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## The Influence of Mordanting with Silver Nitrate on the Dyeability and UV Protection of Cotton Dyed with Green Tea

*Vpliv čimžanja s srebrovim nitratom na obarvljivost in  
UV-zaščito bombaža, pobarvanega z zelenim čajem*

Original Scientific Article/Izvirni znanstveni članek

Received/Prispelo 06–2015 • Accepted/ Sprejeto 08–2015

### Abstract

Silver nitrate ( $\text{AgNO}_3$ ) was used as a mordant for dyeing cotton fabric with natural dyes. Due to the high concentrations of catechin and tannin, green tea was used as a natural dye. The green tea extraction was performed under neutral and alkaline conditions. Four molar concentrations of  $\text{AgNO}_3$  were used, namely 1, 5, 10 and 50 mM. Mordanting was performed during dyeing with green tea extracts. The colour (CIE  $L^*a^*b^*$  values) and UV protection factor (UPF) of the dyed cotton samples before and after ten repetitive washings were measured spectrophotometrically. The results revealed that with the increase of the molar concentration of  $\text{AgNO}_3$ , the samples became darker, redder and bluer. Dyeing with alkaline green tea extract was not as successful as dyeing with neutral green tea extract. Different shades of cotton were achieved when dyeing with neutral green tea extract and mordanting with different molar concentrations of  $\text{AgNO}_3$ . Using 50 mM  $\text{AgNO}_3$  achieved a very dark grey (almost black) colour. Cotton samples dyed with neutral green tea extract had excellent UV protective properties (50+), some even after washing.

Keywords: dyeing, green tea, mordanting, silver nitrate, colours, UV protection.

### Izveček

Pri barvanju bombažne tkanine z naravnim barvilom je bil kot čimža uporabljen srebrov nitrat ( $\text{AgNO}_3$ ). Kot naravno barvilo je bil uporabljen zeleni čaj, saj ima visoko vsebnost katehina in čreslovine. Barvilo zelenega čaja je bilo ekstrahirano pri nevtralnih in alkalnih pogojih. Čimžanje je bilo opravljeno med barvalnim postopkom, z uporabo štirih molarnih koncentracij  $\text{AgNO}_3$ , in sicer 1, 5, 10 ter 50 mM. Barvo (CIE  $L^*a^*b^*$  vrednosti) in UV-zaščitni faktor (UZF) pobarvanih bombažnih vzorcev smo spektrofotometrično izmerili pred 10 zaporednimi prani in po njih. Rezultati kažejo, da z večanjem molarne koncentracije srebrovega nitrata postanejo barve vzorcev temnejše, bolj rdeče in bolj modre. Barvanje z alkalnim ekstraktom zelenega čaja ni bilo tako uspešno kot z nevtralnimi izvlečkom zelenega čaja. Pri barvanju z nevtralnimi izvlečkom zelenega čaja in z uporabo čimže različnih molarnih koncentracij srebrovega nitrata so bili doseženi različni odtenki bombaža. Pri uporabi 50 mM  $\text{AgNO}_3$  so bili doseženi zelo temni (skoraj črni) odtenki. Bombažni vzorci, pobarvani z nevtralnimi izvlečkom zelenega čaja, so dosegli odlično UV-zaščito (50+), nekateri tudi po večkratnem pranju.

Ključne besede: barvanje, zeleni čaj, čimžanje, srebrov nitrat, barve, UV-zaščita

## 1 Introduction

Synthetic dyes are being increasingly used in the process of dyeing, mostly because of the lower price compared to that of their natural counterparts. The fact that their production and use has a tremendous impact on the environment plays an insignificant role. However, natural extracts including dyes have become more popular over recent years because of their better biodegradability and their compatibilities with the environment. Most natural dyes also possess some functional properties [1–4]. Natural extracts have become quite popular amongst environmentally conscious consumers and are receiving more and more attention on the market. Green tea extract, which can be used as a natural dye, has various characteristics such as anti-carcinogenic, anti-oxidant, anti-bacterial and anti-allergic properties [5–10]. Green tea also has high UV protection due to its main component of catechin moiety. The most important ingredient in green tea is tea polyphenol, which is mainly composed of catechin (the main component representing more than 80%), flavones, anthocyan, tannin, and phenolic acid. Catechins' primary four compounds are (–)-epicatechin (EC), (–)-epigallocatechin (EGC), (–)-epicatechin gallate (ECG), and (–)-epigallocatechin gallate (EGCG), which is the predominant compound [11, 12]. Kim T. K. [13] discovered that cotton and nylon fabrics treated with green tea extracts have good antioxidant activities. Hwang et al. [14] discovered the deodorisation function of cotton, silk and wool treated with tea extract. The extract of green tea, used in Shin and Choi's research [15, 16], showed relatively good affinity with protein and polyamide fibres, and low affinity to cellulosic fibres. Cotton's uptake of the tea extract was improved using the cationic agents during the process of pre-treatment. Chun et al. [17, 18] used natural mordants such as camellia ash, bean chaff ash and pyro-lignite of iron for dyeing silk fabrics with green tea extracts. After dyeing, the measurements of the contents of four kinds of catechins absorbed within the silk fabrics were in the following order: EGCG>ECG>EGC>EC. Deo and Desai [19] dyed cotton and jute fabrics with an aqueous extract of tea using three mordant dyeing methods. This resulted in deeper colour shades for jute and medium shades for cotton with good wash and light fastness. Kim [20] treated cotton fabrics with chitosan and dyed them with green tea extracts afterwards. The

results showed good UV protection properties. Catechin contributes to UV protective properties. Kim [21] also focused his research on the ultraviolet protection properties of different kinds of fabrics dyed with green tea extract. The highest UV-A protective properties were shown by silk, followed by wool, nylon and acrylic, PET and cotton; higher UV-B protective properties were also shown by silk, followed by nylon, wool, acrylic, cotton and PET. All of these properties are dependent on several parameters, such as extraction conditions, pH of the extract or dyeing solution, and the temperature of the dyeing solution [22–24]. Kim et al. [22] researched the optimum extraction conditions of green tea. 1 g of green tea was mixed with 100 ml of water, methanol or ethanol using different extraction temperatures. The optimum extraction was shown in water and methanol rather than in ethanol. The extracts showed greater stability under acidic and neutral conditions than in the alkaline region. CIE  $L^*a^*b^*$  results for green tea extract aqueous solutions at different pH (from 3 to 11) showed that the increase of pH of the solution caused the extract to be darker, redder and yellower. The pH of an extract also influences the adsorption properties regarding textiles. Son et al. [23] discovered that the optimum dyeing conditions for dyeing nylon, cotton, rayon and lyocell fabrics with green tea extract was at pH = 5, where the adsorption of green tea extract was the highest. Higher temperatures combined with longer dyeing times caused higher K/S values and deeper shades of colour [24]. As already mentioned, green tea extract has low affinity to cellulosic fibres; therefore the use of mordants is necessary. In the process of dyeing, different mordants can be used, predominantly metallic. However, chitosan can be used as well [25]. Ghaheh et al. [26] carried out an assessment of the antibacterial activities of wool fabrics dyed with five different natural dyes, amongst which was also green tea extract. Aluminium sulphate, used as a mordant, caused the improvement and durability of antibacterial effects after five cycles of washing and 300 min of light exposure. Shin et al. [24] used ferric sulphate as a mordant in their study because tannin and all kinds of metallic mordants react and produce different colour shades. The purpose of our research was to achieve different colour shades of cotton using green tea extract and mordant that is not usually used for dyeing – silver nitrate. Four different concentrations of mordant and two extraction methods were used.

## 2 Materials and methods

### Material

100% bleached cotton plane weaved fabric (Tekstina, Ajdovščina) was used for the research.

### Mordanting

The mordanting of cotton fabric was performed as meta-mordanting (mordanting during dyeing) with different molar concentrations of silver nitrate ( $\text{AgNO}_3$ ), i.e. 1 mM, 5 mM, 10 mM and 50 mM. The volume ratio between mordant and green tea extract was 1:1.

### Extraction of green tea

Green tea was extracted under neutral and alkaline conditions. The neutral extract was prepared in deionised water with 20 g/l of green tea crushed leaves. The leaves were inserted within cold water. The mixture was heated and when it reached 95°C, the extraction took 5 min. Afterwards the mixture was left to cool for 30 min and then filtered to remove any of the solid particulates. The alkaline green tea extract was performed in the same manner however 5 g/l of  $\text{Na}_2\text{CO}_3$  was added to the mixture.

### Dyeing

Dyeing of non-mordanted and mordanted cotton was performed in stainless-steel flasks, at goods to liquor ratio 1:20, at 60°C, for 40 min. After dyeing,

rinsing of the samples was performed in deionised water. The samples were air-dried at room temperature. The samples were marked according to their treatments and are presented in Table 1.

### Wash-fastness

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

### Colour measurements

CIE  $L^*a^*b^*$  colour values and reflectance (R) of the samples were measured using reflectance spectrophotometer Datacolor PLUS. From the CIE  $L^*a^*b^*$  colour values, colour differences were calculated according to equation 1:

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

where  $\Delta L^*$  is the lightness difference,  $\Delta a^*$  is the red/green difference and  $\Delta b^*$  is the yellow/blue difference between standard and batch.

Table 1: Sample marking according to the treatment

Marking of sample	Treatment of sample
Un	Untreated
GT_H <sub>2</sub> O	Dyed with neutral green tea extract (extraction in deionised water)
GT_Na <sub>2</sub> CO <sub>3</sub>	Dyed with alkaline green tea extract (extraction in mixture of deionised water and sodium carbonate)
1 mM + GT_H <sub>2</sub> O	Mordanted with 1 mM AgNO <sub>3</sub> and dyed with neutral green tea extract
5 mM + GT_H <sub>2</sub> O	Mordanted with 5 mM AgNO <sub>3</sub> and dyed with neutral green tea extract
10 mM + GT_H <sub>2</sub> O	Mordanted with 10 mM AgNO <sub>3</sub> and dyed with neutral green tea extract
50 mM + GT_H <sub>2</sub> O	Mordanted with 50 mM AgNO <sub>3</sub> and dyed with neutral green tea extract
1 mM + GT_Na <sub>2</sub> CO <sub>3</sub>	Mordanted with 1 mM AgNO <sub>3</sub> and dyed with alkaline green tea extract
5 mM + GT_Na <sub>2</sub> CO <sub>3</sub>	Mordanted with 5 mM AgNO <sub>3</sub> and dyed with alkaline green tea extract
10 mM + GT_Na <sub>2</sub> CO <sub>3</sub>	Mordanted with 10 mM AgNO <sub>3</sub> and dyed with alkaline green tea extract
50 mM + GT_Na <sub>2</sub> CO <sub>3</sub>	Mordanted with 50 mM AgNO <sub>3</sub> and dyed with alkaline green tea extract
w	10-times washed samples

From the reflectance measurements,  $K/S$  values were calculated according to equation 2:

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

where  $R$  is the reflectance,  $K$  is the absorbance and  $S$  is the scattering.

#### UV protection factor measurements

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

### 3 Results and discussion

During the experiment we noticed that the colour of the green tea extract was much darker when the green tea was extracted under alkaline conditions (in a mixture of deionised water and  $\text{Na}_2\text{CO}_3$ ) than under neutral conditions (only in deionised water). This observation is in accordance with literature [22]. As the extract was darker, the expected result was darker coloration of the dyed cotton. However, the results show differently. The CIE  $L^*a^*b^*$  colour values (Table 2) show that the cotton dyed with alkaline green tea extract was lighter, less red and less yellow than the cotton dyed with neutral green tea extract. The  $K/S$  value of the cotton dyed with neutral green tea extract ( $GT_{H_2O}$ ) was higher than the  $K/S$  value of cotton dyed with alkaline green tea extract ( $GT_{Na_2CO_3}$ ) (Table 2), meaning that dye uptake was lower for the  $GT_{Na_2CO_3}$  sample. Cellulosic fibres, such as cotton, when in contact with water gain slightly negative charge due to the ionisations of the hydroxyl groups [27]. At a pH higher than 8, some of the hydroxyl groups in the hydroxymethyl side chains may also be ionised thus increasing the negative charge significantly [28]. The negative charges on the surface of the cellulose repel anionic dyes and hence the efficiency of dye adsorption on cellulosic fibres is generally low. The increase of the molar concentration of  $\text{AgNO}_3$  increases the dyeability with green tea extract (Table 2). The  $K/S$  values of those cotton samples dyed with neutral green tea

extract increased from 0.61 for non-mordanted to 1.01 for mordanted with 1 mM  $\text{AgNO}_3$ , and up to 10.51 for mordanted with 50 mM  $\text{AgNO}_3$ . The  $K/S$  values of the cotton samples dyed with alkaline green tea extract were lower, ranging from 0.41 for non-mordanted to 1.93 for 50 mM  $\text{AgNO}_3$  mordanted. Cotton samples dyed with neutral green tea extract and mordanted with higher molar concentration of  $\text{AgNO}_3$  were darker, redder and bluer, except the sample mordanted with 50 mM  $\text{AgNO}_3$  that was less yellow (Table 2). Meta-mordanting during alkaline green tea extract dyeing gave darker, redder and yellower colorations of the dyed cotton (Table 2).

Table 2: CIE  $L^*a^*b^*$  colour values and  $K/S$  values of cotton dyed with green tea extract

Sample	$L^*$	$a^*$	$b^*$	$K/S$
Un	95.65	-0.27	2.09	0.02
$GT_{H_2O}$	81.20	3.90	15.32	0.61
$GT_{Na_2CO_3}$	82.95	1.46	13.77	0.47
1 mM + $GT_{H_2O}$	69.58	5.70	16.15	1.01
5 mM + $GT_{H_2O}$	50.65	13.92	29.03	5.77
10 mM + $GT_{H_2O}$	39.63	13.47	18.83	10.19
50 mM + $GT_{H_2O}$	33.84	1.90	7.37	10.51
1 mM + $GT_{Na_2CO_3}$	75.02	2.13	9.62	0.57
5 mM + $GT_{Na_2CO_3}$	69.45	7.14	22.34	1.19
10 mM + $GT_{Na_2CO_3}$	69.08	6.23	29.06	1.66
50 mM + $GT_{Na_2CO_3}$	67.87	7.74	31.73	1.93

The colour differences ( $\Delta E^*$ ) between the non-mordanted and mordanted samples are presented in Figure 1. The differences are higher with increasing mordant molar concentration. Even at low molar concentration (1 mM), the colour difference between the non-mordanted and mordanted samples was higher than 1, meaning that the difference was visibly detectable. Colour differences were higher for samples dyed with neutral green tea extract. The results are in agreement with the results of CIE  $L^*a^*b^*$  values.

In order to demonstrate the colourful qualities of the cotton dyed with green tea extract using different molar concentrations of  $\text{AgNO}_3$ , the samples were photographed and are presented in Figure 2. Mainly brown shading was achieved, varying from very pale to dark brown. Dyeing with

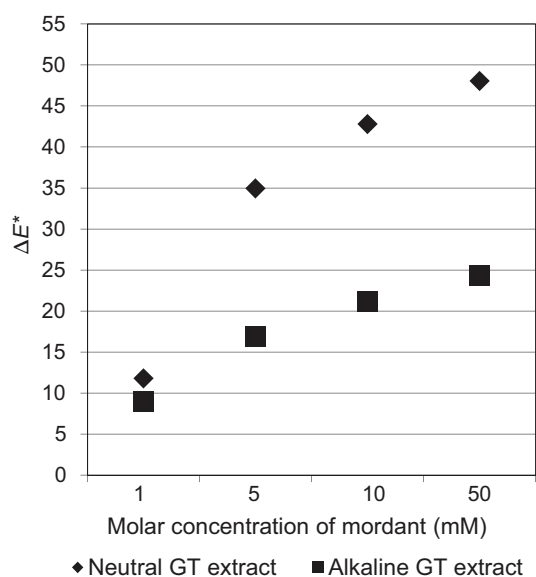


Figure 1: Colour differences ( $\Delta E^*$ ) between the non-mordanted and mordanted samples dyed with green tea (GT) extract

neutral green tea extract and mordanting with 50 mM  $\text{AgNO}_3$  gave dark grey to almost black colorations of the cotton.

Textiles dyed with natural dyes have poor washing stability and mordanting helps to improve the wash fastness. Therefore, samples dyed with green tea extract were repetitively washed 10-times. The CIE  $L^*a^*b^*$  values of the washed samples are presented in Table 3, and the colour differences between non-washed and washed samples in Figure 3. The non-mordanted sample dyed with neutral green tea extract was lighter, redder and less yellow after washing, while the non-mordanted sample dyed with alkaline green tea extract became lighter, less red and less yellow (Table 3). The colour difference was higher for the sample dyed with alkaline green tea extract than for the sample dyed with neutral green tea extract (Figure 3). The reason is in the poor adsorption of the dye onto cotton and is therefore easily removed with washing. Mordanting with  $\text{AgNO}_3$  did not drastically improve the wash fastness of the dyed samples. The colour differences in all cases were higher than 1, meaning that colour difference was detectable (Figure 3). The best results for wash fastness were achieved for the sample dyed with neutral green tea extract and mordanted with 50 mM of  $\text{AgNO}_3$  (sample 50 mM +  $\text{GT}_\text{H}_2\text{O}$ ) (Figure 3).

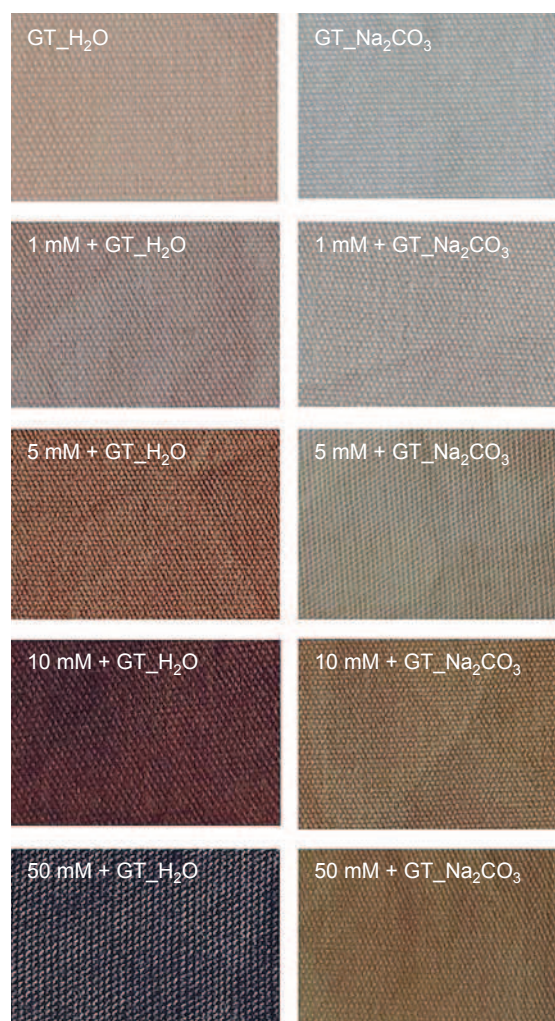


Figure 2: Photographs of samples dyed with green tea extract

Table 3: CIE  $L^*a^*b^*$  values of washed (w) samples of cotton dyed with green tea extract

Sample	$L^*$	$a^*$	$b^*$
$\text{GT}_\text{H}_2\text{O}_\text{w}$	82.03	4.34	10.96
$\text{GT}_\text{Na}_2\text{CO}_3_\text{w}$	89.71	0.91	7.89
1 mM + $\text{GT}_\text{H}_2\text{O}_\text{w}$	72.85	5.93	16.29
5 mM + $\text{GT}_\text{H}_2\text{O}_\text{w}$	51.48	11.84	24.51
10 mM + $\text{GT}_\text{H}_2\text{O}_\text{w}$	41.06	11.46	18.86
50 mM + $\text{GT}_\text{H}_2\text{O}_\text{w}$	39.57	1.13	5.27
1 mM + $\text{GT}_\text{Na}_2\text{CO}_3_\text{w}$	78.88	1.91	5.90
5 mM + $\text{GT}_\text{Na}_2\text{CO}_3_\text{w}$	71.05	9.11	34.22
10 mM + $\text{GT}_\text{Na}_2\text{CO}_3_\text{w}$	71.22	7.67	19.69
50 mM + $\text{GT}_\text{Na}_2\text{CO}_3_\text{w}$	73.47	4.76	26.60

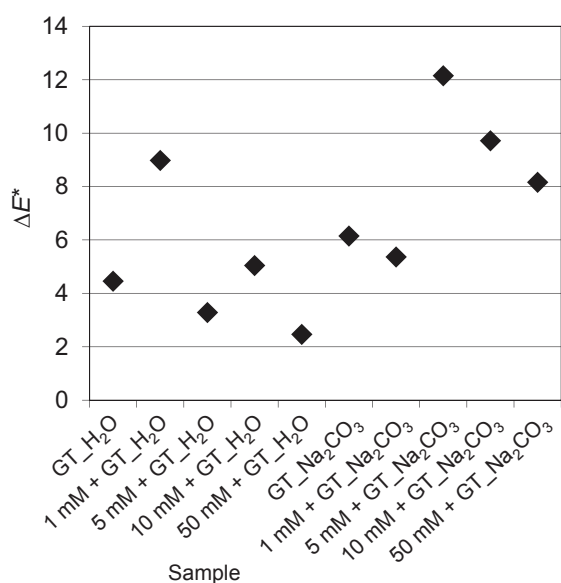


Figure 3: Colour differences ( $\Delta E^*$ ) between the non-washed and 10-times washed samples dyed with green tea extract

Green tea extracts are known for their good UV absorption properties [20, 21]. The UV transmission and UV protection factor of the dyed samples were measured (Table 4). The samples dyed with neutral green tea extract had excellent protective properties against UV radiation (50+), with the UPF values ranging from 66.44 for the non-mordanted sample up to 497.68 for the 50 mM  $\text{AgNO}_3$  mordanted sample. Mordanting with  $\text{AgNO}_3$  greatly increased the UV protection of the cotton dyed with neutral green tea extract. Although the mordanted samples dyed with alkaline green tea extract were darker than the non-mordanted sample, the UPF values are lower than for the non-mordanted sample. The exception was the sample 50 mM +  $\text{GT\_Na}_2\text{CO}_3$ , which had a higher UPF value. We could not find the reason for such results. After washing, the samples dyed with alkaline green tea extract had poor UV protection properties, with UPF values ranging from 8 to 15. The samples dyed with neutral green tea extract had very good UV protection properties even after ten repetitive washings. The non-

Table 4: Ultraviolet protection factor (UPF), UVA and UVB blocking properties, transmission (T) in the UVA and UVB regions and ultraviolet radiation (UVR) of the dyed and mordanted samples, before and after washing (w)

Sample	UPF	UVA blocking (%)	UVB blocking (%)	T (UVA)	T (UVB)	T (UVR)
Un	5.60	77.09	83.57	22.91	16.44	20.69
GT_H <sub>2</sub> O	66.44	96.26	98.85	3.74	1.15	2.94
GT_Na <sub>2</sub> CO <sub>3</sub>	22.15	92.23	96.43	7.77	3.57	6.45
1 mM + GT_H <sub>2</sub> O	41.31	95.96	98.01	4.04	1.99	3.39
5 mM + GT_H <sub>2</sub> O	81.05	98.79	98.95	1.21	1.05	1.14
10 mM + GT_H <sub>2</sub> O	139.65	99.40	99.37	0.60	0.63	0.60
50 mM + GT_H <sub>2</sub> O	497.68	99.82	99.81	0.18	0.19	0.18
1 mM + GT_Na <sub>2</sub> CO <sub>3</sub>	14.11	90.51	94.12	9.49	5.88	8.31
5 mM + GT_Na <sub>2</sub> CO <sub>3</sub>	16.92	94.48	94.90	5.52	5.01	5.28
10 mM + GT_Na <sub>2</sub> CO <sub>3</sub>	20.71	95.92	95.63	4.08	4.37	4.09
50 mM + GT_Na <sub>2</sub> CO <sub>3</sub>	28.30	96.66	96.74	3.34	3.03	3.20
GT_H <sub>2</sub> O_w	21.26	93.73	95.72	6.27	4.28	5.60
GT_Na <sub>2</sub> CO <sub>3</sub> _w	8.63	83.48	89.72	16.52	10.28	14.48
1 mM + GT_H <sub>2</sub> O_w	27.58	92.61	96.75	7.39	3.25	6.10
5 mM + GT_H <sub>2</sub> O_w	53.30	98.50	98.40	1.51	1.61	1.51
10 mM + GT_H <sub>2</sub> O_w	127.23	99.43	99.34	0.60	0.63	0.60
50 mM + GT_H <sub>2</sub> O_w	416.49	99.78	99.77	0.22	0.23	0.22
1 mM + GT_Na <sub>2</sub> CO <sub>3</sub> _w	8.45	86.11	89.61	13.89	10.40	12.67
5 mM + GT_Na <sub>2</sub> CO <sub>3</sub> _w	11.60	92.42	92.41	7.58	7.59	7.46
10 mM + GT_Na <sub>2</sub> CO <sub>3</sub> _w	13.71	93.89	93.37	6.11	6.63	6.15
50 mM + GT_Na <sub>2</sub> CO <sub>3</sub> _w	15.67	94.96	94.49	5.04	5.51	5.09

mordanted sample and the sample mordanted with 1 mM AgNO<sub>3</sub> had lost excellent UV protection properties. Their UPF values were 21.26 and 27.58, respectively. The protection categories for these two samples were good and very good. The samples mordanted with higher molar concentrations of AgNO<sub>3</sub> and dyed with neutral green tea extract maintained their excellent (50+) protective properties against harmful UV radiation after ten repetitive washings.

## 4 Conclusion

Bleached cotton fabric was dyed with green tea extract and mordanted during dyeing with silver nitrate. Green tea was extracted under neutral and alkaline conditions. Silver nitrate was added into a dye bath at four molar concentrations, i.e. 1, 5, 10 and 50 mM. With the increased molar concentration of mordant the dyed cotton was darker, redder and bluer, meaning that different colorations of the cotton were achieved. The dyeability of the cotton was higher when it was dyed with neutral green tea extract than with alkaline. Consequently, mordanting with higher molar concentration of silver nitrate did not have such an influence on the dyeability of the cotton than when it was added into the neutral dye bath (neutral green tea extract). Different brown colorings of cotton fabric can be achieved by choosing the right molar concentrations of mordant. Dark grey coloration of the cotton was achieved when it was dyed with neutral green tea extract and mordanted with 50 mM of silver nitrate. Cotton fabrics had very high UV protection factor (even up to 400). Even after repetitive washing the UV protection of the samples dyed with neutral green tea extract and mordanted was excellent. However, the dyed samples had poor colour fastness to washing.

### Acknowledgements

The research was financially supported by the Slovenian Research Agency (programme Textiles and Ecology P2 – 0213).

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