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INTERNATIONAL ADVANCED RESEARCHES and ENGINEERING JOURNAL

Journal homepage: www.dergipark.org.tr/en/pub/iarej

e-ISSN: 2618-575X

International Open Access

Volume 08
Issue 01

April, 2024

Research Article

Ecological dyeing and UV-protective functionalization of cotton/lyocell blend fabrics designed for high comfort summer clothing

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ARTICLE INFO

Article history:
Received 04 October 2023
Accepted 22 February 2024
Published 20 April 2024

Keywords:
Avocado seed
Clothing comfort
Lyocell
Natural dye
UV-protective clothing

ABSTRACT

Wearing clothes that absorb or block harmful UV radiation is one of the most effective forms of protection against sun damage and skin cancer. In the current study, sustainable processes which will provide high clothing comfort and a sufficient level of UV protection for fabrics used in production of lightweight sun-protective summer clothing were proposed. In accordance with the scope of the study, structural properties, low-stress mechanical properties, surface characteristics, permeability, and UV-protection properties of three woven fabrics produced with different weft settings and lyocell ratios were determined. The fabric type which had the most desirable results was selected for dyeing experiments. Avocado (*Persea americana*) seed was used as an eco-friendly source for dyeing and UV-protective functionalization of cotton/lyocell blend fabric. To obtain different hues, the fabrics were mordanted according to two different recipes using magnesium sulphate and ferrous sulphate and dyed with the natural colorant extracted from avocado seeds. Comfort, color, fastness, and UV-protective properties of dyed samples were evaluated by laboratory tests. Results indicated that it is possible to achieve an excellent level of UV protection (UPF 50+) by dyeing cotton/lyocell blend fabrics with avocado seed extract and produce summer clothing with improved comfort and UV-protective properties, without using any toxic materials.

1. Introduction

Ultraviolet (UV) radiation is a form of electromagnetic radiation that comes from the sun and several man-made sources. The most common types of skin cancer are found on sun-exposed parts of the body, and their occurrence is commonly related to exposure to the UV rays in sunlight. Melanoma is a more serious but less common type of skin cancer, and it is also related to sun exposure [1].

UV radiation is divided into three groups: UVA, UVB and UVC. Exposure to UVA rays can lead to aging of skin cells and cause some indirect damage to cells' DNA. UVB rays are the main cause of sunburn, and they can also damage the DNA in skin cells [1]. UVC reacts with ozone in the atmosphere, consequently, it doesn't reach the ground.

Recent studies indicate that insufficient sun exposure may be responsible for increased incidences of several health problems such as breast cancer, colorectal cancer, hypertension, cardiovascular disease, metabolic syndrome, multiple sclerosis, Alzheimer's disease, autism, asthma, type 1 diabetes, and myopia [2]. Recognition of the beneficial effects of UV exposure has led to a reconsideration of sun avoidance policies [3,4]. As it provides several health benefits, it is not suggested to avoid sunlight completely, but it is necessary to limit the duration and intensity of the exposure. Staying in the shade during midday hours, protecting the skin with proper clothing and using sunscreens are common ways for UV protection.

Thermo-physiological comfort is a very important aspect to be considered, especially for summer clothing [5-9]. Lightweight fabrics made of cotton and regenerated cellulosic fibers such as viscose, modal and lyocell are often preferred in summer clothing because of their good tactile comfort, high air permeability and high moisture vapor transmission rates [10]. However, the level of UV protection provided by textiles greatly changes depending on several parameters such as raw material, fabric construction, color and application of functional finishes [10,11]. Lightweight cotton fabrics with bright colors and low coverages may not provide the required level of protection against UV rays [12]. Researchers previously reported that textiles dyed with natural colorants may

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DOI: 10.35860/iarej.1371104

exhibit increased levels of UV protection [13-21]. Natural dyes are renewable and sustainable bioresource products. In this respect, dyeing fabrics with natural colorants can be a sustainable option for producing sun-protective summer clothing. Using natural sources can also minimize the several negative effects of synthetic dyes [20-28].

Another important aspect of sustainable production is the fiber content. Cotton is a natural fiber which is commonly preferred in summer clothing. However, producing 1 kg of cotton fiber requires more than 20000 L of water [29]. Moreover, cotton is responsible for 11% and 24% of the world's pesticides and insecticides consumption, respectively. Lyocell is a biodegradable regenerated cellulosic fiber which requires a lot less dye than cotton and no bleaching, thus it is a more sustainable option in terms of energy and water consumption [30]. Besides eco-sustainability, using lyocell and its blends in garments provides a series of desirable attributes such as comfort, efficient moisture transportation, vivid colors with high fastness grades, an elegant drape, a strong durability, and antibacterial properties [29-32].

There are several studies investigating the use of natural dyes for sustainable coloration of lyocell fabrics [20-26]. A review of the previous literature indicated that UV-protective properties of lyocell/cotton blend fabrics can be successfully enhanced using aqueous extracts of several plants including kermes oak (*Quercus coccifera* L.) seeds and mango (*Mangifera indica* L.) bio-wastes (seed and peel) [20,21].

The estimated total world production for avocados (*Persea americana*) in 2022 was over 8.9 million tons [33]. In this respect, bio-waste of avocado fruit - which is approximately 27% the of the total mass - can be a favorable source for coloration of textiles. Even though it was previously reported that cotton and wool fabrics can be successfully dyed with pigments extracted from avocado seeds and peels [34-36], coloration and UV-protective functionalization of lyocell blended fabrics using avocado bio-wastes was not discussed. In a study carried out by Cuk and Gorjanc, cotton was dyed with extracts of curcuma, green tea, avocado seed, pomegranate peel and horse chestnut bark [36]. However, the reported UV protection levels achieved by applying avocado seed extract on cotton fabric by the suggested extraction and dyeing methods were quite low.

The aim of the current study was to propose a fabric design and sustainable processes which will provide the desired qualities such as high clothing comfort and a sufficient level of UV protection for producing lightweight sun-protective summer clothing. In accordance with this purpose, several components of clothing comfort were investigated by laboratory tests for three fabrics made of cotton and lyocell fibers. The fabric which provided the most desirable results were dyed with avocado seed extract according to three different recipes. The dyeability of cotton and lyocell fibers with the proposed natural colorant was investigated based on color and fastness results. The efficiency of avocado seed extract as a natural agent for UV-protective functionalization was also discussed.

2. Materials and Methods

2.1 Material

The fabrics used in the current study have a 5-end warp-dominant satin weave pattern, produced with identical yarns and weave parameters (except weft setting). Yarn structure has a large effect on fabric comfort. For instance, woven fabrics produced with compact-spun yarns may possess better air and water vapor permeabilities when compared to fabrics produced with ring-spun yarns [37]. For the fabrics investigated in the current study (LL, ML, HL), compact yarns were used in both warp and weft directions. The warp warns were made of 100% combed cotton and the weft yarns were made of 100% lyocell fibers (Table 1).

Aqueous extract of avocado (*Persea americana*, Hass cultivar) seed was used for dyeing experiments. Fully ripe avocados were procured from a local market. $Mg(SO_4)$. 7 H_2O (magnesium sulfate), $FeSO_4$. 7 H_2O (ferrous sulfate), and $NaHCO_3$ were supplied from Merck (Darmstadt, Germany).

2.2 Extraction

The seeds of avocado fruit were separated from the pulp and rested at room temperature for two hours. The thin outer shell of the seeds was peeled, and the seeds were dipped into the 0.01M $NaHCO_3$ solution with a material to liquor ratio of 1:20.

The extraction was carried out in a beaker at 85 °C and the duration of extraction was three hours. During the extraction, the liquor volume was kept constant by covering the top of the beaker with an elastic cover. When the extraction was finalized, solid residues of avocado seeds were separated from the dye extract by using a 100% cotton filtering fabric.

2.3 Dyeing

For dyeing, samples ML1, ML2, and ML3 were dipped into the prepared avocado seed extract with an M:L ratio of 1:20. Dyeing was carried out by resting the samples in the prepared dye solutions for 3 hours, at room temperature, inside glass bottles with sealed caps. During the 3-hour dyeing process, for each 20 minutes of resting, the dye solution and the sample were mixed by manually shaking the bottles for 5 seconds. After 3 hours of dyeing, samples were rinsed with running tap water for five minutes and rested on a hanger for drying.

Table 1. Production parameters of the studied fabrics

Sample code	Weave pattern	Yarn properties		Setting (cm^{-1})	
		Warp	*Weft*	*Warp*	*Weft*
LL	5-end satin	Cotton (9.8 tex)	Lyocell (9.8 tex)	82	40
ML	5-end satin	Cotton (9.8 tex)	Lyocell (9.8 tex)	82	45
HL	5-end satin	Cotton (9.8 tex)	Lyocell (9.8 tex)	82	50

2.4 Mordanting

To obtain different hues, the fabrics were mordanted with nontoxic metallic salts - magnesium sulphate and ferrous sulphate. Sample ML2 was pre-mordanted with magnesium sulphate and simultaneously mordanted with ferrous sulphate. Sample ML3 was only pre-mordanted with ferrous sulphate. No mordanting process was carried out for sample ML1.

- Pre-mordanting: For pre-mordanting, two separate solutions were prepared. Magnesium sulphate (5% owf) and ferrous sulphate (2% owf) were dissolved in distilled water and the samples (ML2 and ML3 respectively) were rested in prepared mordant solutions at room temperature for 24 hours. No rinsing was carried out after pre-mordanting. Mordanted samples were dried on a hanger.
- Simultaneous mordanting: Simultaneous mordanting was applied to sample ML2 only. For simultaneous mordanting, sample ML2 was removed from the dye bath at the end of the first hour of dyeing. When the fabric sample was removed, 0.35% owf of ferrous sulphate was dissolved in the dye bath, and sample ML2 was dipped back into the mordant added dye solution. The sample was then rested in the mordant added dye solution for two more hours. The total dyeing duration of sample ML2 was three hours, which was equal to the dyeing duration of sample ML1 and ML3.

2.5 Measurements

Structural parameters (mass per unit area and thickness), low-stress mechanical properties (extensibility and bending rigidity), out-of-plane deformation and recovery behavior (deformation, elasticity, plasticity, and hysteresis), surface characteristics (micro and macro-surface variations), tactile comfort, permeability (air and water vapor), color, fastness and UV-protective properties of fabrics were determined by laboratory tests. The samples were conditioned before measurements as prescribed in TS EN ISO 139/A1. All measurements were carried out at standard atmospheric conditions.

- Structural properties: Mass per unit area was measured according to TS EN 12127. The thickness of fabrics was measured under 5 gf/cm^2 pressure using James Heal R&B Cloth Thickness Tester having a circular presser foot with a size of 100 mm^2. Five trials were done for each fabric type.
- Extensibility: Extensibilities in warp and weft directions were determined using Instron 4411 Universal Tensile Tester. The sample size was 250 mm x 50 mm, the test length was 150 mm, the test speed was 25 mm/min, and the pre-tension was equal to zero. Percent elongation values recorded under 100 N/m load were presented as extensibility. Five trials were done for each test direction and fabric type.
- Bending rigidity: Bending rigidity was determined as prescribed in ASTM D1388-18 Option A. Bending properties were determined for warp, weft and two diagonal (-45° and +45°) directions. Five trials were done for each test direction and fabric type.
- Out-of-plane deformation: Deformation and recovery behavior of fabrics in out-of-plane deformation state was investigated using a multifunctional test instrument "Tactile Sensation Analyzer" (TSA) developed by the German company emtec Electronic GmbH [38,39]. For the measurements, the sample (120 mm x120 mm) was fixed to the circular frame and deformed in the out-of-plane direction by the movement of the measuring head of the device. When a load of 600 mN is reached, the magnitude of deformation (D) was recorded. Then the same sample was automatically deformed by the test device for the second time, and the magnitude of deformation observed in this cycle was recorded as elasticity (E). The energy of recovery after the first cycle was recorded as hysteresis (H) and the magnitude of permanent deformation was recorded as plasticity (P) [38]. Five samples were tested for each fabric type.
- Surface characteristics: Macro-surface variations and micro-surface variations of face and back sides of the fabrics were measured by Tactile Sensation Analyzer. For investigating surface characteristics, the test sample was fixed to the circular frame. When the measuring head contacted with the sample, applying a 100 mN load, the head rotated on the fabric surface, and the sound intensity peaks at two frequency regions were recorded using a special sound analysis technique. These two peaks were named as TS7 and TS750 which refer to micro-surface variations and macro-surface variations, respectively. For the measurements, five samples (120 mm x 120 mm) were tested for each sides of the fabrics.
- Nozzle test: Nozzle test is a conventional test method where the force required to pull a fabric sample through a ring, or a nozzle is determined [40-42]. It was previously reported that the pulling forces recorded during nozzle test were significantly correlated with tactile comfort scores of fabrics determined by sensory evaluations [41,42]. In the current study, a nozzle construction with 24 mm diameter and 20 mm height was used. For the measurements, a circular sample with 100 mm diameter was attached to a needle, pulled through the nozzle with a speed of 25 mm/min and the maximum pulling force was recorded. For each fabric type, five trials were done.
- Air permeability: Air permeability was determined according to TS 391 EN ISO 9237 using Textest FX 3300 Air Permeability Tester III. The test area was selected as 20 cm^2 and the test pressure was 100 Pa. 10 measurements were done for each fabric type.
- Water vapor permeability: Water vapor permeability was measured as prescribed in BS 7209:1990 using SDL ATLAS equipment no M261. The duration of the measurements was 24 hours. Three trials were done for each fabric type. Permeability was tested with the principle of measuring the water vapor movement from back side towards the face side.
- Color and fastness properties: Color of samples were measured using Minolta Spectrophotometer CM-3600d (illuminant D65 and 10° observer angle) and presented in

CIE L* a* b*, C*, h° coordinate system. Color strength (K/S) of dyed samples were given as well. Fastness to washing was measured according to ISO 105-C06:2010, method A1S and fastness to rubbing was measured according to ISO 105-X12:2001.

- UV-protective properties: UVA transmittance (%), UVB transmittance (%) and Ultraviolet Protection Factor (UPF) of face side of dyed samples were measured according to the Australian/New Zealand standard AS/NZ4399/1996, using SDL Atlas Camspec M350 Ultraviolet-Visible Spectrophotometer. Based on the classification system suggested by the related standard, the fabrics were categorized according to their UPF ratings [43].

3. Result and Discussion

3.1 Results of Undyed Fabrics

In the current study three - undyed - fabrics were produced with three different weft settings (Table 1). As a consequence of having different settings, these fabrics exhibited different mass per unit area values (Table 2). However, thickness of these fabrics remained within a similar range.

The differences in structural properties may lead to significant changes in low-stress mechanical properties as well. It was observed that the fabric with the lowest weft setting (LL) has the lowest bending rigidity and the highest extensibility results (Table 3).

Higher deformation (D) and elasticity (E) results are usually desired for a better comfort. As can be seen in Table 4, sample ML has the highest D and E values. Similarly, ML has the lowest magnitude of permanent deformation (P) and hysteresis (H) when compared to samples LL and HL. These findings indicate that fabric ML can provide the optimum tactile comfort in terms of deformation and recovery behavior under out-of-plane deformation.

It was also observed that variations of fabric surface increased with the increase in weft setting (Table 5). For micro-surface variations (TS7) this finding was more prominent for face side results. On the other hand, the increase observed in macro-surface variations was more significant for back side of the fabrics. As higher surface variations are associated with a less desirable fabric surface, samples with lower TS7 and TS750 values may provide a more desirable tactile sensation.

The maximum pulling force recorded during nozzle test is commonly used as a measure of tactile comfort where a lower resistance when passing through a nozzle is an indicator of a better tactile comfort [41,42]. It was observed that sample LL has the lowest pulling force. Therefore, in terms of low-stress mechanical properties and nozzle test results, it is possible to state that LL exhibited the most desirable results.

A change in structural parameters may cause significant differences in permeability properties as well. It was recorded that LL has higher air permeability (Table 6). These findings indicated that using less number of weft yarns provided better tactile comfort, more desirable low-stress mechanical properties and better air permeability.

However, as warp yarns were made of 100% cotton and weft yarns were made of 100% lyocell, using different weft settings provided differences in overall cotton/lyocell ratios of fabrics as well, which led to different water vapor permeability results. The transfer of water vapor from back side to face side of fabrics was faster for the fabric which has a moderate level of porosity and lyocell content (sample ML) (Table 6).

Moreover, fabrics with a high coverage may block more of the UV rays. In this respect, producing a fabric with low coverage and high porosity may lead to a less desirable level of UV protection. Results of the study indicated that fabrics with moderate and high weft settings (ML and HL) have similar levels of UV protection, which were higher than the sample with the lowest weft setting (LL) (Table 7). With respect to aforementioned test results, it was concluded that sample ML provided the optimum results. Therefore, sample ML was selected for the dyeing experiments.

Table 2. Structural properties of undyed fabrics

Sample code	Mass per unit area	Thickness
	(g/m²)	*(µm)*
LL	123	254
ML	129	258
HL	135	256

Table 3. Low-stress mechanical properties and nozzle test results of undyed fabrics

Sample code	Extensibility	Bending rigidity	Pulling force
	(%)	*(µJ/m)*	*(cN)*
LL	2.40	7.38	25.2
ML	2.06	7.97	31.3
HL	1.99	8.37	37.4

Table 4. Out-of-plane deformation and recovery results of undyed fabrics

Sample code	Deformation (D)	Elasticity (E)	Plasticity (P)	Hysteresis (H)
	(mm/N)	*(mm/N)*	(µm)	*(J)*
LL	1.81	1.63	175	89.9
ML	1.85	1.75	124	71.8
HL	1.81	1.66	146	77.9

Table 5. Surface characteristics of undyed fabrics

Sample code	Side	Micro-surface variations (TS7)	Macro-surface variations (TS750)
LL	Face	10.9	79.6
ML	Face	11.2	82.7
HL	Face	12.8	85.7
LL	Back	12.7	78.6
ML	Back	12.4	84.9
HL	Back	12.9	95.6

Table 6. Air permeability and water vapor permeability of undyed fabrics

Sample code	Air permeability	Water vapor permeability
	(mm/s)	*(g/m²/day)*
LL	100.6 ± 2.8	786.5 ± 20.2
ML	83.0 ± 2.4	813.2 ± 40.9
HL	60.7 ± 2.0	809.6 ± 33.2

Table 7. UV-protective properties of undyed fabrics

Sample code	UVA	UVB	UPF	UPF rated
LL	25.9	15.4	5.63	5
ML	25.4	14.7	5.86	5
HL	25.5	14.3	5.98	5

UVA: % UVA transmittance, UVB: % UVB transmittance, UPF: UV protection factor, UPF rated: Rated UV protection factor

3.2 Color Properties of Dyed Fabrics

It was observed that the color of the sample dyed without any mordant (ML1) was a moderate shade of yellowish pink (Figure 1). Sample ML2 - which was pre-mordanted with magnesium sulphate and simultaneously mordanted with ferrous sulphate - exhibited a brown color. Meanwhile pre-mordanting the fabric with ferrous sulphate resulted in a grey color (ML3). Results of color measurements indicated that color strength (K/S) was increased from 0.75 to 1.76 and 1.78 by the suggested mordanting processes (Table 8). On the other hand, mordanting caused a noticeable decrease in redness (a*), yellowness (b*) and chroma (C*) values.

All samples exhibited excellent dry rubbing fastness grades (5) and very good wet rubbing fastness grades (4-5) regardless of the mordanting option (Table 9). Washing fastness against staining was also at a very good level (≥4-5). The color change of unmordanted sample during washing fastness test was on a moderate level (3 and 2-3 for face and back sides, respectively), which was significantly improved after mordanting.

3.3 UV-Protective Properties of Dyed Fabrics

According to The Skin Cancer Foundation's Seal of Recommendation, a fabric must have a minimum Ultraviolet Protection Factor (UPF) of 30 to qualify as sun-protective clothing [44].

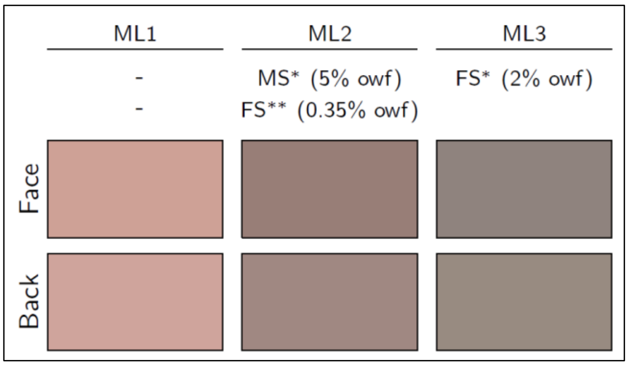

Figure 1. Color of face and back sides of the fabrics pre-mordanted* and simultaneously mordanted**with magnesium sulphate (MS) and ferrous sulphate (FS) and dyed with avocado seed extract

It was observed that sample ML1, which was dyed with avocado seed extract without any mordant has a UPF of 36.9 and a rated UPF of 35 (Figure 2.a), which indicates a very good level of protection against UV light. Even UVA and UVB transmittance values of ML1 slightly increased after washing, it was observed that ML1 still has a moderate level of UV protection after washing fastness test, as the rated UPF of ML1 after fastness test was 20 (Figure 2.b).

Both mordanted samples (ML2 and ML3) exhibited excellent levels of protection against UV light (UPF 50+). It was observed that sample ML3 which was pre-mordanted with ferrous sulphate exhibited the highest UPF result (Figure 2.a). The effective UV radiation transmission (%) values of ML2 and ML3 were lower than 2.5, and UPF values were 119 and 256, respectively.

The decrease in rated UPF of sample ML3 observed after washing fastness test was only 1.6% (Figure 2.b). For sample ML2, the decrease in UPF value after washing fastness test was 29.6%. Even after washing fastness test, the rated UPF of samples ML2 and ML3 remained as excellent (50+).

Table 8. Color results of samples dyed with avocado seed extract

Sample Code	Side*	Mordant	K/S	L*	a*	b*	C*	h°
ML1	Face	-	0.751	70.2	16.0	12.2	20.1	37.3
ML2	Face	Mg, Fe	1.763	55.0	9.61	7.50	12.2	38.0
ML3	Face	Fe	1.781	55.8	4.03	4.84	6.30	50.2
ML1	Back	-	0.653	71.4	15.1	10.7	18.5	35.3
ML2	Back	Mg, Fe	1.333	58.7	8.84	6.46	11.0	36.2
ML3	Back	Fe	1.789	58.8	3.84	7.18	8.14	61.9

*Side: Face side is warp dominant (cotton warp yarns are visible) and back side is weft dominant (lyocell weft yarns are visible).

Table 9. Washing, rubbing and light fastness results of fabrics dyed with avocado seed extract

Fabric code	Side*	Washing fastness							Rubbing fastness	
		Color change	Staining							
			WO	PAN	PET	PA 6.6	CO	CA	Dry	Wet
ML1	Face	3	5	5	5	5	4-5	5	5	4-5
ML2	Face	4-5	5	5	5	5	5	5	5	4-5
ML3	Face	4	5	5	5	5	5	5	5	4-5
ML1	Back	2-3	5	5	5	5	4-5	5	5	4-5
ML2	Back	3-4	5	5	5	5	5	5	5	4-5
ML3	Back	4-5	5	5	5	5	5	5	5	4-5

*Side: Face side is warp dominant (cotton warp yarns are visible) and back side is weft dominant (lyocell weft yarns are visible).

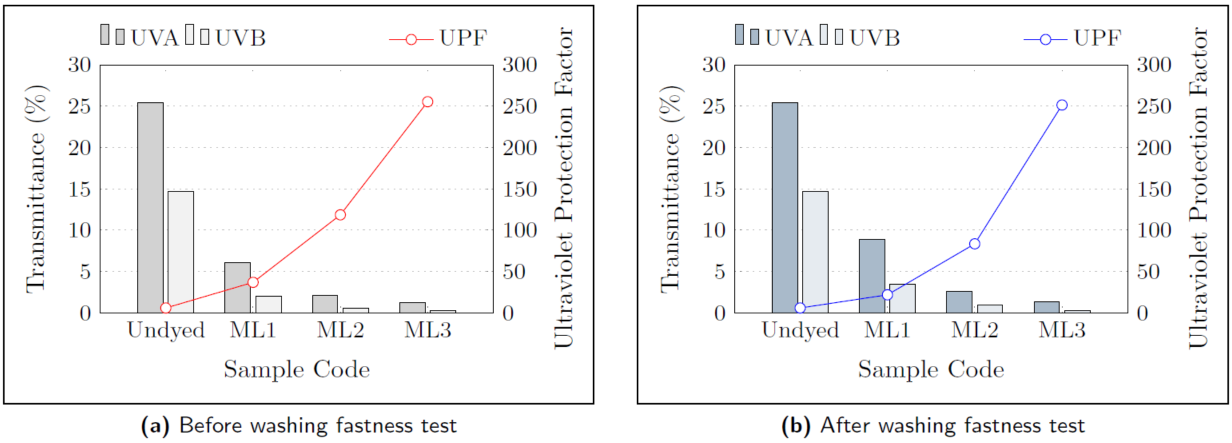

Figure 2. UVA (315 to 400 nm) and UVB (290 to 315 nm) transmittance (%) values and Ultraviolet Protection Factor (UPF) of fabrics dyed with avocado seed extract, measured (a) before and (b) after washing fastness test

4. Conclusions

Sunlight contains ultraviolet (UV) radiation which can negatively affect human skin in several ways. In this respect, developing alternative solutions which would provide protection against the possible harmful effects of UV rays is an important research topic.

In this study eco-friendly production steps for obtaining a UV-protective summer clothing with high clothing comfort were proposed. As lyocell is a more sustainable raw material when compared to cotton and a luxury fiber with several advantages such as high comfort and silk like structure, yarns made of 100% lyocell fibers were used as weft yarns and the effect of using different number of weft yarns on comfort and UV protection was discussed. Avocado seed was proposed as a natural dye source for textiles. Fabrics were mordanted with non-toxic metallic salts and dyed with avocado seed extract. Color, fastness, and UV-protective properties of dyed samples were determined and discussed.

Results of the study indicated that selecting different weft settings can provide significant changes in several fabric characteristics including low-stress mechanical properties and permeability. It was observed that both cotton and lyocell fibers had a good dyeability by the avocado seed extract, as both warp dominant face side and weft dominant back side of the dyed fabric samples exhibited vibrant colors with high K/S values and moderate to excellent fastness grades.

It was recorded that dyeing the studied fabric with avocado seed extract can significantly improve UV-protective properties as the UPF of raw fabric increased from 5.86 to 36.9 after dyeing, even without using any mordants. In the case of using different concentrations of magnesium sulphate and ferrous sulphate for mordanting, UPF was increased to an excellent level (50+) and these samples preserved their UV-protective property even after washing fastness test.

Findings of the study certified that it is possible to produce a light-weight fabric for summer clothing with excellent UV-protective properties and high clothing comfort, using sustainable processes, without including any toxic materials.

Declaration

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article. The author also declared that this article is original, was prepared in accordance with international publication and research ethics, and ethical committee permission or any special permission is not required.

Author Contributions

N. Uren developed the methodology, performed the experiments, measurements and data analysis, and wrote the manuscript.

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