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Modification of Unfinished Textiles by Cu Deposition Developed by Cold Cathode Ion Source for Antimicrobial Application

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THE MAIN goal of the present work is to investigate the influence of depositing copper film on different unfinished textiles, namely; polyester, cotton and polyester/cotton, on their antimicrobial activity. Ion beam of argon gas is used for sputtering of copper target to deposit a thin film of copper on the textiles surface. The samples were characterized by SEM, FTIR, and XRD. The results show that copper nanoparticles are smoothly deposited onto the surface with no change on the chemical or the physical structure of the textiles. Ac conductivity was measured in the frequency range of 50Hz-5MHz and showed a slight increase with Cu nanoparticles deposition. The antimicrobial activity test showed an enhancement of the textiles ability to inhibit gram-positive *S. aureus* and gram-negative *E. coli* after copper deposition and the inhibition is higher for *E. coli*. Moreover, the results show the highest reduction ability of a modified cotton sample for both types of bacteria.

Keywords: Antibacterial activity, Cold cathode ion source, Deposition**,** Sputtering, Textiles.

Introduction:

Textiles are premium surface for microbial generation and bacterial growth under suitable conditions of temperature and humidity. Polluted textiles in hospitals are serious sources of fungi, bacteria and viruses. These polluted textiles may cause transfer of microorganisms to the patients and clinician personnel. *Escherichia coli*, *Staphylococcus aureus*, *Mycobacterium tuberculosis*, *Legionella* and *Pseudomonas* are the most prevalent microorganisms accountable for infections (Breathnach, 2013; Lax & Gilbert, 2015; Khan et al., 2015; Trubiano & Padiglione, 2015; Shukla et al., 2016). Bacterial function limits the applications of textiles. Several types of antimicrobial agents have been integrated into the textiles, and it was found that the nanoparticles on the textiles can control the growth of microorganisms (Montazer et al., 2016; Xu et al., 2018).

The metallic thin films of nanoparticles have been widely studied by many researchers (Akhavan & Ghaderi, 2009; de Faria et al., 2013; Fu et al., 2014). They are used as antimicrobial coatings (Wang et al., 2006; Juan et al., 2010; da Silva et al., 2014), catalysts (Nilius et al., 2011; Angelomé & Liz-Marzán, 2014), and are used in optical (Brandon et al., 2014) and photovoltaic devices (Mubeen et al., 2011; Wu et al., 2011). They have been produced by several methods such as sol-gel (Yliniemi et al., 2007, 2008), vapor-deposition (Lu et al., 2007; Bernardo-Gavito et al., 2013), deposition of metal nanoparticles from colloidal dispersions (Bhatta et al., 2011; Karakouz et al., 2011), electrochemical method, and ion beam sputtering technique (Mech et al., 2011; Attri et al., 2013; Shehata et al., 2017), etc. Due to its advantages over other methods, the ion beam sputtering is used for thin layers depositing. The main advantage of this method is the independent control of many process parameters

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such as vacuum, ion beam energy and ion fluency (Shehata et al., 2017). The ion sputtering is suitable for integrated circuits manufacturing, microsystems or semiconductors interconnects (Merchant et al., 2001), and multilayer thin films (Kucharska & Kulej, 2010).

The aim of the current work is to investigate surface modification of the textiles by coating with Cu nano-particles (Cu-NPs) through cold cathode ion source so as to pick up antibacterial properties of the investigated textiles and to study the antimicrobial effects of the coated textiles.

Experimental:

Cu nano layer is deposited onto standard size (15 mm by 25 mm) textile (cotton, polyester, and polyester/cotton) samples using penning ion source sputtering technique as illustrated in Fig. 1 (Mahmoud et al., 2021). The conditions used for sputtering deposition of copper nanoparticles in this experiment are $1x10^{-3}$ mbar for the pressure. The current density is 200 µA/cm^2. The discharge current equals 1.3 mA and the sputter yield is 13 atoms/ion. The ion energy equals 4 keV and the sputtering time is 40 min with ion fluence equals $2x10^{17}$ ion.cm^{-2}. The copper target is placed by angle 60^0 with the incident beam trajectory.

Characterization techniques:

The Surface morphology of the samples has been studied using Scanning Electron Microscope (JEOL JSM 5600 LV; Tokyo, Japan), and the crystal structure of the samples has been characterized using a fully computerized X-ray diffractometer, (Shimadzu XRD-6000 with Cu radiation λ= 1.54056 Å. FTIR analysis was achieved utilizing BRUKER optik GmbH – VERTEX 70, and the spectra were acquired over the range 400 – 4000 cm^{-1} with a resolution of 0.5 cm^{-1}.

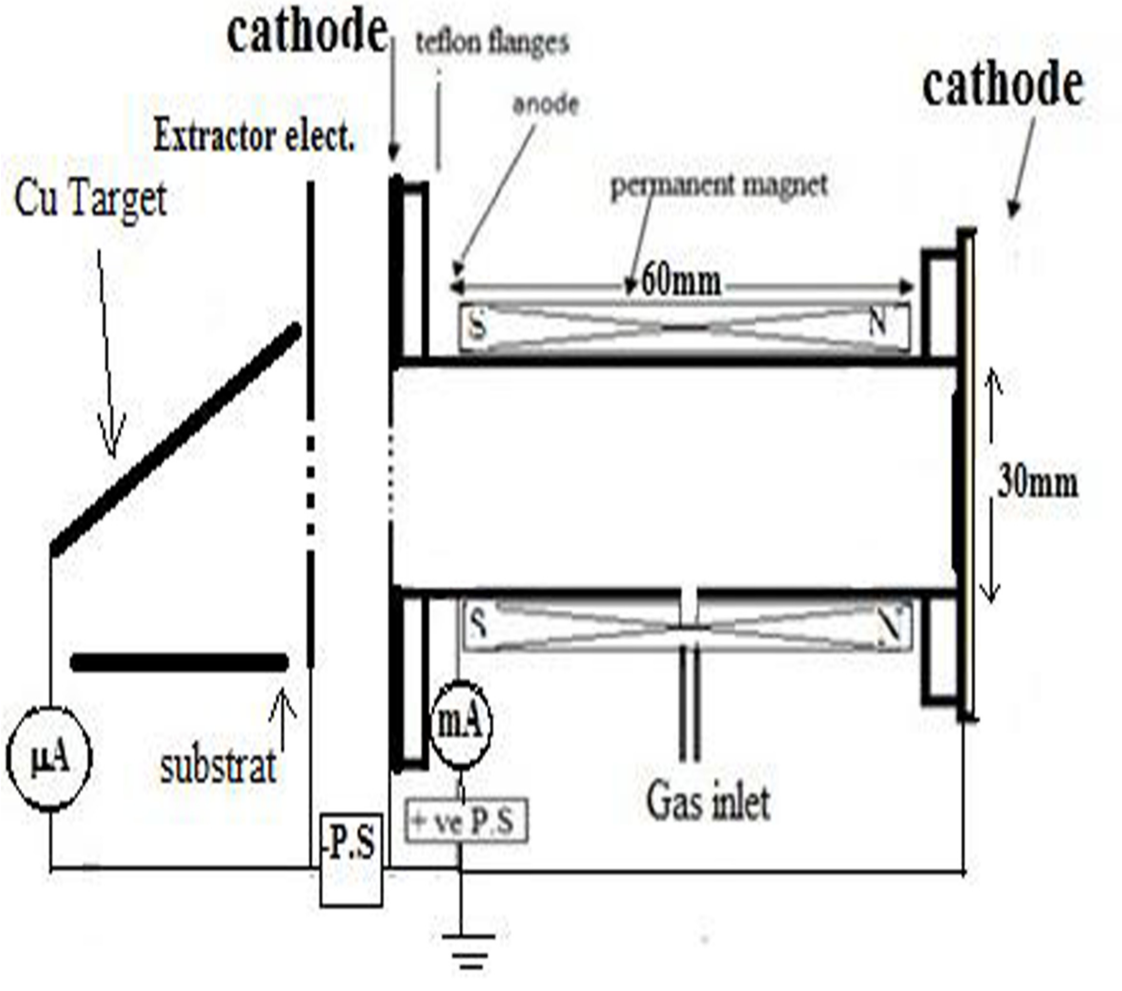

Fig. 1. The cold cathode ion source with electrical circuit

Antibacterial activity testing

Antibacterial activities of polyester, cotton, and polyester/cotton textiles (blank and Cu-coated) were tested against gram-positive *Staphylococcus aureus* and gram-negative bacterium *Escherichia coli*. Briefly, a culture medium (trypton soya broth, TSB) was prepared and distributed (20 mL) in Erlenmeyer flasks (50 mL) before sterilization in autoclave. Loop full of each microorganism was transferred to 20 mL of TSB and the inoculated flasks were incubated in a shaking incubator at 150 rpm at 37°C. After 24 h, 1 mL of the prepared inoculums was added to a flask containing 20 mL of TSB. The control and modified samples were cut into 1 cm x 1 cm under sterile conditions, and the specimen was added to the inoculated flasks before being incubated at 37°C in a shaking incubator at 150 rpm. After an appropriate contact time interval (0, 2 h and 4 h) the bacterial growth or the colony forming count per mL (CFU/mL) was detected by pore plate count method described in bacteriological analytical manual (Food and Drug administration, 2001). Briefly, 1 mL of the bacterial growth was added to a test tube containing 9 mL of sterile saline solution (10^{-1} dilution) and the bacterial suspension was serially diluted up to 10^{-6}. One mL of each dilution was transferred with sterile pipette into sterile Petri dish. About 15 mL of sterile trypton soya agar (TSA, cooled to 45°C) were added to each plate and the medium was carefully mixed with the dilution by gently hand rotating each dish on flat level surface. The solidified plates were incubated for 24 h at 37°C and observed colonies were recorded. The reduction of the bacterial growth was determined using the following equation:

Bacterial reduction percentage= $N_1 - N_2 / N_1 \times 100$ ....(1)

where N_1 is the number of bacterial colonies at the contact time of 0 h and N_2 is the number of bacterial colonies at the contact time of either 2 h or 4 h.

Results and Discussion

Surface morphology

Scanning electron microscopy is performed to investigate the surface morphology of the samples. Figure 2 shows high and low magnifications SEM images of non-coated and coated textile samples. The images declare insignificant changes after Cu modification, which means that Cu mainly coated the textile surface, but did not occupy the voids between the fibers. The small white particles on the modified textile surface which were illustrated in the high magnification images, indicate that Cu-NPs were deposited on the investigated textile surfaces. The surface of the films shows a good uniformity and cracks free, indicating a good adhesion and regularity among the thin film layer on textile substrates.

XRD analysis:

Figure 3 (a, b) shows the XRD pattern of three types of textiles before and after Cu-NPs deposition. The polyester diffraction peaks (Muthukumar & Thilagavathi, 2012) appeared at $2\theta = 18.1^\circ$, 23°, and 26.19°, whereas that of cotton appeared at $2\theta = 15.12^\circ$, 16.8°, 22.92°, and 34.51° (Abd-Elhamid et al., 2019; Zhao et al., 2019; Sun et al., 2021). The polyester/cotton sample shows its diffraction peaks at $2\theta = 16.72^\circ$, 22.89°, and 25.64°. A slight shift of these positions is observed upon coating with Cu, which is an indication that the Cu-NPs were incorporated in the textile (Abou-Elela et al., 2014). However, Very small peaks appear at $2\theta = 43.24^\circ$, 42.79°, and 42.9° in the XRD pattern of Cu-coated polyester sample, Cu-coated cotton textile sample, and Cu-coated polyester/cotton textile sample, respectively. These peaks correspond to the (111) plane of the fcc structure of Cu nanoparticles (Betancourt-Galindo et al., 2014; Suresh et al., 2016).

FTIR spectroscopic analysis:

Figure 4 (a, b) shows the FTIR spectra of the pure and Cu-coated textile samples. For the polyester sample, the band at 3430.14 cm^{-1} is for O–H stretching and the band at 2931.95 cm^{-1} is assigned to the C–H stretching vibration whereas the characteristic ester carbonyl group (C=O) appears at 1711.73 cm^{-1}. The bands from 1339.23 cm^{-1} to 1013.82 cm^{-1} are corresponding to the stretching bands of C-O, and the band at 862.80 cm^{-1} is due to the stretching vibrations of aromatic ring (Hoghoghifard et al., 2016; Koto & Soegijono, 2019; Mousa & Khairy, 2020; Taha et al., 2020). For the cotton sample, the broad band at 3290.51 cm^{-1} is attributed to O–H stretching vibration and the band at 2898.62 cm^{-1} belongs to C–H stretching vibration. The band at 1625.11 cm^{-1} corresponds to O–H bending vibration. The bands at 1427.18 cm^{-1} and 1317.99 cm^{-1} are due to C–H wagging and the bands at 1156.17 cm^{-1} and

1103.53 cm^{-1} are assigned to C–O–C asymmetric stretching, whereas the band at 1021.89 cm^{-1} is attributed to C–O stretching (Abd-Elhamid et al., 2019; Zhao et al., 2019; Sun et al., 2021). For the polyester/cotton samples, the O–H stretching vibration appears at 3332.37 cm^{-1} whereas the C–H stretching vibration is at 2901.09 cm^{-1}, the characteristic band of the ester group appears at 1711.47 cm^{-1}, and the band at 1017.90 cm^{-1} is attributed to C–O stretching. A slight shift in these characteristic bands is only observed upon deposition of Cu-NPs on the textile samples.

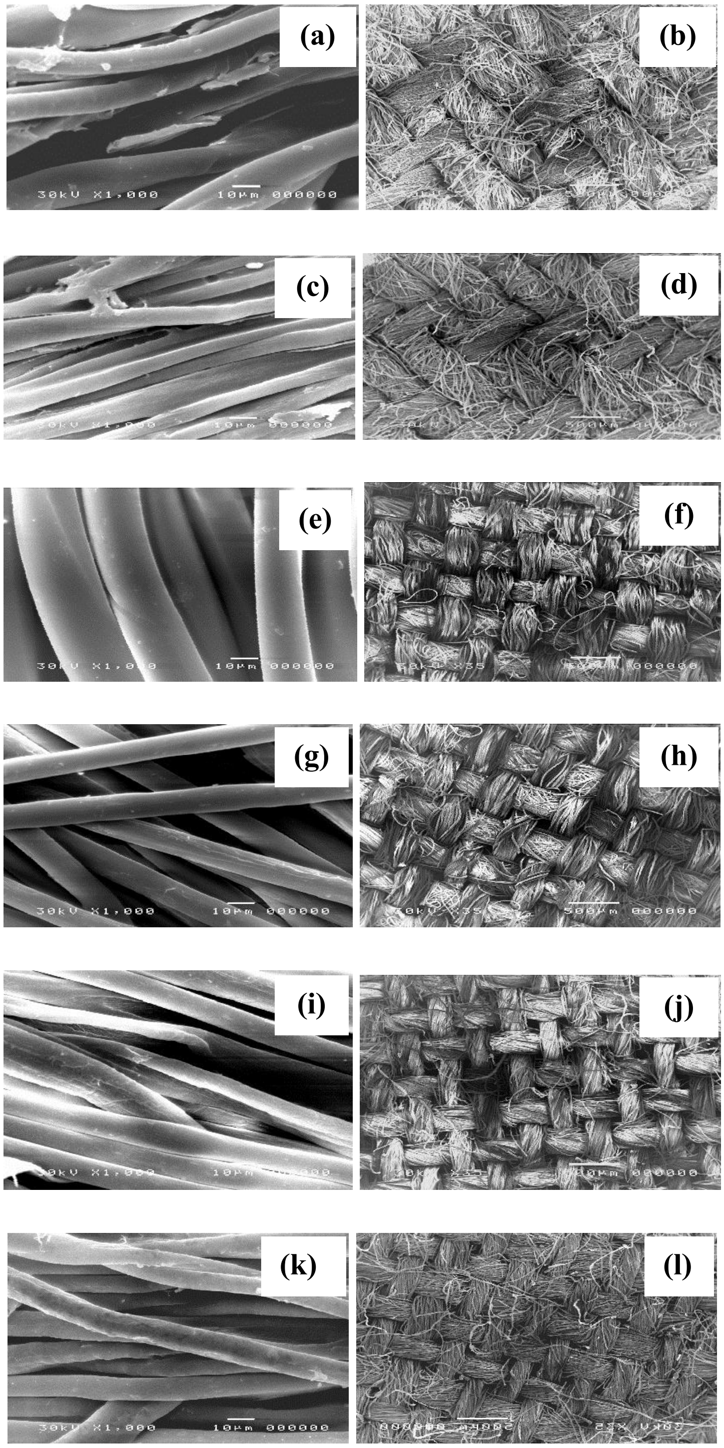

Fig. 2. SEM micrographs of (a, b) cotton, (c, d) modified cotton, (e, f) polyester, (g, h) modified polyester, (i, j) cotton/polyester and (k, l) modified cotton/polyester

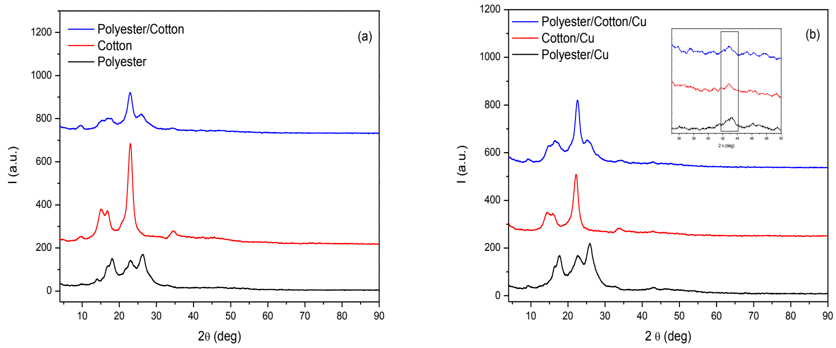

Fig. 3. The XRD patterns of: (a) pure and (b) Cu-coated polyester, cotton, and polyester/cotton textile samples

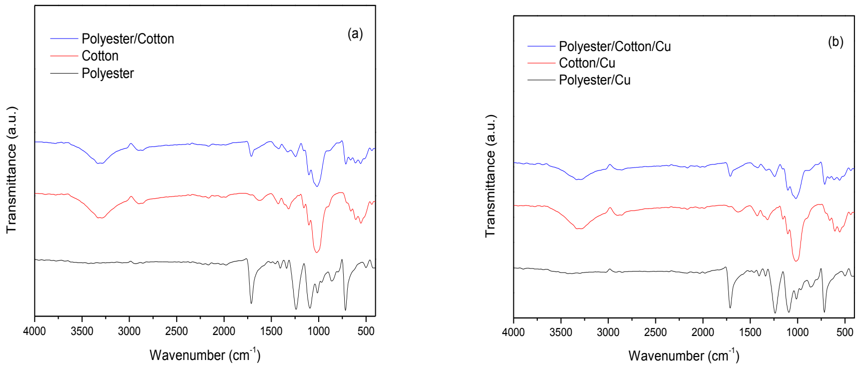

Fig. 4. The FTIR spectra of: (a) pure and (b) Cu-coated polyester, cotton, and polyester/cotton textile samples

Ac conductivity

Ac conductivity has been obtained from the well-known relation (Yadav et al., 2017):

$$\sigma_{Ac}(\omega) = \omega\, \varepsilon_o\, \varepsilon' \tan\delta \qquad (2)$$

where: ω is the angular frequency (ω= 2πf), ε_o= 8.85 × 10^{-12} F/m, ε′ is the real part of dielectric constant, and tan δ is the energy loss of the field into the sample $(\tan\delta = \varepsilon'' / \varepsilon')$.

Table 1 gives $\ln_{Ac}$ values of the non-coated and coated unfinished fibers at certain frequencies in the range 50 Hz up to 5 MHz. From the Table one can notice that Ac conductivity of the pure textiles slightly increased with Cu modification especially in the low frequency region.

TABLE 1. ln σ_{Ac} values of pure and modified investigated textiles at different frequencies, where σ_{Ac} is in $\Omega^{-1}cm^{-1}$ unit

Frequency, Hz	ln σ_{Ac}					
	Cotton		Polyester		Cotton-Polyester	
	Pure	Modified	Pure	Modified	Pure	Modified
50	-15.67	-14.56	-15.08	-14.08	-14.79	-14.64
200	-17.3	-14.71	-15	-14.83	-15.58	-14.27
400	-15.276	-15.22	-15.74	-15.7	-17.8069	-15.99
700	-17.69	-15.53	-15.71	-15.44	-16.16	-16
1000	-16.54	-15.98	-15.05	-15	-18.1361	-15.95
4000	-13.51	-13.15	-13.23	-13.12	-13.45	-13.39
20000	-14.16	-13.02	-12.9	-12.32	-13.39	-13.28
100000	-12.69	-12.58	-11.61	-11.53	-12.59	-12.55
1000000	-9.98	-9.79	-9.16	-9.02	-10.11	-9.92
2000000	-7.43	-7.38	-6.8	-6.75	-7.69	-7.6
3000000	-6.62	-6.57	-6.003	-5.97	-6.87	-6.8
4000000	-5.91	-5.86	-5.3	-5.26	-6.17	-6.11
5000000	-5.55	-5.45	-4.93	-4.89	-5.8	-5.74

Antibacterial activity of modified textiles

The antibacterial activities of the coated polyester, cotton, and polyester/cotton samples were tested against gram-positive *S. aureus* and gram-negative *E. coli* to preliminarily evaluate its use as an antibacterial in different fields. As shown in Table 2, coated polyester and polyester/cotton samples provided (moderate) antibacterial activities to the gram-negative *E. coli* with reduction values 53.12% and 46.88%, respectively after 2 h contact. In addition, the same samples showed lower inhibition of gram-positive *S. aureus* with reduction values reached 35.29% and 29.41%, respectively after 2h contact. Highly and moderate reduction (74.17% and 52.94%) of the coated cotton sample were recorded after 2 h contact for *E. coli* and *S. aureus*, respectively. The reduction count of *S. aureus* by the coated polyester, cotton, and polyester/cotton samples were increased to 73.52%, 87.50% and 64.71%, respectively after 4 h. The highly significant inhibition of *E. coli* was recorded by all coated samples (polyester, cotton, and polyester/cotton) after 4 h with reduction values reached 82.50%, 94.88% and 77.50%, respectively. When comparing gram-positive *S. aureus* and gram-negative *E. coli*, it shows that the antibacterial activity of all samples against *S. aureus* was lower than that against *E. coli*. Furthermore, the coated cotton sample has stronger effect on both bacteria isolates with very highly significant effect after 4 h of contact.

Although the Cu-NPs gained much attention of many research groups espicially because of their strong antimicrobial activity, the mechanism of their action has not been well understood yet (Markovic et al., 2018). It was believed that the direct interaction between the active ingredient compounds (Cu-NPs) in the coated samples and bacterial membrane surface is responsible for the antimicrobial activity of the modified samples towards both types of bacteria, which results in leakage of cell contents and consequent cell death (Stoimenov et al., 2002; Zhang et al., 2007; Wahab et al., 2010). Two possible interactions between the samples' compounds and bacterial cells have been proposed as a result of the different nature of the gram-positive and gram-negative cell wall. Gram-positive bacteria have only one cytoplasmic membrane with a thick cell wall consisting of multilayers of peptidoglycan while gram-negative bacteria possess more complex cell wall structures with a peptidoglycan layer between the outer membrane and the cytoplasmic membrane (Wahab et al., 2010). According to this difference in the cell wall of gram-positive and gram-negative, the variation effect of both samples against the two test bacterial types was observed.

TABLE 2. Antibacterial activities of blank and modified textile samples against *E. coli* and *S. aureus*

Incubation time (h)	Samples			
	Blank (control)	Polyester (modified)	Cotton (modified)	Polyester/ cotton (modified)
E. coli (cfu/mL)				
0	1.6×10^6	1.6×10^6	1.6×10^6	1.6×10^6
2	1.5×10^6	7.5×10^5	4.8×10^5	8.5×10^5
4	1.4×10^6	2.8×10^5	8.2×10^4	3.6×10^5
Reduction (%)	6.25 (12.50)	53.12 (82.50)	74.17 (94.88)	46.88 (77.50)
S. aureus (cfu/mL)				
0	6.8×10^6	6.8×10^6	6.8×10^6	6.8×10^6
2	6.4×10^6	4.4×10^6	3.2×10^6	4.8×10^6
4	6.2×10^6	2.8×10^6	8.5×10^5	2.4×10^6
Reduction (%)	5.88 (8.82)	35.29 (73.52)	52.94 (87.50)	29.41 (64.71)

Data between brackets for reduction % after 4 h

Conclusion

Ion sputtering method is utilized to deposit thin layer of copper nanoparticles (Cu-NPs) onto polyester, cotton, and polyester/cotton textiles. The structure study showed that the structure of the investigated unfinished textile was not affected with Cu deposition. Ac conductivity of the studied textiles slightly increased with Cu deposition. This means that we need to increase the thickness of deposition layer. The antimicrobial activity of the cu-coated textiles against *E. coli* and *S. aureus* was measured using the percentage reduction test. A high inhibition of these organisms was observed in the presence of these coated samples. The investigated textiles coated with nano copper showed high antimicrobial efficiency values of 82.50% and 73.52% (modified polyester), 94.88% and 87.50% (modified cotton), and 77.50% and 64.71% (modified polyester/cotton) for *E. coli* and *S. aureus*, respectively after 4 h, and that modified cotton sample showed the highest antimicrobial efficiency. The results show that the investigated cu-coated antimicrobial textiles could be applicable in the biomedical field.

Disclosure of interest: The authors report no conflicts of interests.

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