text{cm}^{-1}$  were observed indicating the presence of brazilin component in the sappan wood extract.

Black tea extract (b) shows the characteristic

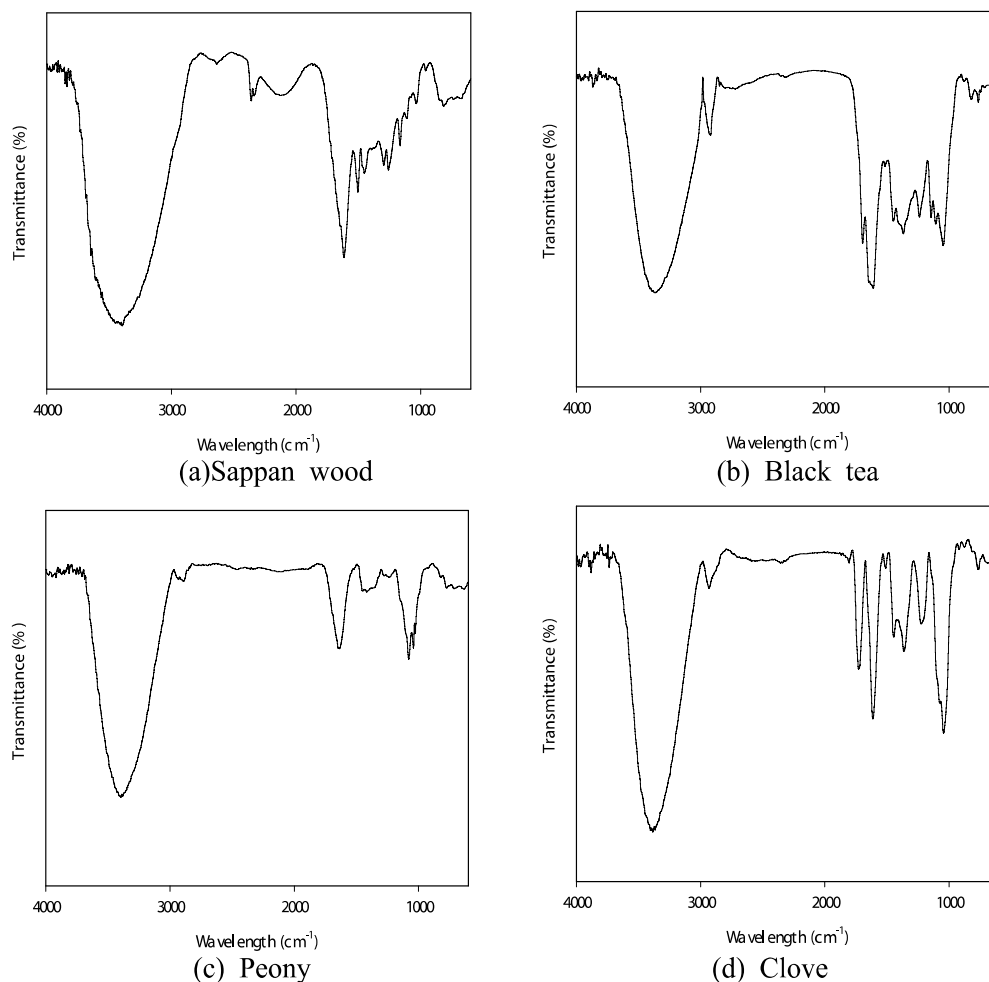


Fig. 1. Infrared spectra of extracted natural colorants : (a) Sappan wood, (b) Black tea, (c) Peony, and (d) Clove extracts.

peaks corresponding to the OH stretch at near  $3372\text{cm}^{-1}$ , CH anti-symmetric and symmetric stretch at  $2926\text{cm}^{-1}$ , C=O stretch at  $1697\text{cm}^{-1}$ , ring stretch (benzene ring in aromatic compounds) at  $1618\text{cm}^{-1}$ , at  $\text{CH}_3$  antisymmetric deformation at  $1451\text{cm}^{-1}$ , O-H deformation and C-O stretch in phenol at  $1372\text{cm}^{-1}$ , C-O-C stretch in cyclic ethers at  $1234\text{cm}^{-1}$  were observed indicating the presence of catechin component in black tea.

Peony extract (c) shows characteristic peaks corresponding to the phenol at  $3200\text{cm}^{-1}$  and  $1238\text{cm}^{-1}$ , CH stretch in phenyl at  $2903\text{cm}^{-1}$ , ring stretch (benzene ring in aromatic compounds) at  $1636\text{cm}^{-1}$ , the C=O stretching at  $1419$ ,  $1086$ , and  $1040\text{cm}^{-1}$  were observed indicating the presence of paeniflorin / paenol in peony extract.

Clove extract (d) have the characteristic peaks corresponding to the =CH stretching in vinyl at

$2931\text{cm}^{-1}$ , overtone of  $\text{CH}_2$  out-of-plane wagging in vinyl and C=O stretch at  $1724\text{cm}^{-1}$ , ring stretch (benzenizing in aromatic compounds) at  $1608\text{cm}^{-1}$ ,  $\text{CH}_3$  antisymmetric deformation at  $1447\text{cm}^{-1}$  and  $1359\text{cm}^{-1}$ , phenol at  $1220\text{cm}^{-1}$ , C-O stretch at  $1044\text{cm}^{-1}$  were observed indicating the presence of eugenol in clove extract.

### 3.2 Concentrations of extracted natural colorants

From the results of our previous study, the optimum liquor ratio was found to be 1:10<sup>18)</sup>.

Therefore, all natural colorants were extracted at a liquor ratio of 1:10. The concentrations of sappan wood, black tea, peony, and clove extracts were found to be 0.19, 3.45, 1.71, and 0.92g/100ml, respectively. The concentration of extract increased in the order of sappan wood <

clove < peony < black tea. The effect of the concentration on the dyeing properties was considered.

### 3.3 Dyeing properties

In this study the dyeing time and temperature were fixed at 60 min and 80°C, respectively. It was because the preliminary experiment found that the optimum dyeing time and temperature at a fixed liquor ratio (1:10) were 60 min and 80°C respectively, to give the highest K/S value for all fabrics.

The colorimetric results (K/S, L\*, a\*, b\*, and HV/C) of the fabrics (cotton, silk, and wool) dyed with sappan wood, black tea, peony, clove extracts are given in Table 2. Generally, the dyeing affinity for fibres is dependent on the content of functional polar groups in fibres. It is well known that the number of functional group in wool is larger than that of silk<sup>19)</sup>, and polarity of protein fibres is higher than that of cellulose fibre. The K/S value was in the order of cotton < silk < wool fabric for all natural colorants. It was found that this order of dyeing affinity matched the order of polarity/functional group content of fabrics very well.

The K/S value of dyed cotton fabric increased in the order of peony < sappan wood < clove < black tea, however, the values of dyed silk and wool fabrics were in the order of peony < sappan wood < black tea < clove.

This result might be related to the concentration and structural feature of these colorants.

The reason why this trend occurred is not clear at the present moment. More detailed studies should be done. From the colorimetric results (L\*, a\*, b\*, and HV), it was found that we could get diverse natural colour tone fabrics dyed with various natural colour materials.

### 3.4 Colour fastness

Colour fastness (light, water, and perspiration fastness) of fabrics dyed with sappan wood, black tea, peony, and clove extracts are shown in Table 3. Colour fastness (light, water, and perspiration fastness) was in the range of 3 - 5 rating with the exception of fabrics dyed with sappan wood. Generally, it is well known that the light fastness of natural dyes was poor. The light fastness of all dyed fabrics was found to be in the range of 3-4 rating with the exception of fabrics dyed with sappan wood.

**Table 2.** Colorimetric data of fabrics dyed with sappan wood, black tea, peony, and clove extracts

Fabric	Natural colorant	K/S	L*	a*	b*	C*	H	V/C
Cotton	Control	0.08	88.97	-0.25	1.10			
	Sappan wood	0.78	93.18	2.52	5.78	6.31	7.7R	9.54/4.90
	Black tea	1.43	95.51	0.80	3.93	4.01	17RP	9.70/3.40
	Peony	0.35	94.48	0.38	1.80	1.84	8.1RP	9.62/0
	Clove	1.32	96.11	0.14	3.13	3.14	13.1RP	9.74/14.7
Silk	Control	0.11	86.87	-0.21	1.03			
	Sappan wood	3.34	89.71	4.65	16.43	17.08	16.3R	9.63/4.2
	Black tea	7.02	91.91	2.09	8.68	8.93	4.3R	9.71/6.3
	Peony	1.06	89.19	1.52	4.59	4.83	12RP	9.6/13.5
	Clove	7.54	94.05	0.15	6.62	6.63	4.5R	9.79/6.0
Wool	Control	0.27	86.38	-1.23	5.00			
	Sappan wood	9.35	84.91	10.20	21.74	24.01	12.2R	9.63/4.8
	Black tea	11.41	89.08	2.79	12.27	12.58	4.4R	9.74/6.2
	Peony	2.90	89.26	0.98	7.40	7.47	13.1RP	9.74/14.6
	Clove	18.70	91.82	0.28	11.59	11.59	7.9RP	9.58/0

**Table 3.** Colour fastness of (a) cotton, (b) silk, and (c) wool fabrics dyed with sappan wood, black tea, peony, and clove extracts

(a) cotton							
Natural colorant			Sappan wood	Black tea	Peony	Clove	
Light			2	3	4	4	
Water	Fade		2	3	5	5	
	Stain	Silk	4-5	4-5	4	4-5	
		Cotton	4-5	4	4	4-5	
Perspiration	Acidic	Fade		4	3	5	
		Stain	Silk	4-5	4-5	4-5	
			Cotton	4	4-5	4-5	
	Alkaline	Fade		2	3	4	5
		Stain	Silk	4	4-5	4	4-5
			Cotton	4-5	4-5	4-5	4-5

(b) silk							
Natural colorant			Sappan wood	Black tea	Peony	Clove	
Light			2	4	4	4	
Water	Fade		1	5	5	5	
	Stain	Silk	3-4	4-5	4	4-5	
		Cotton	4	4-5	4	4-5	
Perspiration	Acidic	Fade		2	5	4	5
		Stain	Silk	4	4-5	4	4-5
			Cotton	3-4	4-5	4-5	4-5
	Alkaline	Fade		1	3	3	4
		Stain	Silk	4-5	4	4	4-5
			Cotton	4	4-5	4-5	4-5

(c) wool							
Natural colorant			Sappan wood	Black tea	Peony	Clove	
Light			2	3	3	4	
Water	Fade		2	5	4	5	
	Stain	Silk	4	4-5	4	4	
		Cotton	4-5	4-5	4-5	4-5	
Perspiration	Acidic	Fade		2	5	4	3
		Stain	Silk	4-5	4	4	4-5
			Cotton	4	4-5	4-5	4-5
	Alkaline	Fade		1	5	5	5
		Stain	Silk	4-5	3-4	4	4-5
			Cotton	4-5	4-5	4-5	4-5

Considering natural dyes generally have low light fastness, fastness of dyed fabrics obtained in this study are considerably good.

### 3.5 Deodorizing performance

The deodorizing performance with the lapse of time for control cotton, silk, and wool fabrics are given in Fig. 2. The deodorizing performance increased with increasing elapsed time up to 5 min and then levelled off. The deodorizing performance of control fabrics was in the order of cotton (length x width x thickness: 10cm x 10cm x 0.27mm, 1.2g) < silk (10cm x 10cm x 0.10mm, 0.3g) < wool (10cm x 10cm x 0.33mm, 1.3g).

This result indicated that the deodorization performance of protein fibres was higher than that of cotton fibre. However, we could not compare the deodorizing performance of silk and wool directly, because the volume and weight of these fabrics were not same in spite of having same surface area (10cm x 10cm).

Table 4 shows the deodorizing performance of cotton, silk, and wool fabrics mordanted with various metallic sulfate salts.

The deodorization performance of mordanted fabrics using mordants ( $\text{CuSO}_4$ ,  $\text{ZnSO}_4$ ,  $\text{Al}_2(\text{SO}_4)_3$ ,  $\text{FeSO}_4$ ,  $\text{NiSO}_4$ ,  $\text{CoSO}_4$ ,  $\text{MnSO}_4$  and  $\text{SnSO}_4$ ) was in the range of 62-99%.

Generally, it was well known that mordanting

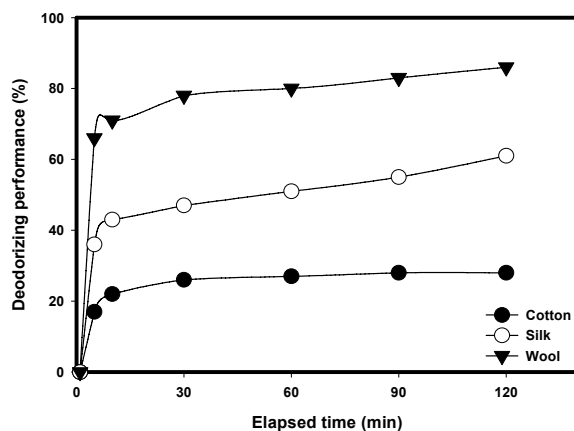


Fig. 2. Effect of elapsed time on the deodorizing performance of fabrics (cotton, silk, and wool) dyed with coffee sludge.

improved the deodorization performance, since the mordant metallic salts could absorb smelling molecules through the formation of coordination bonds between transition metal ions and unshared electron pairs of smelling molecules<sup>7</sup>.

The deodorizing performance of cotton, silk, and wool fabrics dyed with various natural colorant extracts without treatment of mordants are given Fig. 3.

The deodorizing performance of dyed fabrics was in the range of 50-99%. The deodorizing performance increased in the order of peony < black tea < sappan wood < clove. Especially, the deodorizing performance of all fabrics dyed with clove was found to be the highest at 98-99%. The highest value of clove might be related to the action of the main component eugenol of clove.

It is well known that eugenol is used in perfumeries, flavorings, essential oils and in medicine as a local antiseptic and anesthetic. However, the exact cause of the highest deodorizing performance of clove is not clear.

The deodorization performance of dyed fabrics and mordanted fabrics are mostly similar except for few samples. These results clearly demonstrate that utilizing extracted natural colorants as dyeing materials significantly facilitate to obtain prominent deodorizing fabrics.

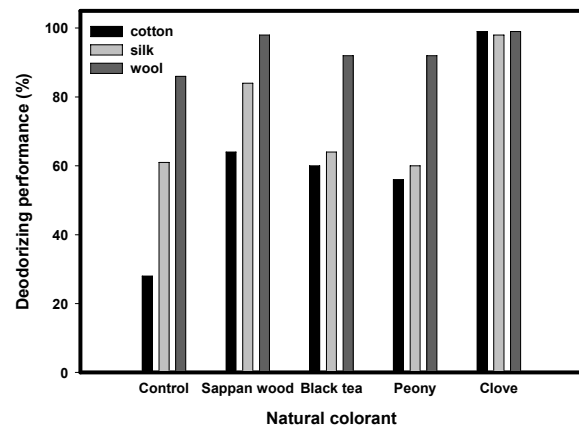


Fig. 3. Comparison of deodorizing performance of undyed fabrics [(cotton, silk, and wool): control] and dyed fabrics using sappan wood, black tea, peony, and clove.

**Table 4.** Deodorizing performance (%) of fabrics mordanted with various metallic sulfate salts

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

Time: 120 min

#### 4. Conclusions

Natural colorants were extracted from sappan wood, black tea, peony, and clove using water as extracting solvent. The optimum extraction temperature, time and liquor ratio were found to be 90°C, 90min and liquor ratio (solid natural colorant material/water, weight ratio) of 1/10, respectively. The effect of natural colorant solution on dyeing, fastness, and deodorizing properties of fabrics (cotton, silk, and wool fabrics) were investigated. For all colorant solutions, the K/S value was in the order of cotton < silk < wool. The K/S value of dyed cotton fabric increased in the order of peony < sappan wood < clove < black tea, however, the values of dyed silk and wool fabrics were in the order of peony < sappan wood < black tea < clove.

Colour fastness (light, water, and perspiration fastness) was in the range of 3 - 5 rating except for sappan wood. The deodorizing performance of fabrics dyed with various natural colorants extracts was in the range of 50-99%.

The deodorizing performance increased in the order of peony < black tea < sappan wood < clove. Especially, the deodorizing performance of all fabrics dyed with clove was found to be the highest at 98-99%. Despite most natural dyes have low fastness, diverse natural colour tone fabrics having high fastness and prominent deodorizing property using various natural colorants were prepared in this study.

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