

DYEING, FASTNESS AND UV PROTECTION PROPERTIES OF COTTON FABRIC DYED WITH MANGROVE BARK EXTRACT

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Cotton fabric was dyed with a natural dye extracted from mangrove bark by the exhaustion process. Optimal results were accomplished when dyeing at 90 °C for 60 min and at pH 5. The dyeing was subsequently conducted with and without metallic salt mordants, using three different mordanting methods, pre-mordanting, meta-mordanting and post-mordanting. The mordanting not only improved color strength of the dyed cotton fabric, but also produced a variety of color shades. The color fastness to washing was very poor to poor due to a change in color shade after washing, but there was no fading of the color. The color fastness to light varied mostly from a fair to a good level. Color fastness to crocking, perspiration and water ranged from fair to very good, depending on the type of mordants used. Cotton fabric dyed with mangrove bark extract also showed very good to excellent UV protection properties.

Keywords: natural dye, mangrove bark, exhaust dyeing, cotton, mordants, UV protection

INTRODUCTION

Natural dyes are colorants that are obtained from plants, animals and minerals and they have been used for coloring textiles made from natural fibers, such as silk, wool and cotton.^{1,2} Various parts of plants, including roots, barks, leaves, flowers and fruits, are the most important sources of natural dyes, offering a wide range of shades.³ In order to obtain high color yield, different color shades and good fastness properties, metallic salt mordants from aluminum, iron, chromium and copper are normally employed.⁴ These metal ions form coordination bonds with the dye molecules, making them insoluble in water. Recently, natural dyes have attracted more interest as a result of the environmental concerns caused by synthetic dyes.^{5,6} Natural dyes are believed to have no environmental impact and to be sustainable, especially if they are extracted from otherwise unused waste products such as dried skin of *Allium cepa* and ash-tree bark.^{7,8} In the past decades, many attempts have been made to improve the dyeing performance and fastness properties of natural dyes.⁹⁻¹¹ Furthermore, other functional finishes provided by natural dyes, for example, UV protection,¹²⁻¹⁶ antibacterial,¹⁷⁻²⁰ and deodorizing^{21,22} properties, have been recently reported.

In addition to their great ecological value as marine habitat and coastal area protection, mangrove forests have long been utilized for firewood, charcoal and construction of dwellings, furniture, boats and fishing equipment.^{23,24} Other applications include the production of tannins, which are used for leather tanning and dyeing. During charcoal manufacture, mangrove bark is removed from timber to reduce the ash content of the charcoal product, and it is regarded as solid waste.²⁵ However, it is reported that these mangrove barks are rich in tannin ranging from 15-36% and can produce reddish brown dyes.^{26,27} The major coloring components of tannin found in mangrove barks are of a condensed type, which are composed of four flavonoid monomers, namely catechin, epicatechin,

epigallocatechin and epicatechin gallate, as illustrated in Fig. 1.²⁸

The aim of the present study is to investigate the exhaust dyeing of cotton fabric with an aqueous extract of mangrove bark, considering the effect of dyeing conditions (temperature, time and pH) on the dye uptake of cotton fabric. The effects of dye concentration and mordanting with metallic mordant salts on color yield, fastness, and UV protection properties were also examined.

EXPERIMENTAL

Materials

A commercially scoured and bleached cotton fabric (twill weave, thickness 0.23 mm, weight 127.81 gm⁻², thread count per inch 140x100) was used. The thread count, thickness and weight of the cotton fabric were determined according to ASTM D 3775-98, ISO 5084-1996 and ISO 3801-1997, respectively. Mangrove bark (*Rhizophora apiculata* Blume) was supplied from a charcoal factory in Samutsongkhram province, Thailand. Four different laboratory grade mordants were used, i.e., aluminum potassium sulfate (AlK(SO₄)₂.12H₂O), ferrous sulfate (FeSO₄.7H₂O), copper sulfate (CuSO₄.5H₂O) and stannous chloride (SnCl₂.5H₂O). A non-ionic soaping agent, Metapon X-80, was supplied by Ilin Enterprise Co. Ltd. (Thailand).

Instruments

The mordanting and dyeing processes were conducted using a Gyrowash machine (James H. Heal & Co. Ltd., England). A UV-visible spectrophotometer (Halo DB-20, UV-Vis Double Beam Spectrophotometer, Australia) was used for absorbance measurement using a quartz cuvette of 1 cm path length. The CIE L*, a*, b* and color strength in terms of K/S values of dyed samples were measured using a spectrophotometer (Hunter Lab Color Quest XE, USA) with illuminant D65 at 10 degree observer. The transmittance and UPF values were measured by a Spectronic Camspec M550 Double Beam Scanning UV-Vis spectrophotometer.

Dye extraction from mangrove bark

The collected mangrove bark was dried in sunlight and later crushed to small pieces before being used for dye extraction. The dye extraction was performed by mixing the plant material and distilled water in the weight ratio of 1:10 and boiling for 1 h. After that, the resulting solution was filtered to remove the residue and the dye solution was separated into two portions: (a) one for evaporating under reduced pressure with a rotary evaporator, and (b) one for dyeing. The crude dye extract obtained from the rotary evaporator was dried and then stored in a desiccator before use. To obtain a standard calibration curve, the crude dye extract was later ground and diluted with distilled water. The dilution of the mangrove bark extract showed a clear solution with a linear dependence on the concentration-absorbance relation at an absorption peak (λ_{max}) of 278 nm. The concentration of 15 g/L was calculated from a standard curve of concentrations of the mangrove bark extract dye solution versus absorbance at the wavelength mentioned.

Mordanting and dyeing

Four dyeing conditions were varied (temperature, dyeing time, pH and dye concentration) to study the mangrove bark dye extract uptake behavior of cotton fabric.

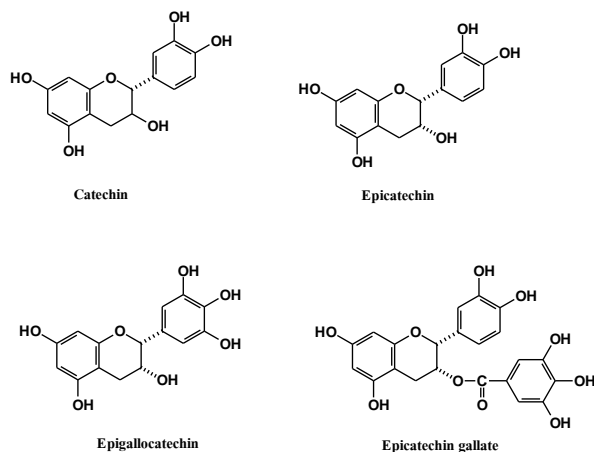


Figure 1: Color components found in mangrove bark

Temperature

To investigate the effect of dyeing temperature, cotton fabric was dyed at seven different temperatures, i.e. 30 °C, 40 °C, 50 °C, 60 °C, 70 °C, 80 °C and 90 °C, using 50% on the weight of fabric (owf) of the mangrove bark extract dye solution at a fabric to liquor ratio of 1:40 and at the original pH (=5.4) of the dye solution for 60 min.

Dyeing time

Cotton fabric was dyed in six sets of 50% owf of the mangrove bark extract dye solution at 90 °C, at a fabric to liquor ratio of 1:40 and pH 5.4, for different time intervals (10, 20, 30, 40, 50 and 60 min).

pH

Cotton fabric was dyed in 50% owf of the mangrove bark extract dye solution in dyebaths at different pH values of 3, 5, 7, 9 and 11, at a fabric to liquor ratio of 1:40, at 90 °C for 60 min. The pH of the dyebath was adjusted to a desired value with acetic acid and sodium carbonate solutions.

Dye concentration

The dye concentration was varied at 20%, 40% and 60% owf and three different mordanting methods were employed, i.e. pre-mordanting, meta-mordanting and post-mordanting, using four types of mordants (aluminum potassium sulfate, ferrous sulfate, copper sulfate and stannous chloride) with a concentration of 40% owf. For the pre-mordanting method, cotton fabric was impregnated in a mordanting solution at a fabric to liquor ratio of 1:40, at 60 °C for 60 min before dyeing. In the post-mordanting method, the dyed fabric was subjected to the same treatment. In the case of meta-mordanting, the mordant was added to the dyebath before dyeing. Cotton fabric was dyed at a fabric to liquor ratio of 1:40 and pH 5.4, at 90 °C for 60 min. After dyeing, the dyed samples were rinsed with cold water, soaped with 1 g/l of Metapon X-80 at a fabric to liquor ratio of 1:40, at 90 °C for 10 min, and finally rinsed with cold water and air-dried.

Evaluation of color strength and fastness properties

To evaluate dyeing performance, the color strength (K/S) and CIELAB of the dyed samples were measured using a Hunter Lab Color Quest XE spectrophotometer with illuminant D65 and 10° observer. The K/S is calculated by the Kubelka-Munk equation, $K/S = (1-R)^2/2R$, where R is the reflectance, K is the absorption coefficient, and S is the scattering coefficient. The L*, a*, b* values refer to the three axes of the CIELAB system. The L* value indicates the perceived lightness in the CIELAB color space. The L* scale runs from 0 (black) to 100 (white); the higher the L* reading the lighter color. The a* value indicates red (+a*) and green (-a*), while the b* value indicates yellow (+b*) and blue (-b*).²⁹⁻³¹ All measured samples showed a maximum absorption wavelength value (λ_{max}) at 400 nm. The color fastness to washing, light, crocking, perspiration, and water of the dyed samples were determined according to AATCC Test Method 61-2010, ISO 105-B02: 1994, AATCC Test Method 8-2007, AATCC Test Method 15-2009 and AATCC Test Method 107-2009, respectively.

Evaluation of UV protection

The transmittance and UPF (Ultraviolet Protection Factor) of the original cotton fabric, and mangrove bark dyed cottons were measured using a Spectronic Camspec M550 Double Beam Scanning UV-Vis spectrophotometer. The UPF of the fabric was determined based on AS/NZ 4399:1996 from the total spectral transmittance as follows:³²

$$UPF = \frac{\sum_{290}^{400} E_{\lambda} S_{\lambda} \Delta_{\lambda}}{\sum_{290}^{400} E_{\lambda} S_{\lambda} T_{\lambda} \Delta_{\lambda}} \quad (1)$$

where E_{λ} is the relative erythemal spectral effectiveness (unitless), S_{λ} is the solar UVR spectral irradiance in $Wm^{-2}nm^{-1}$, T_{λ} is the measured spectral transmission of the fabric, Δ_{λ} is the bandwidth in nm and λ is the wavelength in nm. Fabrics with a UPF value in the range of 15 to 24 are defined as “good UV protection”; from 25 to 39 as “very good UV protection” and of 40 or greater as “excellent UV protection”.³³ There is no rating assigned if the UPF rating is greater than 50.

RESULTS AND DISCUSSION

Effect of dyeing conditions

The effects of dyeing temperature, dyeing time and dye bath pH during dyeing of cotton fabric with the mangrove bark extract on color strength (K/S values) are illustrated in Fig. 2. It is evident that

the color strength increased slowly with an increase in dyeing temperature from 30 °C to 80 °C (Fig. 2(a)). Clearly, higher *K/S* value was observed at 90 °C. The color of the dyed cotton fabric was yellowish-brown. Meanwhile, a longer dyeing time gave successively higher color strength until the dyeing equilibrium was reached at approximately 60 min, where there was no further significant increase in *K/S* values (Fig. 2(b)). In view of pH values, the maximum dye uptake occurred at pH 5, whereas an increase in pH from 5 to 11 resulted in a decrease in dye uptake (Fig. 2(c)). It seems that cotton fabric is dyed with mangrove bark extract mainly by the formation of hydrogen bonding between the phenolic hydroxyl groups of the tannin found in the mangrove bark and the hydroxyl groups of the cellulose fiber.

Based on Tables 1-3, the *K/S* values of cotton fabric dyed with mangrove bark extract increased with an increase of dye concentration. Moreover, mordanting with metallic salt mordants clearly gave higher color strength as compared with the corresponding unmordanted samples. The mordant activity sequence for the pre-mordanting method was $\text{CuSO}_4 > \text{SnCl}_2 > \text{FeSO}_4 > \text{AlK}(\text{SO}_4)_2 >$ without a mordant and $\text{CuSO}_4 > \text{FeSO}_4 > \text{AlK}(\text{SO}_4)_2 > \text{SnCl}_2 >$ without a mordant for the meta-mordanting. In the post-mordanting, $\text{FeSO}_4 > \text{CuSO}_4 > \text{SnCl}_2 > \text{AlK}(\text{SO}_4)_2 >$ without a mordant was observed. Overall, ferrous sulfate and copper sulfate mordants yielded better color strength than aluminum potassium sulfate and stannous chloride, except in the case of the pre-mordanting method where stannous chloride gave higher color strength than ferrous sulfate.

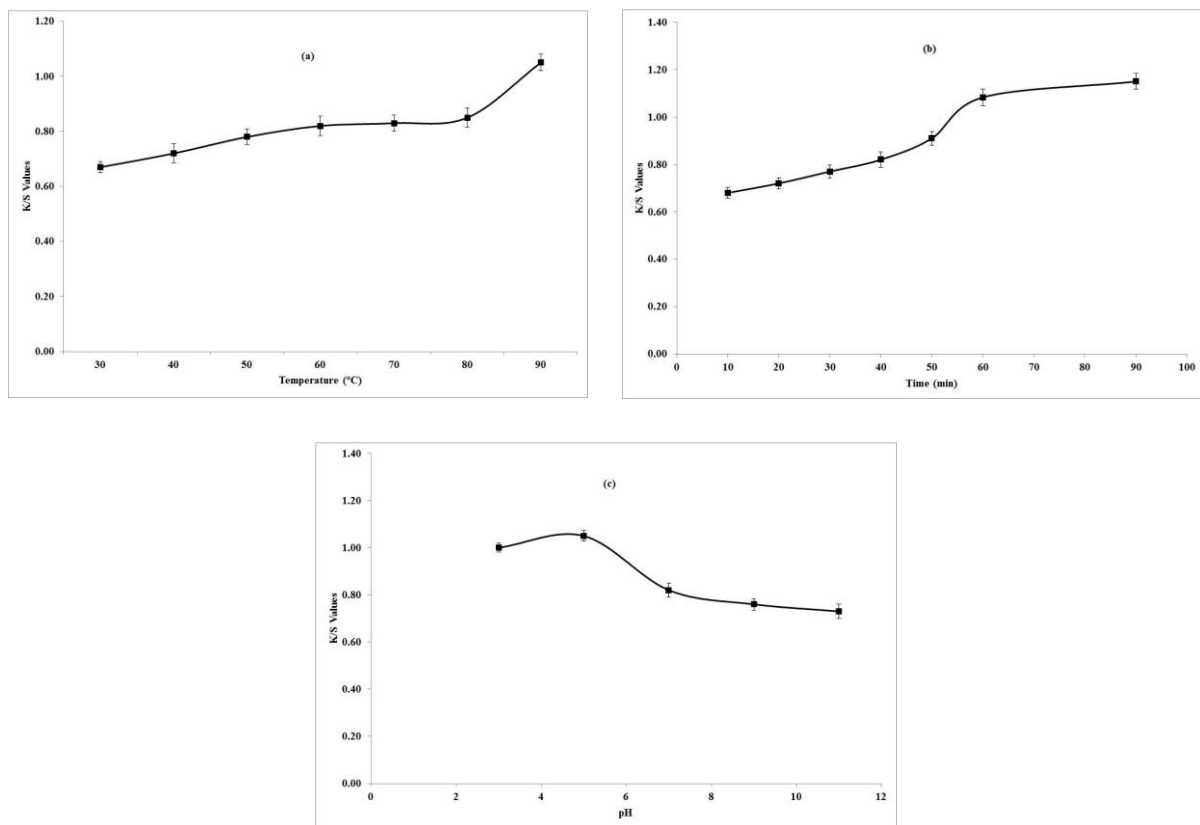
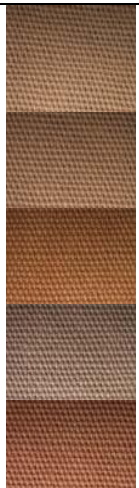


Figure 2: *K/S* values of cotton fabric dyed with mangrove bark extract dye solution (a) at varying temperatures from 30 °C to 90 °C, a fabric to liquor ratio of 1:40 and pH 5.4 for 60 min, (b) for different dyeing times from 10 to 90 min at 90 °C, a fabric to liquor ratio of 1:40 and pH 5.4 and (c) at different pH values (3, 5, 7, 9 and 11), a fabric to liquor ratio of 1:40, at 90 °C for 60 min. The error bars represent the standard deviation of the measurements ($n=5$)

Table 1
Color values at varying dye concentrations (20%, 40% and 60% owf) by pre-mordanting and using 40% owf mordants, a fabric to liquor ratio of 1:40, temperature of 90 °C for 60 min

Type of mordant	Dye conc. (% owf)	Pre-mordanting				Color obtained ^e
		K/S ^a	L ^{*b}	a ^{*c}	b ^{*d}	
Without	20	0.68	74.13	11.48	15.53	
	40	0.76	73.51	12.37	17.21	
	60	0.87	73.24	13.44	19.92	
AlK(SO ₄) ₂	20	0.79	75.17	12.48	20.92	
	40	0.82	73.17	13.56	19.14	
	60	0.93	72.21	13.94	20.80	
CuSO ₄	20	1.84	62.08	17.56	20.24	
	40	2.36	57.34	14.28	18.08	
	60	2.98	57.01	10.74	14.61	
FeSO ₄	20	1.20	62.58	6.71	8.62	
	40	1.28	61.74	7.20	9.26	
	60	1.32	61.52	4.87	7.78	
SnCl ₂	20	1.44	71.94	12.68	23.46	
	40	1.64	67.06	12.83	23.25	
	60	1.90	66.41	13.17	23.08	

^a Represents average value of 4 determinations, standard deviation (%) range ±0.85–1.28

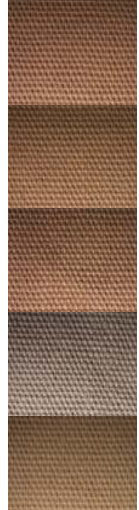
^b Represents average value of 4 determinations, standard deviation (%) range ±0.48–1.14

^c Represents average value of 4 determinations, standard deviation (%) range ±0.58–1.55

^d Represents average value of 4 determinations, standard deviation (%) range ±0.52–1.46

^e 60% owf dye concentration

Table 2
Color values at varying dye concentrations (20%, 40% and 60% owf) by meta-mordanting and using 40% owf mordants, a fabric to liquor ratio of 1:40, temperature of 90 °C for 60 min

Type of mordant	Dye conc. (% owf)	Meta-mordanting				Color obtained ^e
		K/S ^a	L ^{*b}	a ^{*c}	b ^{*d}	
Without	20	0.68	74.13	11.48	15.53	
	40	0.76	73.51	12.37	17.21	
	60	0.87	73.24	13.44	19.92	
AlK(SO ₄) ₂	20	1.18	70.33	13.04	21.69	
	40	1.24	69.56	11.60	20.50	
	60	1.27	68.83	11.68	20.34	
CuSO ₄	20	1.75	64.31	9.07	16.18	
	40	2.15	61.78	9.77	16.54	
	60	2.80	58.02	12.02	18.49	
FeSO ₄	20	1.23	63.46	2.99	6.45	
	40	1.81	56.87	4.19	6.77	
	60	1.85	56.07	3.48	6.49	
SnCl ₂	20	0.46	86.36	4.34	14.72	
	40	0.90	80.40	8.44	21.51	
	60	1.44	74.75	11.46	24.35	

^a Represents average value of 4 determinations, standard deviation (%) range ±0.78–1.14

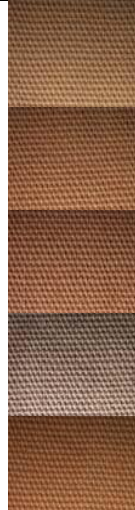
^b Represents average value of 4 determinations, standard deviation (%) range ±0.44–1.26

^c Represents average value of 4 determinations, standard deviation (%) range ±0.59–1.54

^d Represents average value of 4 determinations, standard deviation (%) range ±0.62–1.49

^e 60% owf dye concentration

Table 3
Color values at varying dye concentrations (20%, 40% and 60% owf) by post-mordanting and using 40% owf mordants, a fabric to liquor ratio of 1:40, temperature of 90 °C for 60 min

Type of mordant	Dye conc. (% owf)	Post-mordanting				Color obtained ^e
		K/S ^a	L* ^b	a* ^c	b* ^d	
Without	20	0.68	74.13	11.48	15.53	
	40	0.76	73.51	12.37	17.21	
	60	0.87	73.24	13.44	19.92	
AlK(SO ₄) ₂	20	1.00	69.53	13.24	17.63	
	40	1.28	67.79	14.85	20.64	
	60	1.63	64.71	14.30	21.40	
CuSO ₄	20	1.25	63.64	10.65	12.84	
	40	2.04	57.79	12.40	15.45	
	60	2.51	54.40	14.45	18.35	
FeSO ₄	20	1.40	59.43	5.09	7.00	
	40	2.25	51.93	5.01	6.65	
	60	3.38	46.54	4.95	6.87	
SnCl ₂	20	0.99	72.54	15.14	22.23	
	40	1.62	67.06	16.83	25.25	
	60	1.84	65.83	17.64	26.25	

^a Represents average value of 4 determinations, standard deviation (%) range ± 0.81 –1.32

^b Represents average value of 4 determinations, standard deviation (%) range ± 0.48 –1.34

^c Represents average value of 4 determinations, standard deviation (%) range ± 0.51 –1.26

^d Represents average value of 4 determinations, standard deviation (%) range ± 0.47 –1.44

^e 60% owf dye concentration

The highest *K/S* values obtained with ferrous sulfate and copper sulfate can be explained by the fact that these two mordants can form coordination complexes and readily chelate with the dye. Since the coordination numbers of ferrous sulfate and copper sulfate are 6 and 4, respectively, some coordination sites remain unoccupied when they interact with the fiber. Hence, these metals can form a ternary complex on which one site binds with the fiber and the other site binds with the dye.^{14,34} On the other hand, stannous chloride and aluminum potassium sulfate tend to form quite strong bonds with the dye but not with the fiber, so they block the dye and reduce the dye interaction with the fiber.^{14,34}

Cotton fabric dyed without mordant had relatively pale yellowish-brown color. Mordanting with aluminum potassium sulfate and stannous chloride gave medium to dark yellowish-brown, whereas copper sulfate produced reddish-brown shades. However, a duller and darker shade was obtained with ferrous sulfate mordant. This may be due to the change of ferrous sulfate into a ferric form by reacting with oxygen in the air. Ferrous and ferric forms coexist on the fibers and their spectra overlap, which causes a shift of λ_{max} to a darker shade.^{35,36} Furthermore, the tannins in the mangrove bark extract combine with ferrous sulfate to form complexes, which also results in a darker color shade.³⁷

Effect of dyeing techniques on fastness properties

Fastness properties of the mangrove bark dyed cotton fabric at 60% owf dye concentration and 40% owf mordant concentration are presented in Table 4. The wash fastness ratings of both unmordanted and mordanted dyed samples, irrespective of the mordant types, were very poor to poor (1-2). Although there is no significant fading of the color, the low gray scale rating is caused by the original color of the dyed fabric visually becoming more reddish after the washing test. As illustrated in Fig. 3, a bathochromic effect, i.e., a shift in color towards red in the visible region of the UV-Vis absorption spectra of the mangrove bark extract dye solution, occurred when the pH was adjusted from the starting pH 5.4 to pH 10. This drastic color change may be attributed to (a) the ionization of the hydroxyl groups in the dye molecules under alkaline condition of the standard detergent

solution^{38,39} or (b) the decomposition of the dye itself, resulting in a colorless or a differentially colored compound.³⁹

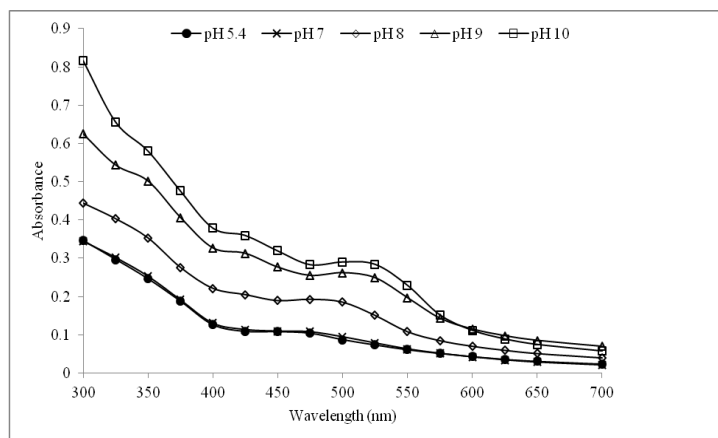


Figure 3: UV-Vis spectra of mangrove bark extract dye solutions

Table 4
Color fastness of cotton fabric dyed with 60% owf mangrove bark extract dye solution, using 40% owf mordants

Fastness	Without	Pre-mordanting				Meta-mordanting				Post-mordanting			
		Al	Cu	Fe	Sn	Al	Cu	Fe	Sn	Al	Cu	Fe	Sn
Washing	1-2	1-2	2	2	1-2	1-2	2	2	1	1-2	2	2	1
Light Crocking	3	3	4	4	1	3	4	4	1	3	4	4	1
Dry Crocking	4-5	4-5	4-5	4-5	4	4-5	4-5	4-5	4	4-5	4-5	4-5	4
Wet Crocking	4	4	4	4	3-4	4	4	4	3-4	4	4	4	3-4
Perspiration	4	3-4	3	2-3	4	3-4	3	2-3	4	3-4	3	2-3	4
Water	4	4-5	4	4-5	4-5	4-5	4-5	4-5	4	4	4-5	4-5	4-5

Note: Al = AlK(SO₄)₂, Cu = CuSO₄, Fe = FeSO₄, Sn = SnCl₂

The light fastness was good (4) for the fabric mordanted with copper sulfate and ferrous sulfate, fair (3) for aluminum potassium sulfate mordanted and unmordanted samples, and very poor (1) for the stannous chloride mordanted sample, irrespective of the mordanting method. Color fastness to dry and wet crocking was good to very good (4-5) and good (4), respectively, except for the samples mordanted with stannous chloride, whose rating was good (4) and fair to good (3-4). Color fastness to acid perspiration was good (4) for stannous chloride mordant, fair to good (3-4) for aluminum potassium sulfate, fair (3) for copper sulfate, poor to fair (2-3) for ferrous sulfate. Color fastness to water was good to very good (4, 4-5) for all samples. The overall ratings of color fastness to washing, perspiration and water in terms of the degree of color staining on adjacent fabrics were good to very good (4, 4-5).

UV protection property

The undyed cotton fabric used in this study had a low UPF of 10.1, as shown in Table 5, which cannot be rated as offering any degree of protection because the UPF value was less than 15. The dyed fabric without a metal mordant gave a UPF value of 26.8, which could be classified as “very good UV protection” (UPF values between 25 and 39). Most of the dyed fabrics with metal mordants provided “excellent UV protection” because their UPF values were greater than 40, except for the samples pre- or post-mordanted with aluminum potassium sulfate and meta-mordanted with stannous chloride, which offered “very good UV protection”. Additionally, it is also clear that an increase in K/S values was accompanied by an increase in the UPF values. For example, in the case of cotton

fabric dyed with the mangrove bark extract with $\text{AlK}(\text{SO}_4)_2$ mordant, the K/S value increased from 0.79 to 1.18 and the UPF value rose from 36.3 to 51.1. Therefore, this result confirms that dyeing the fabrics with natural dyes in deeper color shades can reduce exposure to UV radiation more efficiently than paler ones, which has been previously reported by several works.^{14,15,40}

Table 5
UPF values, protection class, and K/S values of cotton fabric dyed with 20% owf mangrove bark extract dye solution, using 40% owf mordants

Mordants	Mordanting methods	UPF ^a	UPF protection class	K/S ^b
Undyed	-	10.1	No Class	-
Without mordant	-	26.8	Very Good	0.68
$\text{AlK}(\text{SO}_4)_2$	Pre	36.3	Very Good	0.79
	Meta	51.1	Excellent	1.18
	Post	37.9	Very Good	1.00
CuSO_4	Pre	85.7	Excellent	1.84
	Meta	69.0	Excellent	1.75
	Post	57.5	Excellent	1.25
FeSO_4	Pre	55.0	Excellent	1.20
	Meta	60.2	Excellent	1.23
	Post	66.7	Excellent	1.40
SnCl_2	Pre	64.8	Excellent	1.44
	Meta	34.6	Very Good	0.46
	Post	40.5	Excellent	0.99

^a Represents average value of 4 determinations, standard deviation (%) range ± 0.56 –1.62

^b Represents average value of 4 determinations, standard deviation (%) range ± 0.78 –1.30

CONCLUSION

In this research, cotton fabric has been dyed with a natural dye extracted from mangrove bark by the exhaust dyeing method. The optimum dyeing pH, temperature, and time were found to be pH 5 at 90 °C for 60 min. Further improvement in color yield was noticed with increasing dye concentration and additional mordanting. The color obtained on the dyed cotton fabric varied in shade from medium to dark yellowish-brown for aluminum potassium sulfate and stannous chloride, reddish-brown for copper sulfate to a significantly duller and darker shade for ferrous sulfate. All mangrove bark dyed cotton fabrics exhibited good to very good color fastness to water and fair to good light fastness with the exception of the samples mordanted with stannous chloride, whose light fastness was very poor. The color fastness to crocking and perspiration were fair to very good, except for the fabric mordanted with ferrous sulfate, whose color change rating was poor to fair for perspiration fastness. However, the overall wash fastness was rated very poor to poor as a result of changes in color shade, but there was no fading of the color after the washing test in the basic standard detergent solution. Besides, dyeing of cotton fabric with the natural dye extract from mangrove bark in the presence of metal mordants could generally enhance the UV protection property up to very good or excellent levels.

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