

# Characterization of atmospheric pressure plasma treated pure cashmere and wool/cashmere textiles: Treatment in air/water vapor mixture

Stefano Zanini <sup>a,\*\*</sup>, Elisa Grimoldi <sup>a</sup>, Attilio Citterio <sup>b</sup>, Claudia Riccardi <sup>a,\*</sup>

<sup>a</sup> Università degli Studi di Milano-Bicocca, Dipartimento di Fisica "G. Occhialini", p.za della Scienza, 3, I-20126 Milano, Italy

<sup>b</sup> Politecnico di Milano, Dipartimento di Chimica, Materiali ed Ingegneria Chimica "G. Natta", Via Mancinelli 7, I-20131 Milano, Italy

Received 9 March 2015

Received in revised form 30 April 2015

Accepted 1 May 2015

Available online 8 May 2015

## 1. Introduction

Traditional wet-chemical processes used in the textile industry involve high consumption and pollution of water resources. Waste water processing costs are high and drying the wetted fibers is energy-, time- and cost-intensive. Therefore, the textile industry has a great interest in alternative dry processes. Low temperature plasma treatment is a dry and eco-friendly technology which has been widely used to modify the chemical and topographical properties of polymers and textiles surface [1–5]. The application of plasma technologies as a pretreatment and finishing process for textiles has become very popular because this surface modification method changes the outermost layer of the substrate without altering the bulk properties.

Literature abounds of works on low-pressure plasma treatment of textiles for different purposes, as an alternative to wet-chemical processes [6–10]. Low-pressure plasma treatments are known to induce physical and chemical surface changes in textile fibers through several concurrent processes (activation, etching, grafting

of chemical functional groups, cross-linking) [11–14]. For example, low-pressure plasma treatments with gases such as air, helium, nitrogen and oxygen have been used to improve the surface wettability and the dyeing properties of different textiles [15–18]. Moreover, in the case of wool, they have been employed to enhance its shrink resistance [19–24] and its anti-pilling properties [25].

Despite its effectiveness on textiles, the inability to successfully incorporate low-pressure plasma treatment equipment into a continuous textile processing operation has limited the commercial viability of this technique. In the last years, atmospheric pressure plasma treatments have gained considerable popularity for the surface modification of materials [1,26–28]. The main advantages over the low-pressure ones are the possibility to avoid the expensive vacuum systems, to decrease the time of treatment, and to simplify the technological transfer where the processes of production are making in continuous mode. As a matter of fact, the atmospheric pressure plasma treatment devices can be easily integrated with the continuous on-line processing of the textile materials. However, while the effects of low-pressure plasma treatments are well documented, relatively little has been published about atmospheric pressure plasma treatments of textiles [29–35]. In particular, only few papers describe the effect of atmospheric pressure plasma treatments on wool [36–40].

In this work, we performed atmospheric pressure plasma treatments of pure cashmere and wool/cashmere textiles with humid

\* Corresponding author. Tel.: +39 02 6448 2314; fax: +39 02 6448 2367.

\*\* Corresponding author. Tel.: +39 02 6448 2327; fax: +39 02 6448 2367.

E-mail addresses: stefano.zanini@mib.infn.it (S. Zanini), riccardi@mib.infn.it (C. Riccardi).

air. Since it is known that these processes may also cause undesired degradation, treatment parameters have been optimized in order to achieve the surface modifications without changing the bulk properties as well as the touch of the textile.

A deep characterization has been performed to study the wettability, the surface morphologies and the chemical composition of the plasma treated textiles.

## 2. Experimental

### 2.1. Materials

Commercial pure cashmere and wool/cashmere fabrics were supplied by LoroPiana (Quarona, Italy) and used as received. Since the aim of the plasma treatment was the enhancement of the textiles wettability for possible following finishing processes, we used fabrics which had been already dyed. In particular, the pure cashmere textile was black, while the wool/cashmere one was gray.

### 2.2. Plasma reactor

The DBD experimental device (supplied by Tigres GmbH, Germany) has been extensively described elsewhere [41]. It consists of a stainless steel planar electrode coated with a poly(tetrafluoroethylene) sheet (thickness = 1 mm), where textile samples to treat are placed on. Two rod electrodes (220 mm long, 8 mm diameter), coated with pure (>99.7%) synthesized  $\text{Al}_2\text{O}_3$  dielectric (thickness 2 mm), are mounted on a mobile station, at a distance of 20 mm from each other. The two rods are connected through an HV cable to the secondary coil of a transformer, whose primary circuit is connected to a power generator, providing the driving high voltage for the discharge. The distance between the plate electrode and the rod electrodes could be varied from 0.5 mm to few millimeters. In our experiments it is fixed at 2 mm. During the treatments, the station can move horizontally with a speed that can be varied between 0.7 and 7 m/min. The electrical power supplied to the discharge could be varied up to a maximum of 450 W. For each treatment, the energy density, or dose, could be estimated by:  $D = Pn/vl$ , where  $P$  is the electrical power,  $v$  is the speed of rod electrodes,  $l$  is their length and  $n$  is the numbers of passes over the specimens.

The inlet fluxes are controlled by a gas/vapor mixing system. The air flow is regulated using a mass flow controller (El-flow by Bronkhorst) with a maximum flow of 20 L/min. To introduce water vapor in the gas flow, we employed a liquid flow controller with an evaporator/mixer system (Bronkhorst CEM System). The inlet fluxes are injected between the high voltage electrodes through an injection flute that ensures uniform fluxes on the whole length of the electrodes.

### 2.3. Characterization techniques

#### 2.3.1. Wettability measurements

Absorption times of deionized water droplets were measured using a home-made apparatus, as already described [8,10,12]. Water droplet absorption time ( $t_{\text{ads}}$ ) values were obtained as the average absorption time of 12 water droplets of 70  $\mu\text{l}$ , deposited through a standard procedure on different zones of each fabric specimen.

#### 2.3.2. FTIR/ATR spectroscopy

Fourier transform infrared spectroscopy (FT-IR) was performed using a Perkin Elmer 2000 FT-IR fitted with a universal attenuated total reflectance (ATR) sampling accessory (MKGolden Gate) with

a diamond coated zinc selenide window. For each spectrum 150 scans, with a spectral resolution of  $4\text{ cm}^{-1}$ , were recorded.

#### 2.3.3. X-ray photoelectron spectroscopy (XPS)

X-ray photoelectron spectra were recorded using a Vacuum Generator ESCALAB 1 apparatus, with a monochromatic Al-anode  $K_{\alpha}$  source ( $E = 1.487\text{ keV}$ ) working at 350 W and an electron take-off angle of  $45^\circ$ . The pressure in the chamber was around  $10^{-7}\text{ Pa}$ . Unless otherwise noted, samples were mounted in a carousel stage with xyz translational and rotational capabilities. Binding energy scales were referenced to the  $\text{CC/CH}_2$  peak in the C (1s) region at 284.6. A pass energy of 20 eV was used for high-resolution scans of C 1s and S 2p peaks with an acquisition time of 3 min. XPS data fitting was performed using 100% Gaussian peaks.

#### 2.3.4. Scanning electron microscopy (SEM)

SEM analyses were performed with a Scanning Electron Microscope Zeiss EVA 50 EP using a primary electron beam with an energy of 10 kV, coupled with a complete energy dispersive X-ray system (EDX Oxford Inca Energy 200) and with the INCA software program. High sensitivity to topographic features on the outermost surface (<5 nm) is achieved when using a primary electron beam with an energy of <1 kV. Micrographs were obtained in air on aluminum support. No metal was deposited on the surface of the treated samples to minimize the number of treatments. Images were taken at magnifications 100 $\times$ , 300 $\times$ , 500 $\times$ , 1000 $\times$  and 3000 $\times$  with the EDX analyses performed at the highest resolution (spot of  $<1 \times 1\ \mu\text{m}$ ).

## 3. Results and discussion

The first aim of this work was the surface modification of cashmere and wool/cashmere textiles by atmospheric pressure plasma treatments, for the enhancement of their wettability without changing their bulk properties as well as their touch. Since these processes may cause undesired degradation of organic materials, an initial optimization of the treatment parameters has been performed, using an inlet gas mixture with 10 L/min of air and 3 g/h of water vapor. In this first experimental step, we focused on the wettability characterization and on the evaluation of the possible deterioration and/or changes in the touch of the textiles.

Fig. 1 shows the influence of the energy dose on the water droplets adsorption time. The 70  $\mu\text{l}$  water droplets are not adsorbed by the untreated textiles, and remained on their surface until evaporation. The energy dose parameter was varied by fixing the supplied power (270 W) and the speed of the mobile station

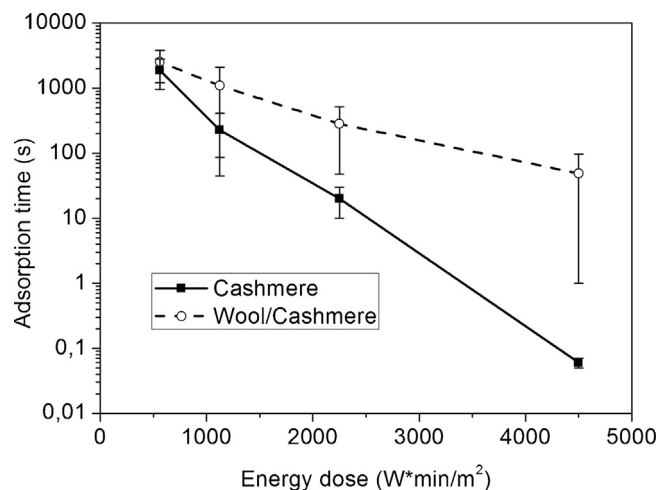
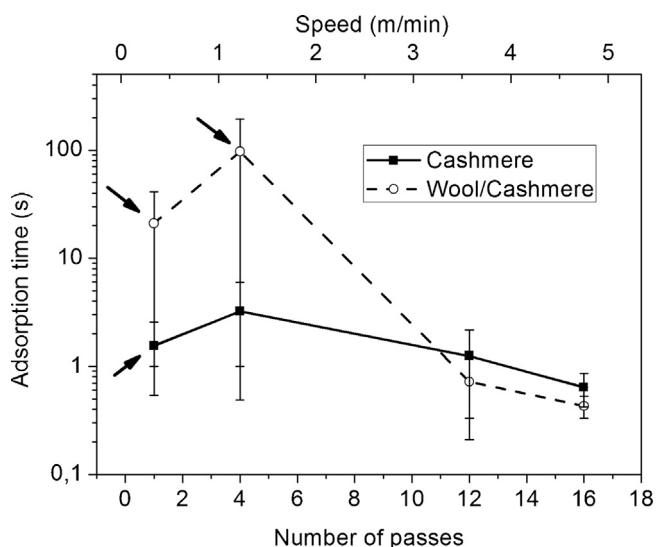


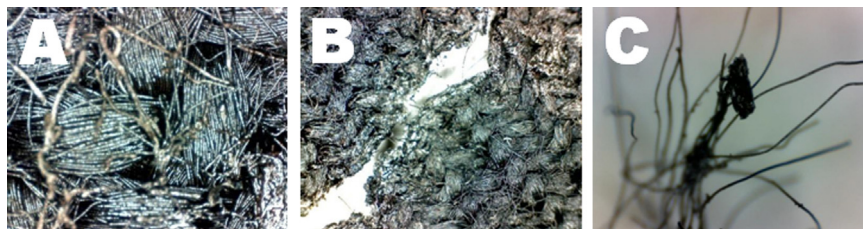
Fig. 1. Influence of the energy dose on the wettability of the treated textiles.



**Fig. 2.** Influence of the number of passes (at constant energy dose) on the wettability of the treated textiles.

(2.5 m/min) and by changing the number of passes over the samples. It can be noticed that, both for cashmere and wool/cashmere fabrics, the wettability increased with the energy dose. Moreover, pure cashmere textiles were more influenced by the plasma treatment than the wool/cashmere ones. None of these samples displays burning or other deterioration after the plasma treatment. However, treated samples appeared more dry at the touch (dry-hand), probably as a consequence of a partial dehydration owing to the plasma treatment. This effect increases with the energy dose, and become important for doses of  $4500 \text{ W min/m}^2$ . Despite of this, it must be pointed out that the treated textiles recovered their original softness in a few day.

Since the energy dose is a composite parameter, the same energy dose can be supplied to the textile in different ways. In order to test if this had influence on the wettability characteristics of the treated samples, we set up an experiment in which we fixed the supplied power ( $270 \text{ W}$ ) and energy dose ( $4500 \text{ W min/m}^2$ ), and varied the speed of the mobile station (from  $0.3$  to  $4.8 \text{ m/min}$ ) and the number of passes (from  $1$  to  $16$ ). Results, in terms of water droplets adsorption time, are displayed in Fig. 2. Despite a general remarkable increase in wettability (which can be explained with the high energy dose and which is particularly evident for the pure cashmere fabrics), we noticed better performances at high number of passes (and hence at high speed of the mobile station). This effect can be better appreciated by observing the curve of the wool/cashmere fabrics. We interpreted this result in terms of uniformity of the treatment: the high number of passes probably allow a more uniform grafting of polar functionalities onto the fibers. Moreover, we observed a deterioration of the samples treated at low speed, with the presence of several burnt fibers (in Fig. 2, three arrows indicate the samples which were irretrievably damaged). Optical microscope images of the damaged fabrics are displayed in Fig. 3.



**Fig. 3.** Optical microscope images of the damaged fabrics treated with high energy dose and low speed of the mobile station.

Carbonized fibers can be observed in Fig. 3a and c, while Fig. 3b shows a laceration of the treated sample. Instead, fabrics treated at high speed do not show any variation of the organoleptic properties (appearance, color), as well as any fibers damage.

The preliminary optimization step allowed us to select the range of treatment parameters for an effective hydrophilic modification without fabric deterioration. The water droplet adsorption time was almost immediate when the energy dose was higher than  $3000 \text{ W min/m}^2$  (Fig. 1). Moreover, no damaging of the treated fibers was observed when the speed of the mobile station was higher than  $2 \text{ m/min}$ . Starting from these considerations, we select a set of parameters (Table 1) for a complete chemical and morphological characterization of both the treated fabrics (treatment A in Table 1). Moreover, we investigated the effect of the water vapor concentration on the chemical composition of the treated textiles by doubling the amount of water introduced in the air flow (treatment B in Table 1). We chose a speed of the mobile station ( $2.5 \text{ m/min}$ ) and a number of passes ( $4$ ) which are a compromise between the necessity of avoiding a fabric damaging and the will of adopting a set of parameters which could be also useful for practical industrial applications.

Pure cashmere and wool/cashmere fabrics were first characterized with FTIR/ATR spectroscopy.

FTIR/ATR analyses of both the textiles do not reveal strong differences between the treated samples and the untreated ones. This result is a consequence of the low penetration of the plasma treatment into the bulk of the fibers, especially when compared to the penetration depth of this analytical technique (a few microns). Most importantly, we noticed no differences in the region  $1500\text{--}1700 \text{ cm}^{-1}$ . This result excludes the formation of carbon-carbon double bonds (which generally cause fabric yellow-ness) from the interaction of the plasma species with the protein fibers and is in agreement with the unchanged textiles color.

However, some differences between the untreated and the plasma treated samples were observed. Fig. 4 displays the ATR spectra of the untreated and the plasma treated wool/cashmere fabrics, in the region  $900\text{--}1300 \text{ cm}^{-1}$ . Significant bands are indicated with arrows. The spectrum of the treated sample refers to the fabric treated with parameters B in Table 1 (with a doubled water vapor flow of  $6 \text{ g/h}$ ).

Bands at  $1041 \text{ cm}^{-1}$ ,  $1079 \text{ cm}^{-1}$  and  $1126 \text{ cm}^{-1}$  can be assigned respectively to the cysteic acid ( $\text{SO}_3^-$  groups), to the cystine monoxide ( $-\text{SO}-\text{S}-$  groups) and to the cystine dioxide ( $-\text{SO}_2-\text{S}-$  groups)- [24,42]. The band at  $1230 \text{ cm}^{-1}$  is the amide III band of the polypeptide chain.

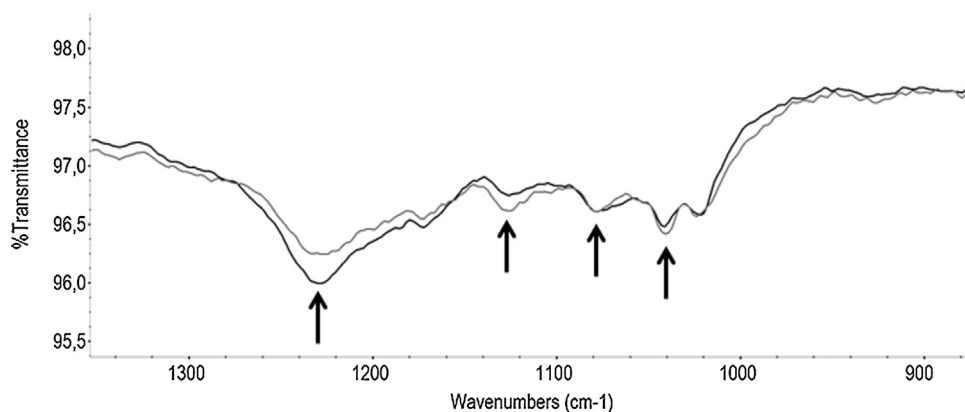
Cysteic acid, cystine monoxide and cystine dioxide were formed as a result of the cleavage of disulphide linkage [42]. Both cystine residues were believed to be intermediate cystine oxidation products (disulphide  $\rightarrow$  monoxide  $\rightarrow$  dioxide  $\rightarrow$  sulphonic acid) [42]. The presence of these groups on the polypeptide chain provides polar characteristics to the fabric, which in turn helps to improve the wettability of the textiles.

Fig. 4 clearly shows that the amount of cysteic acid and of cystine monoxide (bands at  $1041 \text{ cm}^{-1}$  and  $1126 \text{ cm}^{-1}$ ) on the wool/cashmere fabric surface increased after the plasma treatment.

**Table 1**

Operating parameters employed for the treatment of the fabrics which were characterized by FTIR/ATR, XPS and SEM analyses.

Treatment	Inlet gas	Gas flow (L/min)	Water vapor flow (g/h)	Number of passes	Station speed (m/min)	Power (W)	Energy dose (W min/m <sup>2</sup> )
A	Air/water vapor	10	3	4	2.5	450	3600
B	Air/water vapor	10	6	4	2.5	450	3600

**Fig. 4.** FTIR/ATR spectra of the untreated (black line) and the plasma treated (gray line) wool/cashmere fabrics. See text for band assignment.

This indicates a cleavage and an oxidation of the disulphide groups owing to the exposure to the plasma species. A semi-quantitative evaluation of the extent of the plasma modification has been performed by integration of the band at 1041 cm<sup>-1</sup>, 1079 cm<sup>-1</sup> and 1126 cm<sup>-1</sup>, followed by the calculation of the ratio between the integral of these bands with that of the amide III band (1230 cm<sup>-1</sup>) [42]. Results are shown in Table 2.

For the wool/cashmere fabrics, it can be noticed that all these ratios considerably increased as a result of the exposure to plasma (both for parameters A and B in Table 1). This is particularly evident for cysteic acid and of cystine monoxide groups, which almost doubled their presence on the fabric surface. On the contrary, for the pure cashmere fabrics no evident oxidation of the disulphide groups was detected. Cysteic acid and cystine monoxide bands were absent on both the spectra of the untreated and the plasma treated textiles, while the amount of cystine dioxide does not vary after the treatment.

Wool fibers show the presence of covalently bound lipids on the surface of their cuticle membrane [43]. These are a complex mixture of polar and non-polar lipids, with long chain fatty acids being the predominant class. Evidence to date indicates that the majority of the fatty acids are covalently bound to the cuticle surface proteins *via* a thioester bond to cysteine residues with up to 40% of the fatty acids linked as oxygen esters to serine or threonine residues.

It is reasonable to suppose the plasma species mainly interacting with this external layer of lipids, and other authors have described this phenomenon for plasma treatments at both atmospheric and low pressure [11,36,40]. A decrease in the total carbon content on the fibers surface has been detected by XPS analysis on wool treated

**Table 2**

FTIR/ATR bands ratio for cysteic acid, cystine monoxide and cystine dioxide in relation to the amide III band.

Sample	SO <sub>3</sub> <sup>-</sup> /amide III (1040/1230)	SOS/amide III (1071/1230)	SO <sub>2</sub> S/amide III (1121/1230)
Untreated wool/cashmere	0.027	0.037	0.029
Wool/cashmere A	0.065	0.053	0.076
Wool/cashmere B	0.060	0.045	0.053
Untreated pure cashmere	n.d.	0.054	n.d.
Pure cashmere A	n.d.	0.056	n.d.

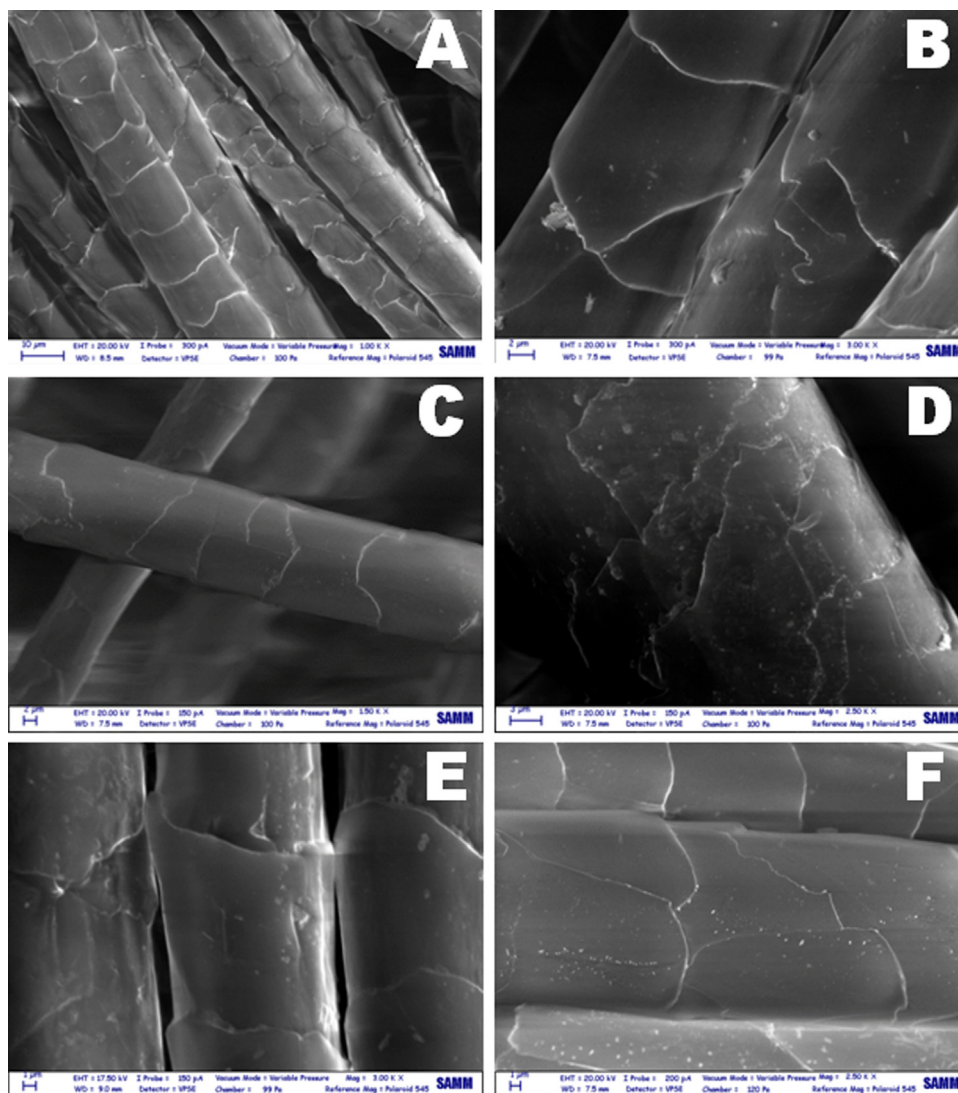
with an atmospheric pressure plasma jet [36]. Molina et al. [11] investigated with XPS the effects of low-pressure plasma treatment with different gases. For treatments with oxygenated gases (pure oxygen, air), they found a decrease of the C–C/C–H component of the C 1s peak, and an increase of the C–O, C=O and O–C=O components. In some cases, the decrease in the surface carbon content has been detected also with FTIR/ATR analysis [40]. In our case, no differences were observed in the region 2800–3000 cm<sup>-1</sup> (the region of C–H stretching). However, XPS analyses allowed us to detect the effects of plasma treatment on the external layers of the fibers. Results are summarized in Table 3. Plasma treated fabrics (both pure cashmere and wool/cashmere) show a decreased carbon content and an increased oxygen content. We believe the increase of the O/C ratio owing to the etching of the external layer of lipids and the insertion of oxygenated species onto the fibers surface. Since the reaction rate of the oxidation (or carbon elimination) induced by atomic oxygen is much higher than that of the nitrification reaction induced by atomic nitrogen, only a slight increase in the nitrogen content has been detected after the plasma treatment. The increase in the nitrogen atomic percentage can be also explained with the removal of the external hydrocarbon chains, which makes more visible the underlying protein material.

By observing the data in Table 3, it can be also noticed that the treatment B (with a doubled water vapor flow of 6 g/h) is more effective than the treatment A in the introduction of oxygenated species and in the removal of the external layers. In fact, in the case of treatment B we observed also a decrease in the sulfur content, which indicates a partial etching also of the cysteine residues of the epicuticle, which lie under the lipids layer. This result helps in

**Table 3**

Atomic percentage of elements measured on untreated and plasma treated pure cashmere and wool/cashmere fabrics.

Sample	Atomic composition (%)				
	C	O	N	S	O/C
Untreated wool/cashmere	74.9	13.8	8.8	2.4	0.18
Wool/cashmere A	66.7	20.6	10.0	2.7	0.31
Wool/cashmere B	65.9	22.7	9.3	2.1	0.34
Untreated pure cashmere	74.4	14.2	9.0	2.6	0.19
Pure cashmere A	69.9	19.4	9.3	2.4	0.28



**Fig. 5.** SEM images of untreated and the plasma treated fabrics. (A) Untreated wool/cashmere 1000 $\times$ ; (B) untreated wool/cashmere 3000 $\times$ ; (C) plasma treated wool/cashmere 1500 $\times$ ; (D) plasma treated wool/cashmere 2500 $\times$ ; (E) untreated pure cashmere 3000 $\times$ ; (F) plasma treated pure cashmere 2500 $\times$ .

the interpretation of FTIR/ATR results. Rather than be ascribed to a lower sulfur oxidation, the lower ratios reported in Table 2 for the treatment B are the consequence of the lower sulfur content.

It is particularly interesting to note that the O/C ratio measured on the plasma treated pure cashmere was slightly lower than that measured on the wool/cashmere sample. This result is particularly unexpected, since plasma treated pure cashmere displayed higher wettability. Rather than deriving from the different surface composition, we ascribed the differences in wettability to the different weaving of the two textiles.

The effects of the plasma treatment on morphological characteristics of the fibers were investigated by means of SEM analyses.

Fig. 5 illustrates the SEM images of the untreated (Fig. 5a and b) and plasma treated (Fig. 5c and d) wool/cashmere fabrics (treatment parameter A in Table 1). Wool fibers (diameter 10–12  $\mu\text{m}$ ) are easily distinguished from cashmere ones (more thinner, diameter around 8  $\mu\text{m}$ ). Plasma treatment seems to have only minor effects on the morphology of the fibers. Comparing Fig. 5a and c, a slight reduction of the scales thickness for both the types of fiber can be noticed. This effect of the plasma treatment is also observed in Fig. 5b and d, and can be attributed to etching phenomenon which contributes to the removal of material from the fibers surface. However, the surface of the scales on both untreated and plasma treated

fiber is smooth (Fig. 5a and c), and only on a minor number of the treated fibers the effect of plasma erosion can be noted (Fig. 5d). The same consideration is valid also for pure cashmere fabrics (Fig. 5e and f): the surface of the scale is smooth, and no erosion effect is visible in SEM images.

Formation of nano-roughness, which can derive from plasma etching, cannot be detected by SEM. However, the minor morphological changes observed with this analysis are one of the reasons of the retention of the touch and softness of the fabrics after the plasma exposure.

#### 4. Conclusions

The effect of plasma treatment of pure cashmere and wool/cashmere textiles with a dielectric barrier discharge (DBD) in humid air (air/water vapor mixtures) was investigated. Optimization of plasma parameters allowed the enhancement of the fabrics wettability without changing the bulk properties as well as the touch of the textiles. Wettability of the fabrics was found to increase by increasing the energy dose supplied during the plasma treatment. Moreover, better results in terms of wettability were obtained by supplying the total energy dose in multiple step, probably as a consequence of a more uniform surface modification.

The observed increment in wettability was found to be ascribable to a surface oxidation of the fibers, as assessed by FTIR/ATR spectroscopy and XPS analyses. Only minor etching effects were observed by characterization with scanning electron microscopy (SEM). This feature is probably essential for the maintenance of the textile softness.

## References

- [1] R. Morent, N. De Geyter, J. Verschuren, K. De Clerck, P. Kiekens, C. Leys, Non-thermal plasma treatment of textiles, *Surf. Coat. Technol.* 202 (2008) 3427–3449.
- [2] G. Buyle, Nanoscale finishing of textiles via plasma treatment, *Mater. Technol.* 24 (2009) 46–51.
- [3] U. Vohrer, M. Muller, C. Oehr, Glow-discharges treatment for the modification of textiles, *Surf. Coat. Technol.* 98 (1998) 1128–1131.
- [4] D. Hegemann, M. Mokbul Hossain, D.J. Balazs, Nanostructured plasma coatings to obtain multifunctional textile surfaces, *Prog. Org. Coat.* 58 (2007) 237–240.
- [5] S. Guimond, B. Hanselmann, M. Amberg, D. Hegemann, Plasma functionalization of textiles: specifics and possibilities, *Pure Appl. Chem.* 82 (2010) 1239–1245.
- [6] X.J. Dai, J.S. Church, M.G. Huson, Pulsed plasma polymerization of hexamethyldisiloxane onto wool: control of moisture vapor transmission rate and surface adhesion, *Plasma Process. Polym.* 6 (2009) 139–147.
- [7] S. Li, D. Jinjin, Improvement of hydrophobic properties of silk and cot-ton by hexafluoropropene plasma treatment, *Appl. Surf. Sci.* 253 (2007) 5051–5055.
- [8] E. Selli, G. Mazzone, C. Oliva, F. Martini, C. Riccardi, R. Barni, B. Marcandalli, M.R. Massafra, Characterisation of poly(ethylene terephthalate) and cotton fibres after cold SF<sub>6</sub> plasma treatment, *J. Mater. Chem.* 11 (2001) 1985–1991.
- [9] S. Sigurdsson, R. Shishoo, Surface properties of polymers treated with tetrafluoromethane plasma, *J. Appl. Polym. Sci.* 66 (1997) 1591–1601.
- [10] R. Barni, C. Riccardi, E. Selli, M.R. Massafra, B. Marcandalli, F. Orsini, G. Poletti, L. Meda, Wettability and dyeability modulation of poly(ethylene terephthalate) fibers through cold SF<sub>6</sub> plasma treatment, *Plasma Process. Polym.* 2 (2005) 64–72.
- [11] R. Molina, J.P. Espinos, F. Yubero, P. Erra, A.R. Gonzalez-Elipe, XPS analysis of down stream plasma treated wool: influence of the nature of the gas on the surface modification of wool, *Appl. Surf. Sci.* 252 (2005) 1417–1429.
- [12] A. Raffaele-Addamo, C. Riccardi, E. Selli, R. Barni, M. Piselli, G. Poletti, F. Orsini, B. Marcandalli, M.R. Massafra, L. Meda, Characterization of plasma processing for polymers, *Surf. Coat. Technol.* 174–175 (2003) 886–890.
- [13] G. Poletti, F. Orsini, A. Raffaele-Addamo, C. Riccardi, E. Selli, Cold plasma treatment of PET fabrics: AFM surface morphology characterisation, *Appl. Surf. Sci.* 219 (2003) 311–316.
- [14] G. Poletti, F. Orsini, C. Riccardi, A. Raffaele-Addamo, R. Barni, Atomic force microscopy investigation of cold-plasma-treated poly(ethyleneterephthalate) textiles, *Surf. Interface Anal.* 35 (2003) 410–412.
- [15] K.K. Wong, X.M. Tao, C.W.M. Yuen, K.W. Yeung, Wicking properties of linen treated with low temperature plasma, *Text. Res. J.* 71 (2001) 49–56.
- [16] A. Raffaele-Addamo, E. Selli, R. Barni, C. Riccardi, F. Orsini, G. Poletti, L. Meda, M.R. Massafra, B. Marcandalli, Cold plasma-induced modification of the dyeing properties of poly(ethylene terephthalate) fibers, *Appl. Surf. Sci.* 252 (2006) 2265–2275.
- [17] C. Riccardi, R. Barni, E. Selli, G. Mazzone, M.R. Massafra, B. Marcandalli, G. Poletti, Surface modification of poly(ethylene terephthalate) fibers induced by radio frequency air plasma treatment, *Appl. Surf. Sci.* 211 (2003) 386–397.
- [18] K. Navaneetha Pandiyaraj, V. Selvarajan, Non-thermal plasma treatment for hydrophilicity improvement of grey cotton fabrics, *J. Mater. Process. Technol.* 199 (2008) 130–139.
- [19] C.W. Kan, C.W.M. Yuen, Effect of low temperature plasma treatment on wool fabric properties, *Fiber Polym.* 6 (2005) 169–173.
- [20] P. Erra, R. Molina, D. Jovic, M.R. Julia, A. Cuesta, J.M.D. Tascon, Shrinkage properties of wool treated with low temperature plasma and chitosan biopolymer, *Text. Res. J.* 69 (1999) 811–815.
- [21] C. Canal, R. Molina, E. Bertran, A. Navarro, P. Erra, Effects of low temperature plasma on wool and wool/nylon blend dyed fabrics, *Fiber Polym.* 9 (2008) 293–300.
- [22] M. Mori, N. Inagaki, Relationship between anti-felting properties and physico-chemical properties of wool treated with low-temperature plasma, *Res. J. Text. Appar.* 10 (2006) 33–45.
- [23] C. Canal, R. Molina, E. Bertran, P. Erra, Study on the influence of scouring on the wettability of keratin fibers before plasma treatment, *Fiber Polym.* 9 (2008) 444–449.
- [24] C.W. Kan, K. Chan, C.W.M. Yuen, M.H. Miao, Low temperature plasma on wool substrates: the effect of the nature of the gas, *Text. Res. J.* 69 (1999) 407–416.
- [25] F. Rombaldoni, R. Mossotti, A. Montarsolo, M. Bianchetto Songia, R. Innocenti, G. Mazzuchetti, Thin film deposition by PECVD using HMDSO-O<sub>2</sub>-Ar gas mixture on knitted wool fabrics in order to improve pilling resistance, *Fiber Polym.* 9 (2008) 566–573.
- [26] R.A. Siliprandi, S. Zanini, E. Grimoldi, F.S. Fumagalli, R. Barni, C. Riccardi, Atmospheric pressure plasma discharge for polysiloxane thin films deposition and comparison with low pressure process, *Plasma Chem. Plasma Process.* 31 (2011) 353–372.
- [27] P. Esena, C. Riccardi, S. Zanini, M. Tontini, G. Poletti, F. Orsini, Surface modification of PET film by a DBD device at atmospheric pressure, *Surf. Coat. Technol.* 200 (2005) 664–667.
- [28] P. Esena, S. Zanini, C. Riccardi, Plasma processing for surface optical modifications of PET films, *Vacuum* 82 (2008) 232–235.
- [29] M.G. McCord, Y.J. Hwang, P.J. Hauser, Y. Qiu, J.J. Cuomo, O.E. Hankins, M.A. Bourham, L.K. Canup, Modifying nylon and polypropylene fabrics with atmospheric pressure plasmas, *Text. Res. J.* 72 (2002) 491–498.
- [30] K.K. Samanta, M. Jassal, A.K. Agrawal, Antistatic effect of atmospheric pressure glow discharge cold plasma treatment on textile substrates, *Fiber Polym.* 11 (2010) 431–437.
- [31] N.A. Ibrahim, B.M. Eid, M.M. Hashem, R. Refai, M. El-Hossamy, Smart options for functional finishing of linen-containing fabrics, *J. Ind. Text.* 39 (2010) 233–265.
- [32] K.K. Samanta, M. Jassal, A.K. Agrawal, Atmospheric pressure plasma polymerization of 1,3-butadiene for hydrophobic finishing of textile substrates, *J. Phys. Conf. Ser.* 208 (2010) 012098.
- [33] Y. Wang, The uniform Si-O coating on cotton fibers by an atmospheric pressure plasma treatment, *J. Macromol. Sci. B* 50 (2011) 1739–1746.
- [34] N. De Geyter, R. Morent, C. Leys, Surface modification of a polyester non-woven with a dielectric barrier discharge in air at medium pressure, *Surf. Coat. Technol.* 201 (2006) 2460–2466.
- [35] G. Borcia, C.A. Anderson, N.M.D. Brown, Surface treatment of natural and synthetic textiles using a dielectric barrier discharge, *Surf. Coat. Technol.* 201 (2006) 3074–3081.
- [36] S.Y. Cheng, C.W.M. Yuen, C.W. Kana, K.K.L. Cheuk, W.A. Daoud, P.L. Lam, W.Y.I. Tsoi, Influence of atmospheric pressure plasma treatment on various fibrous materials: performance properties and surface adhesion analysis, *Vacuum* 84 (2010) 1466–1470.
- [37] C.W. Kan, C.W.M. Yuen, W.Y.I. Tsoi, T.B. Tang, Plasma pretreatment for polymer deposition—improving antifelting properties of wool, *IEEE Trans. Plasma Sci.* 38 (2010) 1505–1511.
- [38] J. Janca, A. Czernichowski, Wool treatment in the gas flow from gliding discharge plasma at atmospheric pressure, *Surf. Coat. Technol.* 98 (1998) 1112–1115.
- [39] A. Ceria, F. Rombaldoni, G. Rovero, G. Mazzuchetti, S. Sicardi, The effect of an innovative atmospheric plasma jet treatment on physical and mechanical properties of wool fabrics, *J. Mater. Process. Technol.* 210 (2010) 720–726.
- [40] C.X. Wang, Y.P. Qiu, Two sided modification of wool fabrics by atmospheric pressure plasma jet: influence of processing parameters on plasma penetration, *Surf. Coat. Technol.* 201 (2007) 6273–6277.
- [41] E.C. Dell'Orto, A. Vaccaro, C. Riccardi, Morphological and chemical analysis of PP film treated by dielectric barrier discharge, *J. Phys. Conf. Ser.* 550 (2014) 012032.
- [42] C.W. Kan, K. Chan, C.W.M. Yuen, Surface characterization of low temperature plasma treated wool fiber – the effect of the nature of gas, *Fiber Polym.* 5 (2004) 52–58.
- [43] S. Hutchinson, D. Evans, G. Corino, J. Kattenbelt, An evaluation of the action of thioesterases on the surface of wool, *Enzyme Microb. Technol.* 40 (2007) 1794–1800.