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RESEARCH ARTICLE

UV-PROTECTION PROPERTIES OF COTTON/SYNTHETIC FABRICS PRINTED/DYED WITH NATURAL PIGMENTS

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ABSTRACT

The UV protection properties of cotton fabrics printed with ten natural colorants of plant and insect origin were investigated. The ability of the printed sample to reduce UV radiation transmittance differed widely from excellent to practically zero depending on the pigment used. The above pigment behaviour was correlated with their chemical structure and their UV-absorption spectra. UV radiation protection provided by synthetic fabrics polyester, acrylic, polyamide dyed with natural dyes has also been studied. UV radiation protection values vary with the fibre and the dye used from insufficient to excellent with the dyed/undyed polyester fabric presenting excellent values apparently due to a UV absorbing agent built-in the mass of polyester.

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INTRODUCTION

Prolonged exposure to excessive classes of UV-radiation can result in skin damage, e.g. sunburn, premature skin ageing, allergies, certain skin cancer etc (Thiry, 2005). UV-radiation is classified into three bands: UVA (315–400 nm), UVB (280–315 nm), and UVC (100–280 nm) (Tomljenovic *et al*., 2005). UVB-rays are only partially absorbed by ozone and have the ability to penetrate into the skin to form stable pigments in the epidermis of the skin as well as to cause skin cancer (Xu *et al*., 2005) and erythema, the most frequent acute UV effect on skin (Grifoni *et al*., 2009). To avoid these health risks, it is important to reduce personal UV exposure. Luckily, the use of sunscreens and UV protective clothing has gained popularity. The safest protection is offered by clothes; measurements of their properties in blocking UV exhibit enormous differences between fabrics due mainly to their structure, composition and colour. Natural cotton fibres are most commonly used for making summer clothes, thus most researches have focused on the UV protection properties of cotton (Gambichler *et al*., 2001; Sarkar, 2007; Hustvedt *et al*., 2005; Kerr *et al*., 2000; Kim, 2006; Dhandapani *et al*., 2007). Fabric colour also has a fundamental importance in UV protection; dyed fabrics protect more than undyed ones and most of these results concern synthetic dyes. Very few studies exist on the UV-protection properties of natural dyes in combination with natural fabrics (Grifoni *et al*., 2009; Sarkar, 2004; Feng, 2007; Kim, 2006; Dhandapani *et al*., 2007; Deepti *et al*., 2007; Iqbal *et al*., 2008; Wang *et al*., 2009) and most concern animal fibres. It is important to learn more about the UV-protection properties of a large number of natural dyes when combined with vegetable fibres, especially cotton. Fabric composition is another important factor in determining UV-protection degree because fibres can have different radiation absorbing properties. Recent studies have demonstrated that synthetic fibres, such as polyester, offer good protection from UV radiation (Sarkar, 2004; Dhandapani *et al*., 2007).

In continuation to previous work on cotton printing with natural pigments (Savvidis, doi: 10.1080/15440478.2016.1212761) the aims of this work were to investigate: i. the UV protection properties of cotton fabric printed with ten natural colorants of plant and insect origin (Table 1, Figure 1). ii The

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UV protection properties of synthetic polymer fabrics polyester, acrylic, polyamide dyed with the two most effective natural pigments selected among the ten already used. The UPF defines the protection efficiency of a fabric from the risks of UV radiation. The higher the UPF value, the greater is the fabric's protection level. UPF is defined as the ratio of the average effective UV irradiance calculated for unprotected skin to the average UV irradiance calculated for skin protected by the test fabric (Driscoll, 2000; Crews, 1999). The higher the value, the longer a person can stay in the sun until the area of skin under the fabric becomes red (Crews, 1999). An effective UVR dose (ED) for unprotected skin is calculated by convolving the incident solar spectral power distribution with the relative spectral effectiveness function and summing over the wavelength range 290-400 nm. The calculation is repeated with the spectral transmission of the fabric as an additional weighting to get the effective dose (EDm) for the skin when it is protected. The UPF is defined as the ratio of ED to EDm and calculated as follows (Gies, 1994):

$$UPF = \frac{ED}{ED_m} = \frac{\sum_{290nm}^{400nm} E_\lambda S_\lambda \Delta_\lambda}{\sum_{290nm}^{400nm} E_\lambda S_\lambda T_\lambda \Delta_\lambda} \qquad \text{Equation 1}$$

where:

$E\lambda$ = erythemal spectral effectiveness
$S\lambda$ = solar spectral irradiance in Wm-2nm-1
$T\lambda$ = spectral transmittance of fabric
$\Delta\lambda$ = the bandwidth in nm
λ = the wavelength in nm
Measured UPF values were also correlated to the colour strength of the dyed fabrics. Colour strength of the printed samples was evaluated using K/S values of the samples.

MATERIALS AND METHODS

Cotton samples (1/24, 150 g/m^2) supplied by KYKE Hellas S.A., polyester samples (Grenter Karre Double-Jersey 100 %, nylon samples CNM70 (Centexbel) and acrylic samples (120g/m^2, KYKE Hellas S.A) were used. In table 1 and figure 1 the pigments used in the present study with their botanical, C.I. names and numbers and their chemical structures are given. All pigments supplied by Alps Industries Ltd, India, were used without further purification for the printing processes. Cotton samples were pretreated with 0.5g L^{-1} Widet MS/V (non ionic surfactant supplied by Prochimica, Italy) and 1g L^{-1} sodium carbonate for 30min at 95 ^{o}C, followed by thorough rinsing in distilled water. The cotton samples were then left to dry in the open air and were used for printing. All constituents for the preparation of the printing paste namely the acrylic binder Cepolbind 1102N, the acrylic thickener Cepolclear HC were supplied by INFA SRL, Italy. The discharging agent used Decrolin was supplied by BASF.

Apparatus

A Werner Mathis flat bed screen printing equipment was used for the printing application. The mesh size of Werner Mathis flat bed screen printing equipment used was 120 mesh. A spectrophotometer UV/Vis JASCO V-550 (JASCO Corporation 2967-5, Tokyo, Japan) was used for obtaining the Ultraviolet Protection Factors. Colour measurements were performed using a Macbeth CE 3000 spectrophotometer under D65 illumination, 10^0 standard observer with UV included and specular component included.

Printing-Dyeing

The printing paste (1 kg) was prepared by adding 40g annatto in 792ml water at 60 C under stirring for 5min. 150g Cepolbind 1102 binder was added to colorant solution under stirring for 5min. 18g Cepolclear HC thickener was added drop wise under stirring in the above mixture, until the appropriate viscosity was achieved. The discharge printing paste was prepared as above by adding 50g Decrolin to 950g of the printing paste. Cotton samples were all pretreated as mentioned in 2.1. For the conventional printing the fixing was done by thermo fixation 4 min at 150^o C. Printed samples were dried and then thermo fixed at 150^o C for 4 min. Dyeing for polyester and polyamide fabrics is given in figure 2a-b respectively in a liquor ratio 1:20, depths of dyeing 0.5, 1.0, 1.5, 2.0, 3.0, 4.0, 6.0% omf, pH 4.

Colour Measurement

This was done according to the CIELAB 1976 and the modified CIELCH system (D65/10^o). Reproducibility was checked by taking four measurements and recording the variation in percentage reflectance values over the range 400-800nm (Mc Donald, 1997).

Measurement of anti-ultraviolet property

Accurate assessment of the amount of UV protection provided by the fabrics printed with the natural pigments (Table 1) required measurement of the spectral transmittance of each fabric across the wavelength range 280-400 nm, which includes UVA and UVB.

RESULTS AND DISCUSSION

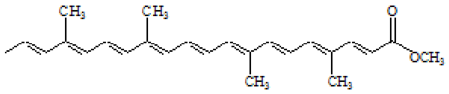
Cotton fibres

The radiation transmittance measured for printed samples with the eleven pigments, unprinted-treated with binder and the reference sample (non-treated, non-printed) are given in Table 2. As it is concluded from nd table 2 the ultraviolet radiation (UVR) transmittance of the blank sample printed directly was very high (both TUVA and TUVB), consequently UPF value was practically zero. The UVR transmittance of the blank sample printed with binder decreased sharply in contrast to that of the original sample, in the wavelength range of UVB (Table 2). The ability of the printed sample to reduce UV radiation transmittance differed according to the dye used. Pomegranate fruit 6 presents excellent UVR protection %UVA and %UVB transmittance being the lowest among the pigments, while cutch 4 and lac dye-S 9 still offer excellent protection.

Annatto 1 presents very good UV protection. The rest of the pigments practically do not offer any UVR protection with their UPF values being in the range 0.00 (indigo)-1.81 (kamala, insufficient protection). In figure 4 the UPF values for the reference, the unprinted-treated with binder and the printed samples are presented. Among the existing classification systems, the Australian ⁄ New Zealand standard was the first and is the most widely adopted; it establishes a classification system of fabrics according to their sun protective properties as shown in Table 3 (AS⁄NZS 4399, 1996).

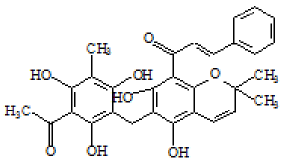
1. ANNATTO (BIXA ORELLANA) - C.I. Natural Orange 4

(Main pigments bixin, norbixin)



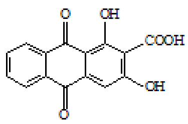
2. KAMALA (MALLOTUS PHILLIPINENSIS) - C.I. Natural Yellow 25

(Main pigment rottlerin)



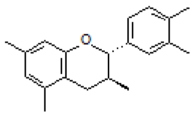
3. MADDER (RUBIA CARTIFOLIA) 1. - C.I. Natural Red 16, 2. C.I. Natural Red 8

(Main pigment munjistin)



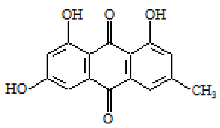
4. CUTCH (ACACIA CATECHU) - C.I. Natural Brown 3

(Main pigments catechin / flavonoids)



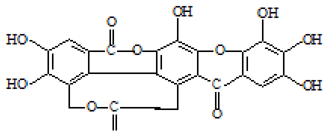
5. HIMALAYAN RHUBARB (RHEUM EMODI) - C.I. Natural Yellow 23

(Main pigment emodin)



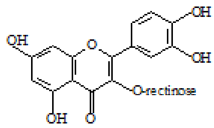
6. POME GRANATE FRUIT (PUNICA GRANATUM) - C.I.Natural Yellow 7

(Main pigment flavogallol)



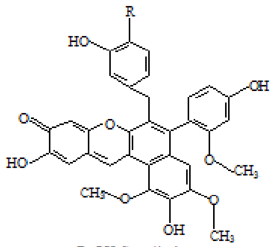
7. GOLDEN DOCK (RUMEX MARITIMUS)

(Main pigments rutin, hyperin)



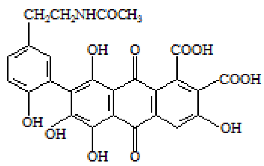
8. RED SANTALWOOD (PHENOCARPUS SANTALINUS) C.I. Natural Red 22

(Main pigments santalin A & B)



9. LAC DYE-S (KERRIA LACCAE) - C.I. Natural Red 25

(Main pigment laccaic acid)



10. INDIGO (INDIGOFERA TINCTORIA) - C.I. Natural Blue 1

(Main pigment indigo)

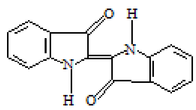


Figure 1. Chemical structures of the pigments used

Table 1. Pigments used

Number	Pigment	Botanical name	C.I. name/number
1	Annatto	Bixa orellana	Natural Orange 4/C.I. 75120
2	Kamala	Mallotus philipinensis	Natural Yellow 25/C.I. 75310
3	Madder	Rubia cardifolia	Natural Red 16/C.I.75340, 75350, 75410
4	Cutch	Acacia catechu	Natural Brown 3/C.I. 75250
5	Himalyan rhubarb	Rheum emodi	Natural Yellow 23/C.I. 75400
6	Pomegranate fruit	Punica granatum	Natural Yellow 7/C.I.
7	Golden Dock	Rumex maritimus	
8	Red santalwood	Phenocarpus santalinus	Natural Red 22/C.I.75510, 75540, 75550, 75560
9	Lac dye-S	Kerria laccae	Natural Red 25/C.I.75450
10	Indigo	Indigofera indica	Natural Blue1/C.I.75780, 75790

Table 2. UV protection properties of the printed textiles

Probes	Pigments	UPF	T(UVA)(%)	T(UVB)(%)
Blank 1	With binder	5.49	234.50	3.69
Blank 2	Without binder	0.00	3498.05	2129.17
1	Annatto	28.75	7.12	3.14
2	Kamala	1.81	575.83	29.38
3	Madder	0.33	1910.32	229.36
4	Cutch	61.11	8.35	7.76
5	Himalyan rhubarb	1.72	585.55	33.89
6	Pomegranate fruit	604.05	2.54	1.06
7	Golden Dock	0.40	1807.39	181.88
8	Red santalwood	1.573	672.91	53.99
9	Lac dye-S	55.61	5.24	3.02
10	Indigo	0.01	2666.47	1298.73

Table 3. UPF categories with relative transmittance and protection level

UPF range	Protection category	$UVBE_{eryt}$ transmittance (%)
<15	Insufficient protection	>6.7
15-24	Good protection	6.7-4.2
25-39	Very good protection	4.1-2.6
40-50, 50+	Excellent protection	≤2.5

Table 4. UPF values, protection class and K/S values of nylon 6.6 fabric dyed with natural colorants at different concentrations

Colorant	Color conc.(dod% owt)	UPF	Protection class	K/S
--	--	5.2	Insufficient	--
2 Red santalwood	1.0	13.2	Insufficient	3.72
	1.5	16.1	Good	4.69
	2.0	19.9	Good	5.26
	3.0	21.7	Good	5.77
	4.0	26.1	Very good	6.60
	6.0	32.3	Very good	7.94
1 Pomegranate fruit	1.0	10.4	Insufficient	4.22
	1.5	13.4	Insufficient	5.17
	2.0	17.1	Good	5.37
	3.0	17.3	Good	6.45
	4.0	23.1	Good	6.72
	6.0	31.6	Very good	7.97

Table 5. UPF values, protection class and K/S values of acrylic fabric dyed with natural colorants at different concentrations

Colorant	Color conc.(dod% owt)	UPF	Protection class	K/S
--	--	3.4	Insufficient	--
Annatto	0.5	16.0	Good	0.54
	1.0	18.7	Good	0.49
	1.5	19.8	Good	0.39
	2.0	22.1	Good	0.65
	3.0	33.5	Very good	0.77
	4.0	37.9	Very good	0.77

Table 6. UPF values, protection class and K/S values of polyester fabric dyed with natural colorants at different concentrations

Colorant	Color conc.(dod% owt)	UPF	Protection class	K/S
--	--	46.3	Excellent	--
3 Red santalwood	1.0	>50	Excellent	1.02
	1.5	>50	Excellent	1.17
	2.0	>50	Excellent	1.22
	3.0	>50	Excellent	1.40
	4.0	>50	Excellent	1.63
2 Pomegranate fruit	1.0	>50	Excellent	0.86
	1.5	>50	Excellent	1.55
	2.0	>50	Excellent	1.94
	3.0	>50	Excellent	2.66
	4.0	>50	Excellent	2.85
	6.0	>50	Excellent	3.18
1 Annatto	0.5	>50	Excellent	0.25
	1.0	>50	Excellent	0.52
	1.5	>50	Excellent	0.70
	2.0	>50	Excellent	0.49
	3.0	>50	Excellent	0.57
	4.0	>50	Excellent	0.62

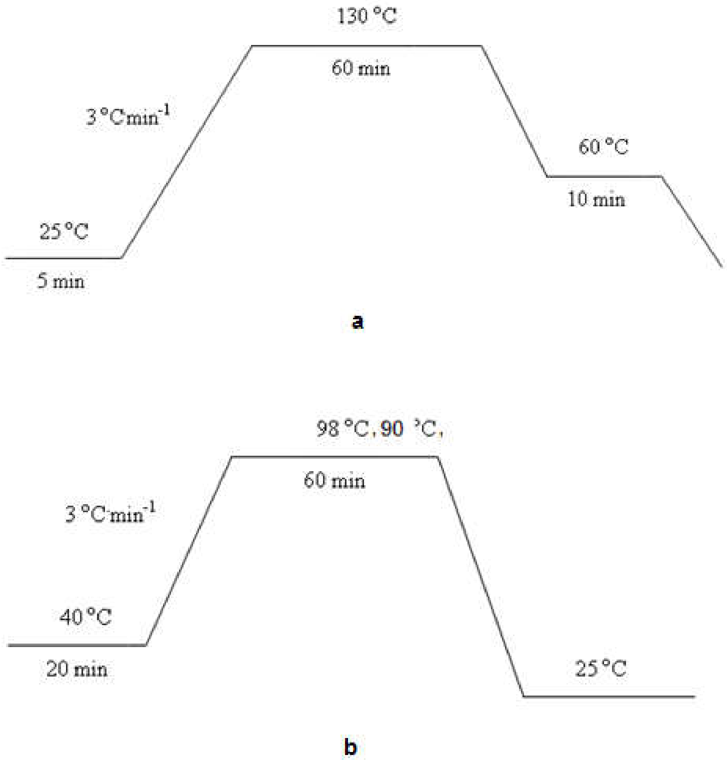


Figure 2. Dyeing process of: a. polyester b. polyamide, acrylic fibres

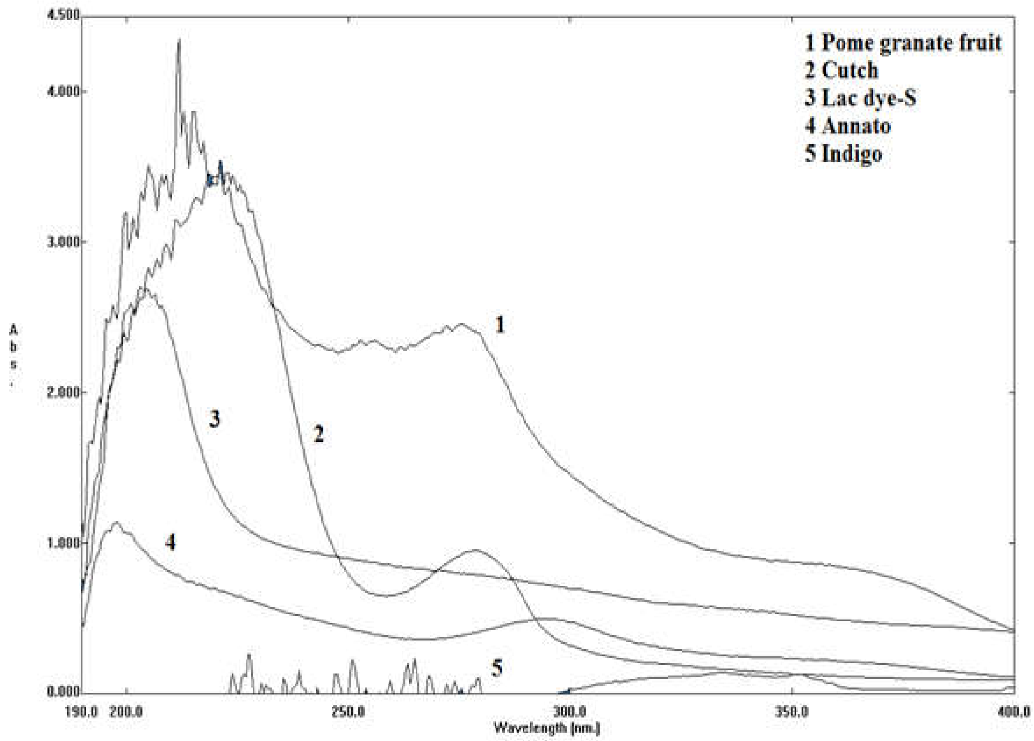


Figure 3. UV spectra of the pigments annatto, cutch, pomegranate fruit, lac dye S and indigo

The samples printed with indigo, madder, golden dock, lac dye-M, himalayan rhubarb, red sandalwood, kamala, as well as the non-printed sample had very low UPF values (Table 2) that did not achieve the minimum UPF rating of 15, corresponding to a good protection level according to the Australian / New Zealand standard. The positive UV-protecting effect of the binder, necessary to bridge the fibre and the pigments was confirmed by measuring the UPF of undyed samples (references) i. treated with an acrylic butadiene binder resulting in the formation of different conjugated p-bonds (blank 1) and ii. without any treatment (blank 2). The presence of binder necessary to bridge the fibre with the pigments results in a slight improvement of the UV protection property (Ibrahim *et al*., 2005). The blank sample treated with binder had higher UPF (Table 2) but still far from offering a sufficient UVR protection (Table 3). Contrarily sample printed with the natural pigment pomegranate fruit 6 presents excellent UV protection its UPF being the highest (604.05) and %UVA and

%UVB transmittance being the lowest (2.54 and 1.06 respectively) among the pigments, while cutch 4 and lac dye-S 9 with their UPF values being 61.11 and 55.61 respectively still offer excellent protection. Annatto presents very good UV protection (UPF value 28.75). The UPF sharp increase observed in the above pigments 4, 6 and 9 can be attributed to the pigments structure (Figure 1): the significant impact of aromatic carboxy groups (lac dye), phenolic hydroxyl groups (pomegranate fruit and cutch) and the carotenoid structure (annatto) present in these molecules can be discussed in terms of the ability of them with these anionic groups to pick up, attract, interact and fix onto the binder/fabric matrix thereby enhancing the extent of blocking the open holes in the fabric structure, i.e. reducing the hole effect, minimizing the extent of UV-transmission and/or acting as UV-absorber (Ibrahim *et al*., 2005). Indigo with nitrogen in its structure (Fig. 1) does not practically offer any UV protection (UPF 0.01) its UPF value being the lowest practically zero among the pigments in accordance with similar studies (Ibrahim *et al*., 2007) on the effect of nitrogen containing additives on cellulose/wool blended fabrics. Contrarily in the molecule of the second N-containing pigment lac dye S the presence of additional hydroxy groups results in a sharp increase in UPF rating up to 50 offering excellent UV-protection as already mentioned before (Ibrahim *et al*., 2007). The UV absorption spectra of the pigments pomegranate fruit, cutch, lac dyes, annatto and indigo with the lowest Ultraviolet Protection Factor given in figure 3 can be correlated with the above pigment behaviour in the UVB region 275-315 nm (radiation reaching the earth's surface).

As it is concluded from Figure 3 the highest pigment absorbance is observed in the UVC region lower than 280 nm. This radiation does not reach the earth's surface. In the UVB region 275-315 nm the pigments absorption follows the range: Pomegranate fruit λ= 275.5nm, abs 2.46> cutch λ= 279.5 nm abs 0.95> lac dye S λ=279.5 nm abs 0.79 >annatto λ=295.5.5 abs 0.50 > indigo =279.5 nm abs 0.11.

Synthetic fibres

Many previous studies have concluded that good UVR protection can be provided by synthetic fibres dyed with high concentrations of synthetic dyes (Gambichler *et al*., 2001; Driscoll, 2000) while dyeing of synthetic fibres polyester, acrylic, polyamide with natural dyes has not been studied. In table 4,5,6 the UV protection factors of polyester, polyamide and acrylic samples dyed with the natural dyes pomegranate fruit, red sandalwood and annatto in various concentrations are presented.

From Tables 4, 5 the following are concluded

UV protection offered by the polyamide and acrylic fabrics is insufficient (UPF values 5.235 and 3.368 respectively). Dyeing with the natural dyes pomegranate fruit, red sandalwood and annatto UPF is increasing to good with increasing dye concentration achieving very good values at the highest concentration 6% omf indicating that UPF values were also correlated to the color strength of the dyed fabrics which is evaluated using K/S values. The structure of the above dyes (absence of ionic sulphonic groups, flexible long carotenoid chain, very low water solubility) favours the dyeing of these synthetic fibres with low hydrophilicity, mainly the of the most hydrophobic polyester fibre. The excellent UV protection offered by the undyed polyester fabric is apparently due to a UV absorbing agent built-in the mass of polyester during its spinning procedure. Dyeing of polyester with the natural dyes improves even more UPF reaching values >50. However increasing UV protection can be considered negligible in this case since protection class of undyed polyester fabric is excellent (Table 6).

The K/S values of the printed fabrics which are a measure of color depth seem to support the claim that higher colour depths increases UPF values. However, the relationship of K/S with UPF is limited to the same fabric type and the results cannot be generalized. A probable reason is the dependence of K/S on the absorbing properties of colorants in the visible region of the spectrum and that may not influence the absorption characteristics of colorants in the UV region.

K/S values were calculated using the equation:

$$K/S = \frac{(1-R)^2}{2R} = a \times c$$

Where: K= absorbance coefficient
S= scattering coefficient
R= % reflectance
c= dye concentration on the fabric
α= constant

Conclusion

The UV Protection Factor (UPF) of cotton fabrics printed with ten natural colorants of plant and insect origin was measured and correlated with their chemical structure and their UV-absorption spectra. The UPF values calculated varied widely from excellent to insufficient depending on the pigment used with pomegranate fruit presenting the highest and indigo the lowest one practically zero. The UV protection factors of polyester, polyamide and acrylic samples dyed with three natural dyes selected from the above colorants in various concentrations were also measured. Fibre composition was found to be the dominant factor affecting the UPF values in the case of synthetic fibres since polyester samples undyed/dyed offered excellent UV protection in all cases probably due to a UV absorbing agent built-in the mass of polyester during its spinning procedure.

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