Scientific paper

The Influence of nano-ZnO Application Methods on UV Protective Properties of Cotton

Mateja Kert,[1] Katja Jazbec,[1] Lidija Černe,[1] Ivan Jerman[2] and Marija Gorjanc[1,*]

[1] *University of Ljubljana, Faculty of Natural Sciences and Engineering, Aškerčeva 12, 1000 Ljubljana, Slovenia*

[2] *National Institute of Chemistry Ljubljana, Hajdrihova 19, 1000 Ljubljana, Slovenia*

** Corresponding author: E-mail: marija.gorjanc@ntf.uni-lj.si*
Phone: +386 1 200 32 55

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Abstract

The influence of different application methods on UV protective properties of white and dyed cotton functionalized with ZnO nanoparticles (nano-ZnO) was investigated. The methods differ in application procedure, time of treatment and auxiliaries used in the treating bath. The ultraviolet protection factor (UPF) was determined for untreated and functionalized samples. The presence of nano-ZnO on fibres was investigated using scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). The content of Zn was determined with energy-dispersive X-ray spectroscopy (EDS) and inductively coupled plasma mass spectrometry (ICP-MS). Dynamic light scattering (DLS) was used for particle size measurements in the prepared solutions. The results show that UV protection of cotton increases with a higher content and uniform distribution of nano-ZnO on the samples and that dyeing increases the loading capacity of cotton towards nano-ZnO. One of the methods (Method IV) gave remarkable results giving cotton an excellent UV protection whether it was dyed or not.

Keywords: ZnO, cotton, dyeing, UV protection, SEM, Ultraviolet protection factor

1. Introduction

Humans are exposed to the Sun and other sources of ultraviolet (UV) radiation for a considerable time of living. Despite the beneficial effects of the UV radiation on skin, the prolonged and frequent exposure can lead to the skin damages.[1] In addition to the solar block creams and lotions that are applied directly on the skin, the use of textile materials as UV protective products is important. The UV protection through textile materials include various apparels, accessories such as hats and scarfs, shade structures such as umbrellas, awnings, baby carrier covers etc.[2] But for a textile material to be able to provide a UV protection, the textile must be properly prepared. Additionally, the UV protective properties of textiles depend on their chemical nature, colour and also the constructional parameters of fabrics.[3,4] Cotton is the most popular fabric used, but its natural fibre comprises only a small barrier to UV radiation, especially when textile garment is white or light colour dyed.[5] For improving the UV protective properties of cotton, different UV absorbers can be used, zinc oxide nanoparticles being one of the most interesting and challenging ones.[6,7] Zinc oxide (ZnO) is a multifunctional crystalline material. Due to its unique properties, which ensure its application in various fields of modern industry, ZnO is an object of intensive scientific studies. In addition to UV blocking property, ZnO nanoparticles have photocatalytic and antibacterial properties.[8] The functional properties of cotton fabric depend on the size of ZnO particles. ZnO particles of smaller size offer a greater protection against harmful UV radiation than larger sized particles.[9] In case of bulk-ZnO coated fabric, about 50% of the UV light was absorbed by the fabric. In case of nano-ZnO coated fabric, a maximum of 75% absorption of UV light was noticed, while in control fabric, an average of 20% of UV light was absorbed. Nano-ZnO coated cotton fabric also had better strength properties and air permeability in comparison to untreated and bulk-ZnO treated fabric. Various methods of applying ZnO onto cotton have been used, i.e. "in situ" formation of nano-ZnO, sol-gel, wet chemical methods and printing, and many of them gave satisfactory but rarely excellent UV protective results.[10–15]

Nano-ZnO has a great potential for use on textile materials in practice. Since textile industry is using commercial products, it is therefore essential to investigate the best application conditions of commercially prepared nano-ZnO onto cotton textiles, in order to achieve high adsorption of nanoparticles and excellent UV protection. In our research, four different wet chemical methods of applying nano-ZnO onto undyed and dyed cotton fabric were used and their influence on UV protective properties of undyed and dyed cotton was investigated.

2. Experimental

2. 1. Dyeing of Cotton

Cotton samples (100% bleached/mercerized cotton fabrics) were dyed with 0.5% bi-reactive dye Cibacron deep red S-B (Ciba, Switzerland), at liquor ratio 20 : 1, 30 g/l Na_2SO_4 (Carlo Erba, Italy) and 8 g/l Na_2CO_3 (Carlo Erba), at 60 °C for 1 hour. After dyeing samples were aftertreated, i.e. rinsed with distilled water, neutralized with 1 ml/l CH_3COOH 30% (Sigma-Aldrich, Italy) and soaped with 1 g/l Cibapon R (Ciba). Samples were finally rinsed with cold distilled water and air dried.

2. 2. Functionalization of Cotton with Nano-ZnO

Four different methods for functionalizing undyed and dyed cotton samples were performed. In all methods 3% of 30 nm ZnO particles (MK Impex Corp., Canada) (nano-ZnO) were used. After functionalization with nano-ZnO all samples were rinsed with cold distilled water and air dried. In Method I cotton samples were functionalized using exhaustion method during dyeing for 60 min at 60 °C,[16] dried at 100 °C for 5 min and cured in the oven for 5 min at 150 °C. In Method II cotton samples were treated in a solution of bidistilled water, nano-ZnO and 1 g/l CHT Dispergator SMS (CHT, Switzerland) for 5 min at a room temperature, foulard wrung with a wet-pick-up of 100%, dried at 100 °C for 5 min and cured in the oven for 5 min at 150 °C. Method III included impregnation of cotton samples in a 40% ethanol dispersion of nano-ZnO, foulard wringing with wet-pick-up of 100%, drying at 100 °C for 5 min and curing in the oven for 5 min at 150 °C. Method IV included treatment of cotton samples in a solution of bidistilled water, nano-ZnO and 1 ml/l CH_3COOH 30% (Sigma-Aldrich) for 30 min, foulard wringing with wet-pick-up of 100%, drying at 100 °C for 5 min and curing in the oven for 5 min at 150 °C.

2. 3. Measurements and Characterization

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

The JEOL JSM 6060LV scanning electronic microscope (SEM) was used to observe the surfaces of untreated and functionalized samples. All samples were coated with a thin layer of gold. The electron accelerating voltage was 10 kV. The magnification of SEM images was 1,500×.

EDS type elemental chemical analyses of cotton samples was performed additionally using JEOL JSM 5610 LV SEM equipped with an EDS system. Samples for EDS analysis were coated with a thin carbon layer to ensure sufficient electrical conductivity and to avoid charging effects.

The quantity of Zn on ZnO functionalized cotton samples was analysed using inductively coupled plasma mass spectroscopy (ICP-MS).[17]

FT-IR spectroscopy was used to analyse the presence of nano-ZnO on cotton fabric by recording the IR spectra in the spectral range of 4000 to 450 cm^{-1}. The IR spectra of undyed sample and samples functionalized using Method I and Method IV, were recorded. Samples were analysed using Tensor 27 FT-IR, Bruker with Specac heated golden gate ATR. On each sample 64 scans were performed.

Particle size measurements were done by Dynamic Light Scattering (DLS) measurement using Zetasizer Nano ZS, Malvern.[18]

Colour and CIE whiteness index measurements of cotton samples were performed using a Datacolor Spectraflash SF 600 PLUS-CT spectrophotometer (Datacolor, USA). The CIELAB colour coordinates were determined for dyed cotton samples. The CIE whiteness index (WI) and tint value ($T_{w,10}$) were determined for undyed samples. All measurements were performed using 4 layers of fabric with a 9-mm aperture, wherein the specular component was included (for determination of CIELAB colour coordinates) or excluded (for determination of CIE whiteness index) under D65 illumination and 10° standard observer. An average of five measurements was recorded for each sample. All samples were exposed to standard conditions according to ISO 139 prior to performing the measurements.

3. Results and Discussion

UPF values, transmission in UVA and UVB region, ultraviolet radiation (UVR), UVA and UVB blocking of cotton samples treated by different methods are presented in Table 1.

The UPF results (Table 1) show that undyed cotton sample has very poor UV protective properties, with UPF value of only 4.481. Dyeing cotton with low concentration of dye increases UPF value to 44.689, giving a fabric an

Table 1: Ultraviolet protection factor (UPF), UV-A and UV-B transmittance (T), UPF rating and UV-R protection category of cotton samples

Sample	Mean UPF	T (UV-A) (%)	T (UV-B) (%)	T (UVR) (%)	UPF rating	UV-R protection
Undyed	4.481	27.312	21.236	25.136	5	Non-rateable
Dyed	44.689	4.235	1.834	3.484	45	Excellent
Method I	9.382	16.155	10.846	14.382	10	Non-rateable
Method I_D	53.702	3.211	1.632	2.710	50+	Excellent
Method II	12.692	14.282	7.430	12.103	10	Non-rateable
Method II_D	60.830	3.132	1.415	2.593	50+	Excellent
Method III	12.703	14.109	7.391	11.970	10	Non-rateable
Method III_D	77.414	2.522	1.118	2.082	50+	Excellent
Method IV	33.631	7.440	2.945	6.043	30	Very good
Method IV_D	125.766	1.701	0.711	1.392	50+	Excellent

Note: D … dyed sample

excellent UV protection. Increasing UV protection of cotton through dyeing process is in correlation to the results obtained by other authors.[19–21] The UV protective properties of cotton therefore highly depend on the treatment of cotton, colour and also the constructional parameters of fabrics.[3,4] Dyeing cotton with reactive dye does not influence the morphological changes of fibres in comparison to undyed cotton (Figure 1). Typical grooved morphology of cotton can be noticeable on SEM pictures of undyed (Figure 1-a) and dyed (Figure 1-b) cotton fibres. The

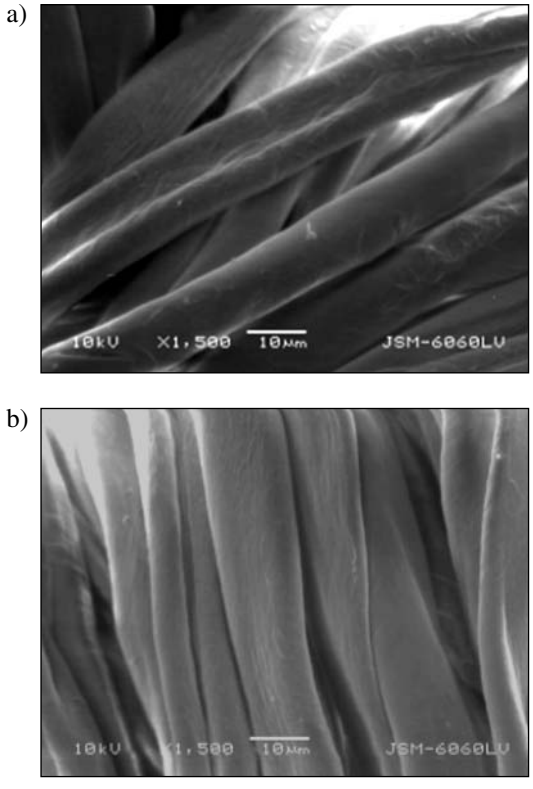


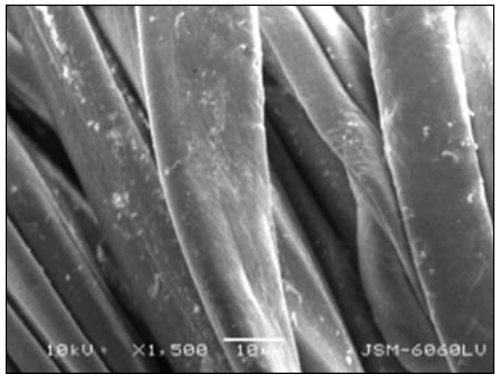
Figure 1: SEM pictures of (a) undyed and (b) dyed cotton fibres

morphological changes of the cotton fibres are noticeable when nano-ZnO is applied onto the fabrics (Figures 4–6). Also when applying nano-ZnO onto cotton, regardless of the method used, increased UV protective properties of cotton (Table 1) were obtained.

Nevertheless, every method of functionalizing cotton with nano-ZnO contributes to the increased UPF value of functionalized cotton differently. The minimum contribution to the increased UV protection is achieved by the functionalization of cotton with nano-ZnO using Method I. The UPF value of undyed functionalized cotton sample increases from 4.481 to 9.382 and UPF value of dyed functionalized cotton sample increases from 44.689 to 53.702. The change in the UPF values between untreated and functionalized cotton samples is very small. From the SEM pictures in Figure 2 it is visible that nano-ZnO particles are unevenly distributed and are present only on some parts of the fibres. EDS analysis (Figure 3) shows low Zn content on fibres, i.e. 7.994 wt.% on undyed and 15.042 wt.% on dyed functionalized sample. Since EDS elemental analysis indicates only the amount of Zn present on the surface of the fibres, additional elemental analysis was performed using ICP-MS where the quantification of total Zn content in the fibres was analysed. The results of ICP-MS analysis (Table 2) corroborate with the results of EDS and UV/VIS analysis. The quantity of Zn on dyed sample is higher by 61.9% in comparison to undyed samples. However, uneven distribution of particles and low content of Zn on the functionalized fibres are reasons for low UPF values. Irrespective to that, the difference in Zn content between undyed and dyed cotton samples is noticeable, indicating higher adsorption capacity of nano-ZnO towards reactive dyed cotton. The similar effect can be noticed for adsorption of nano-Ag particles onto reactive dyed cotton,[22] where Ag^+ ions react with hydroxyl functional cellulosic groups and additional sulfonic acid groups of covalently bound dye on the fibre. In our case, the Zn^{2+} ions react in the same manner. But our

results indicate that the exhaustion method is not as applicable for application of nano-ZnO as it is when nano-Ag is applied onto cotton[23] and therefore Method I has no practical use in increasing UV protective properties of cotton fabric.

a)



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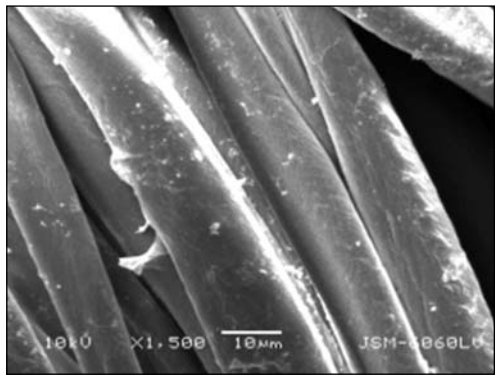


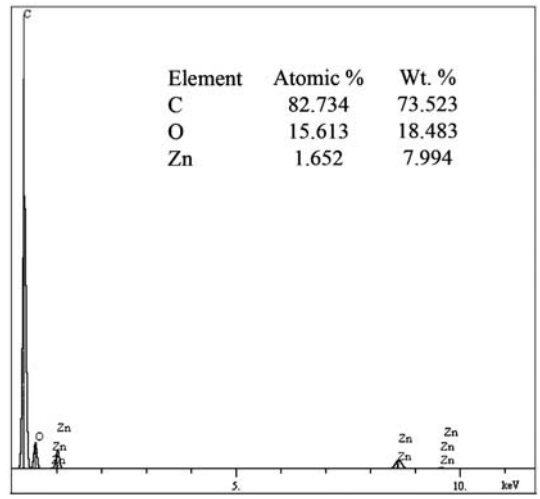
Figure 2: SEM pictures of (a) undyed and (b) dyed cotton fibres functionalized with nano-ZnO by Method I

Table 2: Quantity (ppm) of Zn on undyed and dyed ZnO functionalized samples

Sample	Zn (ppm)
Method I	220
Method I_D	577
Method IV	1097
Method IV_D	1289

In comparison to untreated or samples treated by Method I, higher UPF values were obtained for samples functionalized with nano-ZnO using Methods II and III (Table 1). Although the two methods used are different, similar results of UPF values are obtained. The UPF values of undyed samples are 12.692 (Method II) and 12.702 (Method III). According to Australian / New Zealand (AS/NZS) standard for textile goods to be rated in a category "good protection", the minimum UPF value of sample must be at least 15.00 (good protection UPF 15–24,

a)



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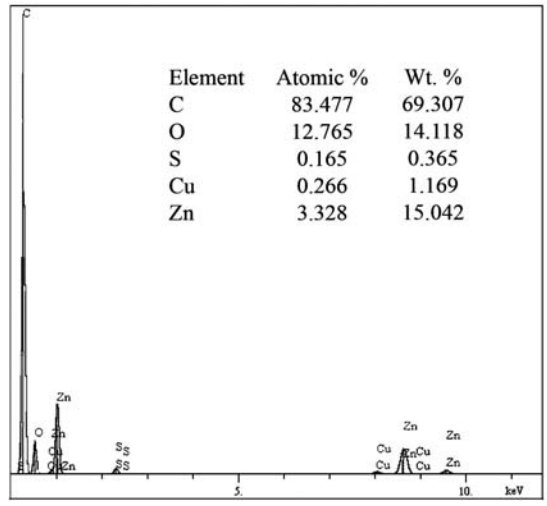
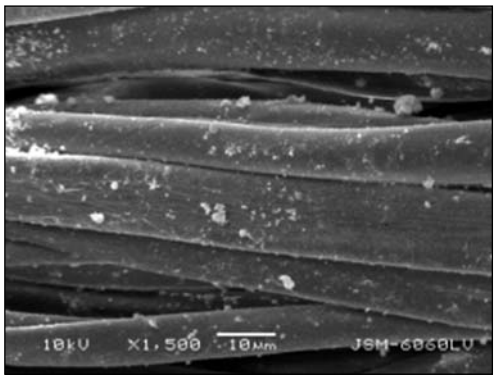


Figure 3: EDS image and element content of (a) undyed and (b) dyed nano-ZnO functionalized cotton by Method I.

very good protection UPF 25–39, excellent protection UPF > 40).[24] Our samples functionalized by these two methods do not meet AS/NZS standard. However, when comparing our results to the results of other authors,[9,11] it can be observed that ours are not significantly different. Although the samples do not meet the standards requirements to be characterized as "good protective" goods, the UPF value difference between untreated and nano-ZnO functionalized sample is obvious. The UV protective properties dramatically increase for dyed samples functionalized with nano-ZnO using Methods II and III. The UPF values increase from 44.689 to 60.860 and 77.414, respectively (Table 1). The UPF values so high, according to AS/NZS standard, place our samples in an "excellent" UV protection category. From the SEM pictures in Figures 4 and 5, where undyed and dyed samples functionalized with nano-ZnO using Methods II and III are presented, it can be observed that nano-ZnO particles are unevenly distributed on the fibres, but the abundance of the particles

a)



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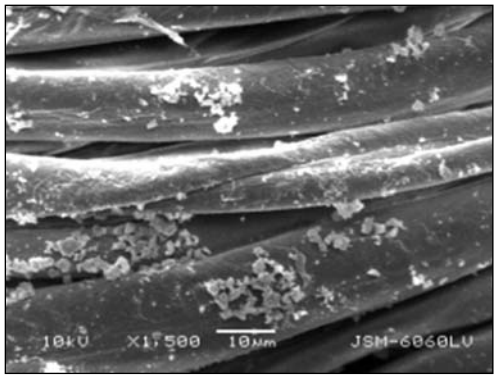

Figure 4: SEM pictures of (a) undyed and (b) dyed cotton fibres functionalized with nano-ZnO using Method II

in comparison to the samples functionalized by Method I is noticeable. Also larger agglomerates can be found on the fibres.

The greatest influence on increased UV protection of cotton is achieved by the application of nano-ZnO using Method IV. The UPF value increases from 4.481 to 33.631 for undyed sample and from 44.689 to 125.766 for dyed sample (Table 1). These high UPF values place the samples into a "very good" and an "excellent" UV protection category. Nano-ZnO particles are evenly distributed over the fibres (Figure 6). On dyed functionalized sample (Figure 6-b) more agglomerates are formed than on undyed cotton sample (Figure 6-a). The EDS analysis of samples functionalized by Method IV shows that Zn content on fibres is the highest among all functionalized samples (Figure 7). The content of Zn on undyed functionalized sample is 79.683 wt.% (Figure 7-a) and on dyed functionalized sample 88.729 wt.% (Figure 7-b). Again the difference in Zn content on undyed and dyed samples is noticeable, which was additionally proven by ICP-MS analysis (Table 2). Despite the high nano-ZnO content on the fibres, the influence of reactive dye on the adsorption capacity of cotton towards ZnO nanoparticles is still present. High content of Zn and an even distribution over the fibres are reasons for such high UPF values of functionalized samples. From the results it can be concluded that Method IV is the most suitable for increasing UV protective pro-

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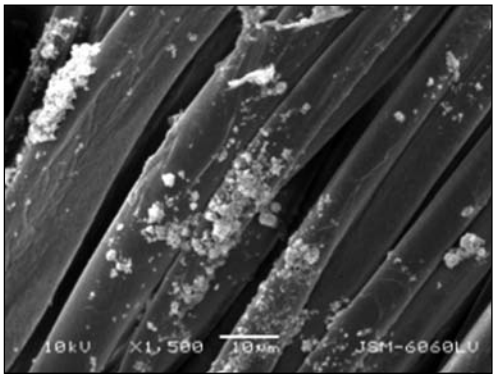

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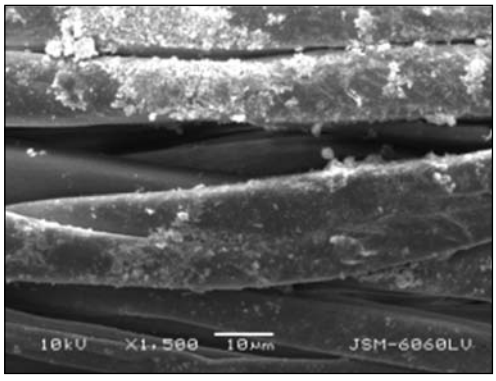
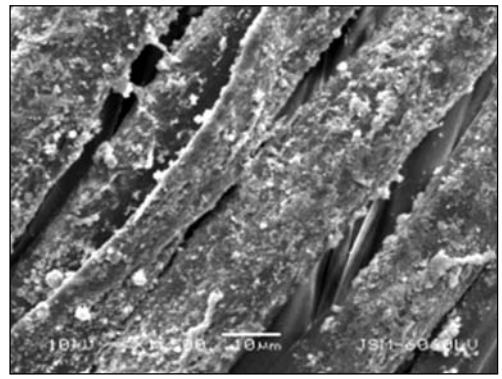


Figure 5: SEM pictures of (a) undyed and (b) dyed cotton fibres functionalized with nano-ZnO using Method III

a)



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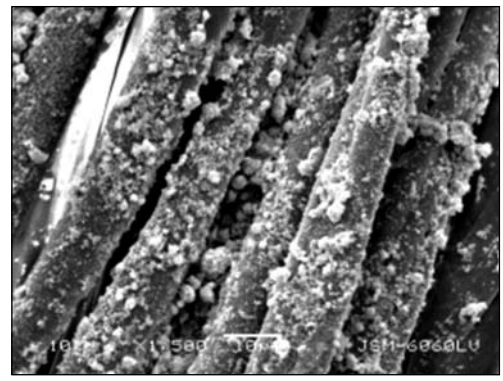
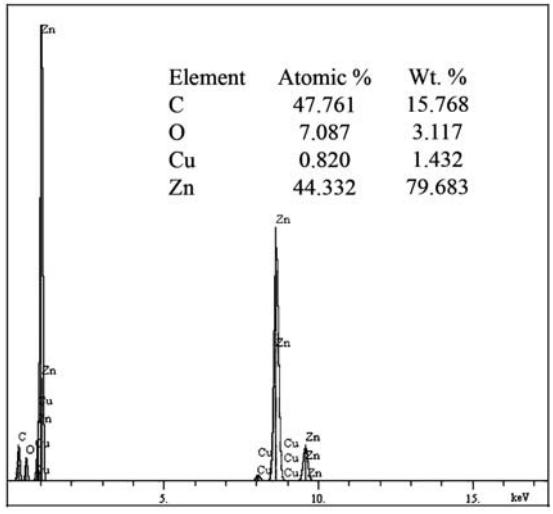


Figure 6: SEM pictures of (a) undyed and (b) dyed cotton fibres functionalized with nano-ZnO using Method IV.

a)



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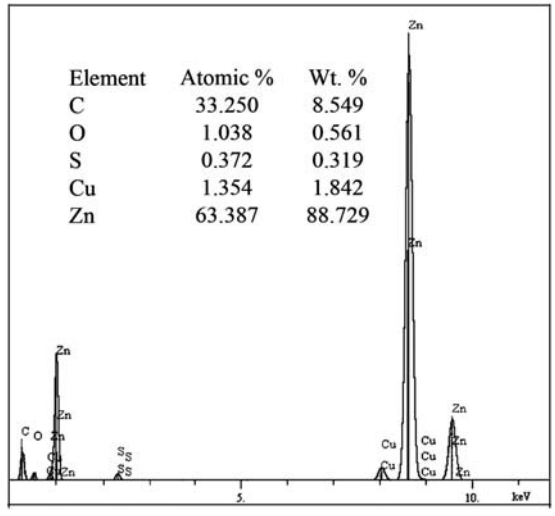

Figure 7: EDS image and element content of (a) undyed and (b) dyed nano-ZnO functionalized cotton using Method IV.

perties of undyed and dyed cotton fabric and achieving excellent UV protective properties of cotton.

Nano-ZnO was not synthetized in this research. As mentioned in the introduction and experimental section, the used nanoparticles were a commercial product intended for industrial use. Nevertheless, DLS analysis was performed to evaluate the size of particles in the treating baths according to Methods I and IV (Table 2). ZnO dispersions were obtained by simple mixing the ZnO nanopowder and the bi-distilled water with addition of alkali (Method I) or with an addition of a small amount of acetic acid. After mixing, samples were exposed for 5 min to ultrasound finger in order to homogenise the dispersion. Samples were prepared without mechanical deagglomeration, like ball milling. Particle size in dispersion was determined with the dynamic light scattering technique (DLS). DLS measurements revealed that the initial (as-prepared) ZnO dispersion consisted of particle or agglomerates with some bigger aggregates. The distribution of the particles on the textile is obtained from SEM micrographs. In a solution prepared by Method I, the majority of particles by the volume have a mean diameter of 1036 nm. However, the solution prepared by Method IV contains smaller agglomerates with the majority of particles by the volume with mean diameter of 230 nm. Nano-ZnO exhibit high ability for aggregation or agglomeration, and the agglomerates can range up to 10 μm when using commercially prepared ZnO powders.[25] The reason for smaller agglomerates in a solution prepared by Method IV is an addition of CH_3COOH, which acts as a thinner.[26] However, higher concentration of CH_3COOH cannot be used when functionalizing cotton fabrics, since cotton is sensitive to medium to strong acidic conditions. Accordingly, the results of DLS analysis are in agreement to those of SEM images.

Table 3: DLS measurements results of particles size diameter distribution for the nano-ZnO using different Methods.

Method	Size distribution by volume	
	Mean diameter (nm)	Percent (%)
Method I	1036	62.2
	192	23.2
	5287	14.5
Method IV	230	51.1
	897	26.5
	5255	22.0

The FTIR spectra of undyed cotton fabric and cotton fabrics functionalized using Methods I and IV are presented in Figure 8. From spectra it can be seen that no changes of spectra in the range 3300–700 cm^{-1} can be observed among studied samples, whilst hardly noticeable differences of spectra are noticed in the range 400–500 cm^{-1}, characteristic for ZnO vibrations.[27] Appearance of absorp-

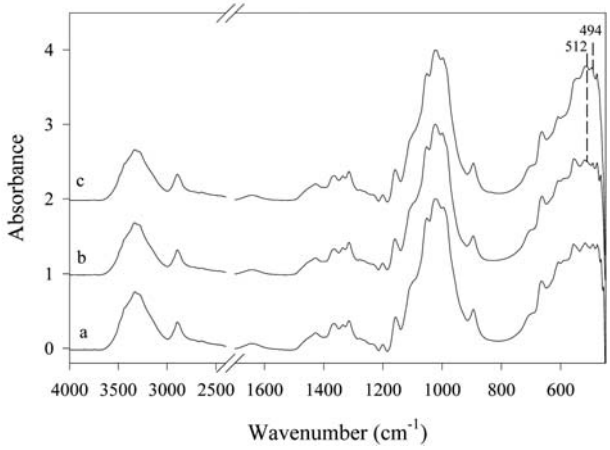

Figure 8: IR spectra of (a) undyed and (b), (c) nano-ZnO functionalized cotton samples, using Method I (b) and Method IV (c).

tion bands at 512 cm^{-1} (spectra b and c, Figure 8) and at 494 cm^{-1} (spectrum c, Figure 8) is attributed to vibrations of Zn–O bond.[28,29]

The results of whiteness and colour measurements of untreated and nano-ZnO functionalized samples by all methods are presented in Tables 4 and 5.

Table 4: CIE whiteness index (WI) and tint value ($T_{w,10}$) of undyed and nano-ZnO functionalized cotton samples

Sample	WI	$T_{w,10}$
Undyed	79.23	–0.17
Method I	67.90	–1.37
Method II	70.94	–0.57
Method III	75.39	–0.37
Method IV	77.26	–0.16

Table 5: CIELAB colour coordinates and colour difference (E^*_{ab}) of dyed and nano-ZnO functionalized cotton samples

Sample	L*	a*	b*	E^*_{ab}
Dyed	45.12	50.43	1.06	–
Method I_D	44.62	49.99	1.02	0.67
Method II_D	44.07	49.15	0.64	2.59
Method III_D	44.35	48.34	0.41	5.63
Method IV_D	45.66	46.79	–3.33	5.73

Note: D … dyed sample

The *Undyed* sample has the highest whiteness index (WI = 79.23) (Table 4). The WI varies according to the method used for loading of nano-ZnO onto cotton. The biggest impact on the whiteness is achieved by a modification by a Method I. The WI of *Method I* sample is decreased (WI = 67.90) and the change in the whiteness of sample was also observed visually. With increasing nano-ZnO content on the fibres, the whiteness of cotton increases. The cotton sample functionalized by Method IV, which has the highest content of nano-ZnO particles, has a WI = 77.26 and a tint value $T_{w,10}$ = –0.16. These values are very close to the values of *Undyed* sample. Therefore, the Method IV has a minimum influence on the whiteness change of functionalized samples while giving them excellent UV protective properties. The calculated colour difference (E^*_{ab}) from CIELAB colour coordinates (Table 5) between untreated (*Dyed*) and nano-ZnO functionalized samples increases with the increased content of nano-ZnO particles on the fibres. The colour difference between *Dyed* and *Method I_D* sample is only 0.67, meaning there is no visible colour change between the two samples. The highest content of nano-ZnO particles on the dyed functionalized cotton gives the colour difference of 5.73 (Table 5). With increasing content of nano-ZnO particles on the fibres, CIE a* and CIE b* coordinates decrease, meaning the dyed nano-ZnO functionalized cotton fabrics are more green and more blue in comparison to the *Dyed* sample. The presence of unintentional impurities in ZnO crystals may shift optical transmission and induce absorption bands in their optical spectra.[30] The nano-ZnO functionalized samples gain the greenish and bluish colour probably due the metal ions impurities present in the ZnO particles such as Cu,[31] which was detected by the EDS analysis (Figures 3 and 7).

4. Conclusions

Four different methods of functionalizing cotton with nano-ZnO particles were investigated for achieving UV protective properties of undyed and bi-reactive dyed cotton. The methods of nano-ZnO application differ in procedure used, time of treatment and used auxiliaries in the treating bath. Regardless of the used method, undyed and dyed cotton samples gain better UV protective properties when functionalized with nano-ZnO particles. Additionally EDS revealed that dyeing with reactive dye increases nano-ZnO content on the fibres. However, not all methods of functionalizing cotton with nano-ZnO gave good results in achieving excellent UV protective properties. Surprisingly, the UPF of samples that were treated in the nano-ZnO bath for one hour was the lowest. On these samples the distribution of nano-ZnO was uneven and the difference in whiteness comparing to the untreated sample was large. The two methods differing in a used medium (water or alcohol) achieved very similar UPF results and good UV protective properties of cotton. Method IV gave remarkable results. The distribution of nano-ZnO on the fibres was uniform and the UPF was high for undyed and dyed samples giving the treated samples excellent UV protective properties. The Method IV can potentially be used as application method of ZnO nanoparticles onto other fibrous polymers, such as keratin fibres, polyesters and polyamides, but the research on those materials will be performed in future research.

5. Acknowledgments

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Povzetek

Za doseganje zaščite pred UV sevanjem so bile proučevane različne metode nanosa nanodelcev ZnO (nano-ZnO) na belo in barvano bombažno tkanino. Metode se med seboj razlikujejo v načinu nanašanja nano-ZnO, času obdelave in dodanih pomožnih sredstev v obdelovalno kopel. Ultravijolični zaščitni faktor (UPF) je bil določen za neobdelane in funkcionalizirane vzorce. Prisotnost nano-ZnO na vlaknih je bila potrjena z uporabo vrstične elektronske mikroskopije (SEM) in z infrardečo spektroskopijo s Fourierjevo transformacijo (FTIR). Vsebnost Zn je bila določena z energijsko disperzijskim spektrometrom rentgenskih žarkov (EDS) in masno spektrometrijo v induktivno sklopljeni plazmi (ICP-MS). Z uporabo dinamičnega sipanja laserske svetlobe (DLS) je bila določena velikost delcev v pripravljenih raztopinah. Rezultati kažejo, da UV zaščita bombaža narašča z višjo vsebnostjo in enakomerno porazdelitvijo nano-ZnO na vzorcih ter da barvanje poveča adsorptivnost bombaža do nano-ZnO. Z eno od metod (Method IV) so bili doseženi izjemi rezultati, saj je imel bombaž odlično UV zaščito neglede ali je bil barvan ali ne.