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The role of natural dyes in the UV protection of fabrics made of vegetable fibres

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ABSTRACT

The safest protection from ultraviolet (UV) radiation exposure is offered by textiles including various apparels, accessories such as hats and shade structures such as umbrellas. Their protectiveness depends on fabric composition, (natural, artificial or synthetic fibres), fabric construction (porosity, weight and thickness) and dyeing (natural or synthetic dyes, dye concentration, UV-absorbing properties, etc.). In this study the UV-protection properties were investigated on fabrics made of vegetable fibres (cotton, flax, hemp and ramie), with different construction parameters (drapery and apparel fabrics), dyed with some of the most common natural dyes. The effect of a tannins-based mordant (the galls of *Quercus infectoria*) on UV-protection capacity was also tested. UV radiation transmittance of fabrics was measured by two methods: one based on the utilisation of a spectrophotometer equipped with an integrating sphere (*in vitro* test), and the other based on outdoor measurements taken by a spectroradiometer. Transmittance measurements were used to calculate the Ultraviolet Protection Factor (UPF).

Our results revealed that thick and dense (cover factor, CF > 94%) drapery fabrics made of vegetable fibres usually showed good UV-protection levels even if undyed. The use of the tannins-based mordant increased, even without dyeing, the UV-protection level up to the very good and/or excellent protection categories when fabric construction was suitable. Dyeing did not further increase the protection level.

Lighter fabrics, usually used for apparel, even showed high UV-protection level after just dyeing, provided that CF was above 94%.

Taking into account the high concentrations of dyes used in the present work and the utilisation of mordants containing tannins, slight differences in UV-protection capacity were detected among natural dyes tested and between the two different methods of transmittance measurement. However, UV-protection category defined from outdoor measurements was often higher than that calculated by the *in vitro* test, indicating an underestimation of the actual protection level of tested fabrics assessed by the latter.

1. Introduction

Fabric colour, as well as its construction (porosity, weight and thickness) and chemical composition [1–3], has a fundamental importance in attenuating solar UV radiation hence protecting human tissues. It is common knowledge that excessive UV exposure can be harmful for human health by inducing acute and chronic effects on skin, eyes and the immune system [4,5]. To avoid these health risks, it is important to reduce personal UV exposure. The safest protection is offered by textiles including various apparels, accessories such as hats, shade structures such umbrellas, awnings and baby carrier covers [6–11].

Dyed fabrics protect more than undyed ones and their protection levels rise with the increase in dye concentration [12]. In general, light colours reflect solar radiation more efficiently than dark ones [13], but part of the radiation penetrates more easily through the fabric thanks to multiple scattering. Most of the studies on this topic concern synthetic dyes.

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, but they are water-repellent and therefore uncomfortable to wear when temperatures are high (generally associated with high UV levels) [12,14]. Natural fibres are most commonly used for making not only summer clothes (lighter fabrics) and hats but also drapery and shade structures (heavier fabrics). Most research on the UV-protection properties of natural fibres has focused on cotton [14–19].

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Very few studies exist on the UV-protection properties of natural dyes in combination with natural fabrics [14,18,20–25] and most concern animal fibres. Taking into account that the use of non-allergic, non-toxic and eco-friendly natural dyes on textiles has become a matter of significant importance due to increased environmental awareness in order to avoid hazardous synthetic dyes [26], and that some natural dyes can also have natural antimicrobial activity [27,28], it is important to learn more about the UV-protection properties of a large number of natural dyes when combined with vegetable fibres, like flax, cotton and hemp. Very few natural dyes give strong colours on vegetable fibres without the aid of mordants, and a more eco-friendly natural dyeing can only be achieved by replacing metal mordants with natural mordants, like vegetable tannins or tannic acid [29]. The latter are water-soluble phenolic compounds that have been used on textiles for several hundred years and they continue to be used both as a pretreatment and after-treatment factor to increase wash fastness [30] and light fastness [31], e.g. on cotton fabrics. Nevertheless metal mordants like potassium alum and aluminium sulphate could also be used in eco-friendly natural dyeing as their environmental toxicity is almost nil [32].

The UV-protection efficiency of a fabric is defined by its Ultraviolet Protection Factor (UPF). The higher the UPF value, the greater is the fabric's protection level.

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 protection properties as shown in Table 1 [33].

The main aims of this work were: i) to investigate the UV-protection properties of natural fabrics made of different vegetable fibres (cotton, flax, hemp and ramie), with different weight/construction and dyed with some of the most common natural dyes; ii) to investigate the effects of the utilisation of tannin-based mordants during the dyeing procedure on UV-protection properties.

2. Materials and methods

The characteristics of the fabrics used in this study are shown in Table 2.

The lighter fabrics used in this experiment (C85, F160 and R130) were chosen among those usually used for summer clothing while the heavier ones (C210, C280, F260, H250 and H300) were drapery fabrics.

The cover factor(CF) of the fabrics, a parameter that measures fabric porosity (fabric porosity is calculated as 100-CF, where CF is defined as the percentage area occupied by warp and weft yarns in a given fabric area), was estimated by an image analysis technique. The fabric sample image was acquired by a high resolution scanner (HP Scanjet 7400C) and then processed by a computer. A suitable cut-off intensity value was used to separate the pixels representing open areas from those representing areas covered by yarns. The number of pixels falling above and below this cut-off value was determined and the percentage of covered area was then calculated.

Table 1
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 2
Parameters of undyed fabrics used in the experiment.

Fabric	Weight (g/m^2)	Count (warp × weft/cm)	Cover factor (%)		Weave	Abbreviation
			Undyed	Undyed-boiled		
Cotton	85	18 × 24	86.6	89.9	plain	C85
Cotton	210	24 × 47	100	100	twill	C210
Cotton	280	17 × 20	99.6	99.9	plain	C280
Hemp	250	13 × 13	98.4	99.5	plain	H250
Hemp	300	13 × 7	98.1	98.8	plain	H300
Flax	160	25 × 15	95	98.2	plain	F160
Flax	260	21 × 10	97.4	97.4	plain	F260
Ramie	130	23 × 24	95.9	97.2	plain	R130

2.1. Dyeing

In order to evaluate the effect of colour on UV transmission, the fabrics were all dyed with some of the most common natural dyes: madder (*Rubia tinctoria* L.), weld (*Reseda luteola* L.) and cochineal (*Dactylopius coccus* L.), except R130 and H250 that were not dyed with cochineal.

In all phases of dyeing the ratio between fabric dry weight and water solution weight was always 1:20.

The first operation of the dyeing procedure was the washing of all fabrics, boiling them with soda (concentration 5% on fabric dry weight) for a few minutes. The fabrics were then wrung out. To verify the influence of probable fabric shrinkage caused by such a high temperature, on CF, the latter was also estimated on these samples before starting the dyeing process. In the text these fabrics are designated "undyed-boiled".

In the dyeing process, dye was applied at 50 and 100% concentration for weld and madder and at 10% for cochineal on fabric dry weight, according to their different dyeable capacity.

As natural dyes do not have affinity for cellulosic fibres, two mordants were used. Fabrics were mordanted prior to dyeing by treating first with *Quercus infectoria* gall extract (10% on fabric dry weight) and then with potassium alum (20% on fabric dry weight) and soda (10% on fabric dry weight). The powder of galls of *Q. infectoria* is a natural mordant containing from 50 to 70% of tannins.

Fabrics were maintained in the bath with *Q. infectoria* for 6 h starting from a temperature of 80 °C to ambient temperature. In the second mordanting process with potassium alum and soda the fabrics were maintained for 3 h at boiling point.

After mordanting, fabrics were wrung thoroughly and dyed. Fabrics were placed in the dyeing solutions at room temperature. The temperature was raised to boiling point and dyeing continued at the boil for 1 h. For madder a dyeing was also performed maintaining the dye-bath at 80 °C for 1 h according to a common practice used by artisans to achieve a different shade to that from a dye-bath at 100 °C.

After dyeing fabrics were rinsed in a solution of acetic acid (2%) for 10 min at 30 °C, then washed using a non-ionic detergent and air-dried.

To verify the possible UV-absorbing capability of mordants, a sample of each fabric mordanted with *Q. infectoria* (in the text "undyed-mordant 1") and a sample mordanted with *Q. infectoria* plus potassium alum (in the text "undyed-mordant 2") were left undyed.

One sample of the two dyed fabrics (C85, F160) was washed with neutral soap at 30 °C and then rinsed to make a first evaluation of the modification of UV-protection capacity simulating the usual domestic cleaning treatments.

2.2. Measurements

In vitro UV-protection factor was determined on three samples (3 cm × 1 cm) cut from the centre of each fabric, undyed, undyed-boiled, undyed-mordant 1, undyed-mordant 2, dyed and washed, fixed in common slide frame and placed in a Jasco UV/VIS Spectrophotometer V-560, equipped with an integrating sphere to measure both direct and diffuse transmitted light. The sample was positioned at right angles to the light beams. Transmission measurements were made in the 250–400 nm range with a 1 nm step. UPF was calculated according to:

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

where S_λ is the solar spectral irradiance at noon for a typical summer's day in central Italy, E_λ is the CIE erythemal spectral effectiveness, T_λ is the spectral transmittance of each fabric sample and Δ_λ is the wavelength step. UPF computed on the basis of this laboratory procedure is designated $\mathrm{UPF_{Lab}}$. The three measurements of each sample were averaged and standard deviation was calculated.

As the *in vitro* test does not completely reflect true outdoor conditions, characterized by direct and diffuse solar radiation as well as radiation reflected by the surrounding environment, outdoor spectral measurements were taken using a double monochromator spectroradiometer (model SR9910-PC, Macam Photometrics Ltd., Livingstone, Scotland) in the 290–400 nm spectral waveband with 3 nm steps.

Measurements were taken around noon (solar zenith angle 44.2°), on a clear day (24th September 2009) at Sesto Fiorentino (Florence, Italy, latitude 43°49′08″ N, longitude 11°12′07″ E; elevation 40 m asl), with the diffuser normally oriented with respect to sunbeams.

Outdoor measurements were performed on all samples already tested in the laboratory except washed samples. Two measurements were taken for each fabric sample: one with the diffuser covered by fabric and one with diffuser uncovered. This approach, already used by Gelsor et al. [34] and by Grifoni et al. [24], allowed the UV transmittance to be calculated as the percentage of incoming solar UV radiation that reached the diffuser through the fabric. A linear interpolation was used to provide values in 1 nm increments.

As each outdoor measurement was very time consuming, it was only taken on one fabric sample to avoid variations in solar radiation characteristics during the set of measurements. However, the spectroradiometer diffuser allowed a wider surface to be detected than the *in vitro* test, minimising the variability due to possible non-homogeneity of colour and weave in the sample.

The UPF was then calculated according to equation (1). UPF computed on the basis of outdoor measurements is designated $\mathrm{UPF_{out}}$.

UPF values higher than 40 were reported as 40 corresponding to the highest UV-protection category (excellent protection, Table 1).

3. Results and discussion

Our results are discussed in respect to the Australian/New Zealand standard [33]. However the differing amount of solar erythemal UV radiation between Australia and Europe should be taken into account. The total daily ambient solar erythemal UV radiation on a cloudless summer's day in Australia is in the order of 20–30 MED (Minimal Erythemal Dose, defined as the threshold dose that may produce reddening of the skin [35]). Thus, clothing with a minimum UPF of 20–30 is required in order to reduce personal exposure to less than 1 MED when most of the day is spent outdoors. In Europe a UPF of 15 and 20 would be sufficient in the UK [36] and central Italy (where values of 20–21 MED are detected on summer days [37]), respectively.

3.1. Cover factor

The CF values of all undyed fabrics examined, except C85, always exceeded 94% (Table 2), corresponding to the threshold below which the UPF value of the fabric assumes values lower than 15 (corresponding to the minimum degree of UV protection according to the Australian/New Zealand standard, Table 1) independently of the UV-absorbing properties of the fibres and/or chemicals applied to them [38]. As already reported in a previous experiment by Grifoni et al. [24], the fabrics treated at boiling point, except for F260, increased their CF due to fabric shrinkage (see undyed-boiled values in Table 2); despite that the CF of C85 remained below the value of 94%. The highest CF value of C210 can be ascribed to the different weave. Indeed, with twill weave it is possible to achieve higher warp/weft density than with plain weave, so the macropores are smaller and UV radiation has less free space to pass through than in plain weaves [39].

3.2. In vitro measurements

Transmittance spectra of undyed samples (Fig. 1) pointed out how drapery fabrics (C210, C280, F260, H250 and H300) were characterized by low transmittance values that ranged between 3 and 14% at 296 nm and between 9 and 21% at 396 nm. On the contrary apparel fabrics showed higher transmittance values mainly at low wavelengths, with values ranging between 25 and 55% and 30 and 40% at 296 and 396 nm, respectively.

Among apparel fabrics the distribution of transmittance spectra, in the UV-B band (280–320 nm), moving from the highest (C85) to the lowest (F160) seemed to be inversely correlated to fabric CF and weight and independent of fibre type. This could also explain the difference between apparel and drapery fabrics. In general terms natural plant fibres such as flax, hemp and cotton do not differ in UV transmittance properties and are not perfect UV filters [18,40,41]. Nevertheless, if CF exceeds 94% and fabric is heavy, as for C280, H250 and H300, the UPF, also in untreated fabrics, can even rise to values corresponding to very good protection category (Fig. 2 and Table 1). Indeed an increase in weight per unit area also decreases fabric porosity [40].

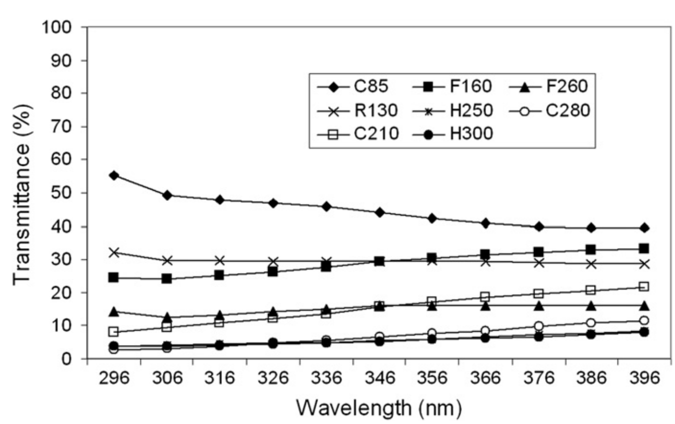

Fig. 1. Transmittance of undyed fabrics obtained by *in vitro* test as the mean of three samples. A subsample of transmission values (10 nm step) is shown just for graphical representation.

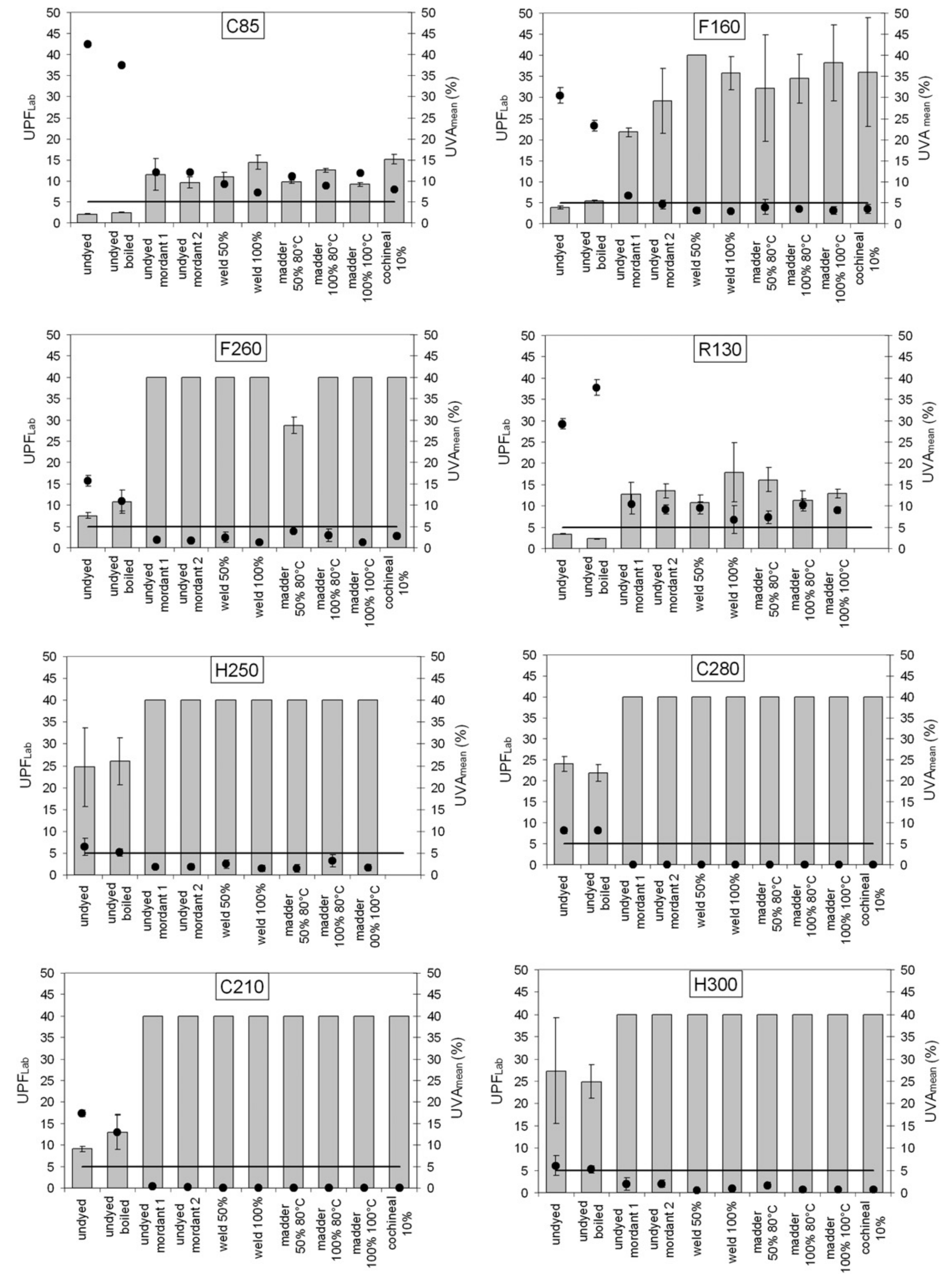

Fig. 2. UPF_{Lab} (bar) and UVA transmittance (black dots) for undyed, undyed-boiled, undyed-mordant 1, undyed-mordant 2 and dyed fabrics. Bars and dots are the means of three measurements. Horizontal black line represents the UVA transmittance threshold established by the European standard for Sun Protective Clothing (Gambichler et al. [45]).

The increase in CF caused by the only boiling treatment did not produce a relevant increase of $UPFs_{Lab}$ and did not usually modify the related UV-protection category (see $UPFs_{Lab}$ of undyed and undyed-boiled fabrics in Fig. 2).

Contrarily to the results obtained by Grifoni et al. [24] and Feng et al. [18] in similar experiments in which the mordants used during the dyeing process did not show any UV-absorbing capacity, in the present experiment the first mordanting markedly affected the UV-protection properties of all fabrics. Apart from C85 and R130, in which the UPF_{Lab} value remains lower than 15 (the minimum protection level as defined in Table 1), the protection category of all other fabrics moved from no protection to good or excellent protection for F160 and C210, respectively, and from good protection to excellent protection for the remaining fabrics. The different effect of mordanting in this experiment in comparison with those previously mentioned was ascribable to the use of a natural mordant containing tannins. Indeed, the main constituents of the galls of *Q. infectoria* are tannins (50–70%), gallic acid and ellagic acid [42]. As already stated by many authors [43,44], tannins absorb UV radiation with an efficiency similar to carotenes and anthocyanins, and provide the same protection from UV damage that accessory pigments do.

Since drapery fabrics (C280, F260, H250, H300 and C210) had already reached the maximum UV-protection category after the mordant-1 application, the further adding of mordant-2 and dyes did not produce any improvement in UV-protection category as defined in Table 1.

In C85 none of the dyes at any concentration allowed the minimum UV-protection category to be reached, even if the mean UPF_{Lab} value of the weld 100% and the cochineal 10% samples was close to 15.

Similar behaviour was also shown by R130in which, however, the mean UPF_{Lab} value of weld 100% dyed fabric and madder 50% 80 °C dyed fabric slightly exceeded the threshold of 15, corresponding to a good UV protection, even if the UPF variability was high.

Regarding F160, a high variability of $UPFs_{Lab}$ values within each dyed fabric can be noted as a consequence of the not homogeneous dye uptake. However all dyes and mordant 2 increased the average UPF_{Lab} values with respect to UPF_{Lab} of undyed-mordant 1 up to the very good or excellent protection category.

In apparel fabrics no relevant differences in $UPFs_{Lab}$ were detected among different dyes, concentrations (weld 50 and 100% and madder 50 and 100%) or dye-bath temperature (madder 100% at 80 °C and 100 °C) of the same dye (Fig. 2).

Generally, in all the cases in which UPF_{Lab} reached at least the good protection level UVA transmittance was also below 5% (Fig. 2), which the European standard for Sun Protective Clothing [45] and also the Chinese National standard GB/T18830-2002 [25] consider the threshold above which photosensitive skin disorders, like chronic actinic dermatitis and solar urticaria, can be aggravated [46]. Nevertheless high UPFs do not necessarily imply low transmission in UVA wavelengths, as already found by Gambichler et al. [16,45] and Grifoni et al. [24]. In this study, for example, F160 undyed-mordant 1, H250 undyed, C280 undyed and undyed-boiled and H300 undyed that had $UPFs_{Lab}$ corresponding to good or very good protection, showed UVA transmittance values slightly above 5%.

Generally, the washing of dyed fabrics C85 and F160 did not significantly modify $UPFs_{Lab}$ (Fig. 3), indicating that at least the first washing did not remove dye or that the dye loss was negligible. Only the F160 dyed with madder 50% 80 °C showed a significant decrease of UPF_{Lab} moving from the very good to good protection category.

3.3. Outdoor measurements

Outdoor measurements are useful for evaluating the protection offered by fabric in natural conditions. UV-protection categories of drapery fabrics identified by $UPFs_{out}$ (Fig. 4) were generally the same as those obtained in the laboratory even if an enhancement of UV protection with respect to laboratory results was detected for C280 and H300 undyed-boiled, which changed from the very good to excellent protection category. With apparel fabrics mean $UPFs_{out}$ of weld 100% and cochineal 100% of C85 were slightly higher than the corresponding UPF_{Lab} and above 15, reaching the lowest protection category, even if the UVA transmittance remained above 5%. The reaching of a UV-protection category of a fabric with a CF < 94% could be ascribed to the different ratio of the diffuse to direct radiation incident on the fabric that occurs outdoors in comparison with laboratory conditions. While

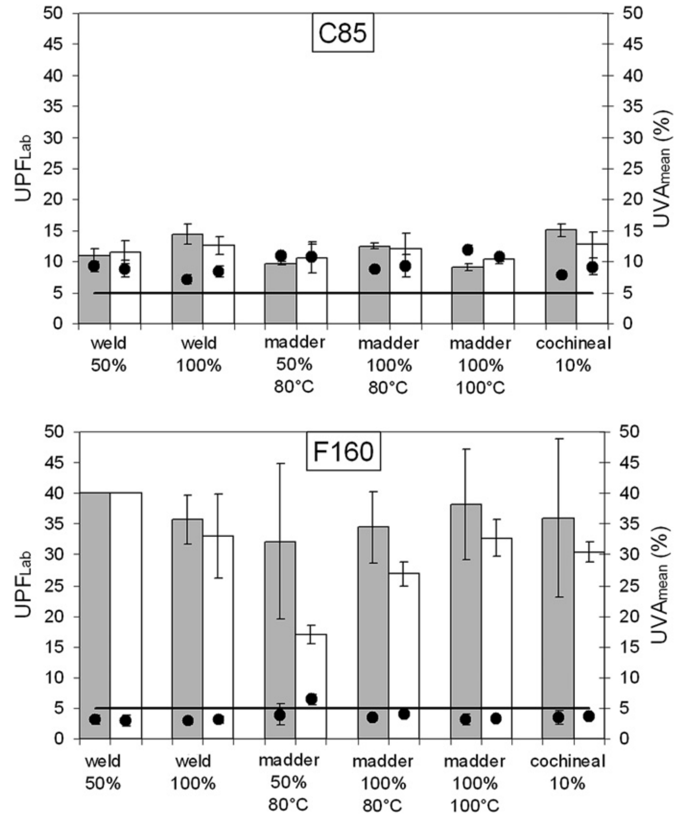

Fig. 3. UPF_{Lab} (bar) and UVA transmittance (black dots), for dyed C85 and F160 fabrics. Grey and white bars represent unwashed and washed fabrics, respectively. Bars and dots are the means of three measurements. Horizontal black line represents the UVA transmittance threshold established by the European standard for Sun Protective Clothing (Gambichler et al. [45]).

transmittance in the *in vitro* test is determined using an artificial direct light source, in the outdoor experiment the fabric received both direct and diffuse sun radiation; the latter component, not taken into account in the *in vitro* test, can modify fabric transmittance because, coming from all directions, it is differently influenced in its pathway through the fabric with respect to direct perpendicular light. This difference in the light source between outdoor and *in vitro* test can also justify the reaching of the good protection category of undyed-mordant 1, undyed-mordant 2, weld 50% and madder 100% 100 °C of R130, classified as no UV protection by the *in vitro* test, and the reaching of excellent category of madder 100% 80 °C and madder 100% 100 °C of F160, classified as very good UV protection by the *in vitro* test. These differences among the UV-protection categories as defined by the UPF_{out} and UPF_{Lab} were also detected by the same authors [24] in a previous work on natural fabrics dyed with natural dyes, confirming that the actual UV protection of a particular textile would always be greater than that obtained using *in vitro* spectrophotometry, which operates under the worst-case conditions, with collimated radiation at right angles to the fabric.

The other samples maintained the same protection category apart from the weld 50% of F160 and weld 50% of F260, which showed an unexpected decrease in outdoor conditions, changing from the very good to good category. This result can probably be ascribed to the not homogeneous dye distribution over the fabric and, therefore, to having performed the *in vitro* test and outdoor measurements on samples with a different UV-protection capacity.

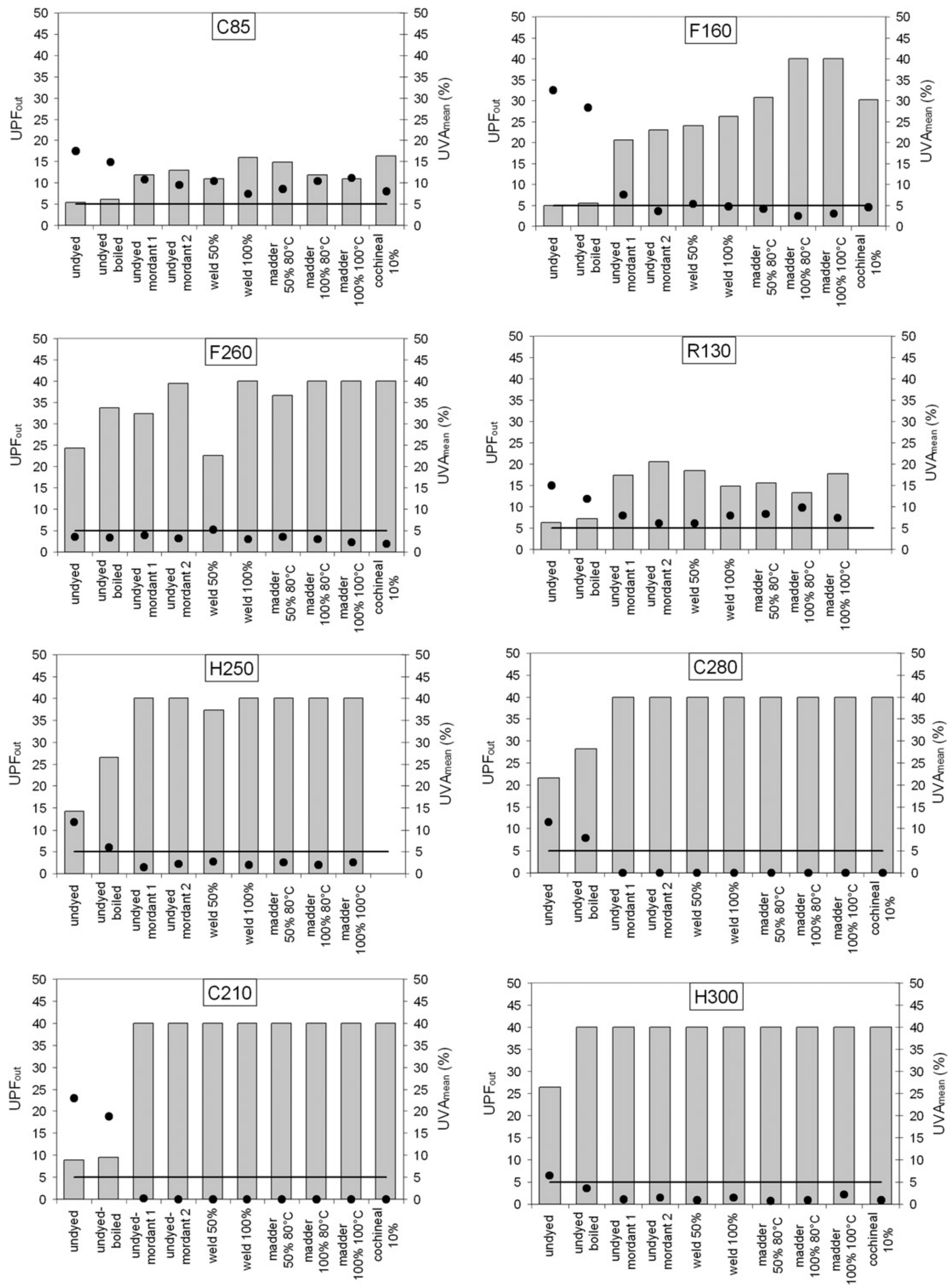

Fig. 4. UPF_{out} (bar) and UVA transmittance (black dots) for undyed, undyed-boiled, undyed-mordant 1, undyed mordant 2 and dyed fabrics. Horizontal black line represents the UVA transmittance threshold established by the European standard for Sun Protective Clothing (Gambichler et al. [45]).

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