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Antifungal efficiency of corona pretreated polyester and polyamide fabrics loaded with Ag nanoparticles

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Abstract This study discusses the possibility of using the corona (electric discharge at atmospheric pressure) treatment for fiber surface activation that can facilitate the loading of Ag nanoparticles (NPs) from colloids onto the polyester and polyamide fabrics and thus enhance their antifungal activity against *Candida albicans*. The laundering durability of achieved effects and the influence of dyeing of fabrics with disperse dyes on their antifungal efficiency were studied. The morphology of fibers loaded with Ag nanoparticles was characterized by SEM whereas X-ray photoelectron spectroscopy was used for the evaluation of surface chemical changes. Corona pretreated polyester and polyamide fabrics loaded with Ag nanoparticles showed better antifungal properties compared to untreated fabrics. The advantage of corona treated fabrics became even more prominent after washing test, particularly for polyester fabrics. Antifungal efficiency of polyester and polyamide fabrics loaded with Ag nanoparticles were almost unaffected by dyeing process.

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Introduction

The interests for antimicrobial textiles aimed for use in long-lasting contact with a skin significantly increased in the last several years [1]. Sportswear, medical, and protective textiles are particularly important in prevention and healing of sensitive skin prone to bacterial and fungal infections. Various agents based on quaternary ammonium salts, metal salts solutions, and antibiotics were used for imparting antimicrobial properties to textile materials [2, 3]. However, poor efficiency or high toxicity made them unsuitable for the long term use. On the contrary, silver in different forms exhibit outstanding antimicrobial activity showing low toxic impact to mammalian cells [4]. The recent progress in synthesis of Ag nanoparticles (NPs) without stabilizers opened up new possibilities for advanced textile finishing. The application of Ag NPs to textile materials is favorable in comparison with other silver forms since the significant portion of Ag atoms on the surface of the NPs is exposed toward surrounding medium, providing a desired antimicrobial effect.

The manufacturing of sportswear, medical, and protective textiles based on polyester (PES) and polyamide (PA) fibers continually increases. These fibers exhibit hydrophobic behavior and low surface energy [5, 6]. Higher accessibility of hydrophobic fibers to various chemical species can be obtained by plasma functionalization and/or plasma etching [7]. Improved loading of Ag NPs from colloids and enhanced interaction between hydrophilic Ag NPs and hydrophobic fabrics can be achieved by plasma treatment [4, 8]. Therefore, plasma treatment can also open new possibility for the introduction of nanotechnologies in textile processing. Yuranova et al. [4] reported that chemical deposition of silver-clusters onto polyamide–polyester based fabric surfaces, which were previously

activated by low pressure RF plasma provides excellent antibacterial activity. Plasmas generated at low pressures remain superior due to excellent control, stability, and uniformity of treatment effects but they require more complex handling of textile materials through the vacuum systems (http://www.kotonline.com/english_pages/ana/basliklar/kiekens.asp). Thus, atmospheric plasma treatments such as corona discharge might be more suitable for textile processing.

The previous studies have been mainly focused on the effect of Ag NPs on antibacterial efficiency of textile materials [9–14]. It is well-known that *Candida albicans* is responsible for a wide range of itching skin infections with yeasts particularly in the skin folds [15]. Hence, the aim of this study was to consider the possibility of using the corona treatment for fiber surface activation (introduction of new reactive groups, oxidation, free radicals, increase of solid surface tension, etc.), which can facilitate the loading of Ag NPs from colloids onto the PES and PA fabrics and thus, improve their antifungal activity against *Candida albicans*. The laundering durability of obtained antifungal effects as well as the influence of dyeing of PES and PA fabrics on antifungal efficiency was also examined.

Experimental

Treatment of fabrics

Desized and bleached polyester (PES, 165 g/m^2) and polyamide (PA, 150 g/m^2) fabrics were cleaned as described elsewhere [16]. Dye C.I. Disperse Blue 3 (DB3) was used for studies on dyeing of PA fabrics and C.I. Disperse Violet 8 (DV8) of PES fabrics. The characteristics of dyes are presented in Table 1.

Corona treatment of fabrics was carried out at atmospheric pressure using a commercial device Vetaphone CP-Lab MK II. Fabrics were placed on the electrode roll, rotating at the minimum speed of 4 m/min. The distance between electrodes was 2 mm. The power was 900 W and the number of passages was set to 30. The Ag loading was always performed 2 h after corona treatment.

$AgNO_3$ (Kemika) and $NaBH_4$ (Fluka) of p.a. grade were used without any further purification for the synthesis of colloid of Ag NPs. Briefly, 8.5 mg of $AgNO_3$ was dissolved in 250 mL of water purged by Ag for 30 min [17, 18]. Under vigorous stirring, reducing agent $NaBH_4$ (125 mg) was added to the solution and left for 1 h in argon atmosphere. The concentration of Ag colloid was 50 ppm.

One gram of fabric was immersed in 65 mL of colloid of Ag NPs for 5 min and dried at room temperature. After 5 min of curing at 100 °C, the procedure was repeated. Subsequently, the samples were rinsed twice (5 min) with deionized water and dried at room temperature.

PES fabrics were dyed in Polycolor (Werner Mathis AG) laboratory beaker dyer in the bath containing 1% (o.w.f.) DV8, 1 g L^{-1} CHT dispergator (Bezema) and 0.5 mL L^{-1} CH_3COOH (30%) at liquor-to-fabric ratio of 25:1 and pH 5. PES fabrics were placed in the dyebath at 20 °C and temperature was raised to 50 °C (heating rate: 2 °C/min), when dye was added. The dyebath was heated up to 130 °C (heating rate: 2 °C/min). After 60 min of dyeing process at 130 °C, the dyebath was cooled down to 80 °C (cooling rate: 5 °C/min).

Dyeing of PA fabrics was carried out in the bath containing 2% (o.w.f.) DB3 at liquor-to-fabric ratio of 60:1 and pH 5. The dyebath was heated up from 20 °C to 50 °C, when the PA fabrics were added. The temperature was then raised to 100 °C (heating rate: 2 °C/min). After dyeing at 100 °C for 60 min, the dyebath was cooled down to 70 °C (cooling rate: 5 °C/min).

The pH values were adjusted with CH_3COOH (30%). Fabrics were subsequently washed in the bath containing 0.5% Felosan RG-N (Bezema) at liquor-to-fabric ratio of 40:1. After 30 min of washing at 40 °C, fabrics were rinsed once with warm water (40 °C) for 3 min and five times (3 min) with cold water. Afterwards, the fabrics were dried at room temperature.

Methods

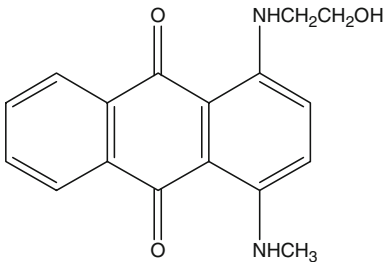
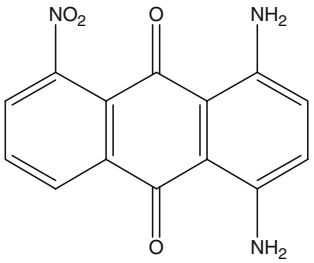
The shape and size of Ag NPs were assessed using transmission electron microscope, (TEM) Philips EM-440 operating at 100 kV. Samples for TEM measurements were prepared by placing a drop of Ag colloid onto a holey carbon-coated standard copper grid (400 mesh) and evaporating the solvent.

The UV/VIS absorption spectra of the Ag colloid were recorded using a Thermo Evolution 600 spectrophotometer.

Fiber morphology was observed by scanning electron microscope (SEM) JEOL JSM 6460 LV. Gold layer was deposited on the samples before the analysis.

X-ray photoelectron spectroscopy was used for the evaluation of surface chemical changes. Samples were analyzed using a PHI Model 5500 Multitechnique System with an Al $K\alpha$ monochromatic X-ray source operating at 350 W. The measurements were taken using a take-off angle of 45°. Survey scans were taken in the range 0–1100 eV, with pass energy of 187.85 eV. High resolution scans were obtained on the C_{1s}, O_{1s}, N_{1s}, and Ag_{3d} photoelectron peaks, with pass energy of 23.5 eV. Binding energies were referenced to the C_{1s} photopeak position for C–C and C–H species at 285.0 eV. Surface composition has been estimated after a linear background subtraction

Table 1 The characteristics of the dyes

Dye	Manufacturer	Molecular structure
DB3 C.I. Disperse Blue 3 (Colliton blue FFR)	BASF	
DV8 C.I. Disperse Violet 8 (Palanil violet 3B)	BASF	

from the area of the different photo-emission peaks modified by their corresponding sensitivity factors [19].

The antifungal efficiency of fabrics was quantitatively evaluated by using a *C. albicans* ATCC 24433. Fungal inoculum was prepared in the tripton soy broth (Torlak, Serbia), which was used as the growth medium for fungi while the potassium hydrogen phosphate buffer solution (pH 7.2) was used as the testing medium. Fungi were cultivated in 3 mL of tripton soy broth at 37 °C and left overnight (late exponential stage of growth). Afterwards, 70 mL of sterile potassium hydrogen phosphate buffer solution was added to sterile Erlenmeyer flask (300 mL), which was then inoculated with 0.7 mL of the fungal inoculum. The zero counts were made by removing 1 mL aliquots from the flask with inoculum, and making 1:10 and 1:100 dilutions in physiological saline solution. 0.1 mL of the 1:100 solution was placed onto a tripton soy agar (Torlak, Serbia) and after 24 h of incubation at 37 °C, the zero time counts (initial number of fungal colonies) of viable fungi were made.

One gram of sterile fabric cut into small pieces was put in the flask (70 mL of sterile potassium hydrogen phosphate buffer solution inoculated with 0.7 mL of the fungal inoculum) and shaken for 1 h. One-hour counts were made in accordance with an above described procedure.

The percentage of fungi reduction (R, %) was calculated using the Eq. 1:

$$R = \frac{C_0 - C}{C_0} \cdot 100 \quad (1)$$

where, C_0 (CFU—colony forming units) is the number of fungi colonies on the control fabric (untreated fabric without Ag) and C (CFU) is the number of fungi colonies on the fabric loaded with Ag NPs [9, 10, 13].

The percentage of fungi reduction after dyeing was determined according to Eq. 1. In this case C_0 denoted the number of fungi colonies on the control fabric (dyed untreated fabric without Ag) and C is the number of fungi colonies on the dyed fabric loaded with Ag NPs.

Laundering durability of antifungal effect was determined after five washing cycles in Polycolor (Werner Mathis AG) laboratory beaker dyer as described elsewhere [16]. The percentage of fungi reduction after five washing cycles was determined according to Eq. 1.

Results and discussion

The surface of PES and PA fabrics were modified using colloidal Ag NPS synthesized without any stabilizer. The absorption spectrum of colloidal Ag NPs in aqueous solution is shown in Fig. 1. The position of symmetric surface plasmon resonance band with maximum at 380 nm and its half-width (*fwhm*, $\Delta\lambda = 46$ nm) indicated the

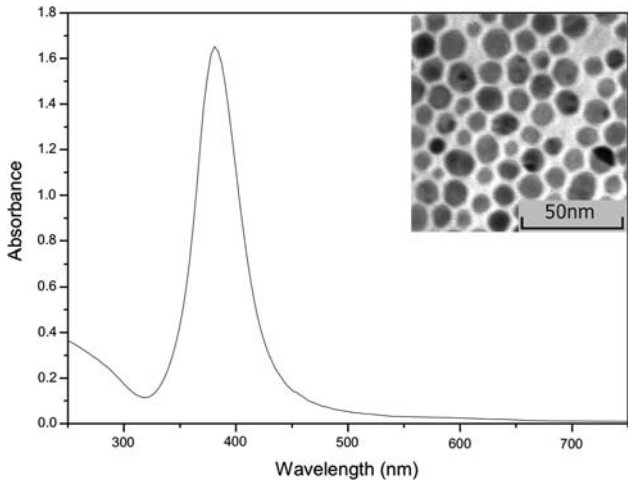


Fig. 1 Absorption spectrum of Ag NPs in aqueous solution; inset: TEM image of Ag NPs

narrow size distribution without undesired aggregation. Characteristic TEM image of nearly spherical Ag NPs with an average diameter of about 10 nm is shown as an inset in Fig. 1.

In order to achieve better accessibility of hydrophilic Ag NPs to hydrophobic fibers, corona pretreatment of PES and PA fabrics was performed. New functionalities introduced to the fabric surface by corona treatment provide better interaction between bare Ag NPs and fibers.

The changes in chemical composition of the surface of PES and PA fabrics caused by corona treatment were analyzed by XPS. Figure 2 shows survey spectra of untreated PES (UPES), corona treated PES (CPES), untreated PA (UPA), and corona treated PA (CPA) fabrics. The photoelectron peaks at binding energies of 285 and 532 eV corresponding to C_{1s} and O_{1s} appear in the spectra of PES fibers. In the spectra of PA fibers emerges an additional peak at 400 eV related to N_{1s}.

Corona treatment of PES fabrics brought about decrease in the intensity ratio between the C_{1s}/O_{1s} peaks. Such changes were also noticeable in the spectra of PA fabrics with additional increase in the intensity of N_{1s} peak. The elemental composition of untreated and corona treated PES and PA fabrics is given in Table 2.

In order to establish the changes of functional groups on the surface of PES and PA fibers induced by corona treatment, high-resolution C_{1s} spectra were analyzed. The C_{1s} spectra were deconvoluted with five components as can be seen in Table 3. The peak at 285.0 eV corresponds to C–C and C–H groups. The peak at 286.6 eV represents

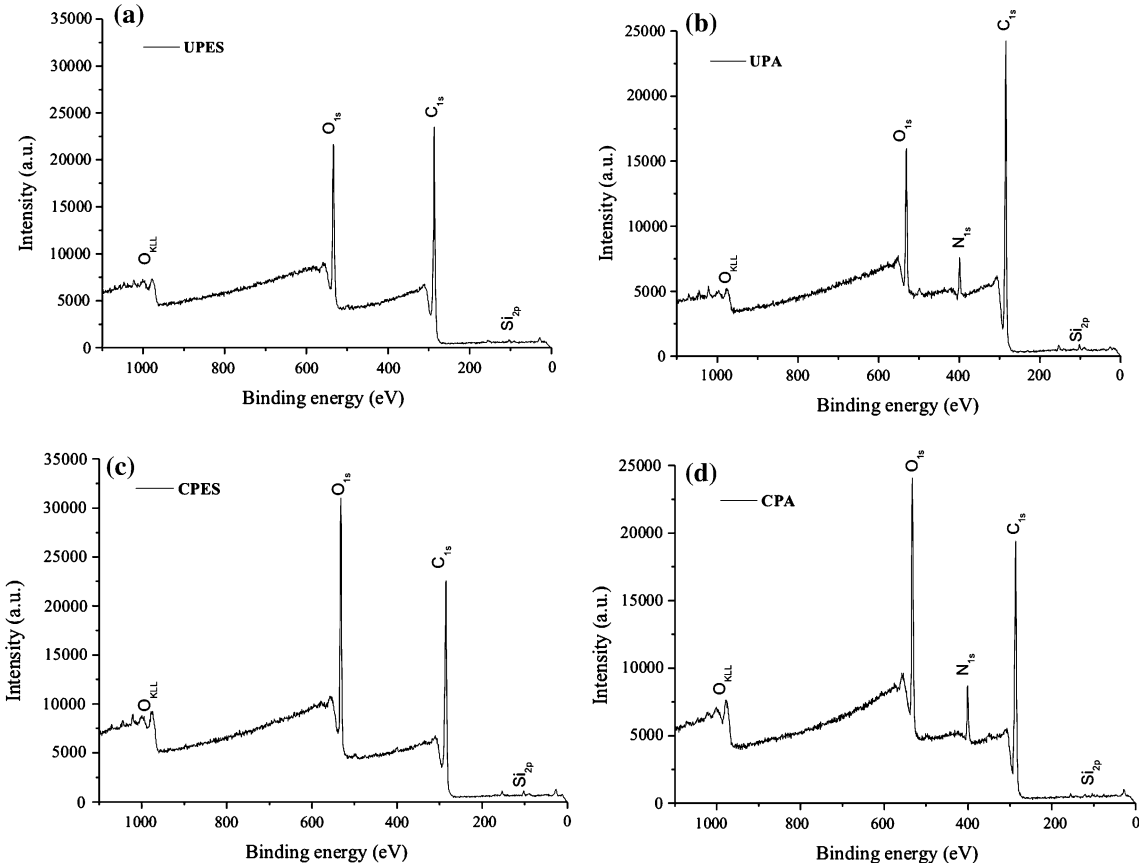


Fig. 2 The XPS survey spectra of **a** untreated PES, **b** untreated PA, **c** corona treated PES, and **d** corona treated PA fabrics

Table 2 Elemental composition of untreated and corona treated PES and PA fabrics

Sample	C (atom %)	O (atom %)	N (atom %)
UPES	76.81	23.19	0.00
CPES	70.69	29.31	0.00
UPA	79.74	14.81	5.46
CPA	68.30	23.67	8.03

Table 3 Relative intensity data of the deconvoluted C_{1s} spectra of untreated and corona treated PES and PA fabrics

Sample	Atomic ratio (%)				
	C_{charge} 283.5 eV	C–C, C–H 285.0 eV	C–N, C–O 286.6 eV	C=O 288.3 eV	O–C=O 289.1 eV
UPES	18.34	51.32	20.23	1.48	8.63
CPES	18.41	45.76	20.45	4.37	11.00
UPA	18.81[a]	65.33	7.86	6.04	1.96
CPA	22.21[a]	48.42	14.41	10.51	4.46

[a] This peak is observed at 283.2 eV on the PA fibers

C–N and C–O groups, whereas the peaks at 288.3 eV and 289.1 eV are assigned to C=O and O–C=O groups, respectively. The component at 283.5 eV for PES fibers and at 283.2 eV for PA fibers could not be attributed to any specific functional group. However, it is suggested that these peaks appeared as a consequence of the broadening of the C_{1s} peak induced by the strong charging of the PES and PA fabrics during the XPS measurements [20–22].

The results from Table 3 indicate that corona treatment led to a significant increase in the content of C=O and O–C=O groups on the surface of PES and PA fibers. The content of C–N and C–O groups was not altered after corona treatment of PES fibers. On the contrary, the content of these groups on the corona treated PA fibers was almost doubled. The decrease in the content of C–C and C–H groups is characteristic for the plasma treatment of polymers, which occurs as a result of chain scission induced by the plasma active particles or as a consequence of a progressive oxidation of the carboxylated groups to release CO_2 [23, 24].

Evidently, corona treatment induced a significant change in the chemical composition of the surface of PES and PA fibers. The formation of new carbonyl and carboxyl groups as a result of the numerous homogeneous and heterogeneous air plasma reactions, led to an increase in hydrophilicity of PES and PA fabrics as was confirmed by decrease in the contact angles of water on the air [16].

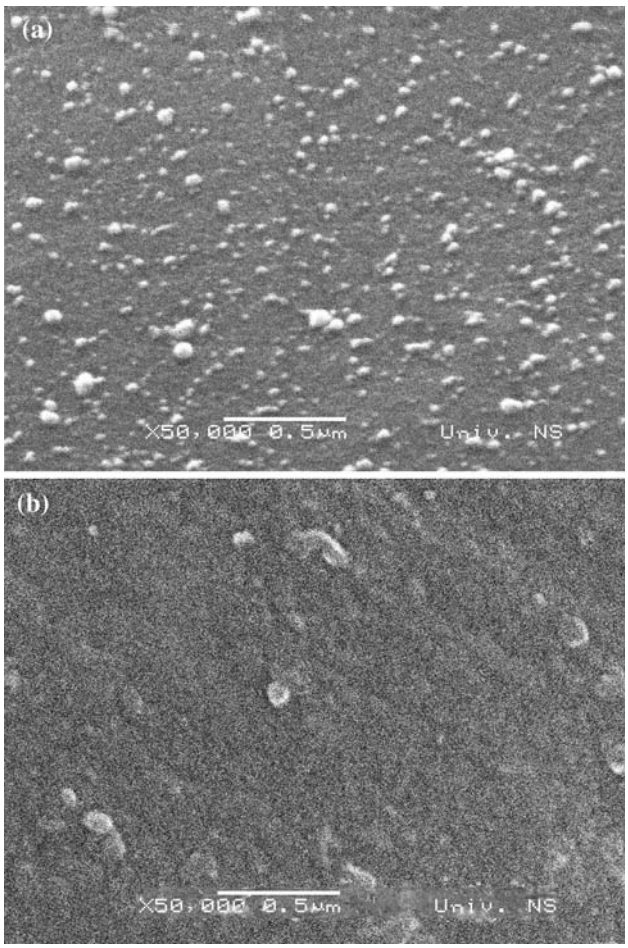


Fig. 3 SEM images of Ag loaded **a** CPES and **b** CPA fibers

Increased hydrophilicity induced by corona treatment led to an enhanced deposition of Ag NPS onto PES and PA fibers, which was followed by SEM analysis (Fig. 3). SEM analysis revealed the higher amount of Ag NPs on the corona treated fibers in comparison to untreated fibers. Corona treated PES fibers are covered with small, uniformly dispersed aggregates of Ag NPs with dimensions mostly ranging from 20 to 60 nm. Almost spherical Ag NPs with diameters around 10 nm can also be noticed which correspond to diameters of single colloidal Ag NPs detected by TEM analysis (Fig. 1). Unlike PES fibers, irregularly shaped bigger assemblies of Ag NPs were scattered over the surface of corona treated PA fibers.

The confirmation of Ag presence on the fiber surface and an investigation of their form were obtained by XPS analysis. Survey spectra of untreated (UPES + Ag, UPA + Ag) and corona treated (CPES + Ag, CPA + Ag) fibers loaded with Ag NPs are shown in Fig. 4. In addition to characteristic photoelectron peaks corresponding to C_{1s}, O_{1s}, and N_{1s}, the peaks related to Ag_{3p} and Ag_{3d} appear at binding energies of 573.6 eV and 368 eV, respectively.

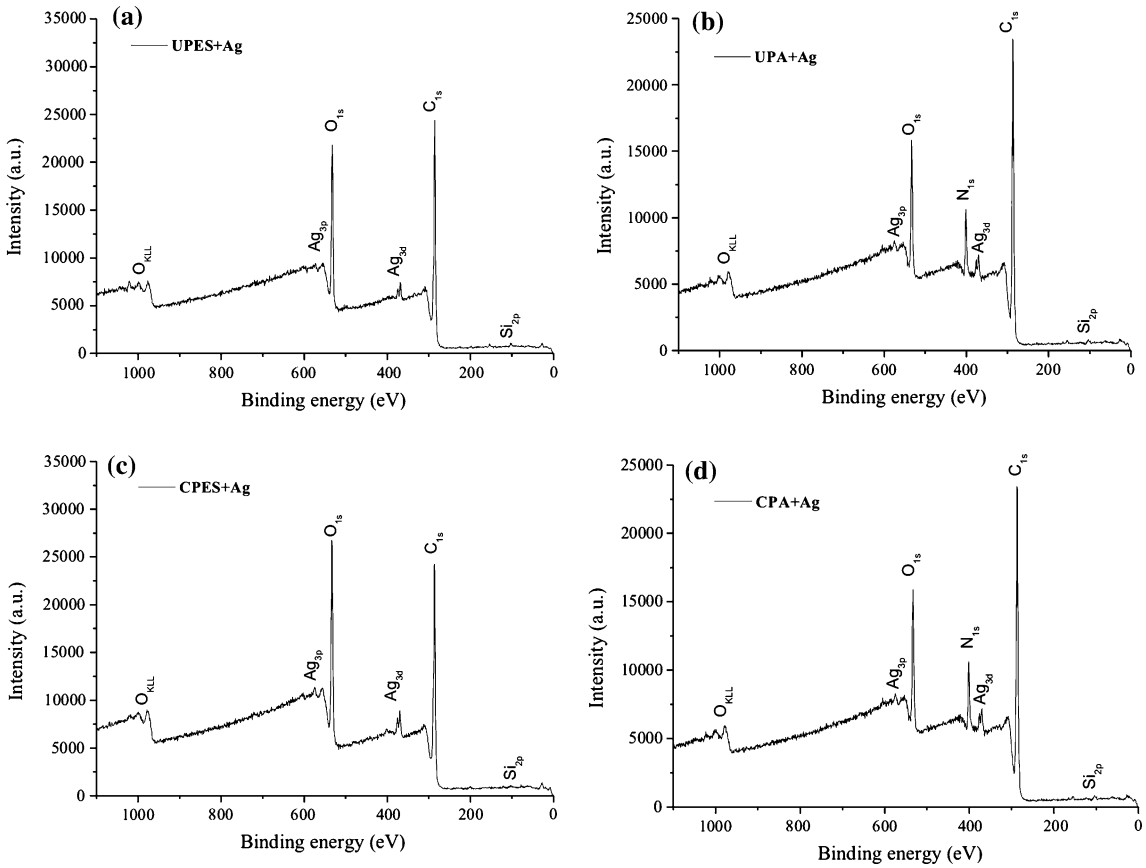


Fig. 4 The XPS survey spectra of untreated and corona pretreated fabrics loaded with Ag NPs

Table 4 Elemental composition of untreated and corona pretreated PES and PA fabrics loaded with Ag NPs

Sample	C (%)	O (%)	N (%)	Ag (%)
UPES + Ag	78.32	21.35	0.00	0.33
CPES + Ag	74.62	24.83	0.00	0.55
UPA + Ag	78.16	17.55	4.16	0.13
CPA + Ag	77.12	13.48	9.03	0.37

Table 4 shows the elemental composition of untreated and corona pretreated fibers loaded with Ag NPs.

Corona treatment positively influenced the binding of Ag NPs to the surface of PES and PA fabrics, leading to an increase in the amount of silver. The content of Ag on the corona pretreated PA fibers was almost three times higher compared to untreated PA fibers, whereas on corona pretreated PES fibers the content is only by 60% higher than on untreated PES fabric. It is also clear that PES fibers contain higher amount of silver compared to PA fibers. The increased number of carboxyl groups confirmed by XPS analysis along with existence of benzene rings in polymer structure indicates the possibility of strong interaction between PES fibers and Ag NPs [25, 26]. It is known from SERS (Surface-Enhanced Raman Spectroscopy) studies of benzoic acid and its derivates on the surface of Ag NPs that the strong interaction between Ag surface and benzene rings and carboxylic groups can be established. In addition to carboxyl groups, the polar amide groups take part in the interaction between PA polymers and Ag NPs [27].

Figure 5 presents the high resolution XPS spectra of fibers surface in the region of binding energies associated to $Ag_{3d\ 5/2}$ core-electrons. The binding energy of the $Ag_{3d\ 5/2}$ core-electrons from Ag loaded UPES and UPA fibers is 368.1 eV. However, the binding energies of the $Ag_{3d\ 5/2}$ core-electrons from Ag loaded corona pretreated PES and PA fibers are shifted to 368.6 and 368.4 eV, respectively. The reason for positive binding energy shift could be explained by the change of oxidation state and/or existence of higher content of bigger agglomerates of Ag NPs on the surface of corona pretreated PES and PA fibers [28]. The larger agglomerates of Ag NPs on the surface of the corona pretreated PA fibers likely induced smaller positive shift of the $Ag_{3d\ 5/2}$ core-electron binding energy compared to the shift of the $Ag_{3d\ 5/2}$ core-electron binding energy of the smaller assemblies of Ag NPs present on the surface of corona pretreated PES fibers [28].

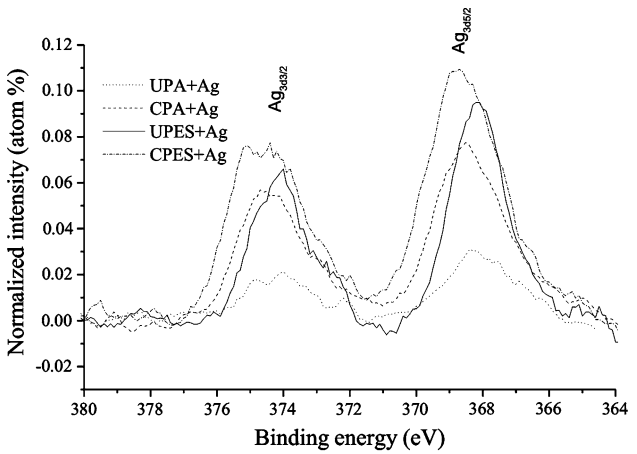


Fig. 5 High resolution XPS spectra of fibers surface in the binding energy region associated to $Ag_{3d\ 5/2}$ core-electrons

To evaluate the antifungal activity of PES and PA fabrics loaded with Ag NPs, fungal tests with *C. albicans* were accomplished. The fungal reduction of untreated and corona pretreated fabrics loaded with Ag NPs is presented in Table 5. Untreated PES fabrics loaded with Ag NPs exhibited better antifungal activity than untreated PA fabrics. Although corona treatment led to an increase in antifungal efficiency of both fabrics, its positive effect was more prominent on PA fabrics.

It is also evident that antifungal efficiency of corona activated fabrics after five washing cycles remained higher in comparison with untreated fabrics (Table 5). Untreated PES and particularly corona pretreated PES fabrics loaded with Ag NPs exhibited remarkably better laundering durability than PA fabrics. This observation is consistent with our previous study on the bacterial reduction of Gram-negative bacterium *Escherichia coli* and Gram-positive bacterium *Staphylococcus aureus* on the same fabrics [16]. The lower amount of Ag NPs on these fabrics could be the reason for poorer laundering durability of PA fabrics.

There are only few articles dealing with the influence of dyeing on antibacterial efficiency of fabrics loaded with Ag NPs [9, 14], but none on antifungal activity have been published to date to the best of our knowledge. The results of studies on antifungal activity of Ag loaded PES and PA fabrics before and after dyeing with disperse dyes are given in Table 6. Both, untreated and corona pretreated PES fabrics loaded with Ag NPs showed extraordinary antifungal efficiency independently of order of the operations, i.e., Ag loading and dyeing. Accordingly, there are no limitations related to order of the operations, which is very important from technological point of view. However, obtained fungal reduction of dyed untreated and corona pretreated PES fabrics loaded with Ag NPs fabrics is higher compared to corresponding fabrics that were not dyed (Table 5). Table 5 reveals that the untreated PES fabric (without Ag NPs) exhibited fungal reduction of only one order of magnitude likely due to adsorption of *C. albicans* on the fabric surface [4]. On the other hand, the untreated PES fabric dyed with DV8 (without Ag NPs) shows the further fungal reduction, indicating that dye itself might inhibit the fungi growth. Reason for such activity of dye DV8 can be found in existence of three amino groups in its structure, which are known to react with cell membrane of microbes, obstructing the fungi growth [29]. Corona pretreated PA fabrics loaded with Ag NPs and dyed with DB3 demonstrated superior behavior if loading was performed prior to dyeing. The opposite order of operations is recommended for untreated PA fabrics loaded with Ag NPs.

Conclusions

Deposition of colloidal Ag NPs onto PA and PES fabrics provides excellent antifungal effect against *Candida albicans*. Untreated PES fabrics loaded with Ag NPs exhibited better antifungal efficiency than untreated PA fabrics. Corona treatment positively affected the antifungal efficiency of both fabrics loaded with Ag NPs. The contribution of corona treatment to improvement of antifungal efficiency of both fabrics became even more prominent after five washing cycles. Significantly better laundering durability of obtained effects on PES fabrics can be attributed to higher content of Ag as detected by XPS.

Table 5 Antifungal efficiency of Ag loaded PES and PA fabrics

Sample	Initial number of fungal colonies (CFU)	Number of fungal colonies on the fabric (CFU) (before washing)	*R* (%)	Number of fungal colonies (CFU) (after washing)	*R* (%)
UPES	4.2×10^5	6.3×10^4		6.3×10^4	
UPES + Ag		1.9×10^3	97.0	9.0×10^3	85.7
CPES + Ag		5.6×10^2	99.1	2.1×10^3	96.7
UPA		1.9×10^5		1.9×10^5	
UPA + Ag		1.3×10^4	93.2	8.4×10^4	55.8
CPA + Ag		1.6×10^2	99.9	6.7×10^4	64.7

Table 6 Antifungal efficiency of Ag loaded PES and PA fabrics before and after dyeing

Dye	Sample	Initial number of fungal colonies (CFU)	Number of fungal colonies (CFU)	*R* (%)
Ag loading before dyeing				
DV8	UPES	2.7×10^5	9.0×10^3	
	UPES + Ag		<10	99.9
	CPES + Ag		<10	99.9
DB3	UPA	3.9×10^5	5.9×10^4	
	UPA + Ag		1.5×10^3	97.5
	CPA + Ag		2.0×10^2	99.7
Ag loading after dyeing				
DV8	UPES	2.7×10^5	9.0×10^3	
	UPES + Ag		<10	99.9
	CPES + Ag		<10	99.9
DB3	UPA	3.9×10^5	5.9×10^4	
	UPA + Ag		1.7×10^2	99.7
	CPA + Ag		8.3×10^2	98.6

Antifungal efficiency of untreated and corona treated PES fabrics is unaffected by dyeing with C.I. Disperse Violet 8. All studied PES fabrics preserved extraordinary antifungal efficiency. In order to achieve optimum antifungal effect, dyeing of untreated PA fabrics with C.I. Disperse Blue 3 before the loading of Ag NPs is recommended. The opposite order of operations is suggested for corona treated PA.

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