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Natural Dyeing and UV Protection of Raw and Bleached/ Mercerised Cotton

UV zaščita surovega in beljenega/merceriziranega bombaža barvanega z naravnimi barvili

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Abstract

Dyeing with natural dyes extracted from curcuma, green tea, avocado seed, pomegranate peel and horse chestnut bark was studied to evaluate the dyeability and ultraviolet (UV) blocking properties of raw and bleached/mercerised cotton fabrics. 20 g/l of powdered plant material was extracted in distilled water and used as a dyeing bath. No mordants were used to obtain ecologically friendly finishing. The colour of samples was measured on a reflectance spectrophotometer, while UV-blocking properties were analysed with UV-Vis spectrophotometer. The results showed that dyeing increased UV protection factor (UPF) to all samples, however much higher UPF values were measured for the dyed raw cotton samples. The highest UPF values were obtained on both cotton fabrics dyed with pomegranate peel and green tea extracts, giving them excellent protective properties (UPF 50+). The lowest UPF values were obtained by dyeing cotton with avocado seed extract and curcumin. Dyeing with selected dyes is not stable to washing, so the UV-blocking properties worsen after repetitive washing. However, raw cotton samples retain their very good UV-blocking properties, while bleached/mercerised cotton fabrics do not provide even satisfactory UV-blocking properties. No correlation between CIE *L*a*b**, *K/S* and UPF values were found.
Keywords: UV protection, dyeing, natural dyes, cotton

Izvleček

*V raziskavi je bilo proučevano barvanje surovega in beljenega/merceriziranega bombaža z naravnimi barvili, ekstrahiranimi iz kurkume, zelenega čaja, avokadovega semena, olupka granatnega jabolka in lubja navadnega divjega kostanja. Pobarvanim vzorcem bombaža sta bila določena obarvljivost in faktor zaščite pred ultravijoličnim (UV) sevanjem. Barvalna kopel je bila pripravljena iz 20 g/l posameznega rastlinskega materiala, ekstrahiranega v destilirani vodi. Da bi bilo barvanje čim bolj okolju prijazno, pri barvanju niso bile uporabljene čimže. Barva vzorcev je bila izmerjena na refleksijskem spektrofotometru, medtem ko so bile UV-zaščitne lastnosti vzorcev analizirane s pomočjo UV-Vis spektrofotometra. Rezultati so pokazali, da je barvanje z naravnimi barvili bombažnim vzorcem povečalo vrednosti UV zaščitnega faktorja (UZF) bombažnih vzorcev, vendar so bile višje vrednosti izmerjene pri surovem bombažu. Najvišje UZF-vrednosti, ki opredeljujejo tekstilije z najboljšo UV-zaščito (UZF 50+), so bile dosežene pri obeh substratih bombaža, barvanega z ekstrakti olupka granatnega jabolka in zelenega čaja. Najnižje UZF-vrednosti so bile izmerjene za bombaž, ki je bil pobarvan z ekstrakti avokadovega semena in kurkume. Ker obarvanja niso bila stabilna na večkratno pranje, se je vzorcem zmanjšala tudi zaščita pred UV-sevanjem. Vseeno pa so vzorci surovega bombaža še vedno zagotovili zelo dobro zaščito pred UV-sevanjem, medtem ko vzorci beljenega/merceriziranega bombaža niso dosegali niti zadovoljive UV-zaščite. V raziskavi ni bilo korelacije med CIE L*a*b*, K/S in UZF-vrednostmi.*
Ključne besede: UV-zaščita, barvanje, naravna barvila, bombaž

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1 Introduction

The art of applying different colours to textile materials has been known since pre-historic times. W. H. Perkins broke supremacy of natural dyes with the first synthetic counterpart in 1856 [1]. Moreover, the development of chemistry and textile industry accelerated the use of synthetic dyes and pigments. Their advantages compared to natural dyes are lower prices, better colour fastness and wider range of shades. On the other hand, the awareness of their harmful, carcinogenic and not bio-degradable properties became present among certain parts of society [2, 3]. Environmentally conscious manufacturers are trying to decrease harmful consequences on human health and environment using natural dyes. Due to their biodegradability and functional properties natural dyes are also used in food, pharmaceutical and cosmetic industry [4]. The major problem associated with dyeing of cotton with natural dyes is their inadequate colour fastness [5]. In addition to their dyeing properties, some natural dyes also have UV-blocking and antimicrobial properties. The UV-blocking properties of textile substrates dyed with natural dyes are of great interest, because the Sun-caused allergies and skin damages (i.e. sunburn, erythema, photo-aging and photo-carcinogenesis) have increased in recent years [6].

The purpose of our research was to dye cotton fabrics with natural dyes, i.e. extracts of curcuma rhizome, green tea leaves, avocado seed, pomegranate peel and horse chestnut bark. The objective of the research was to evaluate the dyeability and UV-blocking properties of dyed raw and bleached/mercerized cotton without using any mordants.

2 Theoretical background

2.1 Curcuma rhizome

Curcuma rhizome (*Curcuma longa*) belongs to the ginger family (*Zingiberaceae*). It probably originates from South or Southeast Asia. Firstly, it was cultivated as a dye, later it was used as a spice, for cosmetic and medicinal purposes. The dyeing constituent of curcuma rhizome is the polyphenolic pigment curcumin (Figure 1) [7]. Due to the keto and enol tautomeric conformations in solid state or liquid, the colour of curcumin depends on a pH of the solution; alkali solution results in red and acid solution in yellow colour [8]. Adeel S. and Osman E. [9] showed that the extract of curcuma might be an alternative for yellow synthetic dyes due to good colour strength and acceptable colour fastness. The best result was obtained when cotton and curcuma powder were UV-irradiated. The acid-treated cotton dyed with cationised curcumin yielded excellent UV protection property and good durability to home laundering [10]. The result is classified as an excellent UV protection due to interaction between cationic curcumin cation and cellulosic citrate anion. The colour fastness of cotton samples dyed with turmeric extract is generally better when using mordants [4]. Their application can also influence on the nuance of the final colour.

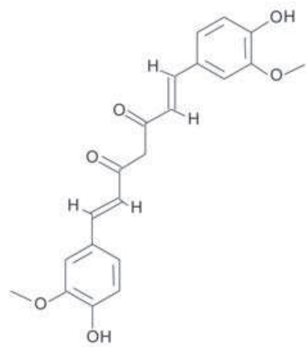


Figure 1: The chemical structure of curcumin [7]

2.2 Green tea

Green tea is produced from *Camellia sinensis* leaves. It originates from China and is nowadays worldwide spread. Tea polyphenol mostly consists of catechin (Figure 2), which is a very effective UV absorber. Catechin is one of the natural phenols and antioxidants [11]. Due to antioxidant and deodorization functions on natural fibres, many researches were carried out using green tea as a dye [12]. It was found that hot extraction of red, black and green tea provided cotton fabrics with UV-blocking properties [13]. The UV-protection factor (UPF) was mostly connected to the method of extraction as well as the colour of the tea. The green tea showed the lowest UPF value. Kim S. [14] increased the adsorption of green tea extract with chitosan mordanting of cotton fabrics, and its ability for UVA and UVB protection. The UV protection generally increased approximately by 7%, which is similar value to a cellulose fabric dyed with green tea and metal mordant.

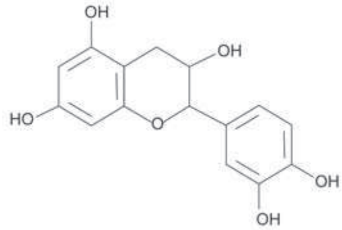


Figure 2: The chemical structure of catechin [11]

2.3 Avocado seed

Avocado (*Persea americana*) belongs to the *Lauraceae* family. The cultivation of this fruit originates from Mexico. It is best known as an important protein and lipid supplier in human diet. The seed accounts for 16% of total avocado weight, and is an under-utilized resource and today classified as a waste which contributes to the environmental pollution [15, 16]. Avocado seeds and pulp are full of hydroxycinnsmin acids, catechins and procyanidins which have antioxidative and antimicrobial properties [17]. The extracts of avocado seed showed antibacterial properties against several strains of *Streptococcus agalactiae* [18]. The seeds could also be used as a source of natural dye. The colour of the extract is supposedly connected to the procyanidins, the oligomeric compounds, formed from catechin (Figure 2) and epicatechin (Figure 3) molecules [19, 20]. Currently no reported research was performed in dyeing of textiles with avocado seed extract. The UV-blocking properties have not been studied yet as well.

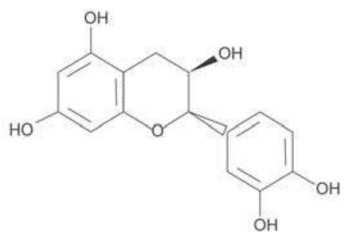


Figure 3: The chemical structure of epicatechin [21]

2.4 Pomegranate peel

Pomegranate (*Punica granatum*) belongs to the *Punicaceae* family. It originates from Persia and is nowadays a common plant in the countries with warm climates. Pomegranate peel contains phenolics, flavonoids and tannins. It also contains pigments called anthocyanidins (Figure 4). Those pigments are further divided into pelargonidins, cyanidins and delphinidins [22–25]. The final colour of 100% cotton samples dyed with pomegranate peel extract can vary from yellow, brown to black and is a result of the used mordant. When using metal salt, better colour strength is obtained [22]. However, it was found that mordanting process was not essential for dyeing cotton with pomegranate peel extract [23]. Cotton, wool and silk fabrics dyed with pomegranate peel extract showed excellent deodorising performance with high wash fastness [24]. The UV-blocking properties of cotton dyed with pomegranate peel have not been evaluated yet.

2.5 Horse chestnut bark

Aesculus hippocastanum is one of the representatives of the *Sapindaceae* family. Native range of the plant is Balkan [30, 31]. Bark contains two different types of coumarins glycosides; aesculine and fraxine.

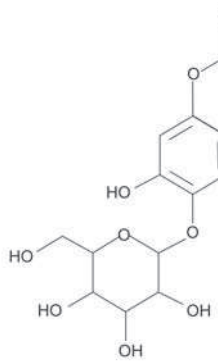
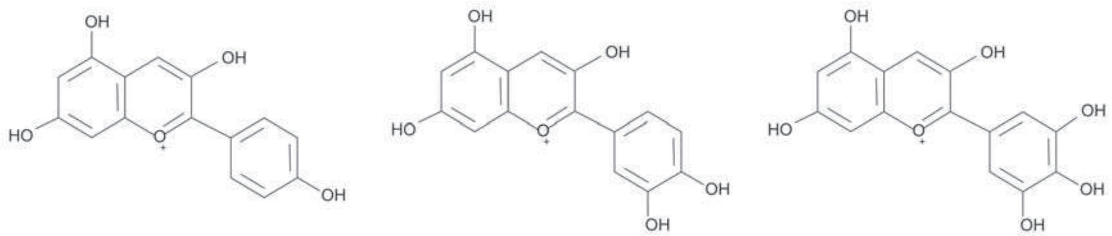


Figure 5: The chemical structure of aesculin [31]



(a) (b) (c)

Figure 4: The chemical structure of anthocyanidins (a) pelargonidin, (b) cyanidin, (c) delphinidin [25–29]

Aesculin (Figure 5) is a good UVB absorber and has the maximum absorbance at 346 nm and another peak at 250 nm [30]. Another important substance is a group of tannins (Figure 2 and 3). They also have good antioxidant and antimicrobial properties [30, 31]. There was no evidence of using horse chestnut bark as a dye or as a UV-blocking substance.

3 Experimental

3.1 Material

Two types of plain weaved 100% cotton fabrics were used for the research: raw cotton (warp 53 threads/cm, weft 29 threads/cm, 136.8 g/m^2) and bleached/mercerised cotton (warp 52 threads/cm, weft 26 threads/cm, 119.2 g/m^2). The fabrics were supplied by Tekstina d. d., Ajdovščina.

3.2 Plant extracts

The extracts for dyeing cotton fabrics were prepared from curcuma rhizome (*Curcuma longa*), green tea leaves (*Camellia sinensis*), avocado seed (*Persea americana*), pomegranate peel (*Punica granatum*) and horse chestnut bark (*Aesculus hippocastanum*). The curcuma rhizome powder and green tea crushed leaves were used as received. The avocado seed, pomegranate peel and horse chestnut bark were cut in smaller pieces, washed in cold deionised water and 0.5 g/l non-ionic surfactant, rinsed thoroughly and dried. The dried plants were then grinded to powder using a blender. The extracts were prepared by immersing 20 g/l of powder from each plant in cold distilled water. The temperature of the solutions was then increased to a boiling and held at that temperature for 5 minutes. Afterwards all solutions were left to cool for two hours and then filtered. The prepared extracts were used for dyeing cotton fabrics.

3.3 Dyeing of cotton fabrics

Dyeing of raw and bleached/mercerised cotton fabrics with prepared plant extracts was performed in stainless-steal flasks of Launder-o-meter apparatus at 60 °C for 60 minutes. The liquor to goods ratio was 20:1. Afterwards dyed samples were rinsed in cold deionised water and air-dried at room temperature.

3.4 Durability to washing

The dyed samples were washed in laboratory apparatus Launder-o-meter in accordance with EN ISO 105-C06 standard. The size of the samples was 100 x 40 mm, the wash bath contained 4 g/l ECE phosphate reference detergent B, the bath volume was 150 ml, the temperature of washing was 40 °C and the washing lasted for 30 minutes. Ten stainless steel globules were added to each bath to perform washing equal to five domestic washings. The samples were rinsed twice after washing in deionised water and air dried at room temperature.

3.5 Colour measurements

A reflectance spectrophotometer Spectraflash 600 PLUS-CT (Datacolor) was used for measuring CIE L^*a^*b values and reflectance (R) of the samples. Colour differences from the CIE $L^*a^*b^*$ colour values were calculated in accordance with the equation 1:

$$\Delta E^*_{ab} = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \quad (1)$$

where ΔL^* is the lightness difference, Δa^* is a difference on red-green component and Δb^* is the difference in blue-yellow component between standard and a batch.

The colour strength (K/S values) from the reflectance measurement was calculated based on equation 2:

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \quad (2)$$

where R represents the reflectance, K means the absorbance and S represents the scattering of the sample.

Dye uptake was determined from K/S values according to equation 3:

$$\Delta\frac{K}{S} = \frac{(1-R_D)^2}{R_D} - \frac{(1-R_O)^2}{R_O} \quad (3)$$

where R_D is the reflectance of dyed sample and R_O is the reflectance of undyed sample.

3.6 UV protection factor measurements

UV protective properties of the cotton samples were analysed on a Varian CARY 1E UV/VIS spectrophotometer containing Solarscreen software and a DRA-CA-301 integration sphere. The transmittance measurements and calculations of the ultraviolet protection factor (UPF) were carried out according to the AATCC TM 183 standard.

4 Results and discussion

The results of the CIE *L*a*b** colour values of dyed raw and bleached/mercerized cotton samples before and after repetitive washings are presented in Table 1. With the increasing number of washings, the lightness of samples is increasing (CIE *L** values), while the colour values on the red-green axis (CIE *a**) and on the

*Table 1: CIE L*a*b* colour values of unwashed and washed raw and bleached/mercerized samples dyed with studied natural dyes*

Cotton sample	Natural dye extract	Number of washing	*L**	*a**	*b**
Raw	Curcumin	0	84.15	0.45	75.28
		1	85.43	−1.19	65.82
		6	86.21	−2.12	61.49
		12	86.56	−1.27	53.31
	Green tea	0	80.02	4.59	19.76
		1	83.52	3.38	14.09
		6	85.02	2.80	13.05
		12	85.08	2.65	12.35
	Avocado seed	0	85.19	4.08	13.36
		1	86.78	2.93	11.62
		6	87.28	2.58	11.61
		12	87.33	2.48	11.37
	Pomegranate peel	0	78.41	3.98	18.51
		1	83.00	1.56	16.66
		6	83.89	1.46	15.55
		12	84.05	1.35	15.84
	Horse chestnut bark	0	78.28	7.29	18.80
		1	80.67	5.73	15.13
		6	80.18	5.58	14.99
		12	82.01	4.68	13.35
Bleached/ mercerized	Curcumin	0	87.98	−0.31	83.22
		1	86.82	−0.70	70.10
		6	90.75	−4.03	69.67
		12	90.60	−3.55	63.36
	Green tea	0	85.27	3.27	18.05
		1	88.96	1.48	7.64
		6	90.68	1.03	6.57
		12	91.22	0.64	5.78
	Avocado seed	0	87.41	4.62	9.59
		1	89.61	2.63	5.51
		6	90.87	1.68	5.84
		12	90.68	1.83	5.54
	Pomegranate peel	0	82.55	5.74	15.17
		1	85.41	4.28	10.13
		6	87.92	3.00	9.44
		12	88.36	2.79	8.66
	Horse chestnut bark	0	80.47	3.77	16.44
		1	84.54	1.72	12.90
		6	86.71	1.61	10.99
		12	86.98	1.28	10.92

Table 2: The colour difference (ΔE_{ab}) and differences in lightness (ΔL*), on red-green axis (Δa*) and on yellow-blue axis (Δb*) between dyed raw and bleached/mercerised cotton samples*

Extract	ΔE_{ab}^*	ΔL^*	Δa^*	Δb^*
Curcumin	8.85	3.83	−0.76	7.94
Green tea	5.68	5.25	−1.32	−1.71
Avocado seed	4.41	2.22	0.54	−3.77
Pomegranate peel	2.93	2.06	−0.21	−2.07
Horse chestnut bark	5.81	4.27	−1.55	−3.63

yellow-blue axis (CIE b^*) are decreasing, thus moving toward green and blue colour axis. With the increasing number of washing, the CIE a^* values of raw and bleached/mercerised cotton samples dyed with curcumin are moving towards negative values, meaning that the colour values move from red to the green axis. All other dyed samples, regardless of the number of washings, are positioned on the positive part of the CIE a^* and CIE b^* axis (on red and yellow axis).

In Table 2 the colour differences (ΔE_{ab}^*) and the differences in lightness (ΔL^*) on red-green axis (Δa^*) and on yellow-blue axis (Δb^*) between dyed raw and bleached/mercerised cotton samples are presented for each dyeing extract (i.e. curcumin, green tea, avocado seed, pomegranate peel or horse chestnut bark). The ΔE_{ab}^* values are higher than 1, meaning that the differences are visible. The calculated ΔE_{ab}^* values for curcumin dyed samples ($\Delta E_{ab}^* = 8.85$) are the highest, and for pomegranate peel extract dyed samples the lowest ($\Delta E_{ab}^* = 2.93$) (Table 2). From the results of ΔL^*, Δa^* and Δb^*, only one common characteristic between dyed raw and bleached/mercerised cotton sample can be found: the bleached/mercerized samples are lighter than raw samples ($\Delta L^* > 0$). This can be expected since the samples of the bleached/mercerised do not contain the natural pigment, which is present in the samples of a raw cotton [32].

The CIE $L^*a^*b^*$ values (Table 1) and the colour differences (Table 2) represent valuable information on the colour of dyed textiles, but cannot give the information on the uptake of the dye on the textile substrate. Although the low CIE L^* values are connected to the higher uptake, when textiles are dyed with synthetic dyes, this relationship is not necessarily true when textiles are dyed with natural dyes [33, 34]. The dye uptake ($\Delta K/S$) results of studied natural extracts are presented in Figure 6. The dye uptake of each natural dye is very similar for both cotton substrates, although the dyed bleached/mercerised cotton is much lighter than the dyed raw cotton (Table 1 and 2). The dye uptake of curcumin and avocado seed extract is slightly higher on bleached/mercerised cotton, while the dye uptake of green tea, pomegranate peel and horse chestnut extracts is slightly higher on raw cotton. These results indicate that raw and bleached/mercerised cotton have very similar uptake of studied natural dyes, and that uptake depends more on the dye structure than on the presence or absence of pigments and other non-cellulosic compounds on the cotton.

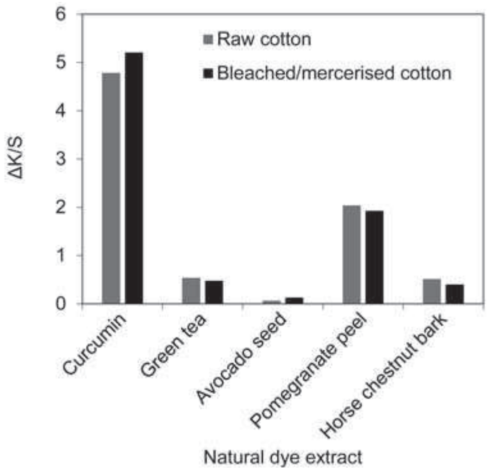


Figure 6: Dye uptake ($\Delta K/S$) of studied natural dyes on raw and bleached/mercerized cotton

In Figures 7 and 8 the colour strength values (K/S) of dyed raw and bleached/mercerized cotton samples before and after repetitive washings are presented. Dyeing with curcumin and pomegranate peel extract gives higher K/S values ($2.30 < K/S > 5.23$) (Figures 7 and 8), while dyeing with green tea, avocado seed and horse chestnut bark extracts gives lower K/S values ($0.43 < K/S > 0.80$). Repetitive washing lowers the K/S values, which indicates not a very stable fastness of natural dyes to washing (Figures 7 and 8). The K/S values greatly reduce on both substrates already after the first wash for the samples dyed with curcumin and pomegranate peel extract (Figure 7 and 8). From Figures 7 and 8 it could be concluded that washfastness is better for the samples dyed with avocado seed, horse chestnut bark and green tea, however the samples have low *K/S* values even at the beginning of the performed washings. From the CIE $L^*a^*b^*$ results (Table 1) it is clear that the latter samples have poor colour wash stability since they become much lighter after repetitive washing.

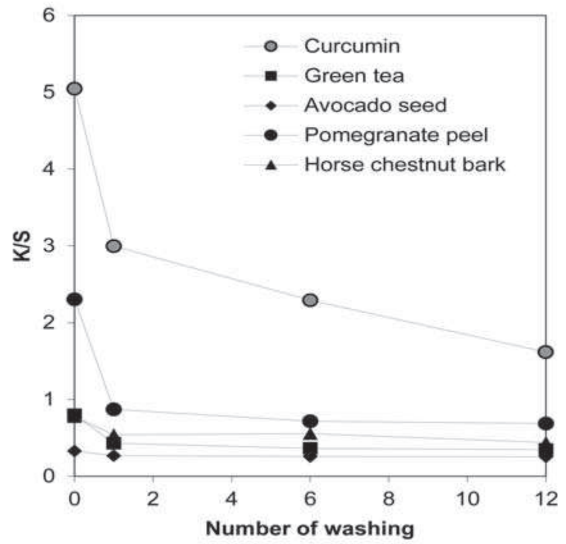


Figure 7: Colour strength (K/S values) of unwashed and washed raw cotton samples dyed with natural dyes

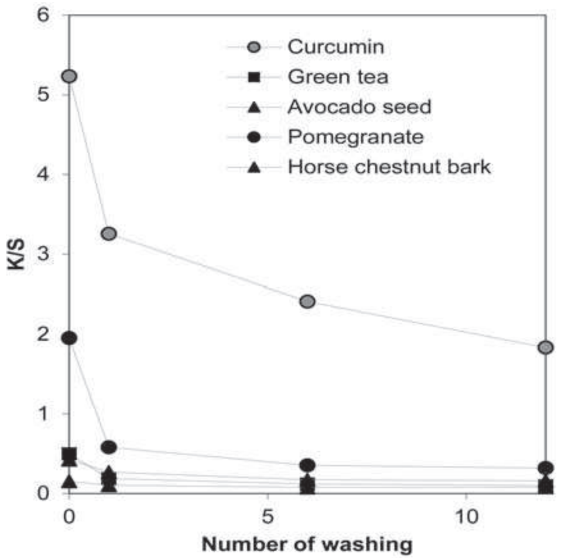


Figure 8: Colour strength (K/S values) of unwashed and washed bleached/mercerised cotton samples dyed with natural dyes

In Figures 9 and 10, the results of the UV protection factor (UPF) are presented for cotton substrates dyed with studied natural dyes (curcumin, green tea, avocado seed, pomegranate peel and horse chestnut bark). Dyed raw cotton fabrics have very high UPF values ranging from 25 to 260 (Figure 9), while the UPF values of dyed bleached/mercerised fabrics are much lower, ranging from 8 to 84 (Figure 10). The UPF values of undyed substrates are 16.50 (raw) and 4.43 (bleached/mercerised). Regardless of the substrate (raw or bleached/mercerised cotton), the highest UPF values are achieved by dyeing with pomegranate peel extract, which yields excellent UV protection factor (UPF_{raw} = 260; $UPF_{bleached/mercerised}$ = 84). These UPF values rate the cotton substrates into the 50+ UV protection category [35]. Raw cotton samples dyed with green tea and horse chestnut bark extracts are also rated into the excellent UV protection category (50+) (Figure 9). Their UPF values are 73 and 55, respectively. However, dyeing the bleached/mercerised cotton with the same extracts does not yield so high UPF values. While dyeing of bleached/mercerised cotton fabric with green tea extract provides very good UV protection (UPF = 26), dyeing with horse chestnut bark extract does not (UPF = 12) (Figure 9). The sample is not even UV rateable. Dyeing cotton substrates with avocado seed extract and curcumin gives the lowest UPF values (Figure 9 and 10). Dyed raw cotton samples have the UPF values 25 and 26, respectively, which rates the samples in the very good UV protection category (Figure 9). The UPF values of avocado seed extract and curcumin dyed bleached/mercerised cotton samples are only 8 and 10, respectively, which is not UV rateable. It is indicated in the literature that dark coloured fabrics with high *K/S* values transmit less UV radiation than lighter shaded fabrics with lower *K/S* values [36–39]. However, the results of our research show that the upper statement cannot be generalised for all dyes, especially not for natural dyes. Our results show that samples dyed with avocado seed extract have the lowest UPF values and are among the lightest samples with low *K/S* values. The samples dyed with pomegranate peel extract that have the highest UPF values are not among the darkest samples and have moderate *K/S* values. For example, the samples dyed with curcumin with the highest *K/S* values and moderate lightness have very low UPF values. The darkest samples with low *K/S* values are those dyed with horse chestnut bark extract and have moderate to low UPF values. From these results no correlation between CIE L^*, K/S and UPF was established. The reason why one or the other natural dye gives higher or lower UPF values to the cotton samples is most likely connected to the chemical structure of dye and its ability to absorb light near UV region than to its dyeing properties. This conclusion is supported by the fact that textiles finished with organic UV absorbers that are colourless compounds that absorb in wavelength from 280 to 400 nm have excellent UV protective properties [40, 41].

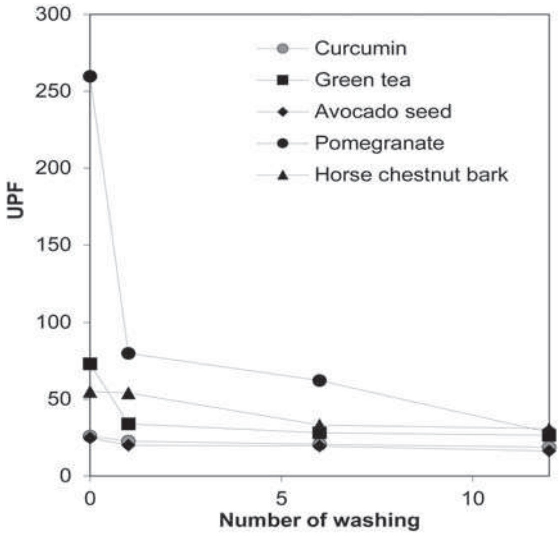


Figure 9: UPF values of unwashed and washed raw cotton samples dyed with natural dyes

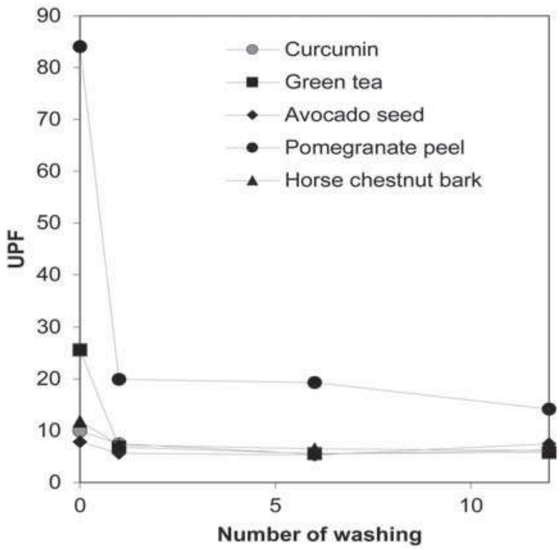


Figure 10: UPF values of unwashed and washed bleached/mercerised cotton samples dyed with natural dyes

Although dyeing cotton samples with pomegranate peel extract gave remarkable UPF results, the effect is not stable to washing (Figures 9 and 10). Even after the first performed washing, the UPF is drastically reduced, i.e. from 260 to 80 for raw cotton (Figure 9), and from 84 to 20 for bleached/mercerised cotton (Figure 10). While the UV protection category of raw cotton dyed with pomegranate peel extract remains excellent until the twelfth wash, this does not apply to dyed bleached/mercerised cotton. The latter is after the first and sixth wash characterised as a fabric with good UV protection properties (UPF ~ 20). After the twelfth performed wash, the UPF values are further decreased to the point where raw cotton is rated as a fabric with very good UV protective properties (UPF = 30), while the bleached/mercerised cotton is not rateable anymore (UPF = 14). The green tea extract performed similar as pomegranate peel extract, with the exception that the decrease in UPF values after the first wash already classifies the dyed bleached/mercerised cotton into the unrateable UV protection category (UPF < 15). The raw cotton dyed with green tea extract remains in the very good UV protection category even after the twelfth wash (UPF = 26). Dyeing with curcumin and avocado seed extract already gave inferior UPF results comparing to other studied dyes, and this is probably why no drastic changes in UPF values were found after repetitive washing.

5 Conclusion

Raw and bleached/mercerised cotton fabrics were dyed with the extracts of plant parts, such as green tea leafs, curcuma rhizome, avocado seed, pomegranate peel and horse chestnut bark. The dye uptake results show similar adsorption of natural dyes onto both substrates. The CIE $L^*a^*b^*$ colour values and colour strength values (K/S) of dyed samples were not in correlation with the UV protection factor (UPF) values. All dye extracts increased the UPF of cotton fabrics, in the following order: avocado seed < curcumin < horse chestnut bark < green tea < pomegranate peel. While all dyed raw cotton samples performed as textiles with at least very good UV protective properties, the bleached/mercerised cotton fabric performed also as a non-rateable. The results showed that extremely high UPF values and UV protection could be achieved by dyeing raw or bleached/mercerised cotton fabric with pomegranate peel or green tea extracts. However, the fabrics as such do not perform well after repetitive washing. Further research should be performed in the area of increasing the fastness to washing by using ecologically friendly compounds and modifications.

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