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Contributed Paper

An Evaluation of UV Protection Imparted by Wool Fabric Dyed with Natural Dye from Eucalyptus Leaf

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ABSTRACT

This research is concerned with natural dye extraction from the leaves of eucalyptus and the application of this dye for wool fabric dyeing using two padding techniques, namely the pad-batch and pad-dry techniques under different conditions. It was observed that with an increase in the dye concentration, the ultraviolet (UV) protection factor (UPF) values ranged between very good and excellent for wool fabric. The colour fastness to light and rubbing after dyeing the wool fabric treated with the mordant was investigated, the results of which showed fair to good fastness, whereas the colour fastness to washing was a good to very good level. The results confirmed that natural dye from eucalyptus leaf extract have potential applications for fabric dyeing and producing UV protective wool fabric.

Keywords: natural dyes, UV-protection, eucalyptus, wool, padding, UPF

1. INTRODUCTION

Recently, consumers have become increasingly aware of the need for sun protection, which is related to the incidence of sun-induced skin damage and its relationship with an increased exposure to UV light. Ultraviolet radiation (UVR) can lead to acute and chronic reactions and damage, such as acceleration of skin ageing [1]. An overdose of UV can cause various skin, eye, and even DNA damage [2]. The UVR band consists of three regions: the UV-A band (320-400 nm), the UV-B band (290-320 nm), and the UV-C band (200-290 nm) [3]. The highest energy region, the UV-C band, is absorbed completely by oxygen and ozone in the upper atmosphere. Of the total solar UV radiation reaching the earth's surface, 6% is in the UV-B region and 94% in the UV-A region [4]. UV-A causes little visible reaction on the skin but has been shown to decrease the immunological response of skin cells. Among the three radiations, UV-B is the most responsible for the development of skin cancers [5]. Therefore, the transmittance of UVR, including UV-A and UV-B, through the fabrics was evaluated in the research.

There are many factors involved in the development of skin cancers and cumulative UV exposure of a patient is an important variable. Besides avoiding the sun, the most frequently recommended form of UV protection is the use of suitable clothes, hats, and sunscreens [6]. The protective properties of suitable clothes depend on fibre composition (natural or synthetic fibres), fabric construction (porosity, weight, and thickness), and the wet processing history of the fabric (using dyes, UV absorbers, and other finishing chemicals) [5-7].

From extensive literature survey, it was observed that there were some recent reports for application of natural dyes on UV protective textile materials. Sarkar [5] evaluated UV protection for different structures of cotton fabrics (plain, twill and sateen weave) dyed with madder, cochineal and indigo at different conditions and reported that dyeing of cotton fabrics with natural colourants increases the ultraviolet protective abilities of the fabrics and can be considered as an effective protection against ultraviolet rays. Hence, the Ultraviolet Protection Factor (UPF) is further enhanced with colourant of dark hues with high concentration of the colourant in the fabric. Kim [2] studied the dyeing characteristics and UV protection property of green tea dyed on cotton fabric using chitosan mordanting condition. The results showed that chitosan mordanting can effectively increase the UV protection property of both UV-A and UV-B of green tea dyed cotton fabrics. Chitosan mordanted undyed cotton and chitosan unmordanted dyed cotton not show an increase in UV protection property. Feng *et al*. [3] reported the ultraviolet protective properties of the cotton and silk fabrics dyed by Rheum and Lithospermum erythrorhizon. Similarly, experiment results revealed that the fabrics dyed with natural dyes had good ultraviolet protective properties. Thus, they could absorb about 80% of the ultraviolet rays. Wang et al. [7] researched the dyeing and ultraviolet protection of silk fabric using vegetable dyes extracted from *Flos sophorae*. It was found that the aqueous solution of this vegetable dye has excellent thermal stability in acid conditions. The optimum extraction conditions were obtained for *Flos sophorae*: extraction temperature of 100°C, extraction time of 60 min, , and material to liquor ratio of 1:10. The UPF and $T\ (UVA)_{AV}$ values for the silk fabric dyed by the optimum dye solution were found to meet the Chinese Standard (UPF = 69 > 30, $T\ (UVA)_{AV}$ = 1.07% < 5%). According to the standard, the silk fabric was claimed to have a "UV-Productive product". Grifoni et al. [8] studied the UV protection properties of flax and hemp fabrics dyed with weld, dyer's woad, logwood lipstick tree, madder, brasil wood, and cochineal as natural dyes. Experiment results revealed that natural dyes could confer good UV protection. While, weld-dyed fabric gave the highest protection level. Mongkholrattanasit *et al.* [9] reported the UV protection properties of silk fabric dyed with eucalyptus leaf extract. Experiment results observed that the ultraviolet (UV) protection factor (UPF) values increases with a increasing of dye concentration. On this note, Punrattanasin [10] green tea dyeing of cotton fabric via azoic combination method was investigated in order to improve ultraviolet protection properties without the application of polutting metal mordants. The result showed that as the strength of electron withdrawing groups of the substituents in the primary aromatic amines increase, both color strength and ultraviolet protection factor (UPF) of dyed fabrics dramatically increased.

Zhou *et al*. [11] studied and reported that cotton fabrics dyed with natural dyes (lithospermum erythrorhizon root, natural indigo, annatto, gardenia, sodium copper chlorophyll and cochineal) could absorb about 80% of the ultraviolet rays. Similarly, it was demonstrated that the UV-protective effects were strongly dependent on the absorption characteristics of natural dyes for UV radiation.

Eucalyptus is one of the most important sources of natural dye that gives yellowish-brown colourants. The colouring substance of eucalyptus has ample natural tannins and polyphenols varying from 10% to 12% [12]. The major colouring component of eucalyptus bark is quercetin, which is also an antioxidant. It has been used as a food dye with high antioxidant properties [13]. In addition, Eucalyptus leaves contain up to 11% of the major components of tannin (gallic acid and ellagic acid) and flavonoids (quercetin, and rutin, etc.) as minor substances [14-16]. Tannins and flavonoids are considered very useful substances during the dyeing process because of their ability to fix dyes within fabrics. Silk dyed with an aqueous extract of eucalyptus leaves and bark possessing a mordant compound displays a yellowish-brown colour. An exception is that when the fabric is dyed with ferrous mordant, the fabric shade becomes dark brownish-grey. Colour fastness to water, washing, and perspiration is good to a very good levels, whereas colour fastness to light and rubbing exhibited fair to good levels [17-18].

In this study, we investigated the UV protection properties of wool fabrics dyed with a eucalyptus leaf extract using pad-dyeing techniques. It is hoped that the data from the present study will provide a good insight into the UV protection properties of clothing made from wool fabrics dyed with a eucalyptus leaf extract.

2. MATERIALS AND METHODS

2.1 Fabric

A commercially produced plain-weave wool fabric (thickness 0.36 mm, weight 193 g/m^2, fabric count per inch 62 × 54) was scoured with an aqueous nonionic surfactant solution at a temperature of 45°C for 30 min, thoroughly rinsed and air dried at room temperature. The thread count, fabric thickness, and fabric weight characteristics of the wool fabric was in accordance with ASTM D3775-98, ISO 5084-1996, and ISO 3801-1997, respectively.

2.2 Mordants and Chemicals

The following laboratory-grade mordants were used: aluminium potassium sulphate dodecahydrate ($AlK(SO_4)_2 \cdot 12H_2O$), iron (II) sulphate heptahydrate ($FeSO_4 \cdot 7H_2O$) and copper (II) sulphate pentahydrate ($CuSO_4 \cdot 5H_2O$). The anionic wetting agent, Altaran S8 (sodium alkylsulphate) and soaping agent, Syntapon ABA, were supplied by Chemotex Decin, Czech Republic.

2.3 Instruments

The mordanting and dyeing processes were carried out in a two-bowl padding mangle machine (Mathis, type number HVF-69805). A drying machine (Mathis Labdryer, type number LTE-2992) was used for drying the dyed fabrics. A GBC UV/VIS 916 (Australia) spectro-photometer and a Datacolor 3890 were employed for the absorbance and colour strength measurements, respectively. The transmittance and ultraviolet protection factor (UPF) values were measured by a Shimadzu UV3101 PC UV-VIS-NIR scanning spectrophotometer in the 190-2100 nm range.

2.4 Dye Extraction from Eucalyptus Leaves

Fresh eucalyptus leaves (Eucalyptus camaldulensis) were dried in sunlight for one month and crumbled using a blender and then were used as the raw material for dye extraction, which was achieved by the reflux technique: 70 g of crumbled eucalyptus leaves was mixed with 1 litre of distilled water and refluxed for 1 hour. It was then filtered and the dye solution was separated into two parts: (a) one for evaporating under reduced pressure (rotary evaporator), and (b) one for dyeing. The rotary evaporator provided a crude dye extract of eucalyptus leaves. Therefore, it was crumbled with a blender and used for obtaining the standard calibration curve. The dilution of the eucalyptus leaf extract gives a relatively clear solution system with a linear dependence on the concentration absorbance, an absorption peak (λ_{max}) at 262 nm [19]. The concentration of 20 g/L was calculated from a standard curve of concentrations of the eucalyptus leaf extract dye solution versus absorbance at the wavelength mentioned.

2.5 Mordanting and Pad-dyeing

A simultaneous padding process was used in this study. To study the effect of dye concentration, three concentrations of the eucalyptus leaf extract dye were chosen: 5, 10, and 20 g/L. Three types of mordants were used at concentration of 10 g/L for each dye concentration and 1 g/L of an anionic wetting agent (Altaran S8) was added to the dye solution. The pH of the dyeing solution (mixed with an acetic acid solution) was adjusted to 4. Nevertheless, this pH condition has been optimised in the previous study [17,18]. The fabric was then immersed in the dye solution at room temperature and padded on a two-bowl padding mangle at 80% pick up. After padding for 2 sec, the samples were dried at 90°C for 5 min for the pad-dry technique. Under the cold pad-batch dyeing technique, the padded fabric was rolled on a glass rod with a plastic sheet wrapped around the rolled fabric. Then, it was kept at room temperature for 24 hours. The samples were then washed in 1 g/L of the soaping agent, Syntapon ABA, at 80°C for 5 min and air-dried at room temperature.

2.6 Evaluation of UV Protection

The transmittance and UPF values of the original wool fabric dyed with the eucalyptus leaf extract were measured using a Shimadzu UV3101 PC (UV-VIS-NIR scanning spectrophotometer, 190-2100 nm range). The UPF value of the fabric therefore is determined from the total spectral transmittance based on AS/NZ 4399:1996 as follows [20]:

$$UPF - \frac{\sum_{290}^{400} E\lambda\, S\lambda\, \Delta\lambda}{\sum_{290}^{400} E\lambda\, S\lambda\, T\lambda\, \Delta\lambda}$$

where E_λ is the relative erythemal spectral effectiveness (unitless), S_λ is the solar UVR spectral irradiance in $W.m^{-2}.nm^{-1}$, while T_λ is the measured spectral transmission of the fabric, whereas Δ_λ is the bandwidth in millimetre and λ is the wavelength in nanometre.

Fabrics with a UPF value in the range of 15-24 were believed to provide "good UV protection", While those in the range of 25-39 provide "very good UV protection", and between 40 or greater provide "excellent UV protection" [5]. Succinctly, there is no assigned rating for the UPF values that is greater than 50.

2.7 Evaluation of Colour Strength and Fastness Properties

The colour strength of the dyed samples were evaluated using the K/S values generated by the spectrophotometer (Datacolor 3890). K/S is a function of colour depth and is calculated by the Kubelka–Munk equation, $K/S = (1\text{-}R)^2/2R$, where R is reflectance of the dyed fabric; K is the sorption coefficient, and S is the scattering coefficient. The colour fastness to washing, light and rubbing properties of dyed samples were determined by standard ISO 105-CO6 A1S: 1994, ISO 105-B02: 1994 and ISO 105-X12: 2001 respectively.

3. RESULTS AND DISCUSSION

3.1 UV Protection Property

Wool fabrics dyed with the eucalyptus leaf extract without a mordant showed a yellowish-brown shade. The samples mordanted with $CuSO_4$ and $AlK(SO_4)_2$ produced medium-to-dark greyish-brown and pale yellow shades, respectively. With $FeSO_4$, the colour was darker and duller. This may be associated with a change of ferrous sulfate into a ferric form by reacting with oxygen in the air. Thus, ferrous and ferric forms coexisted on the fibers and their spectra overlapped, resulting in a shift of λ_{max} and consequent colour change to a darker shade [21]. Additionally, the tannins combined with ferrous salts to form complexes, which also result in a darker shade of fabric [22].

The UV percentage transmittance data of undyed wool fabric and those dyed with and without a mordanting agent are shown in Figures 1 to 3. It can be observed that since the relative erythemal spectral effectiveness is higher in the UV-B region (290-320 nm) than in the UV-A region (320-400 nm), therefore the UPF values depend primarily on transmission in the UV-B region. As can be seen, there is a difference between the dyed fabrics and the undyed fabric for the UV transmittance spectra. The undyed fabric showed a high percent UV transmittance in the range of 4 - 12 % of the UV-B band and about 12-37% in the UV-A band. This indicates that the resistance of undyed fabrics to ultraviolet ray was very poor, as seen in Figure 1. While the UV transmittance of wool fabrics dyed by eucalyptus leaf extract appeared to be lower than 5% in the UV-B region, as shown in Figure 2 and 3. Generally, the UV protection property of fabrics is evaluated good when the UV transmittance is less than 5%, [3, 23].

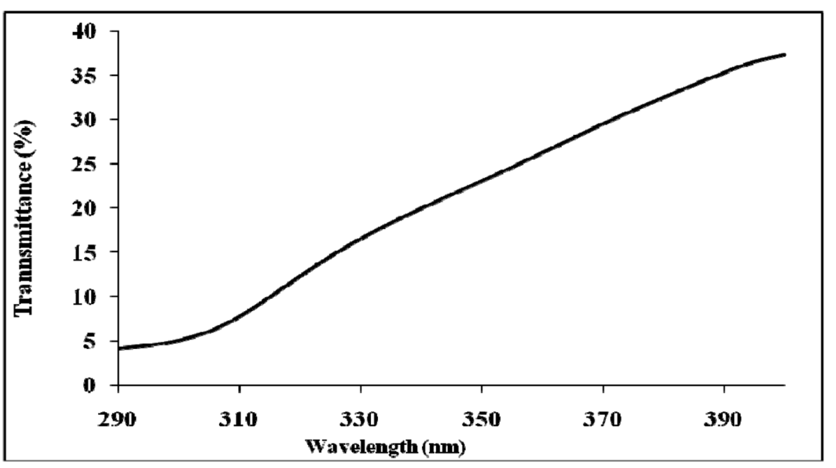

Figure 1. UV transmission of undyed wool fabric.

For the samples mordanted with $AlK(SO_4)_2$, $CuSO_4$, and $FeSO_4$, the percent UV-B transmittance was in the range of 0.06-0.21 %, 0.01-0.06%, and 0.01-0.04%, respectively for pad-batch, as shown in Figure 2 and 0.06-0.26 %, 0.03-0.06 %, and 0.04-0.05%, respectively for pad-dry, as seen in Figure 3. It is clearly seen that the values of the spectral transmittance were decreased with the mordants such as $AlK(SO_4)_2$, $CuSO_4$, and $FeSO_4$ as different mordants had different effects on the spectral transmittance of the fabric dyed [3]. Additionally, the colour and colour depth of the fabric can be related to UV transmittance in which light colours transmit more UV radiation than dark colours [24].

Table 1 to Table 2 show the UPF values, protection class, and K/S values of wool fabrics dyed with the eucalyptus leaf extract with and without metal mordants by pad-batch and pad-dry dyeing techniques. It can be observed that the UPF values increase with an increase in the dye concentration. Nevertheless, little difference was observed between the two padding techniques utilised for this study. The undyed fabric has a high transmittance value and a very low UPF value of 10.8. This indicates that the resistance of undyed fabric to UV rays was very poor. The dyed samples without a metal mordant at different concentrations of the dye using both dyeing techniques showed that UPF values between the range of 32.8 and 50.4, are seen to provide very good and excellent UV protection.

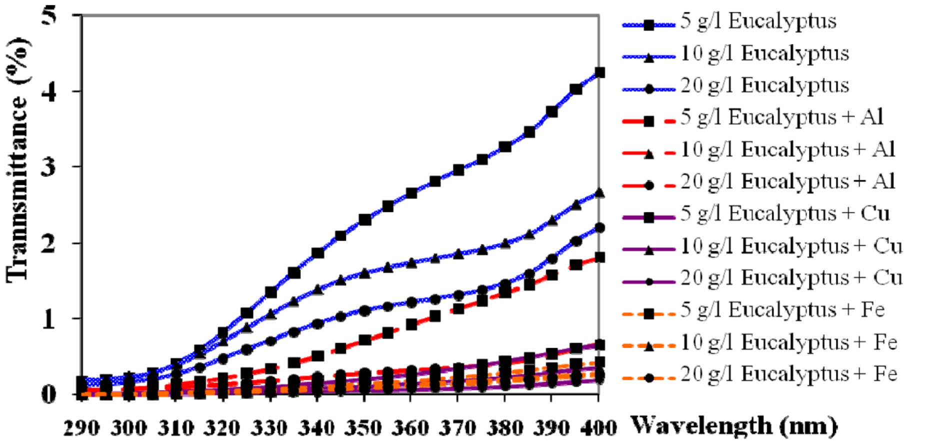

Figure 2. UV transmission of wool fabrics dyed with eucalyptus leaf extract in the absence and in the presence of metal mordants by the pad-batch technique. Al = $AlK(SO_4)_2$, Cu = $CuSO_4$, Fe = $FeSO_4$.

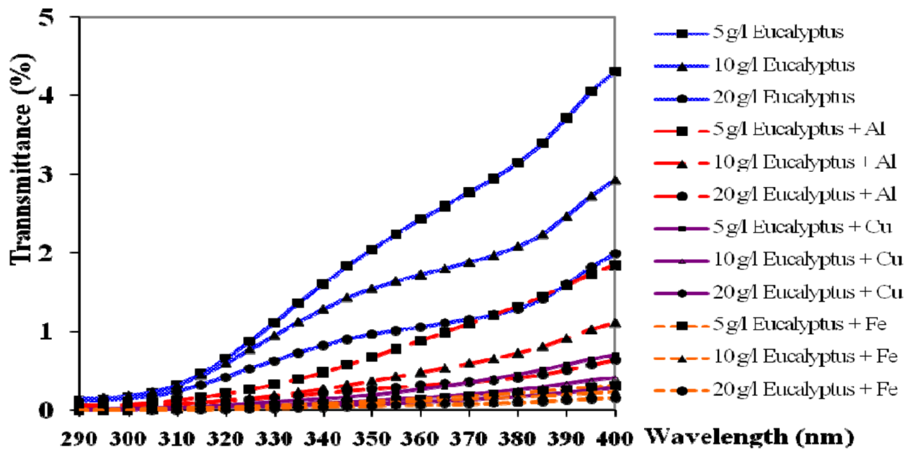

Figure 3. UV transmission of wool fabrics dyed with eucalyptus leaf extract in the absence and in the presence of metal mordants by the pad-dry technique. Al = $AlK(SO_4)_2$, Cu = $CuSO_4$, Fe = $FeSO_4$.

From the transmission data and the corresponding UPF values, it can be observed that with and without metal mordants used in the study caused a reduction in UVR transmission through the wool fabric. Therefore, Wool fabrics dyed with the $AlK(SO_4)_2$, $CuSO_4$ and $FeSO_4$ mordants at different concentrations of the dye using the pad-dry and the pad-batch dyeing techniques could be classified as offering "excellent UV protection" (UPF values equal to or greater than 40). The results also show that the samples dyed with higher concentrations of the eucalyptus leaf extract dye have higher UPF values. For example, the UPF value of the fabric dyed with the eucalyptus leaf extract and the $FeSO_4$ mordant by the pad-dry technique at a dye concentration rate between 5 g/L to 20 g/

Table 1. UPF values, protection class and K/S values of dyed fabrics by pad-batch dyeing.

Mordanting agent	Dye concentration	UPF	UPF Protection class	K/S
-	(g/L)	10.8		0.46
Without	Undyed	32.8	No Class	1.50
	5	43.7	Very Good	1.60
	10	48.5	Excellent	1.86
$AlK(SO_4)_2$	20	59.0	Excellent	1.65
	5	66.8	Excellent	1.91
	10	72.6	Excellent	2.55
$CuSO_4$	20	59.5	Excellent	3.27
	5	67.9	Excellent	3.44
	10	88.4	Excellent	4.12
$FeSO_4$	20	85.3	Excellent	3.93
	5	96.7	Excellent	4.23
	10	102.8	Excellent	4.62
	20		Excellent	

L, the value increased from 81.8 to 104.2.

The K/S values of the dyed fabrics, which were a measures of colour strength, seem to confirm that higher colour strength increases the UPF values. For example, in the case of the wool fabric dyed with the eucalyptus leaf extract using the $CuSO_4$ mordant and the pad-batch technique, the K/S value increased from 3.27 to 4.12 and the UPF value rose from 59.5 to 88.4. For the UPF protection, the mordant activity sequence for the simultaneous mordanting method was $FeSO_4$ > $CuSO_4$ > $AlK(SO_4)_2$ > without mordant for both techniques.

Therefore, it was proven that these results agree with previous data reported by Sarkar [5], who showed that a pale-coloured cotton fabric gives less protection against intense UV radiation. The results also show that the UPF values for colourants applied at higher concentrations are higher compared with those for colourants applied at lower concentrations.

On this note, we agree with Gies et al. [20] and Wilson et al. [24], who indicated that dyeing fabrics in deeper shades and darker colours improves their UV protection properties. Thus, although the studies by Gies et al. [20] and Wilson et al. [24] were done with synthetic dyes, their conclusion seems to hold with natural colourants as well. We also accept the results of Feng et al. [3], who demonstrated that the UV protection properties of cotton and silk fabrics dyed with natural dyes using a metal mordant could effectively protect the skin from solar UVR.

3.2 Effect of Dyeing Technique on Fastness Properties

The fastness rating of wool fabrics dyed with or without mordants at a dye concentration of 20 g/L was presented in Tables 3-5. When comparing the fastness rating of the samples dyed using the two padding techniques, it can be postulated that the pad-batch technique gives nearly the same fastness properties as the pad-dry technique. Table 3 indicates that the washing fastness ratings of wool fabrics dyed with the eucalyptus leaf extract were very good (4-5). However, the rating for light fastness was only fair (3-4), as shown in Table 4. The rating for colour fastness to rubbing was shown to be in the range of 4-5 (good to very good), except for fabrics mordanted with $FeSO_4$, whose rating was in the range of 3-4 (fair to good) when subjected to wet rubbing, as shown in Table 5.

The good fastness properties of wool fabrics dyed with the eucalyptus leaf extract may be attributed to the fact that these dyes contain tannin, which may help in the formation of covalent bonds with the fiber, thereby resulting in good fixation of the dye on the fabric. Also, these tannins, having a phenolic structure, can act as metal chelating agents for different mordants. Hence, after mordanting, these tannins become insoluble in water, ultimately improving

Table 2. UPF values, protection class and K/S values of dyed fabrics by pad-dry dyeing.

Mordanting agent	Dye concentration (g/L)	UPF	UPF Protection class	K/S
-	Undyed	10.8	No Class	
Without	5	35.4	Very Good	1.13
	10	40.3	Excellent	1.54
	20	50.4	Excellent	1.86
$AlK(SO_4)_2$	5	55.1	Excellent	1.65
	10	67.0	Excellent	1.81
	20	71.8	Excellent	2.60
$CuSO_4$	5	65.0	Excellent	2.32
	10	70.1	Excellent	2.62
	20	88.3	Excellent	2.80
$FeSO_4$	5	81.8	Excellent	4.54
	10	94.1	Excellent	4.81
	20	104.2	Excellent	5.14

Table 3. Colour fastness to washing at 40°C (ISO 105-CO6 A1S: 1994).

Fastness	Pad-batch				Pad-batch			
	Without	Al	Cu	Fe	Without	Al	Cu	Fe
Colour change	mordant	4-5	4-5	4-5	mordant	4-5	4-5	4-5
Colour staining	4-5				4-5			
-Acetate	4-5	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	4-5
-Nylon	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
-Polyester	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
-Acrylic	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
-Wool	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5

Note: Al = $AlK(SO_4)_2$ Cu = $CuSO_4$ Fe = $FeSO_4$

Table 4. Colour fastness to light (ISO 105-B02: 1994).

Mordant	Colour change	
	Pad-batch	Pad-dry
Without mordant	3-4	3-4
$AlK(SO_4)_2$	3-4	3-4
$CuSO_4$	4	4
$FeSO_4$	4	4

Table 5. Colour fastness to rubbing (ISO 105-X12: 2001).

Mordant	Colour staining							
	Pad-batch				Pad-batch			
	Warp direction		Weft direction		Warp direction		Weft direction	
	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
Without mordant	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
$AlK(SO_4)_2$	4-5	4	4-5	4	4-5	4	4-5	4
$CuSO_4$	4-5	4	4-5	4	4-5	4	4-5	4
$FeSO_4$	4	3-4	4	3-4	4	3-4	4	3-4

washing fastness [25].

4. Conclusion

In this study, it was observed that wool fabrics dyed with a eucalyptus leaf extract with and without metal mordants ($AlK(SO_4)_2$, $CuSO_4$, and $FeSO_4$) have "very good to excellent UV protection" properties. However, undyed wool fabric cannot be rated as offering any degree of protection. The degree of protection imparted after dyeing was a function of the concentration of the dye in the fabric. In addition, darker colours, such as those obtained using the $FeSO_4$ mordant, provided better protection on account of the higher degree of UV absorption. The ratings for washing and rubbing fastness of the samples dyed by both padding techniques were good to very good (4-5), whereas that for light fastness was fair (3-4). Therefore, it can be concluded that dyeing with a eucalyptus leaf extract can be useful in developing UV-protective wool fabrics.

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