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Priprava bombažne tkanine z nizkotlačno plazmo za boljšo adhezijo nanosrebra

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Low-Pressure Plasma for Pretreatment of Cotton Fabric for Better Adhesion of Nanosilver

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Abstract

In this study, bleached and mercerized cotton fabric was treated with low-pressure plasma; water vapor and CF_4 were used as gases. Some fabrics were treated with plasma and some were not; however, all of the fabrics were blank dyed. Twenty milligrams per liter of nano silver (30 nm and 80 nm in size) was added to the dyebath. A wash fastness test was conducted at 95°C in accordance with the ISO 105-C03 standard. The amount of silver adsorbed onto the fabric was analyzed by the ICP-MS method. The analysis results revealed that the adsorption of nanosilver increased on the surface of the cotton fabric after the plasma treatment while the nanosilver desorbed after ten wash cycles. The amount of adsorbed silver that was 30 nm in size was larger in comparison to the 80 nm-sized silver; nevertheless, after the wash, desorption of the 30 nm-sized silver increased.

Keywords: cotton, low-pressure plasma, water vapour, CF_4, nanosilver particles

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Izvleček

Z nizkotlačno plazmo, pri čemer sta bila kot plina uporabljena vodna para in CF_4, smo obdelali beljeno in mercerizirano bombažno tkanino. S plazmo obdelane in neobdelane tkanine so bile slepo barvane. V barvalno kopel smo dodali 20 mg/l nanosrebra velikosti 30 nm in 80 nm. Opravljen je bil test obstojnosti pri pranju pri 95 °C po standardu ISO 105-C03. Količina srebra, adsorbiranega na tkanine, je bila analizirana z metodo ICP-MS. Rezultati analize kažejo na povečano adsorpcijo nanosrebra na površino bombažne tkanine po obdelavi tkanine s plazmo in na desorpcijo nanosrebra po 10-kratnem pranju. Količina adsorbiranega srebra velikosti 30 nm je večja kot količina srebra velikosti 80 nm, vendar pa se tudi po pranju pokaže povečana desorpcija 30 nm srebra.

Ključne besede: bombaž, nizkotlačna plazma, vodna para, CF_4, nanodelci srebra.

1 Uvod

Plazemska obdelava in njen učinek na tekstilne materiale sta še predmet raziskovanj. Obdelava tekstilij s plazmo prinaša veliko prednosti pred mokrimi kemijskimi obdelavami, saj za obdelavo s plazmo ne potrebujemo vode in kemikalij, s tem pa pozitivno vplivamo na ekonomičnost in ekološki pristop [1]. V primerjavi z mokrimi obdelovalnimi postopki, pri katerih je penetracija globoka, prihaja pri plazemski obdelavi do površinskih reakcij na materialu le v nekaj nanometrih površine [2]. Za obdelovanje tekstilnih materialov se uporablja neravnovesna oz. „hladna“ plazma, ki je del-

1 Introduction

Plasma treatments and their effect on textile materials is still a subject of research. Plasma treating textiles has many advantages over wet-chemical treatments; for instance, plasma-induced treatments do not require water or chemicals, and thus provide economical and ecological benefits. [1] In comparison with wet-treatment processes that have a deep penetration, reactions on the material's surface occur at a depth of only a few nanometers in plasma treatments. [2] To treat textile materials, non-equilibrium or cold plasma is used, where this is partially ionized gas. In plasma, electrons have a higher temperature than ions; therefore, reactions can be performed in cold plasma without inducing substrate degradation. Plasma technology is versatile since it can be used in the treatment of textiles to make the textile more hydrophilic or hydrophobic, to increase its adhesion, or merely to clean or sterilize the textile. [3] The gases that are used in plasma technology can have either an inorganic or organic origin. When using organic gases, e.g., C_2F_6, a product that is similar to a polymer deposits on the surface of the treated material. [4] However, when using inorganic gases, e.g., O_2, the influence of etching and substance removal can be seen on the material's surface. [5] The formation of nanostructure surfaces is important, since the desired functionality of a newly treated surface can be achieved by increasing the surface. [6] Nanostructuring the material surfaces of textile increases the adhesion of metal particles. The use of nano particles has increased in recent years. Due to their extremely small dimensions, nano particles have special physical and chemical properties and biological activity. Thus, nanosilver is used to achieve antimicrobial and anti-inflammatory properties, especially in medicine. [7–11] However, only good conditions on the substrate's surface can provide a qualitative deposition of particles. [12] The substrate's surface can be prepared by using an appropriate plasma system. [13–15] The main reason for forming such structures is the incorporation of nanoparticles into the textile material; in this way, the agglomeration of particles is reduced, their fastness to washing increases,

no ioniziran plin. V plazmi imajo elektroni višjo temperaturo kot ioni in zato lahko v hladni plazmi potekajo reakcije, ne da bi pri tem prišlo do degradacije substrata. Plazemska tehnologija je vsestranska, saj lahko z njo obdelamo tekstilijo, da ta postane na primer bolj hidrofilna, da se ji poveča adhezija, da postane bolj hidrofobna, ali pa jo lahko le čistimo ali steriliziramo [3]. Plini, ki jih uporabljamo pri plazemski tehnologiji, so lahko anorganskega ali organskega izvora. Če uporabimo organski plin, npr. C_2F_6, se na površini obdelovalnega materiala deponira polimeru podoben produkt [4]. Ko pa uporabimo anorganske pline, kot je npr. O_2, se na površini materiala opazi vpliv jedkanja in odstranjevanja snovi [5]. Tvorba in nastanek nanostrukturiranih površin sta pomembna, saj se s povečanjem površine doseže želeno funkcionalnost na novo obdelane površine [6]. Z nanostrukturiranjem površine tekstilnega materiala lahko dosegamo povečanje adhezije kovinskih delcev na material. Nanodelci ekstremno majhnih dimenzij imajo posebne fizikalno-kemijske lastnosti in biološko aktivnost, zaradi česar se je njihova uporaba v zadnjem času povečala. Tako se za doseganje antimikrobnih in protivnetnih lastnosti uporablja nanosrebro, še posebej v medicinske namene [7–11].Vendar pa šele dobri in ugodni pogoji na površini tekstilnega substrata lahko zagotovijo kvaliteto nanosov [12]. Ključna faza je priprava površine substrata, ki jo lahko vzpostavimo z uporabo primerne plazme [13–15]. Glavni razlog za takšno formiranje površin je vključitev nanodelcev na tekstilni material, še posebej zaradi tega, ker pri adheziji nanodelcev na tekstilni material prihaja do njihove aglomeracije, poslabšanja obstojnosti pri pranju ipd. [16]. Iz predhodnih poskusov, ko smo slepo barvali beljeno bombažno tkanino, in to ob dodatku 80 nm srebrovih delcev, smo ugotovili, da se 35 mg/kg teh nanodelcev adsorbira na tkanino [17]. Poskusi funkcionalizacije bombaža z atmosfersko korona plazmo so pokazali povečanje adsorptivne sposobnosti površine bombaža za nanos nanosrebra [18].

Namen našega dela je bil preučiti, kolikšen vpliv ima plazemska obdelava z vodno paro ali s CF_4 na stopnjo adhezije nanosrebra na beljeno in mercerizirano bombažno tkanino. Med seboj so bili primerjani neobdelani in plazemsko obdelani vzorci. S testom obstojnosti pri pranju po standardu ISO 105-C03 smo preverjali tudi stopnjo desorpcije nanodelcev srebra po pranju.

2 Eksperimentalni del

Za raziskavo smo uporabili beljeno in mercerizirano bombažno tkanino v vezavi platno proizvajalca Tekstina, Ajdovščina. 30 cm × 30 cm velike vzorce tkanine smo obdelali v nizkotlačnem plazemskem reaktorju, ki je shematično prikazan na sliki 1. Moč radiofrekvenčnega (RF) generatorja je bila 5 kW in frekvenca 27,12 MHz. Plina, ki smo ju uporabili za funkcionalizacijo vzorcev, sta bila vodna para in CF_4. V sredino razelektritvene posode plazemskega re-

and so on. [16] It has been established in previous research that the addition of silver particles (80 nm in size) when blank dyeing bleached cotton fabric causes 35 mg/kg of the nano particles to be adsorbed onto the fabric. [17] The attempts at functionalizing cotton with atmospheric corona plasma showed an increase in the adsorptive capabilities of the cotton surface for nanosilver coatings. [18]

The purpose of our research was to study the influence of water vapor or CF_4 plasma treatments on the adhesion level of nanosilver to bleached and mercerized cotton fabric. Untreated and plasma treated samples of fabric were compared. The ISO-105-C03 wash fastness test was used to establish the desorption level of nanosilver particles silver after washing.

2 Experimental

Bleached and mercerized cotton fabric in plane weave, which was made by Tekstina, Ajdovscina, was used in the research. Samples with a size of 30 cm × 30 cm were treated in a low-pressure plasma reactor. The diagram of the reactor is presented in Figure 1. An RF generator with a nominal power of 5 kW and a frequency of 27.12 MHz was used. As working gases, water vapor and CF_4 were used. A sample of cotton fabric was put on a glass holder, which was mounted in the centre of the discharge chamber. After closing the chamber, 0.4-mbar pressure was achieved with a two-stage rotary pump and the functionalization of the cotton samples was started. All of the samples were treated with the plasma for 10 seconds and subsequently transported to a silica-gel desiccator.

Untreated and plasma-treated samples were blank exhaust dyed in a Launder-o-meter according to the dye producer's instructions. [19] The dyebath included 20 mg/l of 30 nm or 80 nm silver particles (Ames Goldsmith Corp.). After dyeing and after the treatments, the samples were also treated with 10 g/l of Tubicoat PU 80 (Bezema) and dried at 130°C. Figure 2 presents the dyeing process and the after treatments.

After dyeing and the after treatments, the wash fastness of the samples was tested. The samples were washed in a Launder-o-meter. One wash

aktorja smo postavili naš vzorec. Po zaprtju posode smo s pomočjo dvostopenjske rotacijske črpalke dosegli želeni tlak 0,4 mbar. Ko smo vključili RF-generator, se je plin v razelektritveni posodi spremenil v plazmo in funcionalizacija bombažnega vzorca se je začela. Vzorce smo obdelovali 10 sekund in jih pozneje prenesli v eksikator, ki je vseboval silikagel.

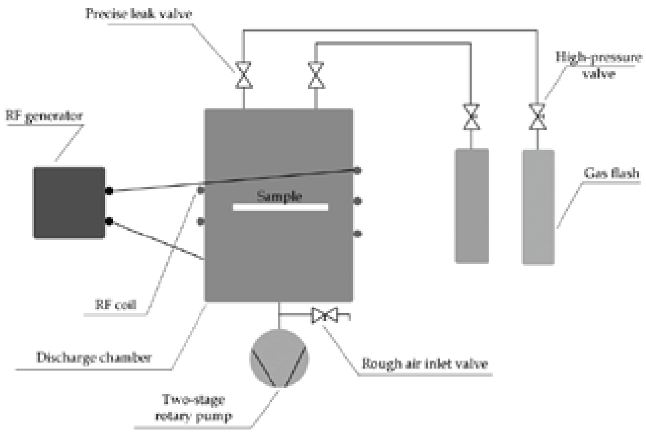

Figure 1: Diagram of plasma reactor

Neobdelane in plazemsko obdelane vzorce smo slepo barvali po izčrpalnem postopku v launder-ometru po navodilih proizvajalca barvil Ciba [19]. V barvalno kopel smo dodali 20 mg/l srebrnih delcev (Ames Goldsmith Corp.) velikosti 30 nm ali 80 nm. Po barvanju in poobdelavah smo vzorce obdelali še z 10 g/l sredstva Tubicoat PU 80 (Bezema) ter jih sušili pri 130 °C. Na sliki 2 je prikazan diagram barvanja z izčrpavanjem s poobdelavami, ki potekajo v sklopu izčrpalnega barvalnega postopka.

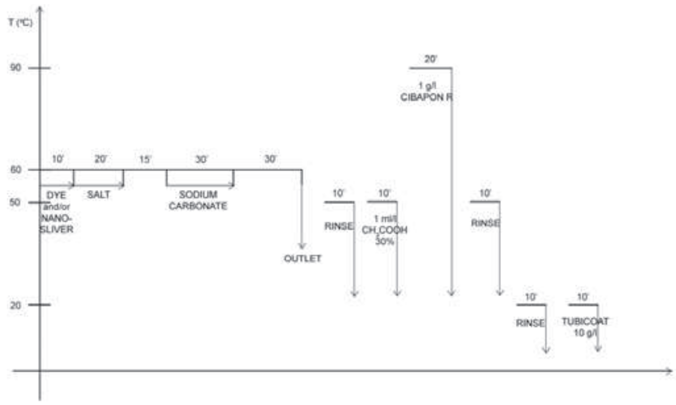

Figure 2: Dyeing process for Cibacron S reactive dyes and aftertreatments

Po končanem barvanju in poobdelavah smo vzorce testirali na pralno obstojnost. Vzorce smo prali v launder-ometru, v katerem eno pranje po standardu ustreza petim pranjem perila v gospodinjskem pralnem stroju. Vzorce smo prali 10-krat zaporedoma *in a Launder-O-Meter provides an accelerated washing treatment that corresponds to five home washings. The samples were washed repetitively 10 times at 95°C in a solution of 5 g/l of SDC standard detergent and 2 g/l of Na_2CO_3 (where 10 globules were added). The duration of the washing cycles was 30 min. After every wash cycle, the samples were rinsed twice in distilled water and then for 10 minutes under a tap water, which was followed by squeezing and air drying. The quantity of silver nanoparticles on the cotton samples was measured with an ICP-MS analysis. [20]*

3 Results and discussion

The results are shown in Table 1 for the ICP-MS analysis of the silver nanoparticles on the untreated and plasma treated cotton samples.

Table 1 shows that the plasma treatment has a great impact on the adsorption of nanosilver onto cotton fabrics. Treating the cotton with water vapour and CF_4 plasma changes its surface, where adsorption of the nanosilver increases in comparison to the untreated cotton; in one case (when 80 nm silver was used on the CF_4 plasma treated cotton), it increased by 100% (from 13 ppm to 26 ppm). The plasma-treated cotton surface, which shows morphological changes [21], is more favorable for the physical adsorption of nanoparticles. Regarding their volume, nanoparticles have a large surface since they have a great ability to travel. By decreasing the size of a particle, its surface distribution rate increases. [22] Silver nanoparticles with a size of 30 nm adsorb onto the cotton surface at a higher concentration than particles with a size of 80 nm. After ten wash cycles (i.e.,

pri 95 °C, in to v raztopini 5g/l ECE phosphate reference detergent B (SDC) in 2 g/l Na_2CO_3 ter ob dodatku 10 kroglic. Po vsakem pranju smo vzorce 2-krat sprali v mrzli deionizirani vodi in 10 minut pod tekočo vodo, jih oželi in sušili na zraku. Količina adsorbiranega srebra na tkaninah je bila določena z ICP-MS [20].

3 Rezultati z razpravo

Tabela 1 prikazuje rezultate analize ICP-MS neobdelanih in plazemsko obdelanih s srebrom barvanih bombažnih tkanin.

Table 1: The quantity (ppm) of silver on cotton (B) samples

Ag \ B	untreated	plasma water vapour	plasma water vapour +10-times washed	plasma CF_4	plasma CF_4 +10-times washed
30 nm	32	50	26	54	36
80 nm	13	17	10	26	22

Iz rezultatov v tabeli 1 je razvidno, da plazemska obdelava površin bombažnih tkanin močno spremeni adsorptivnost le-teh do nano srebra. Obdelava s plazmo vodne pare in s plazmo CF_4 spremeni površino bombaža, adsorbcija 80 nm srebra se glede na neobdelani bombaž v primeru uporabe plazme CF_4 poveča celo za 100 % (s 13 ppm na 26 ppm). Plazemsko spremenjena površina bombaža, ki kaže morfološke spremembe [21], je v tem primeru ugodnejša za potek fizikalne adsorpcije nanodelcev. Nanodelci imajo glede na volumen veliko površino, zaradi česar imajo veliko potovalno sposobnost. Z zmanjševanjem velikosti delca se poveča njegova površinska porazdelitvena stopnja [22]. Srebrni nanodelci velikosti 30 nm se adsorbirajo na površino bombaža v večji koncentraciji kot 80 nm delci. Po 10-kratnem pranju (50-kratnem gospodinjskem pranju) pri 95 °C se koncentracija srebra na bombažnih vzorcih zniža. Koncentracija nanosrebra velikosti 30 nm se po 10-kratnem pranju s plazmo vodne pare spremenjene tkanine zniža za 48 % (s 50 ppm na 26 ppm), medtem ko se s plazmo CF_4 obdelanemu bombažu po 10-kratnem pranju vsebnost srebra zmanjša za 34 % (s 54 ppm na 36 ppm). Padec koncentracije srebra na bombažu po 10-kratnem pranju opažamo tudi v primeru uporabe 80 nm srebra. S plazmo vodne pare spremenjeni bombaž izgubi 41 % (s 17 ppm na 10 ppm), s plazmo CF_4 spremenjeni bombaž pa 15 % (s 26 ppm na 22 ppm) nanosrebra. Površina beljenega in merceriziranega bombaža je po 10-sekundni obdelavi v plazmi CF_4 dovzetnejša za adsorpcijo nanosrebra kot površina, ki je bila funkcionalizirana s plazmo vodne pare. Analiza spremembe morfologije površine beljenega merceriziranega bombaža po obdelavi s plazmo

50 domestic wash cycles) at 95°C, the concentration of silver on the cotton samples decreases. The concentration of 30 nm nanosilver decreases by 48% (from 50 ppm to 26 ppm) after ten wash cycles when using water vapor plasma, while the nanosilver content decreases by 34% (from 54 ppm to 36 ppm) after ten wash cycles for cotton that was treated with CF_4 plasma. The drop in the concentration of silver on the cotton after ten wash cycles is also observed with the 80 nm silver. Cotton that is treated with water vapor plasma loses 41% (from 17 ppm to 10 ppm); however, cotton treated with CF_4 plasma loses 15% (from 26 ppm to 22 ppm) of the nanosilver. After a 10-second treatment with CF_4 plasma, the surface of the bleached and mercerized cotton is more liable to the adsorption of nanosilver than the surface that was functionalized with the water-vapour plasma. After the CF_4 plasma treatment, an analysis of the changes in the surface morphology of the bleached and mercerized cotton was conducted with a scanning electron microscope. [23] The surface changes due to the plasma show incredible homogeneity and thus, it can be assumed that it will play an important role in the future use of nanotechnology.

4 Conclusions

Cotton surfaces that are treated with water vapour or CF_4 plasma are more susceptible to adhesion of silver nanoparticles due to morphological changes of the cotton's surface. The reduction in the silver concentration on the cotton after ten wash cycles is also noticeable; it occurs with both types of plasma and both sizes of nanosilver. However, desorption of the nanosilver is higher when using water-vapour plasma. The results show that the physical forces between the silver nanoparticles and the CF_4 plasma-treated cotton surfaces are stronger than the physical or weak chemical forces between the nanoparticulate silver and the water-vapour plasma-treated cotton surfaces. It is known that the CF_4 plasma-treated surface acquires a hydrophobic character. [24] When using metal nanoparticles, hydrophobic interactions are preferential. Under humid conditions, and especially in the dyeing bath, oxidation of the silver nanoparti-

CF_4 je bila opravljena s SEM (vrstičnim elektronskim mikroskopom) [23]. Plazemsko spremenjena površina kaže neverjetno homogenost in predvidevamo, da bo njena uporaba v nanotehnologiji odigrala pomembno vlogo.

4 Zaključki

Površine bombaža, ki je bil obdelan s plazmo vodne pare ali CF_4, zaradi spremenjene morfologije sprejmejo večje koncentracije srebrnih nanodelcev. Zmanjšanje koncentracije srebra na bombažu po 10-kratnem pranju je prav tako opazno v primerih uporabe obeh vrst plazem na bombažnih tekstilijah in pri obeh velikostih nanosrebra, vendar je desorpcija nanosrebra večja s površine materiala, ki je bil obdelan s plazmo vodne pare. Rezultati jasno kažejo, da so fizikalne sile med nanodelci srebra in med površino bombaža, ki je bil nanostrukturiran s plazmo CF_4, močnejše od fizikalnih oz. tudi šibkih kemijskih vezi med nanodelci srebra in z vodno paro spremenjeno površino bombaža. Znano je, da se s plazmo CF_4 obdelana površina hidrofobira [24]. Pri kovinskih nanodelcih srebra igrajo veliko vlogo hidrofobne interakcije. Ker pa so delci nanovelikosti, se hitro, že v prisotnosti vlage iz ozračja, še bolj pa v barvalni kopeli, obdajo z oksidno plastjo, iz katere se počasi sproščajo ioni Ag^+. Za privlak kationov pa mora površina tekstilije vsebovati anionske funkcionalne skupine. Za detajlno analizo sprememb površine tekstilij je treba izvesti tudi XPS-analizo površin. Pri adheziji nanosrebra na površine tekstilij se prepletajo tako hidrofobne kot hidrofilne interakcije. Primerjava koncentracij različnih velikosti nanosrebra na neobdelanih in s plazmami obdelanih bombažnih tekstilijah jasno pokaže večjo koncentracijo 30 nm srebra glede na 80 nm srebro.

5 Zahvala

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cles occurs, wrapping them with a silver oxide layer from which Ag^+ ions are slowly released. The surface of the textile material that contains anionic functional groups becomes attractive to the cations. A detailed analysis of the changes to the textile's surface is necessary and an XPS analysis is also recommended. Interweaving of the hydrophobic and hydrophilic interactions at the adhesion of the nanosilver particles to textile surfaces is observed. A comparison of the different sizes of nanosilver particles clearly shows that the adsorption as well as the desorption of 30 nm silver particles are more intensive than the adsorption and the desorption of 80 nm silver nanoparticles.

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