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Transmission Reduction for UV and IR Radiation with Dyed Lyocell Knitted Textiles

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Abstract: Sunlight is essential for humans. However, sunlight can be the source of several disadvantageous effects and illnesses, e.g., skin aging, sunburn, and skin cancer. Textiles with functional protective effects can counteract these problems. In the current research, knitted fabrics were produced from Lyocell yarns spin-doped with the inorganic UV absorber titanium dioxide TiO_2. Lyocell yarns without TiO_2 were used as reference materials. The produced knitted fabrics were dyed with different dyestuffs to improve the protective properties against UV light and infrared light. The protective properties are determined by optical spectroscopy in an arrangement of diffusive transmission. With the two dyestuffs Drimaren Yellow HF-CD and Solophenyl Bordeaux 3BLE, dyes were determined which complete UV protection and additionally reduce transmission in the near-IR range (700 nm to 1000 nm). TiO_2 in the fibers enhanced this effect. In the UV range (280 nm to 400 nm), the transmission was almost zero with both dyes. Overall, the Lyocell samples containing TiO_2 exhibit less sensitivity to abrasion and a UV protective effect after washing can be still determined. The weight loss after the abrasion test for these samples is quite low with only around 8.5 wt-% (10,000 rubbing cycles in the Martindale device). It is concluded that the right choice of dye can improve the protective effect of textiles against various types of radiation. Lyocell fiber-based textiles are suitable for the production of summer clothing due to their good moisture management. Compared to other radiation protective materials based on coating application, the presented solution is advantageous, because the textile properties of the realized products are still present. For this, a direct transfer to clothing application and use in apparel is easily possible. This study can be seen as the first proof-of-concept for the future development of light-protective clothing products.

Keywords: Lyocell; TiO_2; transmission reduction; UV irradiation; IR irradiation; reactive dyes; direct dyes

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1. Introduction

The skin is often named the largest organ of the human body [1,2]. It protects against a variety of environmental influences, regulates heat balance, and is an important sensory organ [3,4]. Physical environmental influences, such as ultraviolet (UV) radiation and infrared (IR) radiation, can have a major impact [5–7]. UV radiation covers the spectral range of non-visible electromagnetic radiation in the wavelength range from 100 nm to 400 nm. The UV portion of the sun's electromagnetic spectrum is about 6% and is mainly related to the spectral range of the sun from 280 nm to 400 nm [8]. Moreover, artificial light sources for indoor and outdoor illumination can emit a significant amount of UV light [9–11]. Infrared radiation (IR radiation) is defined as the part of the electromagnetic spectrum in the wavelength range of 700 nm to 1 mm. The human skin is exposed to IR radiation from the sun and artificial sources [12]. Of particular interest is the near-infrared range, also known as

IR-A radiation (700 nm to 1400 nm), as this radiation is richer in energy and the penetration depth of the radiation into the skin is particularly high [13]. Frequent exposure to UV and infrared radiation can cause significant damage to the skin, such as sunburn, premature skin aging, and promotion of the development of carcinomas [14–19]. For this, materials able to support protection against UV and infrared radiation can contribute significantly to human health and wellbeing [20–22]. The use of sun creams to protect against UV radiation is well established. However, the protective properties of these cosmetic products are not permanent and are inferior to a functionalized textile in terms of applicability and benefit [23,24]. In addition to the research on radiation-protective materials, a recent field of research is the development of photoactive materials active under illumination with visible light. These materials show strong potential for applications for disinfection and degradation of pollutants [25–28]. Nowadays, developments in the sustainability of textile production processes are of high importance because textile production is responsible for 5 to 10% of greenhouse gas emissions [29]. Due to this the carbon and water footprint for textile products are intensively investigated during the last years [30–32]. The functionalization of fibers by doping during the spinning process, in-situ growth of metal and metal-organic components, and coating processes on textile fabrics are intensively investigated [23,33–38]. In comparison, the achievement of a UV/IR absorbing effect via the coloring of textiles in civilian use has so far been little researched. In the summer months, the intensity of the sun's rays are particularly high, and many activities take place outdoors. To protect against radiation, light and comfortable textiles made of appropriate materials are an adequate solution and often advantageous compared to cosmetic products. Knitted fabrics made of Lyocell, a cellulose regenerate fiber, are suitable [39]. Compared to cotton, the regenerated fiber has a higher water absorption capacity, which has a positive effect on wearable comfort when sweating [40,41]. A known problem of Lyocell fibers is the so-called "washing greying" (fibrillation), which becomes noticeable after the washing process in the form of a greyish film on the fabric surface and negatively influences the fabric's appearance [42]. However, fibrillation can be reduced by additives such as cross-linking agents or by selecting suitable reactive dyes [43,44]. Textile manufacturing and finishing generally offer a variety of options for reducing transmission in the UV and IR spectral ranges [45–47]. By this, protective properties against UV and IR light can be implemented. Swimwear, outdoor clothing, and technical textiles, for example, awnings and sunshades, focus on reducing the transmission of UV radiation [48,49]. Textiles for reducing the transmission of IR radiation are primarily found in the field of PPE (personal protective equipment) or military technology [50]. Both inorganic (e.g., lanthanum hexaboride LaB_6, titanium dioxide TiO_2 or zinc oxide ZnO) and organic substances (e.g., benzotriazole, coumarin or curcumin) are used [34,51,52]. They can either be applied subsequently as additives or incorporated directly into the fiber by spin doping. The effect of TiO_2 in particular has been proven several times [23,53]. Dyes usually serve aesthetic aspects and a wide range of dyes allows a multitude of design possibilities. Here, the color depends on the chemical structure of the chromophore and its ability to absorb radiation and transfer electrons into the excited state. In principle, compounds with conjugated double bonds are suitable for this [54]. So-called "functional dyes", such as IR dyes used to protect barcodes against forgery [55]. These functional dyes exhibit absorption of radiation in the wavelength range from 700 nm and are preferably not colored. The basic problem in the application for dyes that have transmission-reducing properties in both the UV and IR spectral range is that the absorption and remission properties of the dye components used are dedicated to visible light in the range of 400 nm to 700 nm. For the dyeing of regenerated cellulosic fibers, reactive dyes, and substantive dyes are commonly used [56]. Reactive dyes consist of a coloring chromophore, a solubilizing group, and a reactive group characteristic for this dye class, with which the dye can be covalently bound to the fiber. With the hydroxide groups of cellulose, either a nucleophilic addition, in the case of vinyl sulfones, or a nucleophilic substitution, in the case of aromatic reactive groups, takes place [57,58]. Monoazo dyes, which have narrow and steep absorption bands due to their small molecular structure, are

particularly popular, resulting in very brilliant color shades. Other chromophore systems contain anthraquinone, phthalocyanine, and formazan structural units. Diazo dyes are only rarely used because their greater substantivity has a negative influence on washing-out behavior [59]. Substantive dyes are also named as direct dyes. For these dyes, large-molecular, planar diazo or polyazo chromophores are preferred [60]. The bond between dye and cellulose fibers is based on hydrogen-like bridges between the chromophore of the dye and the hydroxyl groups of the cellulose. As a prerequisite for this, the chromophore must have a planar structure and a high number of conjugated double bonds in order to have a sufficient affinity to the cellulose [61,62].

In the current investigation, dyestuffs from different dye classes were applied on Lyocell knitted fabrics with the purpose to reduce the transmission of UV light and near-infrared light. The main objective of this research is to realize a textile material offering protection against both UV and near IR light while keeping mainly textile properties such as flexibility or hand feel. To guarantee the properties in terms of use the washing and abrasion stability are investigated. A dependence on the protective effect is determined for the chemical structure of the dye and on the presence of TiO_2 in the fiber material. Finally, the proof-of-concept was made, that the transmission for both UV light and near-infrared light can be decreased by developed Lyocell materials. By this, a first step is completed for the development of clothing materials offering additional UV protection also a certain capability for protection against infrared radiation.

2. Materials and Methods

2.1. Textile

2.1.1. Textile Material Production

Raw fibers (38 mm length, 1.7 dtex) of Lyocell, as well as Lyocell with 2% TiO_2 were provided by the Thuringian Institute for Textile and Plastics Research e.V. (TITK, Rudolstadt, Germany). The unit dtex (dezitex) describes the fineness of a fiber or yarn—1 dtex is related to the fiber weight of 1 g per 10,000 m length [63,64]. Yarns with a fineness of Nm40 were spun from these in the spinning technical center of the Department of Textile and Clothing Technology at Niederrhein University of Applied Sciences. The unit Nm describes the fineness of a yarn related to the weight of 1 g—Nm40 means 40 m of this yarn exhibits the weight of 1 g [63,64]. Knitted fabrics were produced from the yarns using a small circular knitting machine type Harry Lucas TK-83-L (Maschinenfabrik Harry Lucas GmbH, Neumünster, Germany) with a gauge of E24. The knitted textile is a single jersey knit with an R/L weave. All knitted fabrics were pre-washed before dyeing at 60 °C, 60 min (Miele Softtronic W1714, Miele & Cie. KG, Gütersloh, Germany) using 4 g/L of a brightener-free detergent (ECE Formulation Phosphate Reference Detergent (B), James Heal, Halifax, UK). After washing line drying at room temperature is performed. The weight per area of the knitted fabrics was 165 g/m^2 for the pure Lyocell fiber and 191 g/m^2 for the Lyocell fiber spin-doped with TiO_2. Photographic images of used Lyocell knitted fabrics are presented in Figure 1.

2.1.2. Washing Experiments

The developed knitted Lyocell fabrics were subjected to washing tests before and after dyeing. In total, each sample was washed over a cycle of 10 consecutive washes. For this purpose, the samples were washed at 40 °C for 60 min each with the same washing machine and detergent as for the prewash. The influence on the transmission and the color change was examined after this washing procedure.

2.1.3. Abrasion Tests

The stability against abrasion of developed samples was tested according to DIN EN ISO 12947-2:216 using a Martindale device (Mesdan Spa, Puegnago del Garda, Italy). The test specimens (diameter 38 mm) were air-conditioned for 24 h under standard conditions (20 °C; 65% humidity) and weighed before the testing. The samples were rubbed over

a standard wool fabric for 10,000 rubbing cycles. Subsequently, the change in the surface, the loss of mass, and the influence on the transmission were examined.

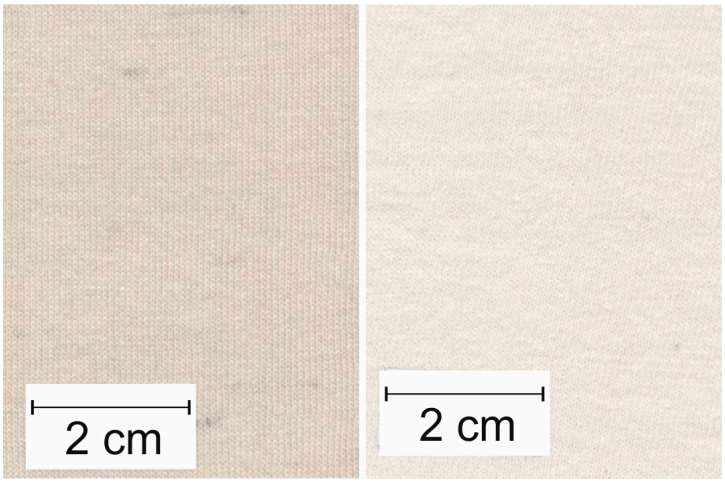


Figure 1. Photographic images of used Lyocell knitted fabrics—(**left**): knit from Lyocell yarn; (**right**): knit from Lyocell yarn spin-doped with TiO_2.

2.2. Dyes and Dyeing Processes

2.2.1. Preparation of Dye Solution

Dye solutions of Remazol Brilliant Red F3B (Dystar, Raunheim, Germany) (monoazo, vinyl sulfone) (reactive dye), Drimaren Yellow HF-CD (Achroma, Wiesbaden, Germany) (monoazo, triflourpyrimidine+vinyl sulfone) (reactive dye), Sirius Orange (Dystar, Germany) (disazo) (direct dye) and Solophenyl Bordeaux 3BLE (Huntsman, Deggendorf, Germany) (disazo) (direct dye) were prepared in different concentrations with distilled water. The mentioned dyes were selected mainly for two reasons. First, the used Lyocell fibers exhibit a cellulosic chemical structure. Typical for dyeing of cellulose fibers are direct dyes and reactive dyes, because with these dyes the fiber dye interaction is strong to lead to sufficient washing and rubbing fastness. Direct dyes are used, because of simple application. Reactive dyes are known for the best washing stability after application on cellulosic fibers. For this, it is reasonable to evaluate both direct and reactive dyes. Second, the optical properties of used dyes are checked on their aqueous solutions by transmission measurements. If for them a decrease in transmission for near-infrared light is determined, these dyes are selected for further dyeing experiments. The dyestuffs are used as received from the supplier without further purification. For each dyestuff, two different concentrations are chosen for application on the textile fabric to present a sufficient proof-of-concept that by application of these dyes, the transmission of UV and infrared radiation can be reduced.

2.2.2. Dyeing of Knitted Fabrics with Drimaren Yellow HF-CD

Knitted fabrics with a fabric weight of around 5 g or 20 g were dyed with the reactive dye Drimaren Yellow HF-CD (Achroma, Germany). The dye liquor was prepared from the dye, soft water, Sarabid MIP (3 g/L, CHT, Tübingen, Germany), sodium chloride (60 g/L) (technical grade, Carl Dicke GmbH & Co.KG, Mönchengladbach, Germany), and sodium carbonate (10 g/L) (technical grade, Carl Dicke GmbH & Co.KG, Germany). The liquor ratio was 1:10. Dyeing was performed by the exhaust method in a Mathis-Polycolor-P dyeing apparatus (Mathis AG, Oberhasli, Switzerland) at 60 °C dyeing temperature and 60 min. process duration. Afterwards, the textiles were rinsed with soft water and then subjected to a prewash with Cotoblanc SEL (CHT, Germany) at 95 °C for 15 min. After rinsing with soft water, the textiles were neutralized with diluted citric acid (2 mL/L, (technical grade, Carl Dicke GmbH & Co.KG, Germany). Finally, line drying is performed at room temperature.

2.2.3. Dyeing of Knitted Fabrics with Solophenyl Bordeaux 3BLE

Knitted fabrics with a fabric weight of around 5 g or 20 g were dyed with the direct dye Solophenyl Bordeaux 3BLE (Huntsman, Germany). The dye liquor was prepared from the dye, soft water, Sarabid MIP (3 g/L), sodium chloride (60 g/L) (technical grade, Carl Dicke GmbH & Co.KG, Germany), and sodium carbonate (15 g/L) (technical grade, Carl Dicke GmbH & Co.KG, Germany). The liquor ratio was 1:10. The dyeing was performed by the exhaust method in a Zeltex Polycolor dyeing apparatus (Zeltex AG, Muttenz, Switzerland) at 98 °C dyeing temperature and 60 min. process duration. Subsequently, rinsing was carried out with soft water for 10 min at 60 °C and then with cold hard water for 10 min. Finally, line drying is performed at room temperature.

2.3. Analytics

2.3.1. Transmission Measurement

The transmission of different dye solutions was measured in an aqueous solution in the wavelength range of 280 nm to 1400 nm. For this purpose, the dye solutions were filled into a quartz cuvette and measured with a UV/Vis spectrometer UV2600 from Shimadzu (Kyōto, Japan). Distilled water was used as a reference. The transmission of the textile samples was measured by clamping the textiles without tension in a special sample holder in the UV/Vis spectrometer UV2600 from Shimadzu and the wavelength range of 280 nm to 1400 nm was measured. For collecting diffusive transmitted light an integrating sphere was used. Each sample was measured at three different points and the mean value over all three measurement curves was determined for evaluation. The used spectrometer UV2600 contains two different light detectors. One detector is sensitive to UV light and visible light. The second detector is sensitive to infrared light. While recording the complete spectral range a change of the detectors is necessary and usually set at a position in the range of 750 nm to 850 nm.

2.3.2. Calculation of Ultraviolet Protection Factor (UPF)

For all textiles, the UPF value was calculated based on the specification set out in the Australia and New Zealand Standard AS/NZS 4399:1996 [65]. The following formula was used for this purpose:

$$\frac{E_{eff}}{E'} = \frac{\sum_{290}^{400} E_\lambda \cdot S_\lambda \cdot \Delta\lambda}{\sum_{290}^{400} E_\lambda \cdot S_\lambda \cdot T_\lambda \cdot \Delta\lambda}$$

where E_λ is the specific erythema effective spectrum as a function of the wavelength and S_λ represents the radiation intensity of the radiation as a function of the wavelength. T_λ stands for the measured transmission value of the respective wavelength and Delta λ for the measurement interval. The values S_λ of the radiation intensity are based on the values prevailing in Melbourne in January and represent a so-called "worst case" scenario. The classification of the UVR protection category is carried out according to Standard AS/NZ S4 399, 1996 [65] in Table 1:

Table 1. UVR protection categories.

UPF Range	UVR Protection Category
<15	bad
15–24	good protection
25–39	very good protection
40–50, 50+	excellent protection

2.3.3. Colorimetry

Developed samples were analyzed colorimetrically using the colorimeter 400 from Datacolor. In order to determine the L*C*h values of the samples, the samples were placed fourfold and measured at three points with D65 standard light. Mean values were calculated. To determine the color change after washing, the color values a* and b* were

additionally determined according to CIE L*A*B*, taking EN ISO 105-J03:2010 into account. For comparison, an unwashed sample was measured as a standard and the washed samples were measured against it. Each sample was placed fourfold and measured three times with the right side of the fabric facing upwards.

3. Results

3.1. Transmission Measurement of Aqueous Dye Solutions

A huge number of dyes are commercially available, from this one yellow/orange dye and one red/purple dye each were selected from the dye classes of direct dyes and reactive dyes. Before starting the application of the dyes to textile fabrics, their optical transmission is determined at aqueous dye solutions. Figure 2 shows transmission spectra for the aqueous solutions of the dyes Sirius Orange and Solophenyl Bordeaux (Figure 2a) and for the dyes Drimaren Yellow and Remazol Red (Figure 2b) for two different dye concentrations.

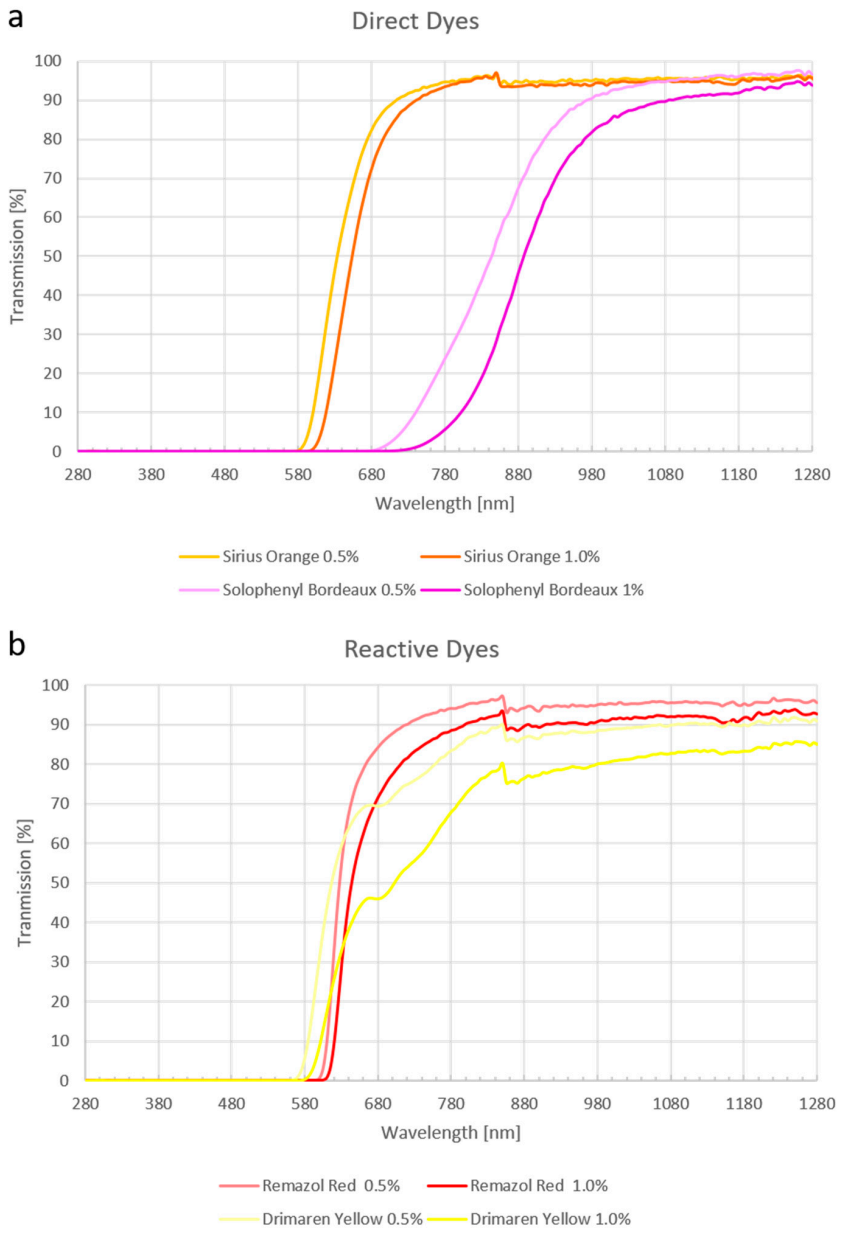


Figure 2. Transmission spectra of dyes in aqueous solution, (**a**) Sirius Orange and Solophenyl Bordeaux, (**b**) Drimaren Yellow and Remazol Red.

For all investigated dyes, it can be seen that a higher dye concentration in the solution leads to lower transmission. Sirius Orange has a high red content. The transmission spectra of the dyes Sirius Orange and Remazol Red are very similar: the transmission of both dyes increases very steeply in the spectral range around 550 nm and runs almost horizontally from 730 nm to 1200 nm.

The transmission spectra of Drimaren Yellow and Solophenyl Bordeaux differ significantly from this. In the case of the Drimaren Yellow HF-CD, the transmission rises strongly at around 595 nm and has a rather linear rise to 700 nm. Due to this, the transmission is reduced in the spectral range of 700 nm to 850 nm. From 870 nm onwards, a slightly rising, horizontal curve follows. The overall transmission is significantly lower. Solophenyl Bordeaux 3BLE is a dark red dye with very high blue content. The transmission increases linearly only at 680 nm, up to around 1000 nm. Thus, this dye shows a reduction of transmission for near-infrared light in the spectral range up to 1000 nm.

All dyes reduce the transmission in the UV range completely. Furthermore, in the NIR range, all dyes exhibit a low to medium potential for transmission reduction. Because the dyes Drimaren Yellow and Solophenyl Bordeaux have the best capability to reduce transmission for near-infrared light, these dyestuffs were chosen in the current investigation to dye the knitted Lyocell fabrics.

3.2. Transmission Spectra of Knitted Fabrics of 100% Lyocell and Lyocell with TiO_2

Knitted fabrics made of Lyocell fibers and Lyocell fibers spin-doped with 2% TiO_2 served as the basis for further tests. Figure 3 shows the transmission spectra of these raw materials without further dye application as reference measurements.

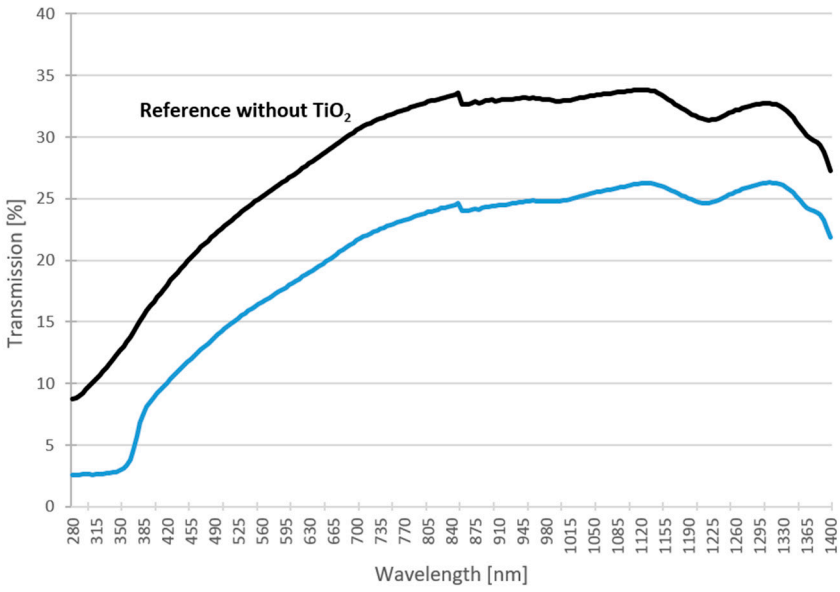


Figure 3. Comparison of transmission spectra of Lyocell knitted fabric with and without TiO_2. The doped Lyocell fibers contain 2 wt-% TiO_2.

The addition of TiO_2 to the fiber material reduces the transmission in the UV range (below 380 nm) drastically. This absorption behavior of the TiO_2 is determined by its band gap energy [66]. However, a reduction in transmission was also observed over the entire measured spectrum, which can be explained by the reflective properties of the TiO_2 white pigment particles. Please remark, in addition to its use as a UV absorber, TiO_2 is one of the most important white pigments with a high reflectivity for visible light.

These knitted fabrics were dyed with the selected dyes Drimaren Yellow and Solophenyl Bordeaux. After dyeing, the optical transmission was investigated. A dye concentration of 1.5% of Drimaren Yellow or Solophenyl Bordeaux was chosen, as the solution tests showed that higher concentrations are more effective. Photographic images of dyed Lyocell

knitted fabrics are presented in Figure 4. The intensive dark yellow-orange and dark violet coloration is presented. Figure 5 shows the spectra of the transmission measurements.

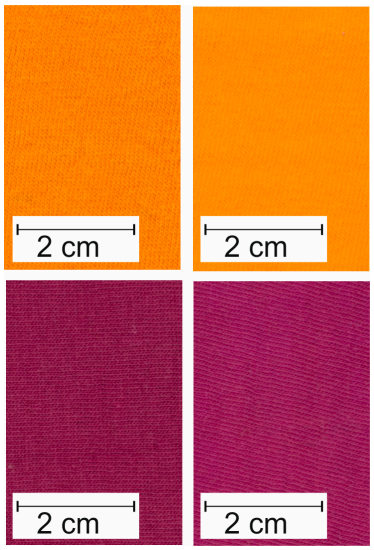


Figure 4. Photographic images of dyed Lyocell knitted fabrics (dyeing with Drimaren Yellow HF-CD on top; dyeing with Solophenyl 3BLE Bordeaus below)—(**left**): knit from Lyocell yarn; (**right**): knit from Lyocell yarn spin-doped with TiO_2.

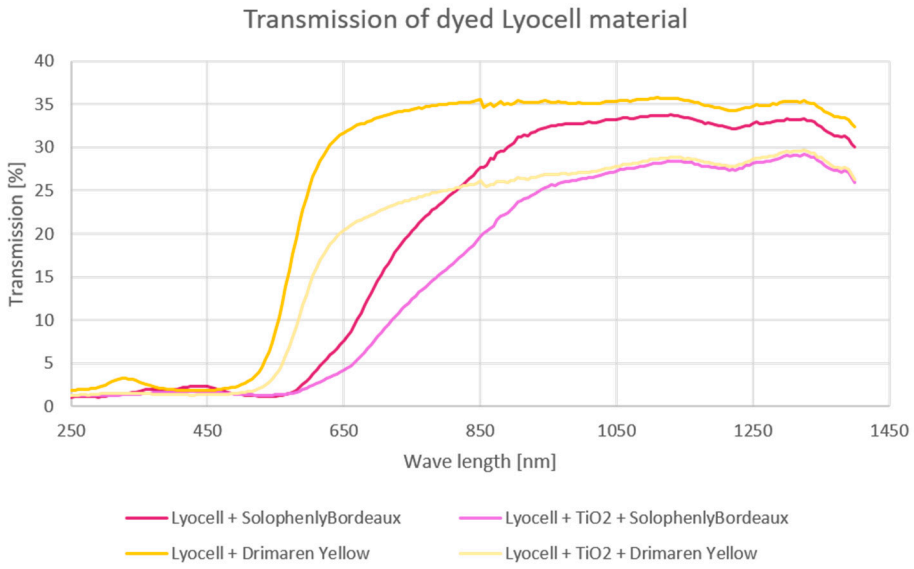


Figure 5. Transmission spectrum of knitted fabrics dyed with Drimaren Yellow HF-CD or Solophenyl 3BLE Bordeaux made of 100% Lyocell and Lyocell with TiO_2.

The influence of the dyeing on the transmission can be determined. In the case of Drimaren Yellow, the transmission is reduced to almost zero in the entire UV range and the spectral range of visible light up to around 480 nm. With increasing wavelength, the transmission increases and approaches the values of the undyed samples for both the pure Lyocell and the Lyocell fabrics containing TiO_2.

In the case of Solophenly Bordeaux, the effect of reducing transmission for visible light is even more pronounced and a slight increase in transmission is determined for the spectral range starting at around 560 nm. Overall, dyeing with Solophenly Bordeaux reduces the transmission for visible range significantly to values below 10%. For the spectral range of near-infrared light up to 920 nm, also a reduction in transmission is observed. An influence of added TiO_2 in the fiber only becomes obvious from a wavelength of around 600 nm and

reduces the transmission by at least 5% from here on. The dyed samples were examined colorimetrically and the LCH values were determined (Table 2).

Table 2. LCH-values of dyed textiles, measured with D65 standard light (before and after washing).

Sample	L*	a*	b*	C*	h
100% Lyocell, undyed	84.36	1.23	6.33	6.45	79.01
Lyocell with TiO_2, undyed	90.99	1.31	4.74	4.92	74.57
100% Lyocell, Drimaren Yellow, unwashed	59.93	34.51	63.72	72.47	61.56
100% Lyocell, Drimaren Yellow, washed	59.57	34.82	63.02	72.00	61.08
Lyocell with TiO_2, Drimaren Yellow, unwashed	64.80	35.01	67.77	76.28	62.60
Lyocell with TiO_2, Drimaren Yellow, washed	63.03	35.71	64.37	73.61	60.98
100% Lyocell, Solophenyl Bordeaux, unwashed	33.89	29.70	−3.39	29.89	353.49
100% Lyocell, Solophenyl Bordeaux, washed	36.22	28.95	−3.79	29.19	352.53
Lyocell with TiO_2, Solophenyl Bordeaux, unwashed	34.57	34.43	−6.11	34.96	349.94
Lyocell with TiO_2, Solophenyl Bordeaux, washed	35.99	33.41	−6.48	34.03	349.02

The presence of TiO_2 in the fiber material increases the brightness (L*) compared to the pure Lyocell fiber material (compare also photographic images in Figure 1). TiO_2 is a bright white pigment with a high refractive index of 2.7, so the increase of brightness of the fiber materials in the presence of embedded TiO_2 particles is expected [67]. This brightening effect remains in a similar order of magnitude for the dyed samples. The Hue values (h) confirm the optical impression of the color tone [46,68]. The samples dyed with Drimaren Yellow show an orange color tone ($a^* \approx 35$, $b^* \approx 64$) with $h \approx 60$. The textiles dyed with Solophenyl Bordeaux have an h-value of about 350 and show a violet color tone ($a^* \approx 30$, $b^* \approx -3$) (see Figure 4). Washing of the samples resulted in small changes in the values. Drimaren Yellow dyed samples became a bit darker (reduced L*) and Bordeaux red-dyed sample became a bit brighter (increased L*). Interestingly, the undyed samples have an h-value of 74–79 and thus exhibit a yellow-red color tone ($a^* \approx 1$, $b^* = 5–6$) (see Figure 1). However, since the saturation (chrome = C*) is very low, these samples appear almost achromatic. In the colored samples, the saturation increases slightly due to the TiO_2 content—the colors thus become minimally more brilliant.

3.3. Usability of the Dyed Knitted Fabrics

In actual investigations, no fastness post-treatment with a cationic auxiliary was carried out following the dyeing. In industrial practice, this process step is usually conducted and state of the art. However, the use of a fastness enhancer was deliberately omitted in the actual investigation to minimize the number of factors influencing the transmission and to be able to determine the influence of the dye. Nevertheless, it can be assumed that a possible post-treatment with a fastness-enhancing agent should have a positive influence on the fastnesses in general and as a result leads to an improved washing fastness. To evaluate the usability of the dyed fabrics for clothing applications, the textiles were washed 10 times in a household washing machine at 40 °C or rubbed up to 10,000 cycles with the Martindale device.

3.3.1. Washing Resistance Analysis

Washing resistance is an important factor in assessing the usability of textiles for clothing applications. Figure 6 compares the transmission of washed and unwashed textiles dyed with Drimaren Yellow (Figure 6, left) and with Solophenyl Bordeaux (Figure 6, right).

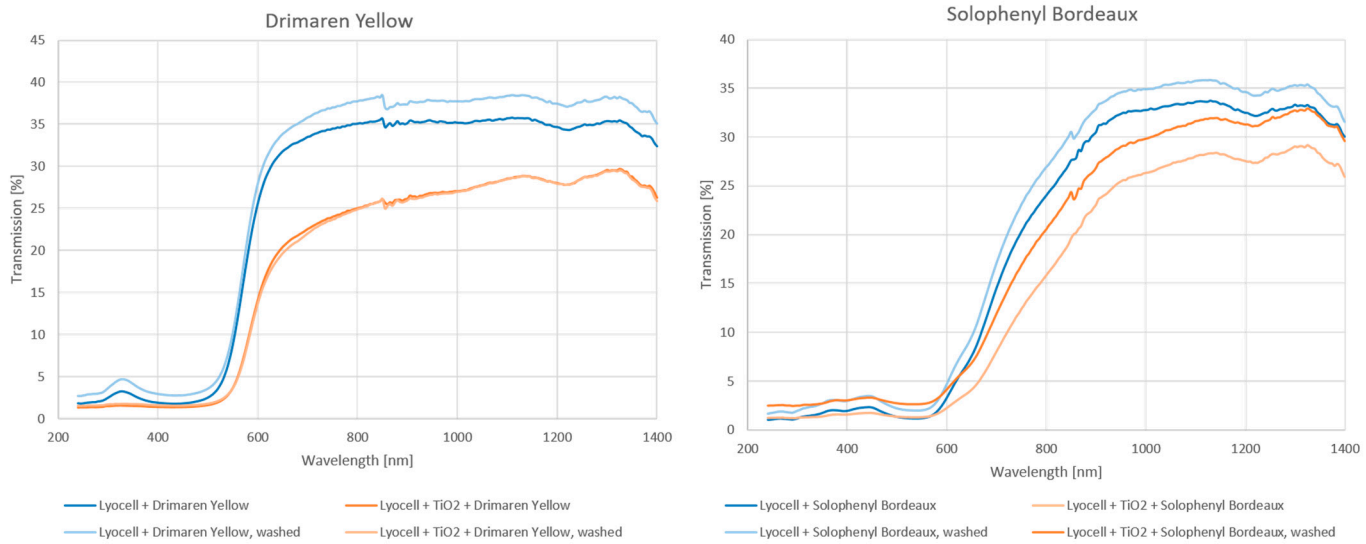


Figure 6. Transmission spectra of knitted fabrics dyed with Drimaren Yellow (**left**) or Solophenyl Bordeaux (**right**) made of 100% Lyocell and Lyocell with TiO_2. Comparison of washed and unwashed samples.

The results of the transmission measurement of Drimaren Yellow HF (washed & unwashed samples) can be seen in Figure 6, left. The influence of washing on knitted fabrics made of 100% Lyocell is clearly visible. The transmission is increased in the UV range as well as in the NIR range. The knitted fabrics with TiO_2, on the other hand, are very resistant to multiple washing and show hardly any deterioration in transmission behavior compared to the unwashed sample. In the UV range, the transmission values are minimally increased, but much lower than with knitted fabrics made of 100% Lyocell.

For samples dyed with Solophenly Bordeaux (Figure 6, right), it is noticeable that multiple washing has an influence on the transmission behavior of both materials. The transmission of knitted fabrics made of 100% Lyocell and those spin-doped with TiO_2 is increased after the washing procedure. This is particularly clear in the NIR range, where the difference between the washed and unwashed samples is more pronounced. The washed Lyocell TiO_2 knitted fabric also shows a noticeable difference in the UV range. The transmission in the UVB range is even higher than that of the washed 100% Lyocell knitted fabric. Both washed samples also show a clear difference in transmission in the visible range. Reasons for this could be a partial dye release and a fiber abrasion.

The color change occurring due to washing was measured colorimetrically (Table 3). The unwashed sample was measured as a standard and the washed samples were measured in comparison. The CIE L*a*b* values and the ΔE* values for the samples at standard light D65 are shown in Table 3 [68].

Table 3. Color values and color distances after washing for dyed samples, with ΔL* (+ = brighter, − = darker), Δa* (+ = red, − = green), Δb* (+ = yellow, − = blue).

Sample	ΔE*	ΔL*	Δa*	Δb*
Solophenyl Bordeaux, 100% Lyocell	2.48	2.33	−0.75	−0.41
Solophenyl Bordeaux Lyocell with TiO_2	1.79	1.43	−1.01	−0.38
Drimaren Yellow, 100% Lyocell	0.85	−0.36	0.31	−0.70
Drimaren Yellow, Lyocell with TiO_2	3.90	−1.77	0.70	−3.40

The measurements show that after the washing procedure, the single color parameters are changed (see Table 2) and that color changes can be determined for all samples. All knitted fabrics dyed with Solophenyl Bordeaux 3BLE show an increase in brightness and a less saturated appearance. The knitted fabric made of Lyocell 100% appears slightly

redder, whereas the knitted fabric containing 2% TiO_2 appears slightly bluer. Overall, the color change by washing the pure Lyocell knitted fabric is more intense, which is shown mainly by the increase in the value L* (see Table 2). This behavior can be explained by the fact that direct dyes usually exhibit a lower wash fastness, because of fiber dye interaction with weaker adhesion.

A color shade difference also occurs with Drimaren Yellow HF. In contrast to Solophenyl Bordeaux 3BLE, the measured values show a decrease in brightness for both knitted fabric types as well as an increase in the red content. The decrease in brightness can be explained by the decrease in the yellow content, which makes the sample appear darker overall. It can also be seen that the multiple washing causes more change in the knitted fabric with 2% TiO_2.

3.3.2. Abrasion Resistance

The abrasion resistance describes the resistance of fabric against mechanical friction and is used to determine changes in the functional and optical properties of use. The dyed samples made of 100% Lyocell and Lyocell with TiO_2 were tested against abrasion, both in unwashed and washed conditions.

After the abrasion procedure, all investigated knitted samples did not exhibit any holes or broken meshes appeared. However, there are clear differences between the two types of knitted fabrics. Figure 7 shows the microscopic images of the samples after the abrasion procedure.

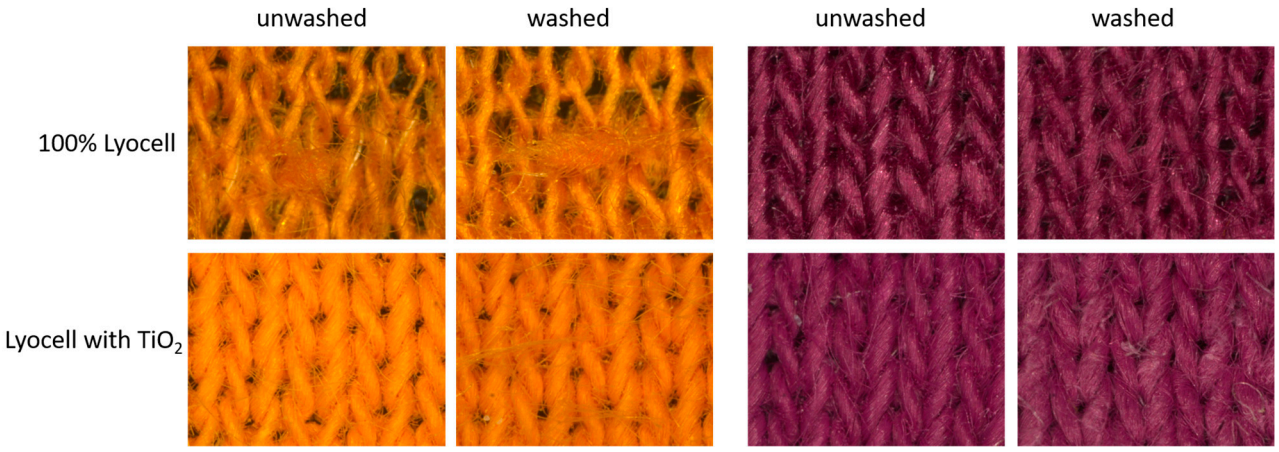


Figure 7. Microscopic images of the samples after the abrasion test (10,000 rubbing cycles in the Martindale device).

The knitted fabrics made of 100% Lyocell tended to form fiber knots and preliminary stages of these knots formed on all variants with varying degrees of severity. In the Solophenyl Bordeaux 3BLE dyed fabrics, this development manifested itself in the form of a greyish veil. Here, the overall fabric appearance was better, and damage to the knitted structure was much less compared to the samples dyed with Drimaren Yellow HF. In addition to the formation of pilling knots, these samples showed strong fiber abrasion in some areas, which caused the knitted fabric to lose a lot of density.

All Lyocell knitted fabrics with TiO_2 were more resistant, although hairiness and accumulations of individual fibers on the surface were also noticeable here, although less pronounced. The loss of mass due to the abrasion load was analyzed and is shown in Figure 8.

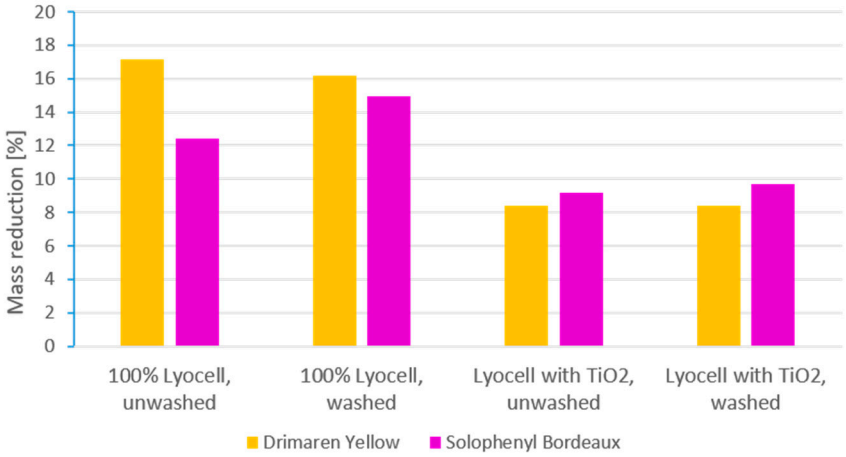


Figure 8. Mass loss after abrasion procedure (10,000 rubbing cycles in the Martindale device).

Furthermore, the influence of abrasion on the transmission was investigated. Figure 9 shows the transmission spectra for the unwashed samples in order to analyze the sole influence of rubbing without the additional influence of washing.

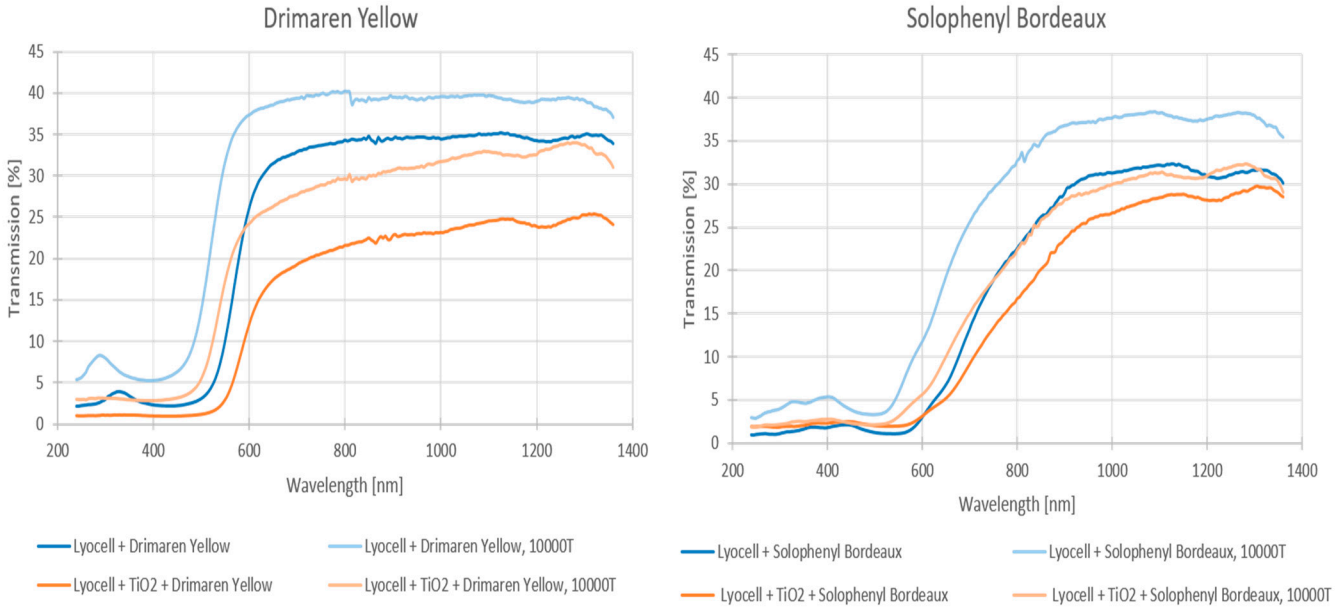


Figure 9. Transmission spectra of unwashed knits dyed with Drimaren Yellow (**left**) or Solophenyl Bordeaux (**right**) from 100% Lyocell and Lyocell with TiO_2. Comparison of non-rubbed and rubbed samples (10,000 rubbing cycles).

After the abrasion test for all samples, a significant loss of mass (Figure 8) and an increase in optical transmission (Figure 9) is determined. The mass reduction most likely results from fiber abrasion. In consequence, the fabrics become thinner and more permeable. The loss of mass is stronger for the knitted fabrics made of 100% Lyocell and in the range between 12 to 17%. The strongest abrasion occurred in the sample dyed with Drimaren Yellow. The increase in transmission of the 100% Lyocell knitted fabrics is comparable for Drimaren Yellow and Solophenyl Bordeaux. The knitted fabrics with TiO_2 showed a significantly lower weight loss, which is in good agreement with the optical assessment (Figure 7). For this, the developed fabrics dyed with Drimaren Yellow exhibited a slight advantage. The transmission values did not confirm the advantage of the Drimaren Yellow dyeing. The scouring here led to a stronger increase in transmission than with the Solophenyl Bordeaux dyed samples.

3.3.3. Ultraviolet Protection Factor (UPF)

The textiles examined here are intended to be used for the production of clothing with a special protective function against UV and IR radiation. The ultraviolet protection factor (UPF) is used to assess UV-protective textiles. The UPF values for the different textiles are shown in Table 4.

Table 4. UPF of different samples, also an evaluation of sample properties according to the UVR protection category is given.

Sample	UPF	UVR Protection Category (See Table 1)
100% Lyocell	6	bad
Lyocell with TiO_2	43	excellent
100% Lyocell, Drimaren Yellow	40	excellent
100% Lyocell, Drimaren Yellow, washed	27	very good
Lyocell with TiO_2, Drimaren Yellow	69	excellent
Lyocell with TiO_2, Drimaren Yellow, washed	59	excellent
100% Lyocell, Solophenyl Bordeaux	67	excellent
100% Lyocell, Solophenyl Bordeaux, washed	42	excellent
Lyocell with TiO_2, Solophenyl Bordeaux	73	excellent
Lyocell with TiO_2, Solophenyl Bordeaux, washed	37	very good

Except for the pure Lyocell material, all textiles have a very good to excellent protective effect against UV radiation. Both the addition of TiO_2 and dyeing strongly enhance the protection. Washing reduces the UV protection effect, but it remains at a high level. It can be stated, that developed dyed Lyocell fabrics are suitable for the production of clothing with UV protection. Depending on the application, it can be considered to do without the addition of the TiO_2 in the fiber and instead, just dye it. Avoiding spin-doping can be reasonable from an economic point of view.

4. Discussion

The aim of the current investigation was to realize a textile material with reduced transmission for UV light and near-infrared light. For this, knitted fabrics from different Lyocell fibers are produced and dyed. It has been shown that the dyes examined are capable of reducing transmission in general (see Figures 2 and 4). This statement is especially valid for UV light. As expected, the reduction of transmission was dependent on the type of dye. The best results were achieved on textiles dyed with Solophenyl Bordeaux 3BLE. In comparison, transmission measurements for samples dyed with Drimaren Yellow HF-CD exhibit less advantageous results. While the measurement results in the UV range (see Table 3) can be easily classified on the basis of comparable studies [69,70]. In comparison, the evaluation of the measurement results in the NIR range is more difficult due to a smaller number of reference data. The behavior of dyes available on the market and frequently used with regard to absorption/reflection properties is relatively unexplored in the IR range and the number of publications, especially with reference to the dye classes mentioned, is low. One reference pointed for a certain classification of the results is the known high reflectivity of indanthrene dyes, whose transmission results serve as a reference point for an assessment in the NIR range [71].

It has been shown that the dyes Solophenyl Bordeaux 3BLE and Drimaren Yellow HF-CD show a comparable behavior in the NIR range as the dye Indanthren Yellow 5GF. Solophenyl Bordeaux 3BLE also shows a significant reduction in transmission values in parts of the NIR range [700 nm to 900 nm], which did not occur in this form with any of the other dyes tested. Compared to the transmission reduction for UV light, the dye has a smaller effect with regard to a reduction in the IR range and a reduction in transmission seems to be more difficult to achieve. Furthermore, it was observed that an increase in dye concentration, and thus a decrease in brightness, leads to a decrease in transmission for

near-infrared radiation. For UV light, the transmission was already significantly decreased by the tested dyes at low concentrations, so an increase in the dye concentration did not lead to further improvements (see Figure 2). The color achieved and the reduction of transmission in the non-visible wavelength ranges (here UV and NIR) is a consequence of the chemical structure of the dyes. However, similar or spectroscopically identical color shades can be achieved by different dye structures. It is often assumed that the darker a color is, the better the protective effect. Based on this statement, the color black should be particularly suitable for reducing transmittance, but this cannot necessarily be confirmed [16]. When comparing the UPF values determined, which ultimately reflect the transmission, with the color values (LCh) (Table 2), Solophenyl Bordeaux 3BLE, the darkest shade, has the highest UPF value (Table 3). Excellent UPF values are also achieved in textiles dyed with Drimaren Yellow HF-CD (Table 3). Thus, the brightness of a shade cannot necessarily be related to the UV protective effect of color. Washing and abrasion increase the optical transmission of investigated fabrics (see Figures 6 and 9). Nevertheless, very good to excellent UPF values are still achieved, which proves the good usability of the textiles. A loss of dye, as confirmed by the color measurement, seems to have a stronger effect on the transmission of Solophenyl Bordeaux 3BLE than on Drimaren Yellow HF-CD, especially as the loss of dye in the latter leads to a decrease in brightness, whereas the Solophenyl Bordeaux 3BLE knitted fabrics became significantly brighter.

If one disregards the clear brightening effect of TiO_2, the use of TiO_2 as a component of the fiber can be assessed as positive. The transmission could be significantly reduced with Solophenyl Bordeaux 3BLE as well as with Drimaren Yellow HF, whereby the absorbing effect of TiO_2 in the UVA and the reduction in the IR range is particularly striking. Furthermore, the knitted fabrics with TiO_2 are much more resistant to mechanical stress, which was shown by a lower increase in transmission and a lower fiber abrasion quantity in the abrasion test.

With regard to the choice of dye to reduce transmission in the various spectral ranges, the factors of dye concentration, color depth, and color shade have an influence, although, with regard to color shade and color depth, no sweeping statements may be made about possible behavior in the optical spectra. Furthermore, the structural composition of the dye is essentially responsible for the transmission. Solophenyl Bordeaux 3BLE has an elongated, two-dimensional character and has two azo groups (disazo) as part of the chromophore. In addition to the aromatic character, this dye has the feature of a copper complex. Possibly this is responsible for a shift of the red spectrum into the NIR range, which may explain the decreased transmission in the spectral range of 700 nm to 900 nm.

Drimaren Yellow HF-CD is a monoazo dye with two reactive anchor groups. The structural formula is not published by the manufacturer and is therefore unknown. In other investigations, the use of dyes with monochlorotriazine anchor groups was described, which positively influenced the transmission in the UV range [72], perhaps similar structures are present here. Although the structure of the dyes shown is quite different, in the case of Solophenyl Bordeaux 3BLE it can be stated that the presence of several aromatic structures and a thus more delocalized π-electron system is possibly a criterion for their good protective effect in the UV and IR spectral range. Furthermore, an elongated structure as in Solophenyl Bordeaux 3BLE also seems to have a positive effect.

In the literature, different approaches are reported to realize textile materials with reduced transmission for UV light and near-infrared light. Prominent examples are based on coatings with pearlescent effect pigments, short basalt fiber, or pigments from indium tin oxide [51,67,73]. The application of pearlescent pigments offers good UV protection due to their content of titanium dioxide. Their capability to reduce transmission for near infrared light depends on interference effects and can yield to reduced transmission values of around 25% at 750 nm [51]. The developed Lyocell materials exhibit lower transmission in this spectral range and are therefore advantageous. The application of short basalt fibers with a high concentration of more than 40 wt-% can lead to textile materials with reduced transmission for near-infrared light with transmission values of around 10% [67].

With a view on this low transmission values, these basalt fiber-containing coatings are advantageous compared to the currently developed Lyocell knitted fabrics. However, due to the high content of inorganic basalt fiber in the coating, the textile properties of these coated materials are lost. Finally, coatings from sol-gel material and indium tin oxide applied on cotton can lead to the low transmission for near-infrared light with transmission values of 10% or less [73]. These sol-gel composite materials are superior compared to the developed Lyocell knitted materials if the capability for protection against IR light is discussed. However, their UV protective properties are not that excellent, and also a change in textile properties as flexibility and hand feeling occurs after sol-gel application. By view of these comparative materials, it can be stated that the currently developed dyed Lyocell materials exhibits comparable or even better protection against UV light and near-infrared light. Especially advantageous is the remaining textile character of the Lyocell knitted fabrics which enables direct transfer to application in the textile and clothing sector.

5. Conclusions

Knitted fabrics produced from Lyocell yarn can be used as an effective basis to realize textile materials offering protective properties against UV and near IR radiation. The use of Lyocell fibers spin-doped with 2% titanium dioxide leads already to a significant transmission reduction for UV radiation. However, in the spectral range of 385 nm to 400 nm for UV light the transmission reduction should be further improved. As well, for the transmission of IR radiation still, optimization is necessary. By application of dyestuffs, the transmission of UV light for these Lyocell knitted fabrics is drastically decreased. Further, the proof-of-concept is complete, that similarly the transmission for near-infrared light can be decreased by dye application. By this, a first step is complete for the development of textile and clothing materials offering additional UV protection also a certain capability for protection against infrared radiation.

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