Danijela Klemenčič[1], Zoran Petrović[2], Nevena Puač[2], Marija Gorenšek[1]
[1] Oddelek za tekstilstvo, Naravoslovnotehniška fakulteta, Univerza v Ljubljani
[2] Inštitut za fiziko, Beograd

Obdelava tkanine poliester/bombaž-Lycra s plazmo Ar/N_2

Izvirni znanstveni članek

Poslano *junij 2009* • Sprejeto *avgust 2009*

Polyester/Cotton-Lycra Fabric Treatment with Ar/N_2 Plasma

Original Scientific Paper

Received *June 2009* • Accepted *August 2009*

Abstract

In the research, a woven polyester/cotton-Lycra (PET/CO-Lycra) fabric was treated with low pressure Ar/N_2 (50%/50%) plasma for two minutes at pressure 26.6 Pa. The fabric was further on coated with the lowest prescribed concentration of Ruco-Bac AgP (RbAg) to obtain antimicrobial properties. The silver content on samples was evaluated with the ICP-MS analysis. The antimicrobial activities of RbAg-impregnated, plasma-treated RbAg-impregnated, and once and ten times washed samples were investigated according to the ASTM Designation E 2149-01. The effects on the mechanical properties and morphological changes of untreated and plasma-treated samples were studied in the research. The results show that the silver content present on plasma-treated RbAg-impregnated samples is slightly higher in comparison to the untreated samples. The results of bacterial reduction of all RbAg-impregnated fabrics show growth inhibition to all four tested microorganisms. Once and ten times washed plasma-treated RbAg-impregnated samples show better resistance to Escherichia Coli and Staphylococcus Aureus, compared to the untreated, washed

Vodilni avtor/corresponding author:
Marija Gorenšek
tel.: +386 1 200 32 34
e-mail: marija.gorensek@ntf.uni-lj.si

Izvleček

Za nanos tržnega protimikrobnega sredstva Ruco-Bac AgP (RbAg) je bila tkanina iz mešanice poliester/bombaž-Lycra (PET/CO-Lycra) dve minuti izpostavljena nizkotlačni plazmi (26,6 Pa) z mešanico plinov Ar/N_2 (50 %/50 %). Koncentracija srebra na tkanini je bila določena z analizo ICP-MS. Bakterijska redukcija RbAg-impregniranih, plazemsko obdelanih in RbAg-impregniranih ter enkrat in desetkrat pranih vzorcev je bila določena na podlagi dinamične stresalne metode ASTM E 2149-01. Opravljene so bile meritve pretržne sile in raztezka ter narejeni mikroskopski posnetki morfoloških sprememb neobdelanih in plazemsko obdelanih vzorcev. Vsebnost srebra na plazemsko obdelanih in RbAg-impregniranih vzorcih je rahlo večja kot pri neobdelanih. Rezultati bakterijske redukcije kažejo, da vse RbAg-impregnirane tkanine zavirajo rast vseh štirih testnih bakterijskih vrst. Enkrat in desetkrat prani plazemsko obdelani in RbAg-impregnirani vzorci kažejo boljšo odpornost proti *Escherichia Coli* in *Staphylococcus Aureus* kot neobdelani prani vzorci. Obdelava tkanine s plazmo nima posebnih učinkov na specifično pretržno napetost in raztezek prej.

Ključne besede: plazma Ar/N_2, Ruco-Bac AgP, srebro, protimikrobna učinkovitost, SEM

1 Uvod

Protimikrobne tekstilije imajo širok spekter uporabe v medicini, v gospodinjstvu, pri izdelkih za šport in prosti čas, za zunanjo uporabo in kot tehnične tekstilije. Tkanina iz poliestra/bombaža-Lycre

samples. Plasma treatment has no influence on specific stress and elongation of yarns.

Keywords: Ar/N_2 plasma, Ruco-Bac AgP, silver, antimicrobial effectiveness, SEM

1 Introduction

Antimicrobial textiles are widely used in medicine, sports and leisure, outdoor, household and technical textiles. Woven polyester/cotton-Lycra (PET/CO-Lycra) fabrics are convenient for bed linen and mattress covers.

The surfaces of textile materials with low adhesion properties to antimicrobial agents cannot provide good antimicrobial effectiveness. However, material surface adhesion properties can be effectively modified by different treatments. Plasma technologies can modify the surface without altering the bulk properties of a material [1, 2]. Such technologies have a strong potential to become a new, surface-engineering tool in the textile industry due to their low temperatures and dry, environment-friendly nature [2].

Plasma has been recently applied in the treatment of living tissues [3–5], sterilization [6, 7], treatment of materials [8, 9], etching processes for nanoelectronics and surface treatment as well as in the growth of structures for nanotechnologies [10]. When plasma is applied to organic and other materials which cannot sustain high temperatures, it is essential to use non-equilibrium, otherwise known as low-temperature or cold plasma [9]. In such a system, the energy of electrons can be controlled by an external field while maintaining the ion and gas temperatures close to the room temperature. Achieving non-equilibrium conditions is relatively easy at low pressure where breakdown voltages are the lowest, since the operation is close to the minimum of the Paschen curve [11, 12]. Under these conditions, relatively large variations in the gap or thickness of the treated material may be accommodated without any effect on plasma. On the other hand, operation at atmospheric pressure is far from the Paschen minimum and thus apart from a higher breakdown voltage; therefore, it is difficult to achieve uniform glow discharges [13]. A stable operation of

(PET/CO-Lycra) je primerna za posteljno perilo in za prekrivala vzmetnic.

Površine tekstilnih materialov imajo navadno slabe adhezijske lastnosti do protimikrobnih sredstev in zato ne dajejo dovolj učinkovite protimikrobne zaščite. Adhezijo površin materialov lahko učinkovito spremenimo z različnimi postopki. Med te spada tudi plazemska tehnologija, s katero lahko spremenimo površino materiala brez velikih sprememb osnovnih lastnosti materialov [1, 2]. Takšne tehnologije imajo priložnost, da postanejo nove metode za površinsko spreminjanje materialov v tekstilni industriji, ker so nizkoenergijske, suhe in okolju prijazne [2].

V zadnjem času so plazmo uporabljali za obdelavo tkiv živih organizmov [3–5], za sterilizacijo [6, 7], za obdelavo materialov [8, 9] in za jedkanje materialov za nanoelektroniko ter za površinsko obdelavo za nalaganje struktur za nanotehnologijo [10]. Pri plazemski obdelavi organskih in drugih materialov, ki ne prenašajo visokih temperatur, je treba uporabiti neravnotežne, znane tudi kot nizkotemperaturne oziroma hladne plazme [9]. V teh sistemih se lahko na eni strani ohranja energija elektronov, medtem ko se ioni in temperatura plina ohranjajo blizu sobne temperature. Pri nizkem tlaku je doseganje neravnotežnih razmer relativno preprosto, ker so razelektritve minimalne glede na to, da poteka delovanje blizu minimuma Paschenove krivulje [11, 12]. Pri teh okoliščinah tudi velika odstopanja v odprtini ali debelini tretiranega materiala nimajo posebnega učinka na plazmo. Na drugi strani je delovanje plazme pri atmosferskem tlaku daleč od Paschenovega minimuma in je težko doseči enakomerne razelektritve [13]. Stabilno delovanje razelektritve v heliju in v nekaterih redkih plinih je mogoče izvesti [14], pogosto pa prevladajo drugi mehanizmi, ki lahko povzročijo poškodbe materiala zaradi iskrenja in sproščanja lokalizirane toplote. Glede na to, da nekatera področja uporabe, kot je obdelava živih celic [15], zahtevajo atmosferski tlak, obstaja relativno stabilno široko področje, ki deluje tako v zraku kot pri atmosferskem tlaku [16]. Kot večina drugih sistemov, ki vključujejo masiven tok helija, kjer pa je gradnja komor vsaj tako kompleksna kot gradnja vakuumske komore z uporabo različnih črpalk [17], je nizkotlačna plazma še vedno možnost za obdelavo materialov, kot so tekstilije in polimeri, saj so le-ti stabilni pri nizkem tlaku [18, 19].

Prednost nizkotlačne plazemske metode je v tem, da je to dobro nadzorovana in ponovljiva tehnika [2]. Cilj nekaterih plazemskih površinskih modifikacij so povečanje adhezije, izboljšava vpojnosti in navzemanje barvila [20, 21]. Površinske spremembe se s pomočjo plazemske obdelave lahko dosežejo na različne načine; uporabljeni so bili že različni plini, kot so zrak, kisik, dušik, argon in helij [2]. Pri plazemski obdelavi z zrakom, argonom, kisikom, dušikom in NH_3 z vključitvijo hidroksilnih (–OH), karboksilnih (–COOH), karbonilnih (>C=O) in amino (–NH_2) skupin so bile dosežene izboljšave vpojnosti [1, 2, 20–32]. Prav tako so poročali o povečanju barvalnih sposobnosti vlaken po plazemski obdelavi z zrakom, kisikom, dušikom, argonom, SF_6 ali akrilatom [2, 23, 25–26].

glow discharges in helium and some rare gases is possible [14]; however, other mechanisms often prevail, which can cause damage to the material due to sparks, localized heating and arcs. While some applications like treating living cells [15] require atmospheric pressure, there is only one relatively stable wide-area system which operates both in air and at atmospheric pressure [16]. Like most other systems involving massive flow of helium, where the building of chambers for helium is as complex as building a vacuum chamber with differential pumping [17], low-pressure plasma is still an option for the treatment of materials such as textiles and polymers which may sustain being subjected to low pressure [18, 19].

The advantage of the low-pressure plasma method is that it is a well-controlled and reproducible technique [2]. The objectives of some plasma surface modifications are adhesion promotion, enhanced surface wettability, and enhanced dyeability [20, 21]. Surface modification with plasma treatment has been achieved in different ways, e.g. with gases such as air, oxygen, nitrogen, argon and helium [2]. An improvement of wettability with the introduction of hydroxyl (–OH), carboxyl (–COOH), carbonyl (>C=O) or amino ($-NH_2$) groups when treated with air, oxygen, argon, nitrogen or NH_3 plasma was observed [1–2, 20–32]. An enhancement of fibre dyeability after the treatment with oxygen, air, nitrogen, argon, SF_6 or acrylate plasma was reported as well [2, 23, 25]. Adhesion intensification, which is a very important property of synthetic fibres, can be obtained by treating fibres with air, oxygen, nitrogen, argon or acrylate plasma [1–2, 28–29, 33–36]. Researchers have also proved an increase of the surface-free energy of synthetic and cellulosic fibres after plasma treatments [1–2, 28–29, 33–36].

According to the above described properties, the purpose of our research was to use low-pressure Ar/N_2 plasma to provide better adhesive surface properties of the PET/CO-Lycra fabric and consequently, a better application of commercial antimicrobial finishing, the activity of which depends on the action of Ag^+ ions. The main purpose of our research was to reduce the concentration of antimicrobial finishing in terms of the amount of silver in the padding bath, while

Povečanje adhezije kot pomembne lastnosti sintetičnih vlaken lahko dosežemo s plazemsko obdelavo vlaken z zrakom, kisikom, dušikom ali akrilatom [1, 2, 28, 29, 33–36]. Raziskovalci poročajo tudi o povečanju površinske proste energije sintetičnih in celuloznih vlaken po plazemski obdelavi [1, 2, 28, 29, 33–36].

Glede na opisane lastnosti je bil namen naše raziskave uporabiti nizkotlačno plazmo Ar/N_2, s katero bi dosegli boljše adhezijske lastnosti površine tkanine PET/CO-Lycra in s tem boljši nanos komercialnega protimikrobnega sredstva, katerega aktivnost je odvisna od delovanja Ag^+ ionov. Glavni cilj naše raziskave je bil zmanjšati koncentracijo protimikrobnega sredstva v impregnirni kopeli, pri čemer naj bi impregniran material dosegal protimikrobne lastnosti skladno z vsebnostjo srebra.

2 Eksperimentalni del

2.1 Tkanina

V raziskavi smo uporabili tkanino PET/CO-Lycra z maso 292 g/m^2, debelo 2,12 mm, z gostoto osnove 42 niti/cm (poliesterna preja, 33,3 tex), gostoto votka 13,5 niti/cm (bombažna OE preja, 33,3 tex) in 4,5 niti/cm (bombaž/Lycra core preja, 41,7 tex), proizvedeno v IBI (Kranj, Slovenija).

2.2 Protimikrobno apreturno sredstvo

Za raziskavo protimikrobne aktivnosti smo uporabili tržni izdelek Ruco-Bac AgP (RbAg) (izdelovalec Rudolf Chemie) [37]. Je anionsko aktivna snov, disperzija, sestavljena iz anorganskih soli in površinsko aktivnih snovi, ki izpolnjuje zahteve Öko-Tex Standarda. Uporabili smo dve različni koncentraciji sredstva, in sicer 2 g/l in 1 g/l. Izdelovalec predpisuje kot najnižjo koncentracijo za doseganje protimikrobnih lastnosti z Ruco-Bac AgP 2 g/l sredstva v impregnirni kopeli.

2.3 Obdelava s plazmo

Uporabljen induktivno sklopljen plazemski reaktor (CCP) za obdelavo vzorcev tkanin PET/CO-Lycra je deloval pri 13,56 MHz v popolnoma nesimetrični obliki. Namen je bil zmanjšati energijo ionov, ki padajo na osnovno elektrodo. Pri tem so poškodbe zaradi bombardiranja ionov minimalne in temperatura plina je blizu sobne temperature. Sistem smo že pred tem uporabili za obdelavo polimerov [18], konoplje [38] in netkanih tekstilij, materialov iz reciklirane volne [19], kakor tudi za obdelavo semen [39, 40], da bi izboljšali kalitev.

Nizkotemperaturna RF razelektritev pri 13,56 MHz se ustvari s pomočjo napajalnika Dressler Caesar 1010 RF v kombinaciji z omrežjem Variomatch. Za obdelavo vzorcev tkanin PET/CO-Lycra smo uporabili komoro, ki je bila narejena iz nerjavečega jekla, s premerom 370 mm in dolgo 500 mm. Osrednja elektroda s premerom 14 mm je bila napetostna elektroda, medtem ko so stene komore *still producing a treated material with the desired antimicrobial properties.*

2 Experimental

2.1 Material

A woven blended PET/CO-Lycra fabric with the mass 292 g/m², thickness 2.12 mm, warp density 42 threads/cm of polyester filaments (33.3 tex), weft density 13.5 threads/cm of cotton open-end yarn (33.3 tex) and 4.5 threads/cm of cotton/lycra core yarn (41.7 tex) obtained from IBI (Kranj, Slovenia) was used for the research.

2.2 Antimicrobial finishing agent

For the examination of the antimicrobial activity, Ruco-Bac AgP (RbAg) produced by Rudolf Chemie was used [37]. This commercial, anion active dispersion is composed of inorganic salts and surfactants, which fulfils the Öko-Tex Standard. Two concentrations of RbAg were used, i.e. 2 g/l and 1 g/l, the former being the lowest concentration prescribed by the producer of Ruco-Bac AgP.

2.3 Plasma treatment

A capacitively coupled plasma (CCP) reactor used for the treatments of PET/CO-Lycra samples was designed to operate at the frequency 13.56 MHz in a highly asymmetric mode. The idea was to reduce the energies of ions that fall on the ground electrode. Therefore, the damage caused by the ion bombardment is minimal and the gas temperature is close to the room temperature. This system was previously used for the treatment of polymers [18], hemp [38] and non-woven, recycled wool material [19] and also for the treatment of seeds [39, 40] in order to improve germination.

The low-temperature RF discharge at 13.56 MHz is generated using the Dressler Caesar 1010 RF power supply in combination with the Variomatch matching network. For the treatment of woven blended PET/CO-Lycra samples, a cylindrical chamber made of stainless steel of 370 mm in diameter and 500 mm in length was used. The central electrode of 14 mm in diameter represented the powered electrode, while the chamber wall represented the ground electrode.

ozemljena elektroda. Na dnu in stran od sten komore je vgrajena ravna plošča za vzorce PET/CO-Lycra. Shematski prikaz uporabljene plazemske enote je na sliki 1.

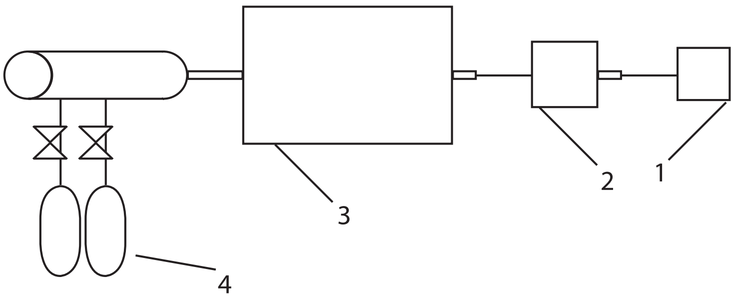

Figure 1: Schematic representation of the plasma set-up.

1 – RF power supply and matching network, 2 – home-made voltage transformer, 3 – chamber with electrodes and sample holder, 4 – Ar and N_2 flask.

Pri tej raziskavi uporabljena tkanina je bila obdelana v nizkotlačni plazmi pri naslednjih pogojih: Ar/N_2 plazma s 50 % argona in 50 % dušika, čas obdelave dve minuti, moč obdelave 100 W in tlak 26,6 Pa. Obdelava tkanine s plazmo je bila izvedena na Inštitutu za fiziko v Zemunu (Beograd, Srbija).

2.4 Protimikrobno apretiranje

Neobdelane in plazemsko obdelane vzorce tkanin z maso 10 g smo impregnirali z RbAg po impregnirno-sušilni metodi. Vzorce tkanin smo najprej omočili v vodi pri sobni temperaturi, oželi na dvovaljčnem fularju s 75-odstotnim ožemalnim učinkom in sušili pri 130 °C. Na naslednji stopnji smo tkanine impregnirali s protimikrobno impregnirno kopeljo, ki je bila sestavljena iz sredstva proti navzemanju nečistoč (Sevatex SRH) in protimikrobnega sredstva (RbAg) v rahlo kislem mediju (0,5 g/l ocetne kisline 80 % (Fluka)) pri sobni temperaturi. Sledilo je ožemanje na dvovaljčnem fularju s 56-odstotnim ožemalnim učinkom in končnim sušenjem pri 160 °C. V preglednici 1 so podane oznake vzorcev in koncentracije

Table 1: Sample marks and agent concentrations used in the research.

Sample	Sevatex SRH (g/l)	Ruco-Bac AgP (g/l)
U-2RbAg	30	2
U-1RbAg	30	1
P-2RbAg	30	2
P-1RbAg	30	1

U – untreated; P – plasma-treated.

trode. At the bottom of the chamber and clear of the sidewalls, there was a flat platform where woven blended PET/CO-Lycra samples were placed. A schematic representation of the plasma set-up is shown in Figure 1.

The samples were treated with low-pressure plasma at the following conditions: Ar/N_2 plasma of the mixture 50% argon and 50% nitrogen for two minutes at the power 100 W and pressure 26.6 Pa. The plasma treatments of fabrics were conducted at the Institute of Physics Zemun, Belgrade, Serbia.

2.4 Antimicrobial finishing

The fabrics of 10 g in mass were impregnated with RbAg with the pad-dry method. In the first stage, the fabrics were wetted with water at room temperature with the wet pick-up of 75% and dried up at 130 °C. In the second stage, the fabrics were impregnated with the mixture of a soil release and hydrophilic agent (Sevatex SRH) and antimicrobial agent in slightly acidic media (0.5 g/l of 80% solution of acetic acid (Fluka)) at room temperature with the wet pick-up of 56%. Afterwards, the fabrics were dried up at 160 °C. The marks of samples and concentrations of agents used in the research are presented in Table 1. The fabrics were impregnated two months after being plasma-treated.

2.5 ICP-MS analysis

A quantitative analysis of silver was performed on the RbAg-impregnated samples and on the washed RbAg-impregnated samples using the ICP-MS method based on the plasma effect. The latter is formed when argon passes through the radiofrequency field in which gas particles become partly electrically discharged and emit light of characteristic wavelengths. The analysis was conducted by a certified laboratory. For each sample, three measurements were made and the Ag concentration was given as the mean value.

2.6 Antimicrobial effectiveness

The untreated reference sample, the RbAg-impregnated samples and the washed RbAg-impregnated samples were tested for bacterial reduction according to the ASTM Designation E 2149-01 [41]. This test method was designed for

uporabljenih sredstev v tej raziskavi. Plazemsko obdelane tkanine so bile impregnirane dva meseca po obdelavi s plazmo.

2.5 Analiza ICP-MS

Kvantitativna analiza srebra na RbAg-impregniranih in pranih RbAg-impregniranih vzorcih je bila določena z uporabo metode ICP-MS. Metoda temelji na učinku plazme, ki se ustvari, ko argon potuje skozi radiofrekvenčno polje, kjer delci plina dobijo delni električni naboj in oddajajo svetlobo karakterističnih valovnih dolžin. Analiza je bila opravljena v laboratoriju s certifikatom. Za vsak vzorec so bile opravljene tri meritve in koncentracija Ag je podana kot srednja vrednost treh meritev.

2.6 Protimikrobna učinkovitost

Bakterijska redukcija referenčnih RbAg-impregniranih in pranih RbAg-impregniranih vzorcev je bila izvedena z dinamičnim stresalnim testom po ASTM E 2149-01 [41]. Testna metoda je bila razvita za ocenitev odpornosti neizluženih protibakterijsko obdelanih vzorcev na rast mikrobov pri dinamičnih kontaktnih pogojih. Testna metoda je bila opravljena na štirih različnih bakterijah: *Staphylococcus Aureus* (ATCC 25923), *Escherichia Coli* (ATCC 25922), *Streptococcus Faecalis* (ATCC 29912) in *Pseudomonas Aeruginosa* (ATCC 27853). Analiza je bila opravljena v laboratoriju s certifikatom. Bakterijska redukcija (R) je izračunana po naslednji enačbi (enačba 1):

$$R\ (\%) = \frac{B - A}{B} \times 100 \qquad (1)$$

kjer je A število kolonij bakterij (CFU/ml) po eni uri stresanja apretiranega vzorca in B število kolonij bakterij (CFU/ml) po enominutnem stresanju (času „0“) apretiranega vzorca [41].

2.7 Pralne obstojnosti

RbAg-impregnirane vzorce smo enkrat in desetkrat prali pri 60 °C v Launderometru po standardni metodi ISO 105-C03 [42]. Po vsakem pralnem ciklusu smo vzorce temeljito sprali pod tekočo vodo in jih posušili na zraku.

2.8 Pretržna sila in raztezek

Pretržno silo in raztezek neobdelanih in plazemsko obdelanih prej, ki smo jih prej izvlekli iz tkanine, smo merili na dinamometru Instron 6022 skladno s SIST ISO 2062:1997 [43]. Metoda je bila spremenjena tako, da je bila vpeta dolžina 100 mm. Specifična pretržna napetost je bila izračunana po naslednji enačbi (enačba 2):

$$\sigma \left(\frac{\mathrm{cN}}{\mathrm{dtex}}\right) = \frac{F}{T_t} \qquad (2)$$

kjer je F pretržna sila (cN) in T_t dolžinska masa preje (dtex).

the resistance evaluation of non-leaching antibacterial-treated specimens to the growth of microbes under dynamic contact conditions. The test method was conducted for four different bacteria, i.e. Staphylococcus Aureus (ATCC 25923), Escherichia Coli (ATCC 25922), Streptococcus Faecalis (ATCC 29912) and Pseudomonas Aeruginosa (ATCC 27853). The analysis was performed by a certified laboratory. The bacterial reduction (R) was evaluated according to Equation 1, where A is CFU per millilitre for the flask containing the treated substrate after 1 hour contact time and B is CFU per millilitre for the flask to determine A before the addition of the treated substrate (time '0') [41].

2.7 Wash fastness determination

RbAg-impregnated samples were subjected to a single wash cycle and ten wash cycles at 60 °C in the Launder-ometer according to the ISO 105-C03 standard [42]. At the end of each wash cycle, the washed samples were rinsed thoroughly under tap water and were allowed to dry at room temperature.

2.8 Tensile stress and elongation

The tensile stress of untreated and plasma-treated yarns, which were previously unravelled from the fabrics, was measured on the Instron 6022 dynamometer according to the SIST ISO 2062:1997 standard [43]. The method was modified in the gauge length, which was 100 mm. The specific breaking load was calculated with Equation 2, where F is tensile stress (cN) and Tt is linear density of a yarn (dtex).

The specific stress σ (cN/dtex) and elongation ε (%) of yarns are the mean value of ten measurements of breaking load and elongation in warp (polyester) and weft (cotton, cotton-lycra) direction. Before the testing, the samples were conditioned as prescribed by ISO 139.

2.9 SEM

The microscopic evaluation of the morphological changes occurring after the plasma treatment of the PET/CO-Lycra sample was undertaken with the JEOL JSM 6060 LV scanning electron microscope. Prior to the observation, all samples were covered with carbon and an

Rezultati specifične pretržne napetosti σ (cN/dtex) in raztezka ε (%) prej so podani kot povprečne vrednosti desetih meritev pretržne sile in raztezka v smeri osnove (poliester) in votka (bombaž, bombaž-Lycra). Pred meritvijo so bile preje klimatizirane skladno z ISO 139.

2.9 SEM

Morfološke spremembe po plazemski obdelavi tkanine PET/CO-Lycra smo opazovali z vrstičnim elektronskim mikroskopom JEOL JSM 6060 LV. Vzorci so bili pred opazovanjem površine naparjeni s približno 10 nm tanko plastjo ogljika in zlitino Au/Pd (90 % / 10 %). Pospeševalna napetost elektronov je bila 10 kV, delovna razdalja je bila 20 mm in velikost opazovanega področja 32 × 32 μm^2 pri 8000-kratni povečavi.

3 Rezultati z razpravo

3.1 ICP-MS analiza

Kvantitativna analiza srebra na RbAg-impregniranih, plazemsko obdelanih RbAg-impregniranih in pranih RbAg-impregniranih vzorcih je bila določena z uporabo metode ICP-MS. Rezultati kvantitativne analize vsebnosti srebra na impregniranih vzorcih so podani v preglednici 2.

Table 2: Silver content on treated samples.

Sample	Ag (mg/kg)		
	W0	W1	W10
U-2RbAg	9.3 ± 1.9	3.1 ± 0.6	2.4 ± 0.5
U-1RbAg	6.2 ± 1.2	2.4 ± 0.5	1.6 ± 0.3
P-2RbAg	9.6 ±1.9	4.1 ± 0.8	2.7 ± 0.5
P-1RbAg	6.7 ± 1.3	2.4 ± 0.5	1.6 ± 0.3
W0 – unwashed; W1 – single-washed; W10 – ten times washed.			

Vzorci, impregnirani z 2 g/l RbAg, kažejo večjo vsebnost srebra v primerjavi z vzorci, ki so bili impregnirani z 1 g/l RbAg. Vsebnost srebra na plazemsko obdelanih vzorcih je za približno štiri odstotke večja kot na neobdelanih vzorcih. Takšni rezultati so bili pričakovani, saj so bili plazemsko obdelani vzorci impregnirani šele dva meseca po plazemski obdelavi. Primerjava vsebnosti srebra na blagu med plazemsko obdelanimi in neobdelanimi vzorci kaže, da se vsebnost srebra po enkratnem pranju zmanjša na približno tretjino, po desetem pranju pa na četrtino.

3.2 Protimikrobna učinkovitost

Protimikrobna aktivnost referenčne, plazemsko obdelanih, RbAg-impregniranih, plazemsko obdelanih in RbAg-impregniranih ter

Au/Pd (90%/10%) alloy in an about 10 nm thick layer. The electron accelerating tension was 10 kV, the working distance was 20 mm and the analyzed area was 32 × 32 μm² in size at 8,000× magnification.

enkrat in desetkrat pranih RbAg-impregniranih vzorcev je bila določena skladno z metodo ASTM E 2149-01. Rezultati v preglednici 3 so prikazani kot bakterijska redukcija, R (%).

Table 3: R (%) of RbAg-treated and of washed fabrics.

Sample	Wash cycles	Bacterial reduction (%)			
		Staphylococcus aureus ATCC 25923	Escherichia coli ATCC 25922	Streptococcus faecalis ATCC 29912	Pseudomonas aeruginosa ATCC 27853
U	0	30	8	1	–6
U-2RbAg	0	100	100	100	88
	1	–[a]	5	–[a]	2
	10	–[a]	–[a]	–[a]	–[a]
U-1RbAg	0	100	100	100	100
	1	–[a]	–[a]	–[a]	0
	10	–[a]	–[a]	4	3
P	0	–[a]	–[a]	2	–[a]
P-2RbAg	0	100	98	100	100
	1	7	51	2	–[a]
	10	43	23	–[a]	–[a]
P-1RbAg	0	92	82	100	100
	1	–[a]	–[a]	2	–[a]
	10	–[a]	6	1	1

[a] *no reduction of bacteria*

3 Results and discussion

3.1 ICP MS analysis

The quantitative analysis of silver was performed on the RbAg-impregnated, plasma-treated RbAg-impregnated and once and ten times washed RbAg-impregnated samples using the ICP MS method. The results of the quantitative analysis of the silver content on the impregnated samples are presented in Table 2.

The samples impregnated with 2 g/l of RbAg show a higher silver content than the samples impregnated with 1 g/l of RbAg. The amount of silver on the plasma-treated samples is approximately 4% higher than on the untreated

Rezultati vseh RbAg-impregniranih vzorcev kažejo odlično odpornost proti vsem štirim testiranim bakterijam. Vsi RbAg-impregnirani vzorci kažejo več kot 60-odstotno bakterijsko redukcijo. Prvo pranje uniči protimikrobno aktivnost apreturnega sredstva. Po prvem pranju nekaj protimikrobne aktivnosti na *E. coli* ohrani le P-2RbAg vzorec (R = 51 %) in po desetih pranjih na *S. aureus* (R = 43 %).

Opazna je korelacija med koncentracijo srebra s protimikrobno aktivnostjo impregniranih vzorcev. Vsebnost srebra na vzorcu P-2RbAg je po desetem pranju 2,7 ± 0,5 mg/kg, vendar pa vzorec ne doseže predpisane mejne protimikrobne aktivnosti 60 odstotkov. Na splošno rezultati pranih vzorcev kažejo boljšo redukcijo bakterij na plazemsko obdelanih in RbAg-impregniranih vzorcih (preglednica 3).

Iz rezultatov bakterijske redukcije in vsebnosti srebra vzorcev je razvidno, da je adhezija protimikrobnega sredstva na plazemsko obdelanih vzorcih boljša, predvidevamo pa lahko, da bi lahko bila odlična, če bi impregniranje z RbAg sledilo takoj po obdelavi tkanine s plazmo.

samples. Such results were expected, since the fabric surface was treated with Ar/N_2 plasma two months before the impregnation. If drawing a comparison between the plasma-treated and the untreated samples, the amount of silver after one wash cycle reduces to about on a third of the value and after the tenth wash cycles to about on a quarter.

3.2 Antimicrobial effectiveness

The antimicrobial activity of the untreated, plasma-treated, RbAg-impregnated, plasma-treated RbAg-impregnated and once and ten times washed RbAg-impregnated fabrics was determined according to the ASTM Designation E 2149-01 method [41]. The results in Table 3 are presented as bacterial reduction, i.e. R (%).

The results show excellent resistance of all RbAg-impregnated samples to the tested bacteria. All RbAg-impregnated samples show a reduction of bacteria higher than 60%. The first wash cycle destroys the antimicrobial activity of the RbAg-impregnated samples. Only the P-2RbAg sample retains some effectiveness against E. coli (R = 51%). After ten wash cycles, the P-2RbAg retains its activity to S. aureus (R = 43%). The dependence of antimicrobial activity on the concentration of silver is evident. The amount of silver on the P-2RbAg sample remains also after ten wash cycles 2.7 ± 0.5 mg/kg of silver; however, the sample does not reach the prescribed antimicrobial activity of the prescribed limit of 60%. In general, the washed P-2RbAg samples display a better antimicrobial effect (Table 3).

The results of antimicrobial activity and concentration of silver on the samples show higher adhesion of the antimicrobial agent on the plasma-treated fabric. A better effect would be obtained if the impregnation with RbAg followed directly after the plasma treatment.

The influence of the silver-based commercial antimicrobial finishing applied to cotton fabric was studied by Tomšič and Simončič [44]. 3 g/l of iSys AG (CHT) and 15 g/l of iSys MTX (CHT) was applied to the fabric using the pad-dry-cure method. In comparison to our research, we achieved the same bacterial reduction on E. coli with a five times lower silver

Vpliv protimikrobnega tržnega sredstva prav tako na osnovi srebra, nanesenega na bombažno tkanino, sta proučevali Tomšič in Simončič [44]. Protimikrobna kopel s 3,0 g/l iSys AG (CHT) in 15,0 g/l iSys MTX (CHT) je bila nanesena na tkanino po impregnirno-sušilni metodi. V primerjavi z našo raziskavo smo s petkrat nižjo koncentracijo srebra na blagu dosegli enako bakterijsko redukcijo na *E. coli*. V obeh raziskavah se koncentracija srebra na blagu znižuje po pralnih ciklusih za približno polovico, vendar pa v njihovem primeru po desetem pralnem ciklusu bakterijske redukcije niso dosegli. V naši raziskavi pa smo z uporabo najnižje predpisane koncentracije RbAg po desetem pranju dosegli 23-odstotno bakterijsko redukcijo na *E. coli*.

3.3 Pretržna sila in raztezek

Rezultati specifične pretržne napetosti (cN/dtex) neobdelanih in plazemsko obdelanih prej so prikazani na sliki 2.

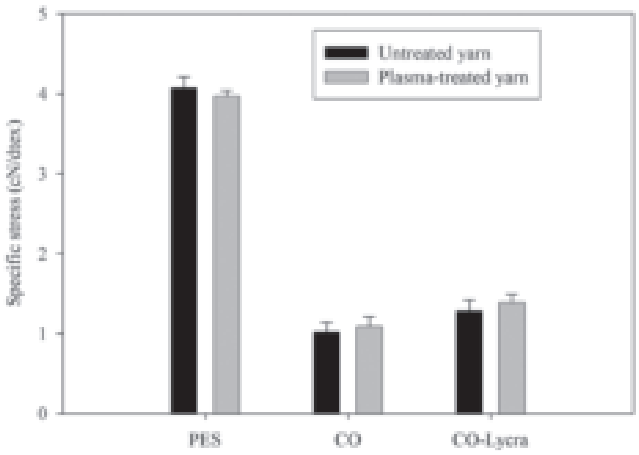

Figure 2: Specific stress (cN/dtex) of untreated and plasma-treated yarns.

Rezultati raztezka (%) neobdelanih in plazemsko obdelanih prej so prikazani na sliki 3.

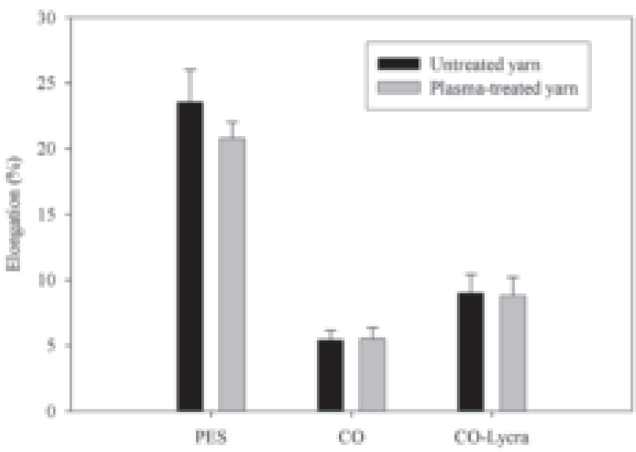

Figure 3: Elongation (%) of untreated and plasma-treated yarns.

content on the fabrics. In both researches, after the first and tenth wash cycles, the silver content on the fabrics decreased by about half the value; however, with Tomšič and Simončič, there was no bacterial reduction on the impregnated fabric after the tenth wash cycle. In our research, we achieved a 23% bacterial reduction after ten wash cycles on E. coli despite using a lower concentration of RbAg.

Rezultati specifične pretržne napetosti (slika 2) in raztezka (slika 3) kažejo zanemarljivo majhne razlike med neobdelanimi in plazemsko obdelanimi vzorci prej. Obdelava s plazmo zelo voluminozne tkanine PET/CO-Lycra ni vplivala na spremembo mehanskih lastnosti tkanine.

3.4 SEM

SEM-mikroposnetki neobdelanih in plazemsko obdelanih vzorcev so prikazani na slikah 4 in 5.

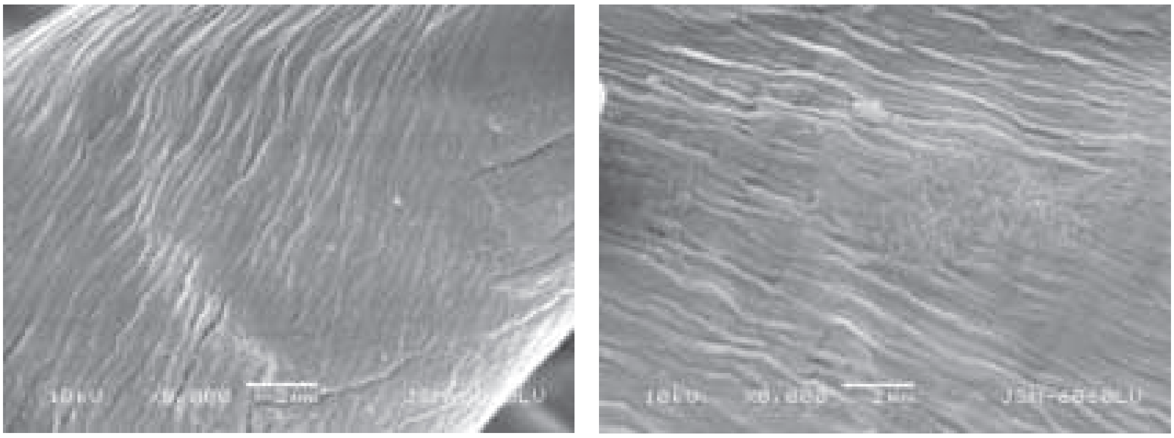

Figure 4: SEM micrographs of untreated cotton (a) and plasma-treated cotton (b).

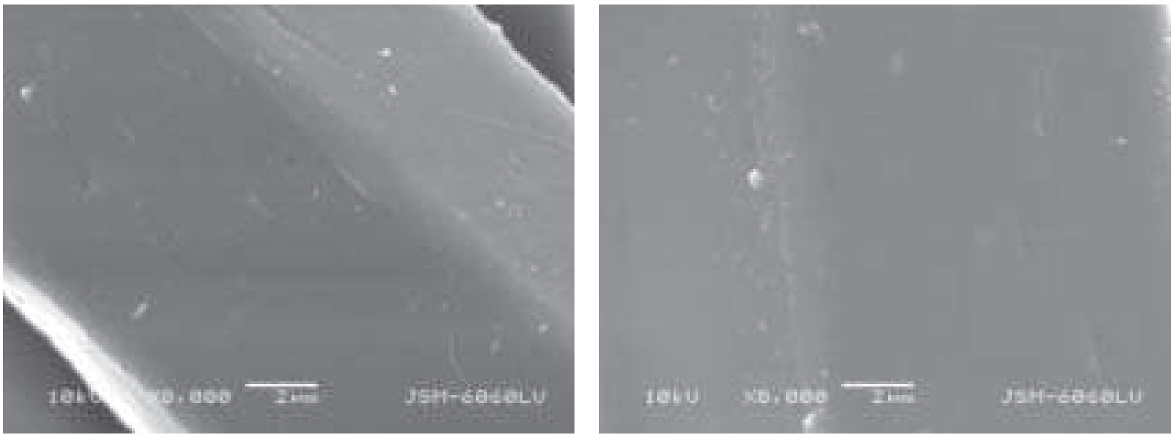

Figure 5: SEM micrographs of untreated polyester (c) and plasma-treated polyester (d).

3.3 Tensile stress and elongation

The results of the specific stress (cN/dtex) of the untreated and plasma-treated yarns are demonstrated in Figure 2.

The results of elongation (%) of the untreated and plasma-treated yarns are shown in Figure 3.

The results of the specific stress (cf. Figure 2) and elongation (cf. Figure 3) between the untreated and plasma-treated yarns show negligibly small

Glede na to, da je tkanina voluminozna in da je obdelava s plazmo trajala le dve minuti, SEM-mikroposnetki plazemsko obdelanih vzorcev poliestra (PET) in bombaža (CO) ne kažejo zaznavnih morfoloških razlik.

Nobenih vidnih površinskih sprememb po daljšem delovanju plazme z argonom na bombažna vlakna (do 90 minut), potrjenih s SEM-posnetki površin, niso opazili niti Jung, Ward in Benerito [30]. Hkrati pa se hidrofilnost bombažnih vlaken poveča, kar pomeni, da se površinsko odvisne lastnosti dramatično spremenijo. Prav tako je Wakida s sodelavci [32] dokazal, da obdelava PET-

differences. A two-minute low-pressure plasma treatment of a very voluminous PET/CO-Lycra fabric did not destroy the mechanical properties of the fabric.

3.4 SEM

The SEM images of the untreated and plasma-treated samples are shown in Figure 4 and Figure 5.

Considering that the fabric is voluminous and that the exposure time of the plasma treatment lasted only two minutes, the SEM images of both plasma-treated polyester (PET) and plasma-treated cotton (CO) fibres do not show any detectable morphological differences.

Jung, Ward and Benerito [30] established no visible surface changes after a long-time exposure of cotton fibres to argon plasma (up to 90 minutes), which was confirmed by the SEM images. At the same time, an increase in the hydrophilicity of the cotton fibres shows that the surface-dependent properties of fibres changed dramatically. Moreover, Wakida et al [32] established that no physical damage was caused to the PET fibre surface when the fibres were treated with low-temperature plasma of helium/argon or acetone/argon under atmospheric pressure for 10 to 180 seconds.

Despite the CO and PET surfaces after the plasma treatment not showing evident difference, the surface properties of both parts changed, which is evident from the results of silver content of the plasma-treated samples.

4 Conclusions

The purpose of this research was to provide better adhesive properties of the PET/CO-Lycra fabric to the durable hygienic finish Ruco-Bac AgP after the Ar/N_2 plasma treatment.

The concentrations of silver present on the RbAg-impregnated samples show that silver adsorbs better on plasma-treated samples in comparison to the untreated samples. Although the SEM analysis does not show any detectable changes of the surface morphology of the plasma-treated cotton and polyester fibres at 8,000× magnification, the plasma-treated samples do show better adhesive properties to silver.

The P-2RbAg sample shows a higher silver con-

vlaken v nizkotemperaturni plazmi helij/argon ali aceton/argon pri atmosferskem tlaku v 10 do 180 sekundah fizično ne poškoduje površine vlaken.

Čeprav površini CO in PET po obdelavi ne kažeta razlik, so površinske lastnosti spremenjene, kar je razvidno iz rezultatov vsebnosti srebra na plazemsko obdelanih vzorcih.

4 Sklepi

Namen raziskave je bil po plazemski obdelavi tkanine PET/CO-Lycra z Ar/N_2 zagotoviti boljše adhezijske lastnosti do obstojnega higienskega sredstva Ruco-Bac AgP.

Koncentracije srebra na RbAg-impregniranih vzorcih kažejo, da plazemsko obdelani vzorci adsorbirajo nekoliko več srebra kot neobdelani vzorci. Čeprav analiza SEM pri 8000-kratni povečavi ne kaže opaznih površinskih sprememb, kažejo plazemsko obdelani vzorci boljše adhezijske lastnosti do srebra.

Vzorec P-2RbAg kaže večjo vsebnost srebra po prvem in tudi po desetem pranju v primerjavi z drugimi vzorci. Na podlagi rezultatov vsebnosti srebra lahko povzamemo, da kažejo plazemsko obdelani vzorci boljšo bakterijsko redukcijo. Vzorec P-2RbAg ohrani po prvem pranju aktivnost na *E. coli* (R = 51 %) in po desetem pranju na *S. aureus* (R = 43 %). Ti rezultati, četudi niso odlični (R > 60 %), kažejo možnost uporabe plazme Ar/N_2 kot fazo predobdelave tkanine PET/CO-Lycra za dosego boljših adhezijskih lastnosti. Tkanine so bile obdelane s plazmo dva meseca pred nanosom protimikrobnega sredstva. Izkušnje kažejo, da se aktivnost površine, ki izvira iz novih funkcionalnih skupin, formiranih s plazmo, s časom zmanjšuje, seveda pa izjedkanine in s tem povečana površina in spremenjena morfologija površine vlaken ostanejo.

Raziskava je pokazala tudi, da obdelava tkanine z nizkotlačno Ar/N_2 plazmo ne vpliva na mehanske lastnosti tkanine.

5 Zahvala

Raziskavo sta finančno podprli Agencija za raziskovalno dejavnost RS v okviru programa P2-0213 in MVZT v okviru projekta Eureka E!4043 Nanosilver for multipurpose textiles.

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tent after the first and the tenth wash cycle as well. It can be concluded that this behaviour is connected with the generally better bacterial reduction of the plasma-treated washed samples. The P-2RbAg sample retains after the first wash cycle some activity to E. coli (R = 51%) and after the tenth wash cycles, activity to S. aureus (R = 43%). These results, although not excellent (R>60%), show the possibilities of using the Ar/N_2 plasma as a pre-treatment media for achieving better adhesive properties of PET/CO-Lycra fabrics. The plasma treatment of fabrics was conducted two months before the impregnation with the antimicrobial agent. Experiences show that the surface activity springing from the new functional groups, which originate in plasma application, reduces in time. Nevertheless, an increase in the surface area caused by surface etching and changed morphology remain unchanged.

The results of this research also show that the low-pressure Ar/N_2 (50%/50%) plasma treatment does not affect the mechanical properties of the treated material.

5 Acknowledgements

The research received grants from the Eureka E!4043 project and P2-0213 ARRS research programme.

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