



Functionalization of cellulose-containing fabrics by plasma and subsequent metal salt treatments

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ARTICLE INFO

Article history:

Received 18 April 2012

Received in revised form 21 May 2012

Accepted 10 June 2012

Available online 19 June 2012

Keywords:

Plasma treatment

Metal salts

Cellulose containing fabrics

UV-protection

Antibacterial functionality

Surface morphology

ABSTRACT

In order to upgrade the UV-protection and antibacterial functional properties of cotton/polyester (80/20), cotton/linen (50/50) and linen/viscose–polyester (50/50) fabric blends, they were treated with different plasma gases (oxygen, air, and argon) followed by subsequent treatment with certain metal salts namely Zn-acetate, Cu-acetate, Al-chloride, and Zr-oxychloride. The obtained results show that the type of plasma gas, the kind of metal salt as well as the nature of the treated substrate play an important role in the extent of enhancing the demanded functional properties. Oxygen plasma treatment followed by Cu-acetate or Zn-acetate treatment gives the best UV-protection or antibacterial activity respectively, keeping other parameters constant. The surface morphology of some untreated and plasma-treated samples was also analyzed by SEM.

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1. Introduction

Blending of textile fibers such as flax/cotton, cotton/polyester and flax/polyester/viscose is carried out to enhance the performance and quality properties, to achieve economic advantages, to develop newer textile properties, to attain esthetic color effects, and/or to improve the durable and handle value (Ibrahim, 2011).

It is well recognized that conventional chemical processes used for modification of textile substrates are inherently very energy and material consuming, costly, not eco-friendly, and may adversely affect the performance properties of the modified substrates (Pandiyaraj & Selvarajan, 2008). As an alternative to conventional chemical processes, plasma based processes are frequently used for desirable modification and surface engineering processes which could positively affect both product and environment quality. Plasma-based treatments have been successfully used to enhance or replace wet-finishing processes for incorporating functional properties such as hydrophilicity, hydrophobicity, dyeability, water and oil repellent, antimicrobial, and anti UV-finish processes (Ceria & Hauser, 2010; El-Zawahry, Ibrahim, & Eid, 2006; Gorenssek et al., 2010; Hossain, Hermann, & Hegemann, 2006; Ibrahim, Eid, Hashem, Refai, & El-Hossamy, 2010; Karahan &

Ozdogan, 2008; Kostic et al., 2009; Yaman, Ozdogan, Seventekin, & Ayhan, 2009; Zhang et al., 2003).

Our previous studies demonstrated the positive role of plasma treatment on enhancing the performance and functional properties of linen-containing fabrics (Ibrahim, Eid, et al., 2010; Ibrahim, El-Hossamy, Hashem, Refai, & Eid, 2008; Ibrahim, Hashem, et al., 2010). The aim of this paper is to impart anti-UV and antibacterial functionalities to cellulose-containing fabrics via plasma treatment followed by subsequent treatment with certain metal salts.

2. Experimental

2.1. Materials

The cellulose-containing fabrics used in the present study were plain weave: cotton/linen (50/50, 320 g/m²), and cotton/polyester (80/20, 245 g/m²) and linen/polyester–viscose (50/50, 250 g/m²). Before the plasma-treatment, the mill-scoured and bleached blended fabrics were washed at 90 °C for 30 min, thoroughly rinsed and then dried before treatment. Zirconium oxychloride octahydrate, copper acetate, zinc-acetate, and aluminum chloride were of reagent grade. Hostapal® CV-ET (a non-ionic wetting agent based on alkylaryl polyglycol) was kindly supplied by Clariant. C.I. Basic Blue 9 (Merk) was used as supplied.

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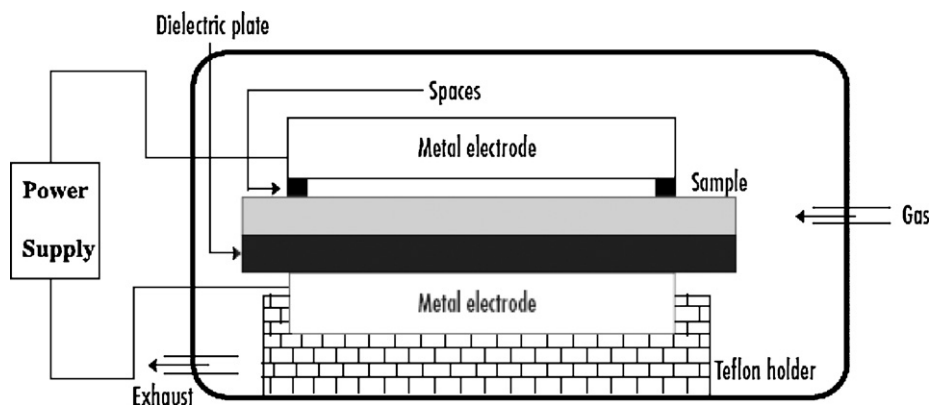


Fig. 1. A schematic diagram of the dielectric barrier discharge plasma system.

2.2. Methods

2.2.1. Plasma-treatment

Plasma treatment of cellulose containing fabric samples was carried out using APDBD (atmospheric pressure dielectric barrier discharge) reactor (Fig. 1). The flow rate of the working gas was kept constant at 3 L/min. Process parameters such as gas type (oxygen, air or argon) and exposure time were varied to attain the best treatment conditions for incorporating certain functional properties such as hydrophilicity, anti-UV and antibacterial finish.

2.2.2. Post-treatment with metal salts

The plasma-treated fabric samples were then immersed in the metal salt solution (0–5 g/L) along with a nonionic wetting agent (2 g/L) with material to liquor ratio, ML, of 1/20 for 15 min, followed by squeezing to a wet pick-up of 80%, and drying at 120 °C for 5 min. Subsequently, the fabric samples were thoroughly washed to remove excess and unfixed metal salt, then dried in air and conditioned for evaluation.

2.2.3. Post-basic dyeing

Post dyeing with nominated basic dye was carried out using the dye (1% owf), nonionic wetting agent (2 g/L), pH (9), LR (1/20) for 30 min at 95 °C followed by thoroughly washing and drying.

2.3. Fabric evaluation

The wettability was assisted using AATCC Test Method 79–1992.

The chemical changes that occur during the plasma treatment were analyzed using Nexess 670 FTIR Spectrophotometer from Nicolet USA. The spectra was recorded in the range of 4000–400 cm^{−1} with a resolution of 4 cm^{−1}.

The surface morphology of some untreated and treated fabric samples were observed using Scanning Electron Microscope (SEM, JEOL JZA 480A–Japan), after the samples plated with gold.

Metal content of the treated samples was quantitatively determined by using Flame Atomic Absorption Spectrophotometer, GBC-Avanta Australia.

UV-protection factor (UPF) was determined according the Australian/Newzealand Standard (AS/NZS 4399–1996). Fabric can be ranked as providing good, very good and excellent UV-protection if their UPF values range from (12–24), (25–39), and above 40 respectively.

The color strength (K/S) values were determined from the reflectance measurements using the Kubelka–Munk equation (Judd & Wyszecks, 1975)

$$\frac{K}{S} = \frac{(1 - R)^2}{2R}$$

where K/S is the ratio of absorption and scattering coefficient, R is the reflectance at the wave length of maximum absorbance of the used basic dye.

The antibacterial activity against G+ve bacteria (*Staphylococcus aureus*) and G−ve bacteria (*Escherichia coli*) was evaluated using colony counting method (AATCC 100–1999). The percent reduction in number of colonies for treated sample (B) as compared to untreated sample (A) gives the antibacterial activity or % reduction.

$$\text{Antibacterial activity or \% reduction} = \left[\frac{A - B}{A} \right] \times 100$$

3. Results and discussion

This investigation is directed toward enhancing the protection capacity of cellulose-containing fabrics especially against harmful-UVB and bacteria via plasma-pretreatment followed by metal-salts treatment under proper conditions. Results obtained along with appropriate discussion follow.

3.1. Exposure time

For a given set of O₂-plasma treatment conditions, Fig. 2 demonstrates that: (i) prolonging of O₂-plasma treatment from zero up to 45 s is accompanied by a significant decrease in wetting time of the

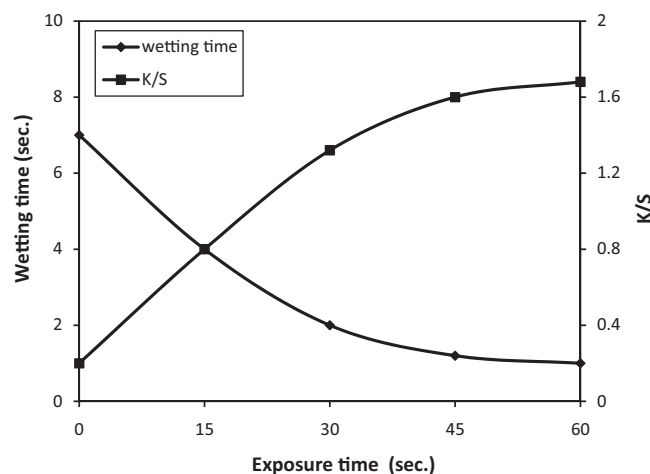


Fig. 2. Effect of exposure time on the wettability and subsequent basic dyeing. Substrate: cotton/polyester (80/20). Plasma treatment: O₂-gas, APDBD plasma, power supply with 20,000 Hz frequency, 50 W, and output of 5 kV/20 mA. Post-basic dyeing: C.I. Basic Blue 9 (1%), pH (9), LR (1/20) at 95 °C for 30 min.

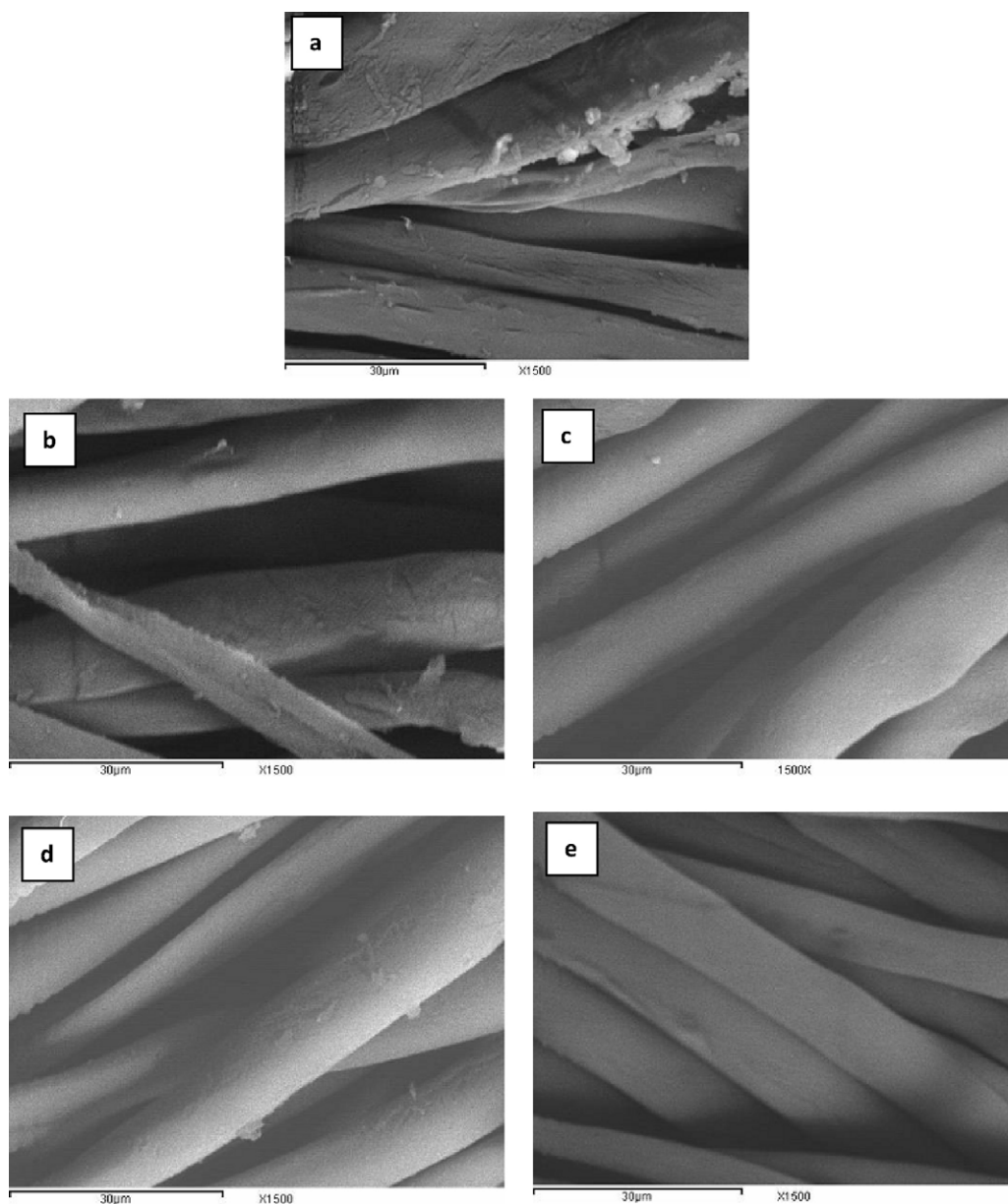


Fig. 3. SEM of untreated and plasma treated cotton/polyester (80/20) fabrics. Untreated cotton/polyester; (b) O₂-plasma treatment; (c) O₂-plasma followed by metal salt treatment; (d) Ar-plasma treatment; (e) Ar-plasma followed by metal salt treatment.

treated cotton/polyester blend (80/20) from 7 s to 1 s, (ii) increasing the exposure time intervals up to 45 s results in a remarkable increase in the extent of picking-up and dyeing with the used basic blue dye, expressed as *K/S*, from 0.2 up to 1.68, (iii) further increase in exposure time, i.e. beyond 45 s, has practically no effect on both the wetting time and extent of post-basic dyeing, and (iv) the remarkable improvement in the abovementioned properties is the results of morphological changes of the fabric surface (Fig. 3a and b) with etching as well as creation of an additional functional groups based on oxygen such as –COOH groups due to surface oxidation along with removal of any remnant hydrophobic impurities or additives, which enables greater hydrophilicity and dyeability with the used basic dye (Ibrahim, Eid, et al., 2010; Ibrahim, Hashem, et al., 2010; Ibrahim et al., 2008; Pandiyaraj & Selvarajan, 2008).

The FTIR spectra of untreated and oxygen plasma treated cotton/polyester (80/20) are demonstrated in Fig. 4(a and b) respectively. The untreated substrate spectrum (a) shows a broad band around 3330 cm^{−1} corresponds to alcoholic –OH stretching, two

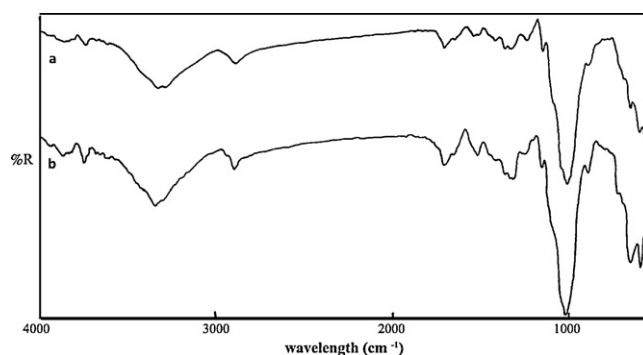


Fig. 4. FT-IR spectra of untreated (a) and oxygen plasma treated of cotton/polyester blend (b).

bands were observed around 2895.5 cm^{-1} and 1424 cm^{-1} due to CH_2 stretching and CH_2 bending respectively. The presence of carboxyl groups in the bleached fabric is confirmed with two strong bands at around 1710 and 1220 cm^{-1} corresponds to $\text{C}=\text{O}$ and $\text{C}-\text{O}-\text{H}$ stretching respectively. While the oxygen plasma spectrum (b) show the same bands but with higher intensity especially for bands corresponding to carboxyl groups ($-\text{OH}$ at 3330 cm^{-1} , $\text{C}=\text{O}$ at 1710 cm^{-1} and $\text{C}-\text{O}-\text{H}$ at 1220 cm^{-1}). This further affirms the creation of an additional $-\text{COOH}$ groups due to surface oxidation by plasma.

3.2. Subsequent-treatment with metal salts

As far as the changes in the UPF values of the cotton/polyester blend (80/20) as a function of exposure time to O_2 -plasma as well as subsequent treatment with different metal salts namely Cu-acetate, Zn-acetate, Al-chloride and Zr-oxychloride, the data demonstrated in Fig. 5 signify that (i) the extent of improvement in the UPF values is governed by both the exposure time as well as the type of metal salt used in the subsequent treatment, (ii) the enhancement in the UPF values, keeping the exposure time constant, follows the descending order: Cu-acetate > Al-chloride > Zn-acetate > Zr-oxychloride > none, (iii) the longer the exposure time, the better are the UPF values, keeping the metal salt constant, (iv) prolonging the exposure time up to 45 s is accompanied by the interactions among the O_2 -plasma active species and the fabric surface, etching of surface/contaminated layer, and formation of new polar groups, e.g. $-\text{COOH}$ groups due to surface oxidation along with chemical and morphological alteration, (v) the net effect of the aforementioned surface modifications is increasing the extent of picking-up and enhancing fixation of metal cations onto/within the fabric matrix via coordination according to the following tentative mechanism, Scheme 1 (Ibrahim, Refai, Youssef, & Ahmed, 2005a; Sun & Stylios, 2005; Wong, Tao, Yuen, & Yeung, 2000) and (vi) the variation in UPF values upon subsequent treatment with the nominated salts reflects the differences among the metal salt ions in: molecular size, extent of fixation onto/within the modified structure, its ability to interact with the

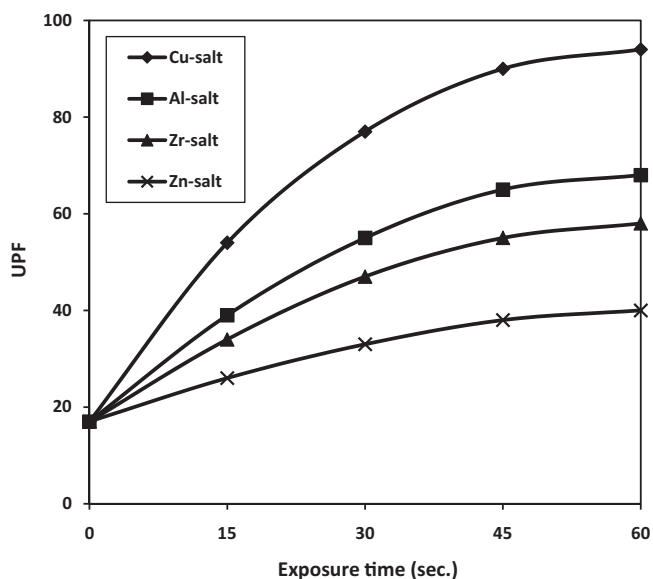


Fig. 5. Effect of exposure time and post-treatment with certain metal salts on the UPF. Substrate: cotton/polyester (80/20). Plasma treatment: O_2 -gas, APDBD plasma, power supply with 20,000 Hz frequency, 50 W, and output of 5 kV/20 mA. Post-treatment: metal salt (5 g/L); nonionic wetting agent (2 g/L); wet pick-up 80% (owf); drying at $100^\circ\text{C}/5\text{ min}$; followed by afterwashing to remove excess and unfixed metal salt.

new functional groups, its ability to entrap into the created empty spaces or fiber porosity, extent of mechanical adhesion, as well as the ability of the metal-loaded structure to scatter and block the hazardous UV-B radiation (Ibrahim, Refai, Youssef, & Ahmed, 2005b).

Obviously, the best UV-protection properties were obtained by O_2 -plasma pretreatment for 45 s followed by subsequent treatment with Cu-acetate as shown in Fig. 5.

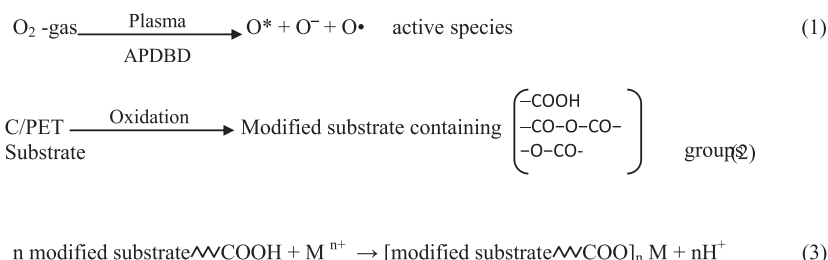
3.3. Type of cellulose-containing fabric

Fig. 6 shows the relation between the UPF values and variation in substrate type, keeping other parameters constant. For a given set of plasma-treatment and subsequent treatment with metal salts condition, it is clear that: (i) post-treatment with any of the used metal salts is accompanied by a significant improvement in the UPF values and follows the decreasing order: Cu-acetate > Al-chloride > Zr-oxychloride > Zn-acetate > none, irrespective of the used substrate, (ii) the extent of improvement in the imparted UV-protection functionality is governed by the type of cellulose-containing fabric and follows the descending order: linen/viscose-polyester (50/50) > cotton/polyester (80/20) > cotton/linen (50/50), keeping other parameters constant, (iii) the variation in the UPF values upon using different substrates reflects the differences among them in: fiber type and content, e.g. cellulosic fibers, polyester fibers, etc., fabric construction, e.g. mass, thickness, porosity, etc., chemical characteristics, surface morphology, amorphous/crystalline regions, extent of modification by plasma-treatment, accessibility to the metal salt cations, extent of picking-up and anchoring metal cations physically/mechanically and/or chemically via the generated polar groups, e.g. $-\text{COOH}$ groups (Ibrahim, El-Zairy, & Eid, 2010; Ibrahim et al., 2005a, 2005b; Ibrahim, EL-Badry, Eid, & Hassan, 2011), (iv) incorporation of polyester fibers, an aromatic polyethylene terephthalate, as a fabric component enhances UV-absorption and blocking, i.e. better UPF compared with the cellulosic one (Schuierer, 1997), and (v) the most significant UV-protection properties were achieved by O_2 -plasma treatment followed by treatment with Cu-acetate, irrespective of the used substrate.

3.4. Type of gas

Fig. 7 shows the relation between the imparted UV-protection functionality, expressed as UPF, and variation in plasma-gas, keeping other parameters constant. As shown in Fig. 7, the cellulose-containing substrates that received the plasma-pretreatment and subsequent Cu-acetate treatment have a significant improvement in their UV-protection abilities compared with the untreated ones. The extent of improvement in their UPF values is governed by type of substrates as discussed earlier, and type of gas: oxygen- > air- > argon-gas, which confirmed that both oxygen and air are more active than the inert argon gas (Ibrahim, Eid, et al., 2010; Krump, Hudec, Jasso, Dayss, & Luyt, 2006). It seems that both oxygen and air gases have more positive impacts on physical, morphological and chemical properties of the treated substrates compared with the argon-argon, thereby enhancing the extent of picking up and loading of Cu-cations onto/within the substrate, via creating more etched/hydrophilic surfaces with accessible active functional groups, e.g. $-\text{COOH}$ groups, which in turn improves the extent of anti-UV functionality.

Fig. 3 illustrates the surface morphology changes of samples treated with oxygen and argon plasma, Fig. 3(b and d), followed by metal salt treatment, Fig. 3(c and e). Fig. 3(b and d) shows that contamination and fibrils at the upper layer of fiber surface are removed producing a clean surface, comparing with the untreated



Scheme 1. O₂-plasma and subsequent metal salt treatments.

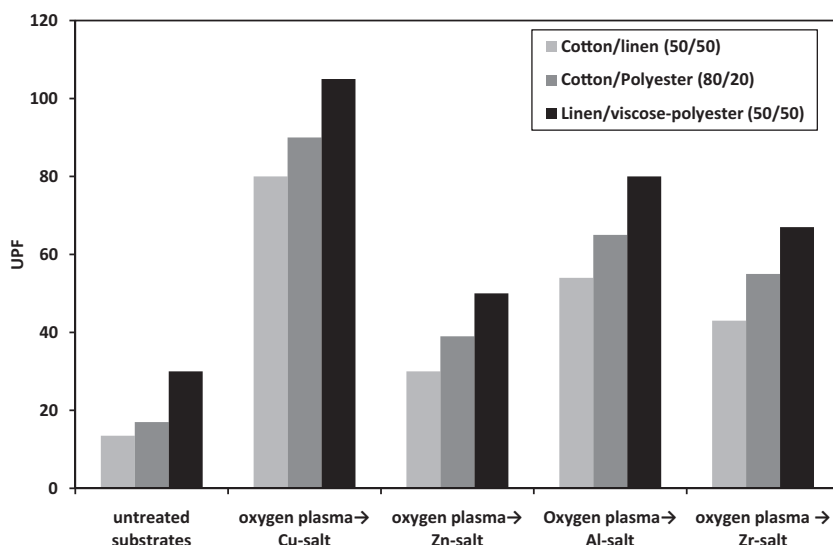


Fig. 6. Effect of type of substrate and kind of loaded metal ion on the anti-UV function. Plasma treatment: O₂-gas, APDBD plasma, power supply with 20,000 Hz frequency, 50 W, and output of 5 kV/20 mA, exposure time 45 s. Post-treatment: metal salt (5 g/L); nonionic wetting agent (2 g/L); wet pick-up 80% (owf); drying at 100 °C/5 min; followed by afterwashing to remove excess and unfixed metal salt.

sample, due to plasma ablation and etching (Fig. 3a). However, oxygen plasma causes more cracks and pits than argon plasma this may be due to the relatively slow rate of physical etching introduced by the argon plasma. Fig. 3(c and e) shows a smooth and homogenous coating layer after post treatment with metal salt, however the coating layer in case of argon plasma is thinner than oxygen plasma.

3.5. Multi-functional properties

We examined the extent of loading the metal ions and enhancing the anti-UV and antibacterial function as well as the durability of the imparted properties to wash as a function of the gas type, the kind of metal salt and the nature of cellulose containing fabric. The results are shown in Table 1.

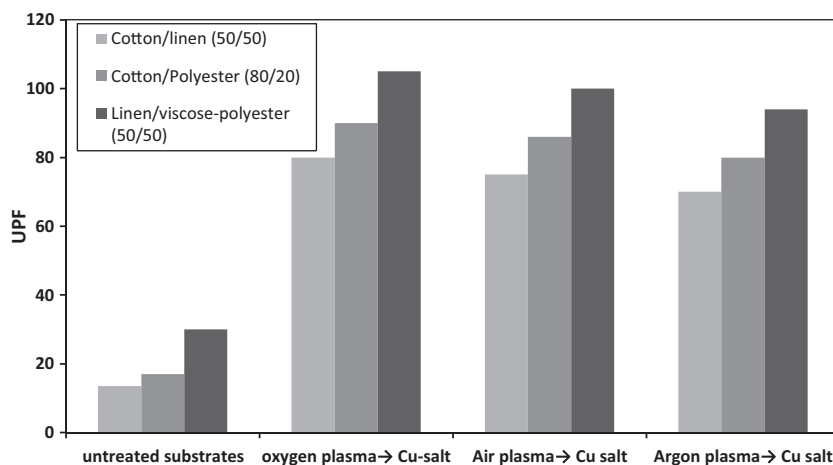


Fig. 7. Effect of using different plasma gases followed by subsequent treatment with Cu-acetate on the anti-UV function of the treated substrates. Plasma treatment: APDBD plasma, power supply with 20,000 Hz frequency, 50 W, and output of 5 kV/20 mA, exposure time 45 s. Post-treatment: Cu-acetate (5 g/L); nonionic wetting agent (2 g/L); wet pick-up 80% (owf); drying at 100 °C/5 min; followed by afterwashing to remove excess and unfixed metal salt.

Table 1
Anti-UV and anti-bacterial properties of treated cellulose containing fabrics.

Metal salt (5 g/L)	Plasma gas	Substrate	Metal content (%)	UPFWashing cycles		Antibacterial activity (RPC%)	
				1	10	G+ve	G–ve
Cu-acetate	Oxygen	I	0.491	80	66	88.70 (81.50)	86.09 (79.32)
		II	0.352	90	75	81.69 (75.18)	77.95 (72.01)
		III	0.378	105	90	83.52 (76.24)	81.06 (74.22)
	Air	I	0.299	75	60	80.67 (74.80)	77.01 (71.46)
		II	0.182	86	74	72.30 (67.01)	70.08 (65.30)
		III	0.263	100	87	75.82 (70.14)	72.85 (67.84)
	Argon	I	0.272	70	56	78.75 (72.09)	75.05 (68.85)
		II	0.129	80	65	69.68 (65.10)	64.96 (62.91)
		III	0.239	94	80	76.09 (70.08)	73.20 (68.02)
Zr-oxychloride	Oxygen	I	0.274	43	35	94.23 (86.56)	92.49 (84.85)
		II	0.103	55	44	84.68 (78.09)	82.59 (84.85)
		III	0.115	67	55	88.96 (82.01)	86.32 (79.72)
	Air	I	0.213	38	30	91.14 (83.95)	88.66 (81.97)
		II	0.073	45	39	80.25 (74.38)	76.26 (70.84)
		III	0.123	60	48	86.18 (80.26)	85.20 (79.28)
	Argon	I	0.120	32	25	87.88 (80.93)	85.21 (78.36)
		II	0.025	39	34	75.96 (70.08)	72.48 (67.13)
		III	0.061	55	44	80.49 (74.45)	76.92 (71.23)
Zn-acetate	Oxygen	I	0.288	30	24	96.24 (89.02)	94.56 (87.28)
		II	0.140	38	30	86.01 (78.92)	84.15 (77.14)
		III	0.160	50	41	90.20 (83.04)	87.76 (80.54)
	Air	I	0.260	24	16	95.05 (87.15)	93.80 (86.12)
		II	0.094	32	25	84.34 (77.11)	82.03 (74.80)
		III	0.153	45	37	89.85 (82.62)	87.41 (80.42)
	Argon	I	0.233	27	18	92.43 (85.16)	91.22 (84.26)
		II	0.96	36	27	83.22 (76.52)	80.61 (74.35)
		III	0.142	40	35	88.66 (81.15)	85.20 (77.85)
Untreated		I	–	12	5	–	–
		II	–	17	12	–	–
		III	–	30	22	–	–

Substrate I: cotton/linen (50/50); substrate II: cotton/polyester (80/20); substrate III: linen/viscose–polyester (50/50).

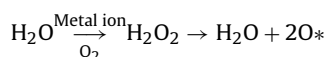
Plasma treatment: APDBD plasma, power supply with 20,000 Hz frequency, 50 W, and output of 5 kV/2 mA, exposure time 45 s.

Salt-treatment: metal salt (5 g/L); nonionic wetting agent (2 g/L); wet pick-up 80% (owf); drying at 100 °C/5 min; followed by afterwashing.

Values in parentheses indicate retained functional properties after 10 laundering cycles.

From Table 1, it can be clearly seen that: (i) the metal content of the treated substrates is governed by: type of substrate: substrate I > substrate II > substrate III, plasma gas: oxygen > air > argon, and metal salt: Cu-acetate > Zn-acetate > Zr-oxychloride, keeping other parameters fixed, (ii) the extent of improvement in the UPF values of the treated substrates is governed by type of substrate: substrate III > substrate II > substrate I, plasma gas: oxygen > air > argon, and metal salt: Cu-acetate > Zr-oxychloride > Zn-acetate, keeping other parameters constant, (iii) the enhancement in the imparted anti-bacterial property is determined by type of substrate: substrate I > substrate III > substrate II, plasma gas: oxygen > air > argon, and metal salt: Zn-acetate > Zr-oxychloride > Cu-acetate, keeping other parameters constant, (iv) the variation in the abovementioned chemical and functional properties of the treated substrates in presence of the aforementioned metal ions, reflects the differences among these metal ions in: molecular weight and size, content, location and extent of distribution, extent of fixation onto/within substrate and durability of loaded metal cations to severe washing (10-laundering cycles), UV-blocking property and absorbing capacity, as well as ability to inhibit the multiplication of micro-organisms (Ibrahim, El-Gamal, Gouda, & Mahrous, 2010; Ibrahim, Gouda, Hussein, El-Gamal, & Mahrous, 2009), (v) the antibacterial activity of the metal ion-loaded substrates could be discussed in terms of its ability to: inhibit the multiplication of the harmful bacteria via destroying or passing through the cell membrane and subsequent bonding to –SH of cellular enzyme thereby altering the micro-organism metabolism and suppressing of their growth up to cell death and/or via catalization of

the production of oxygen radicals according to the following reaction:



thereby destroying molecular structure of bacteria (Ibrahim, Eid, & El-Batal, 2012; Ibrahim, Eid, Youssef, Ameen, & Salah, 2012), (vi) the extent of inactivation of G+ve bacteria was better than G–ve bacteria, regardless of the used metal salt, which could be explained by the differences between them in their molecular structure, outer membrane as well as in the amenability to suppression their growth, up to cell death and/or destruction of their molecular structure by the produced oxygen radicals as discussed before, and (vii) after 10 laundering cycles, the imparted functional properties are maintained without a significant changes.

4. Conclusions

The present work aimed to enhance the anti-UV and antibacterial functionalities of cotton/linen (50/50), cotton/polyester (80/20) and linen/viscose–polyester (50/50) blends via plasma pretreatment followed by post-treatment with certain metal salts namely Cu-acetate, Zn-acetate, Al-chloride and Zr-oxychloride. From the obtained results, it was possible to conclude that:

- Plasma treatment for 45 s and subsequent metal salt treatment (5 g/L) resulted in a significant improvement in the imparted

functional properties, regardless of the used plasma–gas, metal salt, and type of substrate.

- The best UPF values were obtained by using O₂-plasma followed by subsequent treatment with Cu-acetate, irrespective of the used substrate.
- The best antibacterial activity was obtained by using O₂-plasma followed by subsequent treatment with Zn-acetate, regardless of the used substrate.
- The effectiveness of plasma treatment was governed by the type of gas, i.e. oxygen > air > argon.
- The extent of surface modification by plasma as well as fixation of metal ions onto/within the substrate, which in turn affects the imparted functional properties, were determined by the type of substrate.
- The imparted UV-protection and the antibacterial functionalities showed no significant changes after 10 laundering cycles.

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