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Dyeing Properties and Colour Fastness of Cotton and Silk Fabrics Dyed with *Cassia tora L.* Extract

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Abstract: A natural colorant was extracted from *Cassia tora L.* using buffer solutions (pH: 2-11) as extractants. The dyeing solution (*Cassia tora L.* extract) extracted using pH 9 buffer solution was found to give the highest K/S values of dyed fabrics. Cotton and silk fabrics were dyed with *Cassia tora L.* extract at 60 °C for 60 min with pre-treatment of various metal salts as mordants. It was found that *Cassia tora L.* extract was polygenetic dyestuffs and its major components were anthraquinones. Studies have been made on the effects of the kind of mordant on dyeing properties and colour fastnesses of cotton and silk fabrics. The K/S of cotton fabrics increased in the order of the dyeing using $FeSO_4 > CuSO_4 > ZnSO_4 > MnSO_4 \cong Al_2(SO_4)_3 > NiSO_4 >$ none, however, the K/S of silk fabrics increased in the order of the dyeing using $FeSO_4 > CuSO_4 > ZnSO_4 \cong Al_2(SO_4)_3 > MnSO_4 \cong NiSO_4 >$ none. It was found that the K/S values of dyed fabrics were largely affected by the colour difference (ΔE) between mordanted fabric and control fabric. However, they were not depended on the content of mordanted metal ion of the fabrics. Mordants $FeSO_4$ and $CuSO_4$ for cotton fabric, $FeSO_4$, $CuSO_4$, and $Al_2(SO_4)_3$ for silk fabric were found to give good light fastness (rating 4).

Keywords: Natural dye, *Cassia tora L.*, Mordant, Dyeing, Fastness

Introduction

The colouring 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 coloured with natural dyes may be one or a combination of factors, including a preference for naturalness, environmental friendliness, lower toxicity, antibacterial/anti-allergic/deodorizing/anti-cancer properties, harmonising 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 America 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 non-toxic aluminium formulation and biodegradable auxiliary substances. In Germany, Livos Pflanzenchemie Forschungs- und Entwicklungs GmbH 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 possible use of natural dyes in textile dyeing processes have been performed by various research groups. In general, in order 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 co-ordinate 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 nad Desa [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, 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 using various mordants [15]. However, research in the natural dyes obtained from *Cassia tora L.* can be hardly found

The *Cassia tora L.* is described under the class of Leguminosae, its common name is Senna or Chinese, and it is annual semi-shrub. The major chemical constituents of *Cassia tora L.*

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seeds are emodin(4,5,7-trihydroxy-2-methyl anthraquinone), rhein (1,8-dihydroxy anthraquinone-3-carboxylic acid), and chrysophanol (1.8-dihydroxy-3-methyl anthraquinone) [16].

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 improved fastness properties or exhaustion and develop different shades with same dye [17-20].

In this study, the effects of the kind of various mordants ($FeSO_4$, $CuSO_4$, $NiSO_4$, $ZnSO_4$, $Al_2(SO_4)_3$, and $MnSO_4$) on dyeing property and colour fastness (light, water, washing, and perspiration fastness) of cotton and silk fabrics dyed with *Cassia tora L.* extract were investigated. The K/S values and the apparent colour, and the colour fastness of pre-mordanted and un-mordanted cotton and silk fabrics were compared.

Experimental

Materials

Cotton and silk fabrics (Standard Adjacent Fabrics for Staining of Fastness: KS K 0905) were used without further purification. Aluminium sulfate hydrate ($Al_2(SO_4)_3 \cdot 13\text{-}14H_2O$, MW: 585, Junsei Chemical Co. Ltd.), Manganese (II) sulfate hydrate ($MnSO_4 \cdot 5H_2O$, MW: 241, Junsei Chemical Co. Ltd.), Zinc sulfate hydrate ($ZnSO_4 \cdot 7H_2O$, MW: 287.56, Junsei Chemical Co. Ltd.), Nickel sulfate hydrate ($NiSO_4 \cdot 6H_2O$, MW: 262.87, Junsei Chemical Co. Ltd.), Copper sulfate hydrate ($CuSO_4 \cdot 5H_2O$, MW: 249.68, Junsei Chemical Co. Ltd.), and Iron (II) sulfate hydrate ($FeSO_4 \cdot 7H_2O$, MW: 278.03, Junsei Chemical Co. Ltd.) were used without further purification.

Extracting/Mordanting/Dyeing

Extracting: The *Cassia tora L.* seed was washed and dried. And then the *Cassia tora L.* seed was extracted using various pH buffer solutions (pH 2-11) for 3 days at room temperature in material-liquor ratio 1:10. The *Cassia tora L.* extract solution (natural dye solution) obtained in this study was a yellowish red solution.

Mordanting: Cotton and silk fabrics were pre-mordanted at 40 °C for 60 min using various aqueous mordant solutions (0.01 M) with liquor ratio 1:100.

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

Characterization

A FT-IR (computerized Nicolet Impact 400D Fourier Transform Infrared spectrometer, USA) was used to identify the structure of *Cassia tora L* extract. For each samples 32 scans at 4 cm^{-1} resolution were collected in the transmittance mode. The content of mordanted metal ion of pre-mordanted fabrics was determined using a Induced Coupled Plasma Atomic Emission Spectrometer (Optima 4300 DV, Perkin Elmer, USA). The reflectance values and the corresponding CIE L*, a*, b*, H V/C, and K/S values for the dyed samples were measured using a CCM (Gretag Macbath Color-Eye 7000A, USA) interfaced to a digital PC under illuminant D_{65}, with a 10^0 standard observer. Colour 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 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. Colour difference (ΔE) based on CIE L*, a*, b* determined using Hunterlab Colour Quest II. The light fastness, the wash fastness, the water fastness, and the perspiration fastness were determined according to KS K 0700, KS K 0430 B method, KS K 0645, and KS K 0715, respectively. Fade and stain in colour were assessed using grey scales.

Results and Discussion

Identification of *Cassia tora L.* Extract

Figure 1 shows the IR spectrum of *Cassia tora L.* extract solution (solution extracted with pH 9 buffer solution). Generally, it is known that the extract of *Cassia tora L.* seed contains emodin (4,5,7-trihydroxy-2-methyl anthraquinone), rhein (1,8-dihydroxy anthraquinone-3-carboxylic acid), and chrysophanol (1.8-dihydroxy-3-methyl anthraquinone) [16].

The characteristic peaks corresponding to the C=O stretching vibration overtone at 3418 cm^{-1}, the C=O stretching of quinone at 1640 cm^{-1}, the OH group in carboxylic acids (in-plane OH bending) at 1410 cm^{-1}, CH-O-H in cyclic alcohols (C-O stretch) at 2927 cm^{-1}, and CH_3 (CH stretch in C-CH_3 compounds) at 1350 cm^{-1} were observed indicating the presence of anthraquinones.

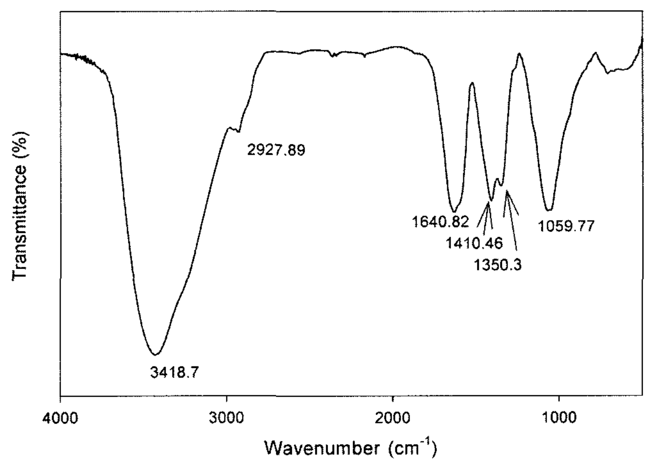

Figure 1. Infrared spectrum of *Cassia tora L.* extract.

Generally, in the absence of hydroxyl and amino-groups, anthraquinone absorbs strongly in the region 1680-1650 cm^{-1} due to the stretching vibration of carbonyl group. However, the presence of hydroxyl and amino-groups results in the lowering of this frequency [21]. The stretching vibration peak of quinone C=O group of *Cassia tora L.* extract component obtained in this study was appeared at 1640 cm^{-1}. From the frequency shift of C=O stretching vibration peak, it was concluded that the anthraquinone of *Cassia tora L.* extract contained the hydroxyl- and amino- groups.

Extracting and Mordanting

The influence of the pH of extractant (buffer solution) on the colorimetric data (L*, a*, b*, and H V/C) and K/S value for the cotton and silk fabrics dyed with *Cassia tora L.* extract is shown in Table 1. The K/S value of dyed cotton fabrics was mostly increased with increasing the pH of buffer solution up to about 9, and thereafter it levelled off. The K/S value of dyed silk fabrics was not depended on the pH of buffer solution. However, the pH 9 gave the highest K/S value for cotton and silk fabrics. The reason of the different effect of pH on K/S for cotton and silk fabrics is not clear at the present moment. More detailed studies should be made. From these results, it was suggested that pH 9 buffer solution gave the highest natural dye concentration of *Cassia tora L.* extract.

The colorimetric data (L*, a*, b*, and HV/C), colour difference (ΔE), and the content of mordanted metal ion of pre-mordanted cotton and silk fabrics are shown in Table 2. The contents of mordanted metal ion of silk fabrics were higher values than those of cotton fabrics. This may be due to the easier formation of complex between metal ions and ligands of silk compared to cotton. The ΔE value was not directly related with the content of mordanted metal ion, but

Table 1. The effect of extracting pH on the colorimetric data of the cotton and silk fabrics dyed with *Cassia tora L.* extract

Fabric	pH	L*	a*	b*	H	V/C	K/S
Cotton	2	74.86	−0.19	15.60	2.81Y	7.33/2.17	0.8
	3	74.11	−1.17	16.46	4.01Y	7.25/2,24	0.9
	4	62.15	11.70	14.29	1.81YR	6.03/3.56	1.8
	7	69.22	6.03	15.82	7.52YR	6.75/2.87	1.2
	8	60.74	11.64	13.65	1.56YR	5.89/3.46	1.8
	9	59.67	12.84	16.81	2.38YR	5.78/4.01	2.3
	10	58.82	14.83	16.03	0.24YR	5.70/4.27	2.3
	11	58.64	13.87	14.40	10.00YR	5.68/3.92	2.1
Silk	2	70.14	−1.82	36.80	4.66Y	6.85/5.23	4.2
	3	70.97	−2.51	44.77	4.94Y	6.93/6.34	5.5
	4	52.86	16.68	26.54	4.36YR	5.11/5.60	6.6
	7	64.25	6.55	28.89	0.02Y	6.24/4.71	3.8
	8	57.83	12.84	26.77	6.25YR	5.60/5.13	4.6
	9	48.38	22.43	24.98	1.23YR	4.67/6.25	6.9
	10	56.83	14.36	23.57	4.58YR	5.50/4.97	4.3
	11	54.35	15.81	25.97	4.57YR	5.25/5.42	5.3

Table 2. The colorimetric data and mordanted contents in mordanted fabrics

Fabric	Mordant type	L*	a*	b*	ΔE	Mordanted content (ppm)
Cotton	None	80.45	−0.20	−0.44	-	-
	$Al_2(SO_4)_3$	80.13	−0.10	−0.75	0.46	346
	$MnSO_4$	80.37	−0.23	−0.32	0.15	649
	$ZnSO_4$	80.60	−0.07	−0.92	0.52	941
	$NiSO_4$	80.53	−0.04	−1.07	0.65	738
	$CuSO_4$	79.28	−0.85	−0.77	1.37	878
	$FeSO_4$	77.25	0.05	9.22	10.18	559
Silk	None	80.73	−0.22	0.65	-	-
	$Al_2(SO_4)_3$	78.89	−0.23	0.10	1.87	1110
	$MnSO_4$	79.52	−0.32	1.29	1.37	1650
	$ZnSO_4$	78.55	−0.21	0.73	2.18	2260
	$NiSO_4$	79.76	−0.60	1.37	1.27	2140
	$CuSO_4$	76.42	−2.77	1.53	5.23	2010
	$FeSO_4$	71.26	1.33	9.02	12.73	1950

Table 3. The effect of mordant type on the colorimetric data of cotton and silk fabrics dyed with *Cassia tora L.* extract

Fabric	Mordant type	L*	a*	b*	H	V/C	K/S
Cotton	None	75.73	6.42	11.68	4.77YR	7.42/2.48	0.5
	$Al_2(SO_4)_3$	70.90	13.26	8.24	6.35R	6.92/3.61	0.7
	$MnSO_4$	72.29	9.28	9.00	9.57R	7.07/2.76	0.7
	$ZnSO_4$	66.04	8.66	1.99	9.89RP	6.43/2.37	0.8
	$NiSO_4$	72.70	8.00	8.89	0.68YR	7.11/2.50	0.6
	$CuSO_4$	56.49	12.21	0.81	7.38RP	5.46/3.08	1.5
	$FeSO_4$	55.37	2.33	−0.59	2.99RP	5.35/0.63	1.6
Silk	None	62.63	6.77	18.48	8.06RY	6.08/3.30	2.5
	$Al_2(SO_4)_3$	57.21	16.69	16.18	9.42R	5.54/4.60	2.7
	$MnSO_4$	60.02	8.59	14.82	5.01YR	5.82/3.09	2.6
	$ZnSO_4$	55.62	8.64	9.90	1.75YR	5.38/2.52	2.7
	$NiSO_4$	59.62	7.31	14.60	6.09YR	5.78/2.86	2.6
	$CuSO_4$	54.77	9.72	12.94	2.88YR	5.29/3.02	3.2
	$FeSO_4$	39.67	4.32	3.96	1.45YR	3.82/1.02	5.6

it depended on the type of mordants. The ΔE value of the cotton fabrics increased in the order of $FeSO_4 > CuSO_4 > NiSO_4 > ZnSO_4 > Al_2(SO_4)_3 > MnSO_4$, however, ΔE of the silk fabrics increased in the order of $FeSO_4 > CuSO_4 > ZnSO_4 > Al_2(SO_4)_3 > MnSO_4 > NiSO_4$.

Dyeing Properties

From the preliminary experiment, it was found that the optimum dyeing time and temperature were 60 min and 60 °C, respectively, to gave the highest K/S values for cotton and silk fabrics. Therefore, the pre-mordanted cotton and silk fabrics were dyed with *Cassia tora L.* extract solution at 60 °C for 60 min. The effects of mordant type on the colorimetric data (L*, a*, b*, and HV/C) and K/S are shown in Table 3. It was found that the K/S values of pre-mordanted cotton and silk fabrics were higher than those of un-mordanted fabrics. Generally, it is known that the K/S values of cotton fabrics dyed with natural dyes are very low. However, the pre-mordanted and un-mordanted cotton fabrics dyed with *Cassia tora L.* extract obtained in this study have high K/S values. This result might be attributed to the higher affinity of anthraquinone dye with cellulose. The K/S values of silk fabrics were higher than that of cotton fabrics. This might be due to the higher mordanted metal ion content (see Table 2).

The K/S value of the cotton fabrics increased in the order of dyeing using $FeSO_4 > CuSO_4 > ZnSO_4 > MnSO_4 \cong Al_2(SO_4)_3 > NiSO_4 >$ none, however, the K/S of the silk fabrics increased in the order of dyeing using $FeSO_4 > CuSO_4 > ZnSO_4 \cong Al_2(SO_4)_3 > MnSO_4 \cong NiSO_4 >$ none. Therefore, the K/S value seemed to depend primarily on the ΔE rather than the content of mordanted metal ion in fabrics (see Table 2). The best mordants were found to be $FeSO_4$ and $CuSO_4$ for improving the colour strength (K/S) of cotton and silk fabrics.

Figure 2 shows the apparent colour change of un-mordanted cotton and silk fabrics dyed with *Cassia tora L.* extract solutions (the dye solutions extracted by the different pH of buffer solution: 2-11). It was found that the colours of un-mordanted dyed fabrics were changed from yellow to yellowish red.

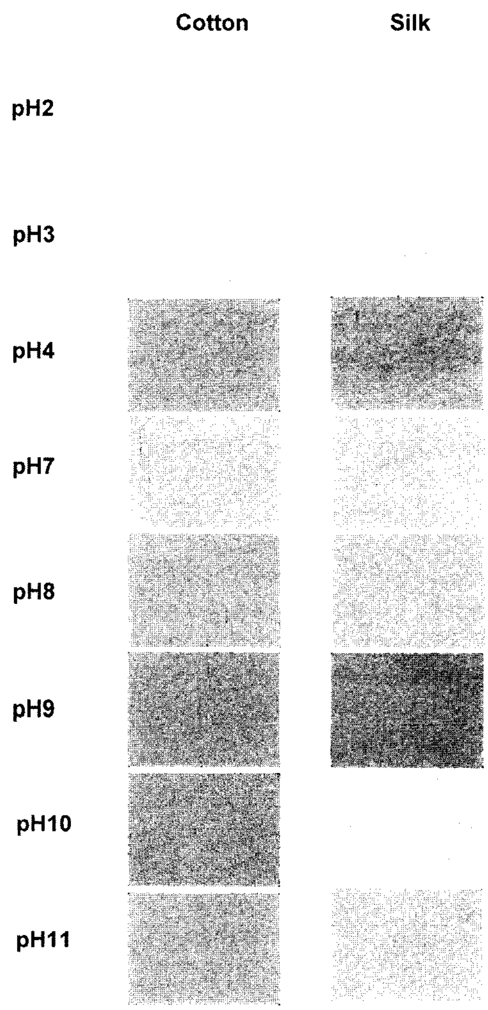

Figure 2. The effect of extracting pH on apparent colour.

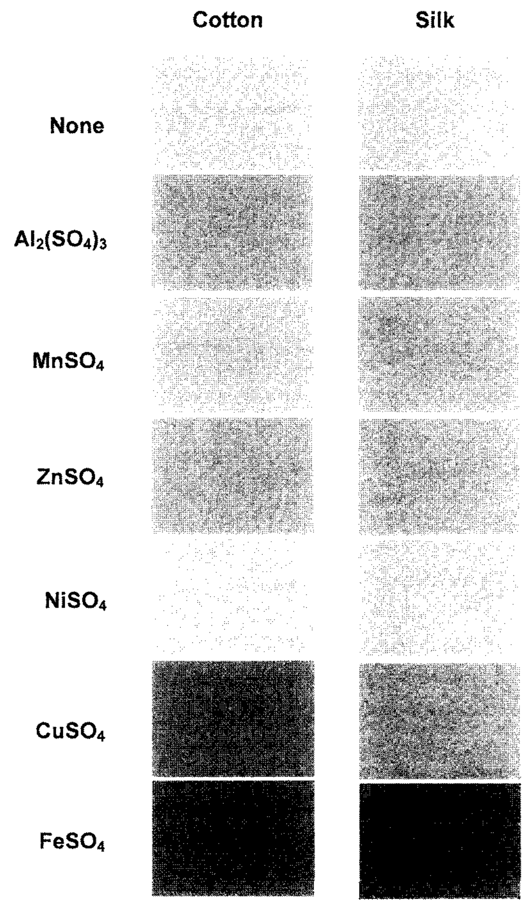

Figure 3. The effect of mordant type on apparent colour.

The apparent colour of un-mordanted and pre-mordanted cotton and silk fabrics dyed with *Cassia tora L.* extract solutions (the dye solutions extracted by the buffer solution of pH 9) are shown in Figure 3. The apparent colours of cotton fabrics pre-mordanted using $Al_2(SO_4)_3$, $MnSO_4$, $ZnSO_4$, $NiSO_4$, $CuSO_4$, $FeSO_4$, and none, were 6.35R, 9.57R, 9.89R, 0.68RP, 7.38RP, 2.99RP, and 4.77YR, respectively. And the apparent colours of silk fabrics pre-mordanted using $Al_2(SO_4)_3$, $MnSO_4$, $ZnSO_4$, $NiSO_4$, $CuSO_4$, $FeSO_4$, and none, were 9.42R, 5.01YR, 1.75YR, 6.09YR, 2.88YR, 1.45YR, and 8.06YR, respectively. It was found that the colour of pre-mordanted fabrics was deeper than the colour of un-mordanted fabrics. With varying the kind of mordant, the colour of dyed cotton fabrics was changed from yellow-red to red-purple, however, the colour of dyed silk fabrics changed from yellow-red to red. From these results, it was concluded that the apparent colours were depended on the extractant (buffer solution) pH and the kind of mordant used in this study.

Colour Fastness

The rating of fastness (light, water, washing, and perspiration fastness) of pre-mordanted and un-mordanted cotton and silk fabrics dyed with *Cassia tora L.* extract are shown in Table 4 and Table 5, respectively. It was found that water, washing, and perspiration fastness of un-mordanted cotton and silk fabrics showed considerably good. This result might be attributed to the high affinity of anthraquinone dye with cellulose and protein. However, the light fastness of un-mordanted cotton and silk fabrics was bad. Generally, it is well known that the poor light fastness was a problem for natural dyes. The light fastness of pre-mordanted cotton and silk fabrics was significantly found to increase from rating 2 to rating 4, and rating 3 to rating 4 by using $FeSO_4$ and $CuSO_4$ for cotton, and by using $FeSO_4$, $CuSO_4$ and $Al_2(SO_4)_3$ for silk, respectively. However, the other mordants among mordants used in this study did not affect significantly the light fastness of pre-mordanted cotton and silk fabrics. From these results, it was concluded that mordants $FeSO_4$ and $CuSO_4$ for cotton,

Table 4. The fastness of cotton fabrics dyed with *Cassia tora L.* extract

				None	$Al_2(SO_4)_3$	$MnSO_4$	$ZnSO_4$	$NiSO_4$	$CuSO_4$	$FeSO_4$
Light				2	2	2	2	2	4	4
Water		Fade		3-4	4-5	4	4	3-4	4-5	4-5
		Stain	Silk	3-4	3-4	3-4	3-4	3-4	3-4	3-4
			Cotton	3	4	3-4	3	3	3	4
Washing		Fade		3	3	3	3	3	4	4
		Stain	Silk	4-5	4-5	4-5	4-5	4-5	4-5	4-5
			Cotton	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Perspi-ration	Acidic	Fade		3	4	3	3	3	3	3
		Stain	Silk	4	3-4	3-4	3-4	4	3	3
			Cotton	3	3	3	3	3-4	2	3
	Alkaline	Fade		4	4	4	4	4	4-5	5
		Stain	Silk	4	3-4	3-4	3-4	4	3-4	3-4
			Cotton	4	4	3-4	3-4	3-4	3-4	4

Table 5. The fastness of silk fabrics dyed with *Cassia tora L.* extract

				None	$Al_2(SO_4)_3$	$MnSO_4$	$ZnSO_4$	$NiSO_4$	$CuSO_4$	$FeSO_4$
Light				3	4	2	3	2	4	4
Water		Fade		4	4	4	4	3-4	4	4
		Stain	Silk	3	3	3	3	3	3	3
			Cotton	3	4	4	4	4	3-4	3-4
Washing		Fade		3	3	3	3	3	3	4
		Stain	Silk	4-5	4-5	4-5	4-5	4-5	4-5	4-5
			Cotton	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Perspiration	Acidic	Fade		3	3	3	3	3	3	4
		Stain	Silk	3	2-3	3	2-3	2-3	2	2
			Cotton	3	1-2	2	1-2	1-2	1	1-2
	Alkaline	Fade		4	4	4	4	4	4	4
		Stain	Silk	2-3	3	3	3	3	3	2-3
			Cotton	3	3-4	3-4	3	3	2-3	3-4

and mordants $FeSO_4$, $CuSO_4$ and $Al_2(SO_4)_3$ for silk were the best mordants to improve the light fastness of cotton and silk fabrics.

Conclusions

The purpose of this study is to investigate the effect of mordant type on dyeing properties and colour fastness (light, wash, water, and perspiration fastness) of cotton and silk fabrics dyed with *Cassia tora L.* extract solutions. It was found that *Cassia tora L.* extract was polygenetic dyestuffs and its major components were anthraquinones. The K/S value of dyed cotton fabrics increased in the order of dyeing using $FeSO_4 > CuSO_4 > ZnSO_4 > MnSO_4 \cong Al_2(SO_4)_3 > NiSO_4$ > none, however, the K/S value of silk fabrics increased in the order of the dyeing using $FeSO_4 > CuSO_4 > ZnSO_4 \cong Al_2(SO_4)_3 > MnSO_4 \cong NiSO_4$ > none. The K/S value seemed to depend primarily on the colour difference (ΔE) between mordanted fabric and control fabric rather than the mordanted metal ion content of pre-mordanted fabrics. Mordants $FeSO_4$ and $CuSO_4$ for cotton fabric, $FeSO_4$, $CuSO_4$, and $Al_2(SO_4)_3$ for silk fabric were found to give good light fastness (rating 4). It was found that $FeSO_4$ and $CuSO_4$ were the best mordants for the improvements of colour strength (K/S) and light fastness.

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