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COMMUNICATIONS IN DEVELOPMENT AND ASSEMBLING OF TEXTILE PRODUCTS

UV-protective and IR-protective properties of natural dyed textiles – an evaluation on the impact of metal mordanting

Submitted 20 April 2026, accepted 8 July 2026, published 23 November 2026
https://doi.org/10.25367/cdatp.v7.a244

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Abstract

The ability of natural dyeing to implement UV and IR protective properties on textile fabrics is investigated. For this, dyeing is performed on three different types of fabrics made from wool, cotton and Lyocell fibers. As natural dyes, commercially available extracts from madder root and logwood are used, which are combined with three different types of metal mordanting agents containing iron, copper or titanium. The dyeing procedure is done as pre- or meta-mordanting procedure. Optical transmission of dyed textiles is determined for a spectroscopic range from 220 nm to 1400 nm by using a photospectrometer equipped with an integrating sphere. It was demonstrated that by selected combinations of natural dye with a specific metal mordant, the optical transmission in the near IR range can be significantly decreased. Together with an as well decreased UV transmission, textiles with a combined protection for UV light and IR light can be realized. The modified interaction with IR light might by also of interest for camouflage applications. In conclusion, the presented results can be the starting point for future development of light protective and camouflage textiles and clothing.

Keywords

UV protection, Light protection, Natural dyes, Madder, Logwood, UPF, Optical spectroscopy, Optical transmission, Lyocell

Mahltig, B., Ostheim, A., Ratovo, K., Klinkhammer, K., Fischer, M., Bendt, E. (2026). UV-protective and IR-protective properties of natural dyed textiles – an evaluation on the impact of metal mordanting. *Communications in Development and Assembling of Textile Products*, Vol. 7. 124-141.
https://doi.org/10.25367/cdatp.v7.a244
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ISSN: 2701-939X

1 Introduction

Sunlight contains additionally to visible light significant amounts of UV light and near infrared light (NIR light). Both types of those non-visible radiation can have significant impacts on human skin health [1-3]. The negative impact of UV light is widely known and can be described by the following buzz words – sunburn, skin cancer and skin ageing [4]. The impact of infrared light is usually smaller compared to the impact of UV light and infrared light is also less present in daily discussion [5,6]. However, even for infrared light a certain negative impact on skin health is reported. These reports are often related to accelerated skin ageing [3,7,8]. With this background, textiles offering both UV and IR light protective properties are demanded products [9-11]. Protective properties are reached by low transmission values for these types of radiation, so the amount of radiation reaches the skin is drastically reduced. For UV-protective textiles, several well-established methods are available using different types of UV-absorber [12]. Most prominently is the use of the inorganic UV absorber titanium dioxide TiO_2 [12,13]. Other inorganic UV absorbers are e.g. zinc oxide ZnO or cerium dioxide [13-15]. Also, organic UV-absorbers can be used which can be applied in wet-processes similar to dyeing processes [16,17]. In contrast, for protection against IR light only fewer effective materials are available [18]. Some IR protective systems are based on reflective pigments as aluminum effect pigments or pearlescent effect pigments [19-21]. Other effective IR protective systems are based on sol-gel coating [22]. As IR light absorptive materials, pigments or special types of dyes can be used [18,23]. Often IR manipulating compounds are mentioned in relation to camouflage materials and applications [24,25]. An interesting approach to reach UV protective properties on textiles is the dyeing with natural dyes and especially in combination with bio-based mordanting agents new environmentally friendly textile materials can be realized [26,27]. The application of natural dyes is often connected to the production of environmental and bio-based products [28,29]. Nevertheless, several disadvantages in the use of natural dyes should be mentioned, as cost structure, availability, limitation in color shades and limited fastness properties [30]. By application of natural plant dyes in combination with tannin containing mordants, textiles with camouflage properties covering a broad spectral range from UV to near infrared light can be realized [31]. The application of natural dyes for creating UV protective textiles is broadly investigated and well reported in literature [32,33]. Nevertheless, the use of natural dyes for realization of protective textile against both UV light and near-infrared light is less investigated and reported. With this background, the current investigation aims on the evaluation, if the application of natural dyes in combination with different metal mordant agents can be used to introduce both UV and IR protective properties to different textile fabrics in one application step. In the current study, special focus is set on the influence of the metal mordant and the mordanting procedure on the absorption of near-infrared light. As textile fabrics, woven materials from wool and cotton and a knitted Lyocell fabric are considered. While the natural dye application is widely reported on various textile materials, the dyeing of Lyocell using natural dyes is less investigated [34-37]. Wool fibers are protein based and exhibit different interactions to dye molecules and mordanting agent compared to the cellulosic based cotton and Lyocell fibers. For metal mordanting agents mainly an interaction of the metal ions with the amide unit of the protein structure by complexation is proposed [38,39]. In contrast, on cellulosic fibers the interaction to the metal mordant is based on the complex formation of hydroxy group to the metal ions [40,41]. Cotton and Lyocell have as main component cellulose and are for this in many properties quite similar. However, the dyeing behavior of those two fibers is not totally the same due to different crystallinity, surface structure and porosity [42,43]. As natural dyes, two commercially available types are considered – extracts from madder root (*Rubia tinctorum L*) and logwood (*Haematoxylum campechianum*). Red madder (C.I. Natural Red 8) from madder root con-

tains several color components based on the chemical structure of anthraquinone. Main color components of madder root are alizarin, purpurin, munjistin and pseudopurpurin (compare their chemical structures in Figure 1 [44-46]. The main color component from logwood (Natural Black 1; C.I. 75290) is Hematein which is formed by oxidation from Hematoxylin [44,47,48]. The chemical structure of Hematein is given in Figure 2. By view on the chemical structures of these color components, metal ions from mordanting agents can bond to different functional groups. Possible is the complex formation to two neighboring hydroxy groups in the dye molecule. Alternatively, the complex formation to a keto group and neighboring hydroxy group is possible [40,41,49]. As mordant agents, three different water soluble salts from iron Fe, copper Cu and titanium Ti are investigated in their application in pre- or meta-mordanting. Reference samples are produced by dye applications without mordanting. By this, 15 dye recipes are evaluated in their effectivity on three types of textile fabrics from wool, cotton and Lyocell fibers. Finally, it is shown that with specific combination of natural dye and metal mordant the transmission of UV light and near IR light of the textile fabric can be simultaneously decreased. The realization of a certain IR protective effect by application of natural dyes is possible and this can be a promising starting point for future development of UV/IR light protective textile materials.

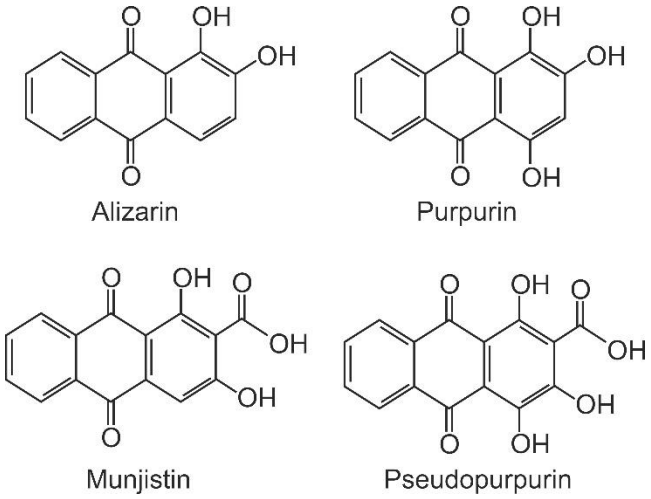

Figure 1. *Chemical structures of main color components from madder root.*

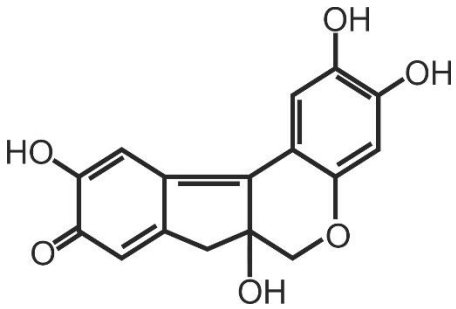

Figure 2. *Chemical structure of Hematein as main color component in logwood.*

2 Experimental Section

2.1 Materials and Sample Preparation

As textile materials woven fabrics from wool and cotton are used. The weight/area of these fabrics 230 g/m2 for the cotton fabric and 180 g/m2 for the wool fabric. Further, investigated is a knitted fabric from Lyocell fibers with a weigh/area of 180 g/m2. The fabric structure is shown by SEM images of different magnifications in Figure 3. The more open fabric structure of the Lyocell knitted fabric is clearly visible in the microscopic image.

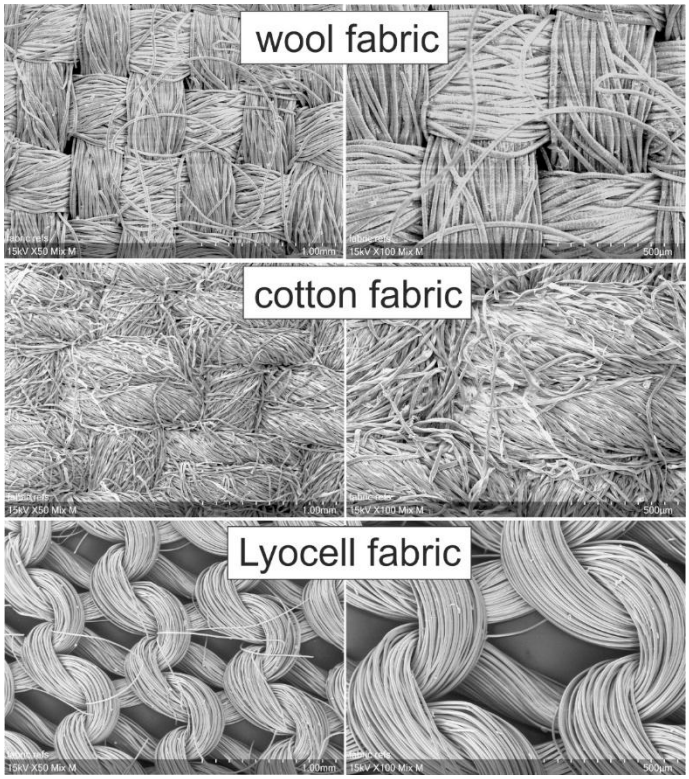

Figure 3. *Microscopic images of used fabrics. Shown are the fabric images recorded in different magnifications.*

As natural dyes, extracts from madder root (*Rubia tinctorum*) and logwood (*Haematoxylum campechianum*) are used. Both dye extracts are supplied as dry powders by the company Pflanzenfärbershop (Hückelhoven-Baal, Germany). The coloration of both powders can be described as red/brown to brown and a good water solubility is reported. Further information on these products together with detailed dyeing recipes are available on the supplier web-pages [50-53]. The IR spectra of used dye extracts are reported earlier and are public available [54,55]. Optical spectra recorded from aqueous solution of the dye extracts are presented in Figure 4. In aqueous solution both dye extracts exhibit no absorption of light in the range of 700 nm to 1400 nm. In the range of visible light, the madder extract solution exhibits an absorption maximum at 505 nm and for the logwood extract an absorption maximum is observed at 557 nm.

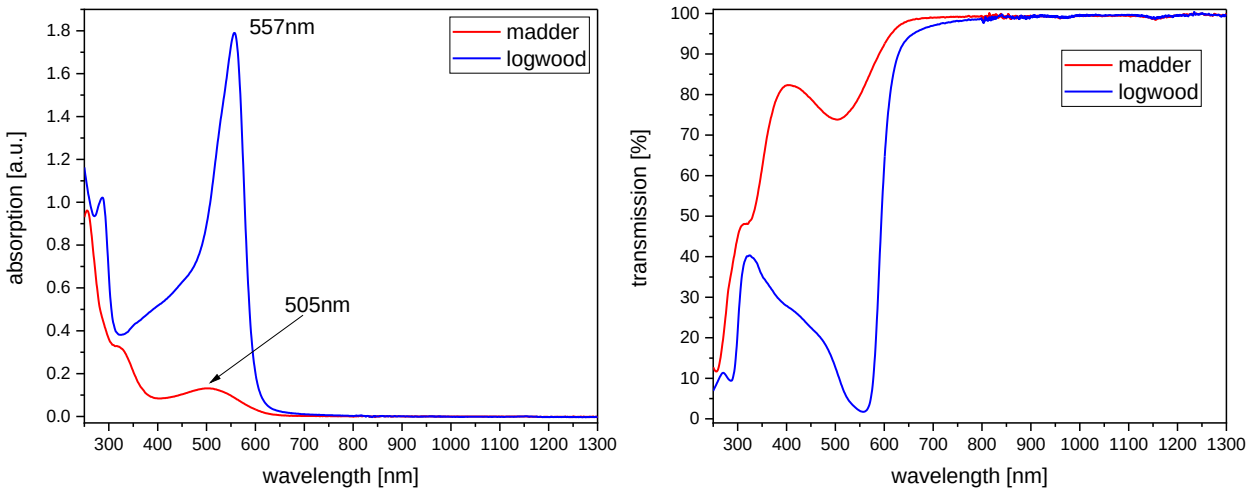

Figure 4. *Optical spectra of aqueous solutions of the used dye extracts. The aqueous solutions are prepared with a concentration of 0.1 g/L dye extract. The optical spectra are recorded and presented as absorption spectra and spectra of transmission.*

The dye extracts are applied in a concentration of 5%-ofw and in combination with three different metal mordant agents (Table 1). The mordanting agents are applied in a concentration of 3%-ofw in pre- or meta-mordanting procedure. As process temperature 80°C is set for a duration of 60 minutes. As dyeing machine an Ahiba Polymat from Datacolor GmbH (Marl, Germany) is used. A detailed description of used dyeing procedures, dyeing recipes, pre-treatment, after-treatment and drying procedure is published recently [56].

Table 1

Overview on used metal mordanting agents.

No.	*Name*	*Formula*	*Supplier*	*Molecular weight M_w (g/mol)*
1	Iron (II) sulphate	$FeSO_4$ $7H_2O$	Merck (Darmstadt, Germany)	278.0
2	Copper (II) sulphate	$CuSO_4$ $5H_2O$	Carl Roth GmbH (Karlsruhe, Germany)	249.7
3	Titanium (IV) dioxalate	TiC_4O_8	Jeromin Shop (Mannheim, Germany)	223.9

2.2 Analytics

The optical transmission spectrum of the textile fabrics is determined by using a photospectrometer UV-2600 (Shimadzu), which is equipped with an integrating sphere. For measurement of the optical transmission of textile samples, the use of such an integrating sphere is absolutely necessary, because additionally to the directly transmitted light also the diffusively transmitted light has to be recorded [57]. The transmission spectrum is recorded for the spectral range from 220 nm to 1400 nm. By this, the spectral range of UV light, visible light and the near infrared light (NIR) area is covered. The wavelength interval is set to 1 nm. For signal detection two different types of detectors are used. These detectors

are switched at a wavelength of 800 nm. To visualize the UV protective properties of the fabrics additionally to the evaluation of the transmission spectra also the UPF – protection factor is calculated by using equation (1) [58]. For this calculation, the transmission values recorded in the spectral range from 290 nm to 400 nm are used, which are determined in that range with a wavelength interval of 5 nm.

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

The values E_λ (solar UVR spectral irradiance and S_λ (relative erythemal effectiveness) are parameters at the wavelength λ according to standard AS/NZS 4399:1996. Δ_λ is the wavelength interval of the optical transmission measurement. T_λ is the determined optical transmission of the fabric at wavelength λ. The same photospectrometer is used to record the optical spectra of aqueous solutions of dye extracts (presented in Figure 2). For these measurements dye solutions with a concentration of 0.1 g/L are prepared and a quartz glass cuvette is used as vessel during the optical measurement. For recording of microscopic images (presented in Figure 3), a scanning electron microscope SEM TM4000 Tabletop (Hitachi, Japan) is used.

3 Results and Discussion

For sample evaluation, optical spectra of transmission with a spectral range from 250 nm to 1300 nm are compared. First, as reference materials the transmission of undyed fabrics are compared to dyed fabric without mordanting application. Following, samples dyed with madder extract in combination with different metal mordants are presented. Finally, the results for logwood dyed samples with different mordants are shown. All prepared fabric samples exhibit a strong coloration which is documented broadly in a previous publication [57]. This earlier publication supports a full documentation on coloration by sample photographs, K/S-measurements and CIELab measurements. These data are supported for dyed fabrics but also for reference fabrics without any treatment or a treatment with mordanting agents only. By application of mordanting agents a certain coloration of the samples can be reached but this coloration is clearly weaker compared to the coloration reached after dyeing procedures. Due to this earlier publication, in the following sub-sections mainly the interaction of the prepared fabrics with UV light and IR light is discussed while the interaction with visible light is not considered in detail again. The comparison of optical spectra recorded from aqueous dye solution (Figure 2) with the following presented spectra recorded from dyed textile samples exhibits significant differences. In fact, the chemical surrounding of a dye molecule solved in water is clearly different from the surrounding on a textile fiber. This different surrounding leads to a clearly different absorption property. However, over-interpretation of optical spectra should be avoided in this respect.

3.1 Optical transmission without metal mordanting

The optical transmissions of wool fabrics untreated and after dyeing with logwood or madder extract are compared in Figure 5. The dyeing is performed without usage of metal mordant agents. The undyed wool fabric exhibits nearly zero transmission for UV light below 300 nm corresponding to a quite high UPF value of 37 which is in good correspondence to values reported for wool in literature [59]. By dye application, the transmission for UV light is decreased to values <5% over the complete UV range corresponding to UPF values > 50. The transmission for infrared light is for all three compared wool samples around 35%, so an additional IR protective effect can not be implemented by this dyeing application.

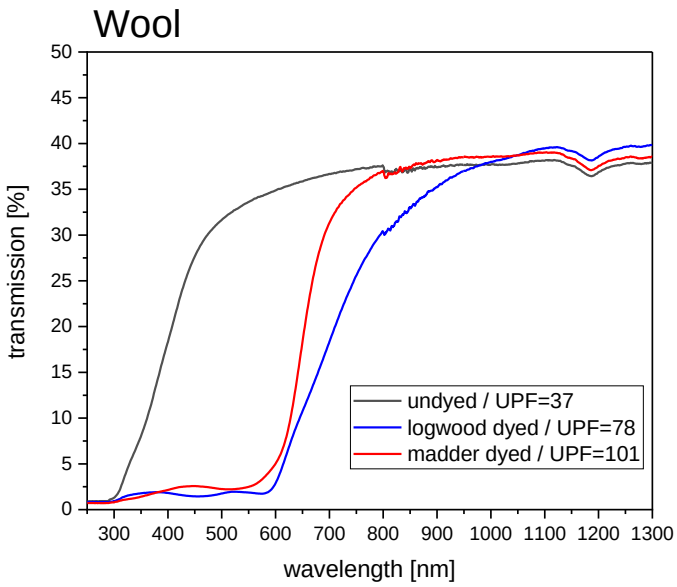

Figure 5. *Optical transmission of investigated wool fabric and related UPF values. Compared is the undyed wool fabric, with wool fabrics dyed with logwood extract and madder extract. The dyeing is done without addition of metal mordanting agents.*

The figure 6 presents the optical transmission of the undyed cotton fabric and dyed cotton fabrics without mordant application. Due to the denser structure and the higher weight/area of the cotton fabrics compared to the investigated wool fabric, the overall transmission of the cotton fabric is lower and does not exhibits a transmission of more than 25%. Nevertheless, for the undyed cotton fabric a quite high UV transmission is determined. For the UVB range below 300 nm a UV transmission of around 5% is determined. According to this, the UV protective properties of the investigated undyed cotton fabric can be considered to be in the range of low to medium, which is also presented by the UPF value of 11. This low UV protective property of cotton compared to wool is well known and is related to the different chemical composition of the cellulosic cotton compared to the protein based wool fibers [60]. After dyeing with the natural dye extracts, the UV transmission of the cotton fabric is drastically decreased and excellent high UPF values of >50 are reached (Figure 6). This result is in good accordance with published results for dyeing cotton knit fabrics with several natural dyes under special consideration of madder [61]. However, the optical transparency or IR light is nearly not changed by the applied natural dyes. Only for the application of logwood extract, a small decrease in transmission in the spectral range of 800 to 1000 nm is determined (Figure 6).

Due to the open knitted structure of the Lyocell fabric, the undyed Lyocell fabric exhibits higher optical transmission values in the range of 25% up to nearly 50% (Figure 7). The open structure of the investigated used knitted fabric is related to a lower cover factor tor this fabric. For this, here no UV protection can be determined corresponding to a low UPF values of only 2. By dyeing with madder or logwood, the transmission of the Lyocell fabric for UV light can be decreased to a certain amount but also by this decrease the UV protection is not significantly improved with UPF values increased to 5. The transmission of near infrared light is mostly not affected by dyeing the Lyocell fabrics with both investigated natural dye extract without addition of mordanting agent.

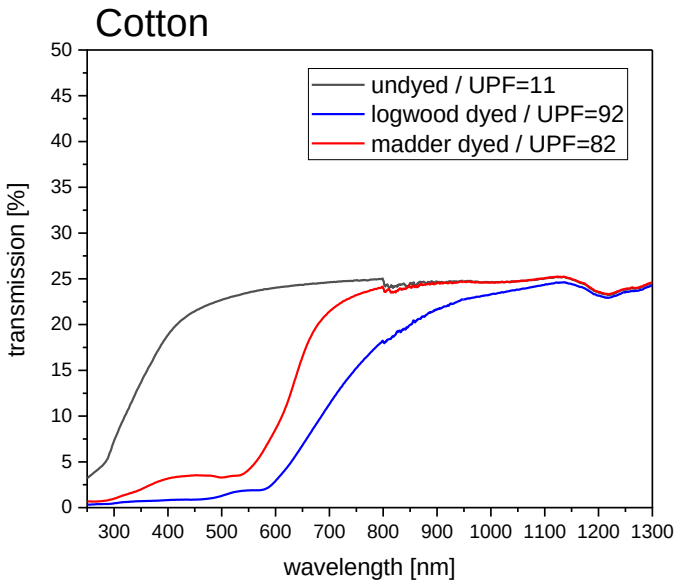

Figure 6. *Optical transmission of investigated cotton fabric and related UPF values. Compared is the undyed wool fabric, with wool fabrics dyed with logwood extract and madder extract. The dyeing is done without addition of metal mordanting agents.*

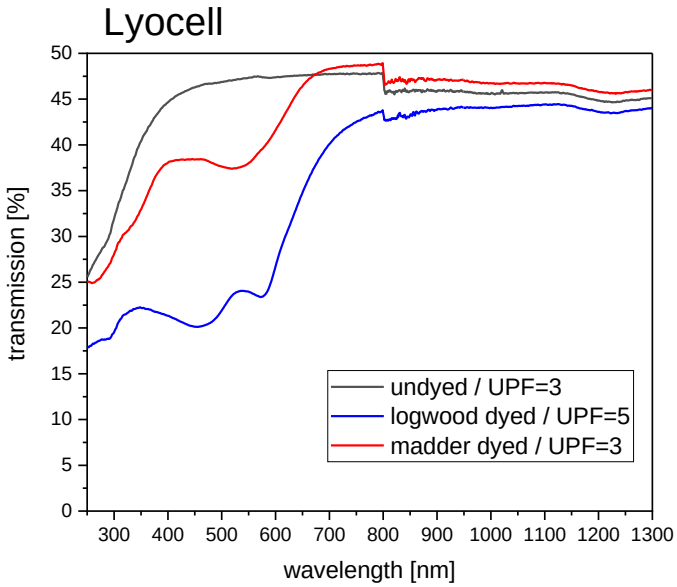

Figure 7. *Optical transmission of investigated Lyocell fabric and related UPF values. Compared is the undyed wool fabric, with wool fabrics dyed with logwood extract and madder extract. The dyeing is done without addition of metal mordanting agents.*

3.2 Optical transmission for madder dyeing and metal mordanting

The optical transmission for madder dyed wool fabrics in combination with different metal mordant agents are compared in Figure 8. For all samples over the complete UV range transmission values <5% are determined leading to high UPF values >50, so a sufficient UV protection can be stated for all

wool/madder samples. In contrast to this, the interaction with near infrared light differs clearly depending on the type of used mordanting agent and applied mordanting procedure (pre- or meta-mordanting). By combination with copper mordant, the transmission for IR light is clearly decreased in the IR range up to 1100 nm, in comparison to the wool sample dyed with madder extract without mordant. This effect of copper mordant is stronger for the meta-mordanting procedure.

Also, for the combination of iron mordant, a certain effect on IR transmission can be stated but this effect is weaker compared to the effect with copper mordant. The combination with titanium mordant does not lead to a decrease in IR transmission of the dyed wool fabric. However, by combination with mordants containing copper or iron, the interaction of the treated fabrics with IR light can be influenced. The observed decrease of IR transmission might not be sufficient for IR protective applications but useful in the field of IR camouflage materials.

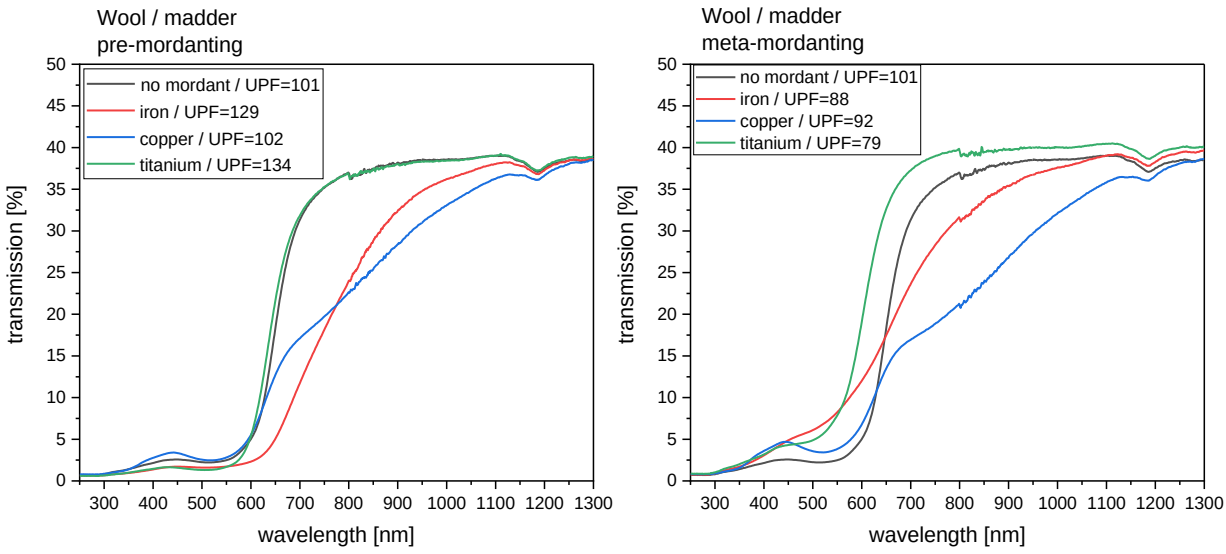

Figure 8. *Optical transmission and related UPF values of investigated wool fabric dyed with madder extract. Compared is the dyed wool fabric without mordanting agents, with wool fabrics dyed with madder extract and different metal mordants. The mordanting procedure is done as pre-mordanting (left) and meta-mordanting (right).*

Figure 9 summarizes the transmission spectra of cotton fabrics dyed with madder in different combinations with different metal mordants. Like the wool fabrics, also for cotton with application of the mordant agents based on copper or iron a decrease in IR transmission in the spectral range till 1100 nm can be reached. However, this effect is less significant as for wool. Especially with the pre-mordanting process with iron mordant, the decrease in IR transmission is stronger compared to the effect with copper mordant. Beside type of dye extract and mordant, also the fiber type has in this procedure an influence on the reached interaction with infrared light.

A totally different result is gained for the madder application on Lyocell knitted fabrics (Figure 10). After dyeing in presence of mordant, the transmission for UV light is clearly decreased but no values below 15% are reached. Accordingly, the UPF values are only slightly increased to values of 4 to 5, so even after dyeing no sufficient UV protection is gained with the knitted Lyocell fabric. Further, no influence on the transmission of IR light can be determined for the dye/mordant combinations on the Lyocell fabric.

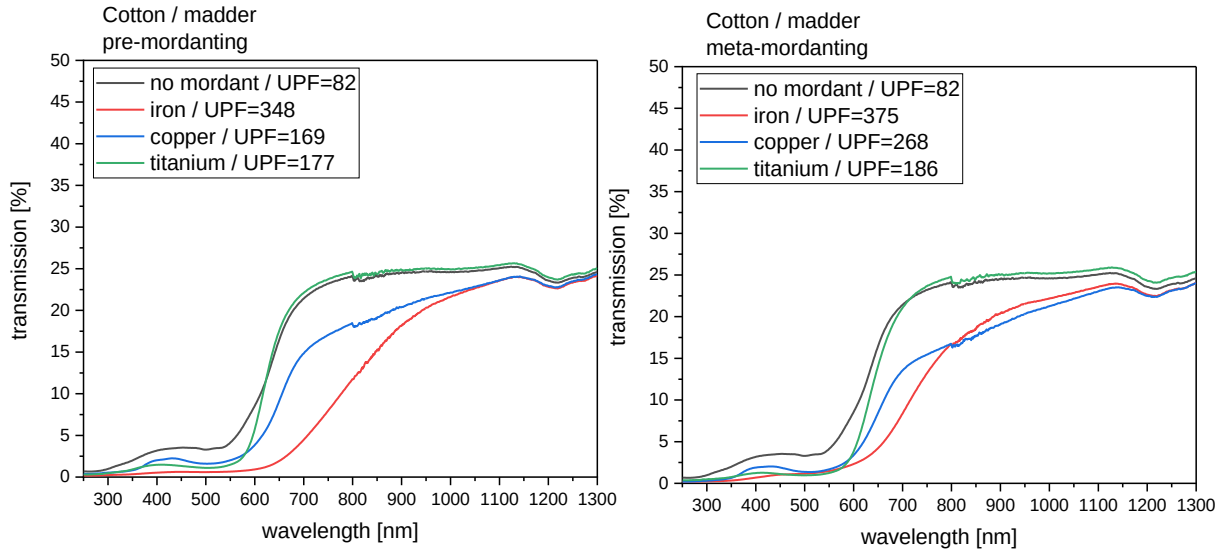

Figure 9. *Optical transmission and related UPF values of investigated cotton fabric dyed with madder extract. Compared is the dyed wool fabric without mordanting agents, with wool fabrics dyed with madder extract and different metal mordants. The mordanting procedure is done as pre-mordanting (left) and meta-mordanting (right).*

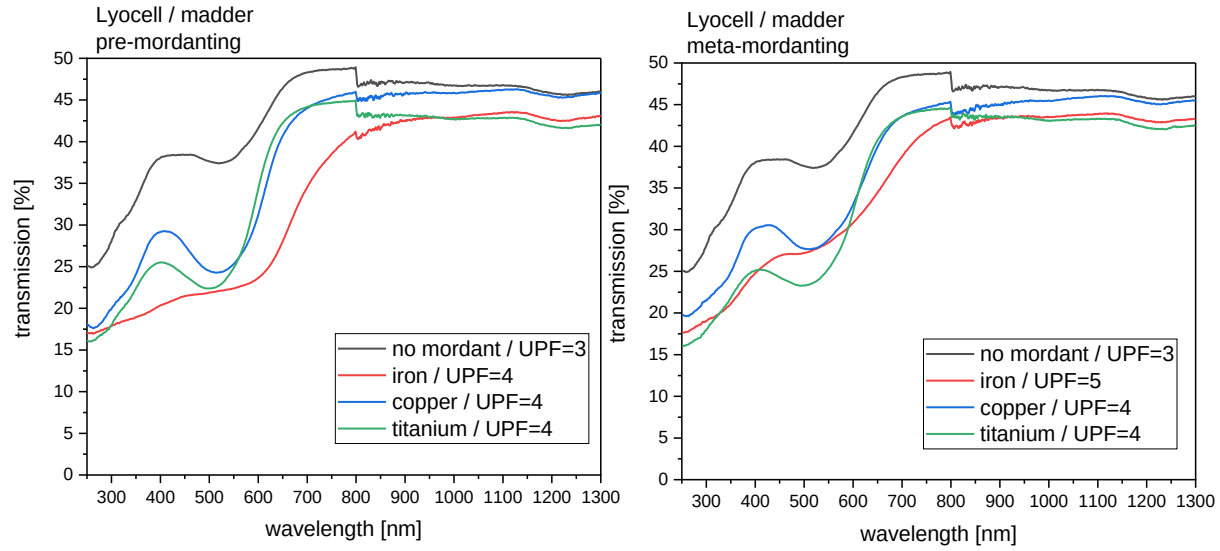

Figure 10. *Optical transmission and related UPF values of investigated Lyocell fabric dyed with madder extract. Compared is the dyed wool fabric without mordanting agents, with wool fabrics dyed with madder extract and different metal mordants. The mordanting procedure is done as pre-mordanting (left) and meta-mordanting (right).*

3.3 Optical transmission for logwood dyeing and metal mordanting

The optical transmission of wool fabrics dyed with logwood extract in presence of different metal mordants are presented in Figure 11. For all applied logwood/mordant combinations, low UV transmission and according high UPF values >50 are observed. In comparison, the interaction with infrared light is strongly influenced by the type of mordant applied together with the logwood extract on wool. By application with the titanium mordant, mostly no change in the transmission of IR light is determined.

However, the application of logwood with copper or iron mordants leads to a broad decrease in IR transmission. Here, strongest decrease is reached with the copper application in pre-mordanting procedure. In comparison with iron mordant lower IR transmission is gained by the meta-mordanting procedure.

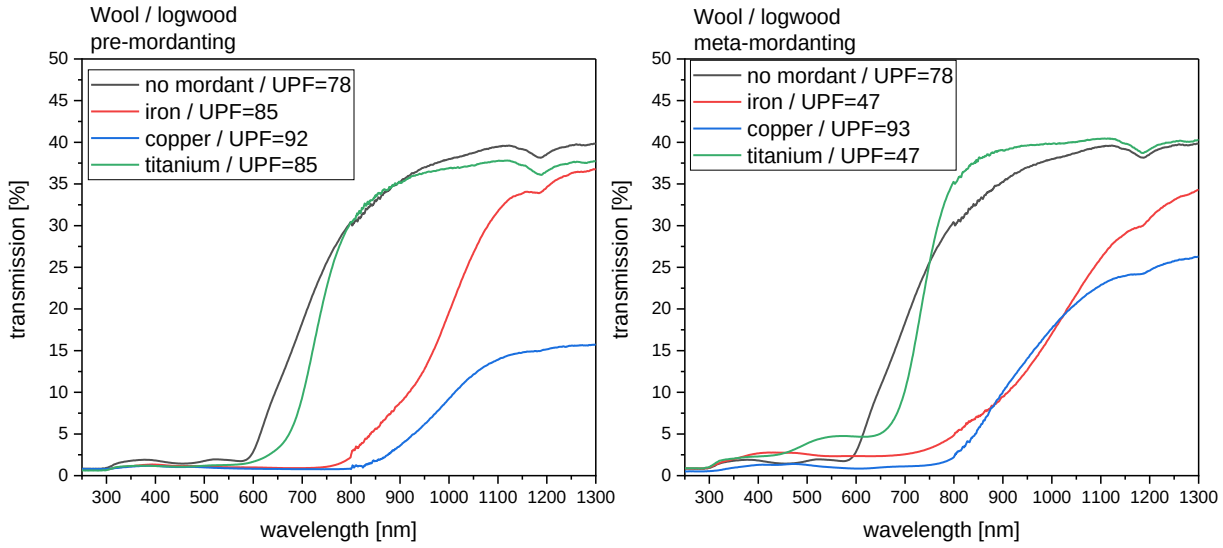

Figure 11. *Optical transmission and related UPF values of investigated wool fabric dyed with logwood extract. Compared is the dyed wool fabric without mordanting agents, with wool fabrics dyed with madder extract and different metal mordants. The mordanting procedure is done as pre-mordanting (left) and meta-mordanting (right).*

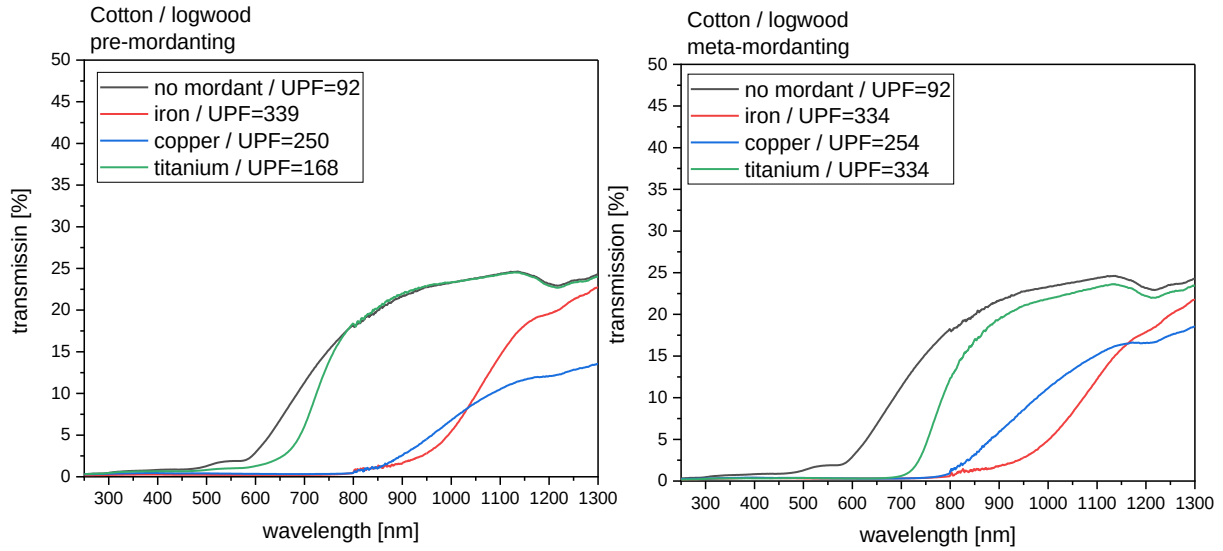

Figure 12. *Optical transmission and related UPF values of investigated cotton fabric dyed with logwood extract. Compared is the dyed wool fabric without mordanting agents, with wool fabrics dyed with madder extract and different metal mordants. The mordanting procedure is done as pre-mordanting (left) and meta-mordanting (right).*

The spectroscopic results for the logwood/mordant combinations on cotton fabrics are presented in Figure 12. Also, on cotton the realized UV protection is excellent. The UV transmission is below 2% over the complete spectral range of UV light and the related UPF values are much higher than 50. Similar to

the application on wool, also on cotton the applied titanium mordant nearly does not change the transmission for IR light. However, by combination of logwood with copper or iron mordants the IR transmission is strongly decreased. Some as for the application on wool, on cotton the combination logwood/copper exhibits the strongest effect in pre-mordanting procedure, while the combination logwood/iron is more effective in the meta-mordanting procedure.

The logwood/mordant applications on Lyocell knitted fabrics are presented in Figure 13. Also, for this combination, no UV protective property are reached on the Lyocell fabrics, because of the open structure of the knitted Lyocell fabric. However, the IR transparency of Lyocell fabrics can be strongly modified by the application of logwood combined with different metal mordants. Strongest effects are gained for the meta-mordanting procedure. Here the decrease of IR transmission can be clearly ranked for the investigated metal mordants in the following order iron > copper > titanium. Even if for the investigated Lyocell fabrics the transmission values are not low enough to support a protective function, the change in the interaction with infrared light might be attraction for applications in the field of IR camouflage. However, for development of clothing with camouflage properties, the full spectral range form UV light, visible light and IR light have to be considered and set in relation to the optical properties of the surrounding area [62-64].

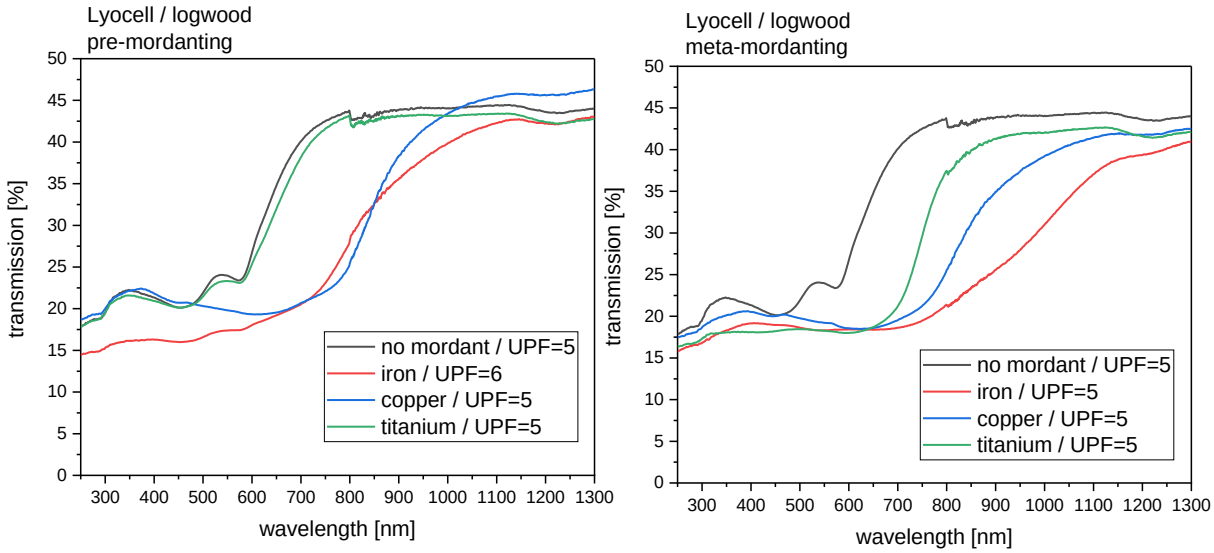

Figure 13. *Optical transmission and related UPF values of investigated Lyocell fabric dyed with logwood extract. Compared is the dyed wool fabric without mordanting agents, with wool fabrics dyed with madder extract and different metal mordants. The mordanting procedure is done as pre-mordanting (left) and meta-mordanting (right).*

4 Conclusions

The combination of natural dyes with different metal mordant agents is investigated for their capability to realize UV and IR properties on natural fiber based textile fabrics. While the UV protection is quite easily reached by the dyeing process, the property to filter near IR light is strongly determined by the type of metal mordant and its application in a pre- or meta-process. On wool fabrics for this procedure the investigated copper based mordant is advantageous. For cellulosic based fibers – cotton and Lyocell – the application of iron mordant in the meta-procedure is advantageous to reach a reduction of IR transmission. Finally, the proof-of-concept is made that with natural dyes UV/IR protective textile materials can be reached, which might be also of interest for clothing with IR camouflage properties.

Author Contributions

B. Mahltig: supervision, writing, optical transmission measurements, microscopic measurements; A. Ostheim: sample preparation, dyeing experiments; K. Ratovo: fabric preparation, initial idea; K. Klinkhammer: validation; M. Fischer: validation; E. Bendt: original project concept & idea. All authors have read and agreed to the published version of the manuscript.

Acknowledgements

The authors owe many thanks to Dipl.-Ing. Simone Wagner for help during dyeing experiments. Funded by the European Union. Views and opinions expressed are however those of the authors only and do not necessarily reflect those the European Union or the European Research Executive Agency (REA). Neither the European Union nor the granting authority can hold responsible for them. Grant agreement ID: 101135042.

Conflicts of Interest

The authors declare no conflict of interest.

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Communications in Development and Assembling of Textile Products is a Diamond Open Access Journal supported by PUBLIA – SLUB Open Publishing.



DOI https://doi.org/10.25367/cdatp

ISSN 2701-939X

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