

## Research Article

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# Enhancing the surface properties of linen by non-thermal atmospheric air-plasma treatment

**Abstract:** In this research, a diffuse coplanar surface barrier discharge (DCSBD) type plasma reactor was used for the surface modification of raw linen fabric. Changes in physical properties and chemical composition of the fiber surface as well as color of the fabric were measured as a function of time of the atmospheric air plasma treatment. Furthermore, ageing of the effects created on the fiber surface by plasma treatment was also characterized in a period of 0-14 days elapsed after the plasma treatment. Significant differences were found between the properties of the raw and plasma treated linen fabrics, including increase of wettability, wickability, surface energy and O/C ratio, and decrease of water contact angle and deterioration of the waxy outer layer of the fibers. Most of the parameters depended on the time of plasma treatment (0–180 s). O/C ratio increased steadily with the increase of duration of the plasma treatment, which was explained by destruction of the waxy surface layer, creation of polar groups and exposure of cellulosic components. Most of the properties tested were found to be stable during two weeks of storage after the plasma treatment, indicating that the surface ‘topography’ created by plasma remained almost unaltered and the recovery of the etched waxy coverage of the fiber did not occur.

**Keywords:** Hydrophilicity, Wetting, Wicking, Surface chemical composition, Ageing of plasma effects

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## 1 Introduction

In the last couple of years, there has been a growing interest in the use of plasma pretreatment for activation and modification of the surface layers of textile materials in order to increase their functionality. Enhancement of functionality of fibers and textiles with innovative finishes is an excellent opportunity for creating high-added value products. The non-thermal atmospheric plasma can be characterized by atomic temperatures close to ambient, but electron temperatures reaching values up to orders of magnitude higher. Plasma with such characteristics readily interacts with solid surfaces, causing reactions that would otherwise occur only at elevated temperature. In the field of textiles, plasma pretreatment represents a well-controlled and environmentally-friendly method to prepare reactive fiber surfaces [1-3].

It was proved that improved hydrophilicity and sorption properties of natural and synthetic fibers (i.e. wool, cotton, flax, silk and PET, PA6, PP) can be achieved by treatment with plasma. Results showed that the plasma modified the outer surface layers of the fiber and degraded the impurities on the surface and, as a consequence, the plasma treatment accelerated and intensified the subsequently applied chemical processes [4-9].

Linen is mainly composed of cellulose, which is located principally in the secondary cell walls. The non-cellulosic substances such as hemicelluloses, pectin, lignin, and some fats and waxes, present in raw linen constitute approximately 30% of the total fiber weight. They can be detected mainly in the middle lamella and the primary wall. The waxy outer layer makes the fibers hydrophobic and some of the non-cellulosics especially pectic materials act as cementing materials of the large numbers of ultimate cells [10]. It is well known that plasma treatment can etch and attenuate the outer layer of the fiber surface up to the depth of about 200 nm [4]. It means that plasma can partially destroy and remove the thin waxy outer layer of the fiber (which is about 12 nm

for raw cotton [11]), increasing the hydrophilicity of the plasma treated fabrics.

Although a number of shorter studies and comprehensive reviews have been published regarding the effects of plasma treatment on the surface properties of natural fibers, most of them concentrated on cotton and only a very few publications focused on the plasma-aided surface modification of linen [12,13]. Furthermore, little has been published on the ageing of the effects achieved by plasma treatment.

In this research, non-thermal atmospheric air-plasma was used for surface modification of raw linen fabrics. Linen is a multicellular fiber derived from the stem of the flax plant. It consists of long elementary fibers (approx. 2–5 cm) with a diameter of 10–25  $\mu\text{m}$ , which are ‘glued’ together by pectins into bundles [10]. Changes in physical properties and chemical composition of the fiber surface as well as color of the fabric were measured as a function of time of the atmospheric air-plasma treatment. Wettability and wickability were measured and the surface free energy of the fibers was calculated. Changes in the chemical composition of the fiber surface were detected by X-ray photoelectron spectroscopy (XPS) and infrared spectroscopy (FT-IR ATR). The ageing of the surface modifications created by plasma treatment was also tested by characterizing the permanency of the surface hydrophilicity and chemical composition as well as color of the fabric in a period of 1–14 days.

## 2 Experimental Procedure

### 2.1 Materials

100 per cent raw linen fabric, plain-weave, with a fabric weight per unit area of 250  $\text{g m}^{-2}$  was selected for the experiments. The fabric samples were conditioned at 65% relative humidity and 20°C for 24 hours prior to any of the treatments and testing. All chemicals used were of analytical grade and were purchased from Sigma-Aldrich Co. LLC. and Reanal Private Ltd.

### 2.2 Plasma treatment

Non-thermal plasma treatment was performed in ambient air, by a diffuse coplanar surface barrier discharge (DCSBD) type equipment (Roplass s.r.o., Czech Republic). Both sides of the textiles were treated, applying a power of 300 W and treatment times of 30, 60, 120, 180 s.

### 2.3 Analysis

Wettability of the plasma treated fabrics was characterized by water drop test, counting the elapsed seconds between the contact of the water drop with the fabric and the disappearance of the drop into the fabric. Ten readings were taken from different locations on the samples subsequent to the plasma treatment and the average was calculated.

Contact angle measurements were carried out at room temperature using a goniometer (Ramé-Hart Instruments Co., USA) with drop image standard software of DT-Acquire, using distilled water as a test liquid. For the experiments 2 cm  $\times$  4 cm of linen fabrics was used and the measurements were repeated five times.

Wickability was evaluated by the thin-layer wicking experiments. A strip of the plasma-treated fabric (2 cm  $\times$  10 cm) was vertically dipped to a depth of 1 cm into the test liquids and the time for wicking height of 2 cm was measured at  $20 \pm 0.2^\circ\text{C}$ . In the experiments, n-heptane was used as a total wetting liquid. Water and  $\alpha$ -bromonaphthalene were used as high-energy liquids. Wicking rate was measured and the apparent (effective) pore radius ( $R$ ), the dynamic contact angle, the dispersion and polar components of the solid surface and the free energy of the fabric ( $\gamma_s^{\text{total}}$ ) were calculated [14–15]. At least eight wicking measurements were made for each fabric in warp direction and the averaged wicking time was used for the calculations.

XPS studies were done by a Kratos XSAM 800 spectrometer (Kratos Analytical Ltd., UK) using  $\text{Mg K}\alpha_{1,2}$  radiation at a power of 225 W and fixed analyzer transmission mode (80 and 40 eV pass energies for survey and detailed spectra, respectively). Survey spectra were recorded at kinetic energy of 100–1300 eV. Average spectra were calculated of four parallel measurements. Data collection and deconvolution analyses of C1s peaks were performed using Kratos Vision 2 software.

FT-IR measurements of the fabrics of area of 1 mm  $\times$  1 mm were carried out applying a Tensor 27 (Bruker) spectrophotometer with a diamond ATR cell (Bruker Platinum ATR) in the wavenumber range of 4000–400  $\text{cm}^{-1}$  using 2  $\text{cm}^{-1}$  resolution and 32 scans. Relative peak intensities were also calculated in comparison to 609  $\text{cm}^{-1}$ , which can be assigned to the OH out-of-plane bending in cellulose [16].

Color measurement was performed using a Hunterlab Color QUEST (D65/10°) colorimeter. Color evaluation was done according to the CIE Lab color space. Each sample measurement represents the average of five readings from different positions on the swatch. Effect of plasma treatment was characterized by the color difference

values ( $\Delta H_{ab}^*$ ,  $\Delta L_{ab}^*$ ,  $\Delta C_{ab}^*$  and  $\Delta E_{ab}^*$ ), which were based on the color of the untreated fabric as well as the Berger whiteness ( $WI_{Berger}$ ) and E313 yellowness ( $YI_{E313}$ ) indices of the linen samples.

In order to characterize the ageing of the effects created by plasma (for 30, 60 and 180 seconds), the plasma treated fabrics were stored at 65% relative humidity and 20°C for 0–14 days and the surface properties and their changes with the time elapsed after the plasma treatment (0, 1, 7 and 14 days) were evaluated by some of the methods (i.e. water drop test, contact angle, and color measurements) described above.

### 3 Results and Discussion

#### 3.1 Surface hydrophilicity

Changes induced by air-plasma treatment in the hydrophilicity of surface of the linen were characterized

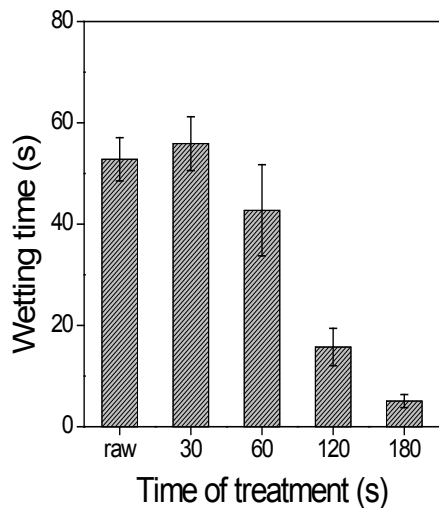


Figure 1: Wettability of raw and plasma-treated linen fabrics as a function of time of plasma treatment.

by measuring the wetting and wicking properties as well as water contact angle of the fabrics. For the untreated raw fabric, a wetting time of 52.8 s was obtained (Fig. 1), which decreased to 5.1 s, when the plasma treatment was applied for 180 s. These results are in qualitative and quantitative agreement with the corresponding water contact angle data, which are presented in Fig. 2 for various times. Thus, the water contact angle decreases strongly upon plasma treatment, reflecting an increase in surface hydrophilicity.

In order to differentiate the effectiveness of the applied plasma treatments in improving the surface properties of raw linen fabric more precisely and to calculate the surface energy, the wicking properties of the plasma treated fabrics were also evaluated by the thin-layer wicking experiments, using n-heptane, water and  $\alpha$ -bromo-naphthalene solvents. The wicking rate values of the untreated and plasma treated fabrics were determined by measuring the time for wicking height of 2 cm. Table 1 presents the apparent pore radius (R) and the surface energy ( $\gamma_s^{total}$ ) data. Results measured after the plasma treatment reveal that changes in surface properties induced by plasma treatment are accompanied by the changes in capillary spaces and pores in the fabric. The apparent capillary radius increased from 4.95  $\mu\text{m}$  to 7.45  $\mu\text{m}$  following plasma treatment (for 180 s), and a similar increase in surface free energy can also be observed. Thus, the untreated raw linen with a surface covered by waxy materials can be characterized by a  $\gamma_s^{total}$  value of 26.2  $\text{mJ m}^{-2}$ , which increased to 56.1  $\text{mJ m}^{-2}$  after 180 s plasma treatment.

Ageing of the plasma effects was characterized by water drop and water contact angle tests. Fig. 3 shows the relative wetting time as a function of time elapsed after plasma treatment. Results reveal that the relative wetting time values of the plasma treated fabrics (30, 60, 180 s) did not vary significantly in a period of 1–14 days and remained near the original value measured immediately after the plasma treatment (100%). This means that

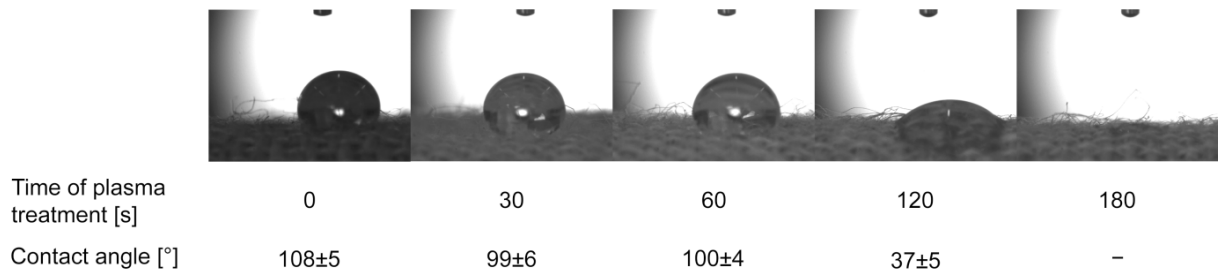


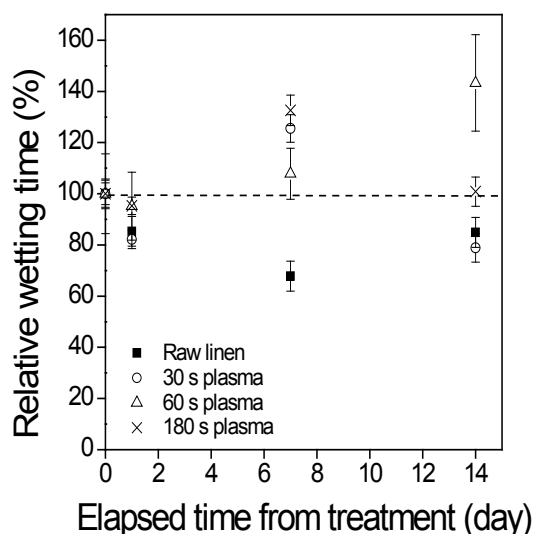
Figure 2: Water contact angle of raw and plasma treated linen fabrics as a function of time of plasma treatment.

**Table 1:** Effect of air-plasma treatment on the wickability of the linen fabrics. Changes in apparent capillary radius ( $R$ ) and surface free energy ( $\gamma_s^{\text{total}}$ ) as a function of time of plasma treatment.

Time of plasma treatment (s)	0	30	60	120	180
$R$ ( $\mu\text{m}$ )	$4.95 \pm 0.4$	$6.09 \pm 0.5$	$6.09 \pm 0.2$	$5.85 \pm 0.3$	$7.45 \pm 0.1$
$\gamma_s^{\text{total}}$ ( $\text{mJ m}^{-2}$ )	$26.2 \pm 1.1$	$44.9 \pm 2$	$48.4 \pm 3.7$	$52.6 \pm 4.2$	$56.1 \pm 6.3$

**Table 2:** Ageing of the plasma effects characterized by changing in water contact angle (CA) as a function of time elapsed from the plasma treatment.<sup>a</sup>

Time elapsed after plasma treatment (day)								
	0		1		7		14	
Sample	raw	plasma-treated	raw	plasma-treated	raw	plasma-treated	raw	plasma-treated
CA ( $^\circ$ )	$101 \pm 6$	$81 \pm 5$	$88 \pm 13$	$67 \pm 12$	$108 \pm 6$	$75 \pm 10$	$115 \pm 4$	$90 \pm 5$
$\Delta\text{CA}$ ( $^\circ$ )	$20 \pm 5.5$		$21 \pm 12.5$		$33 \pm 8$		$25 \pm 4.5$	

<sup>a</sup> Time of plasma treatment of linen fabric: 60 s**Figure 3:** Relative wetting time versus time elapsed after plasma treatment of linen fabrics.

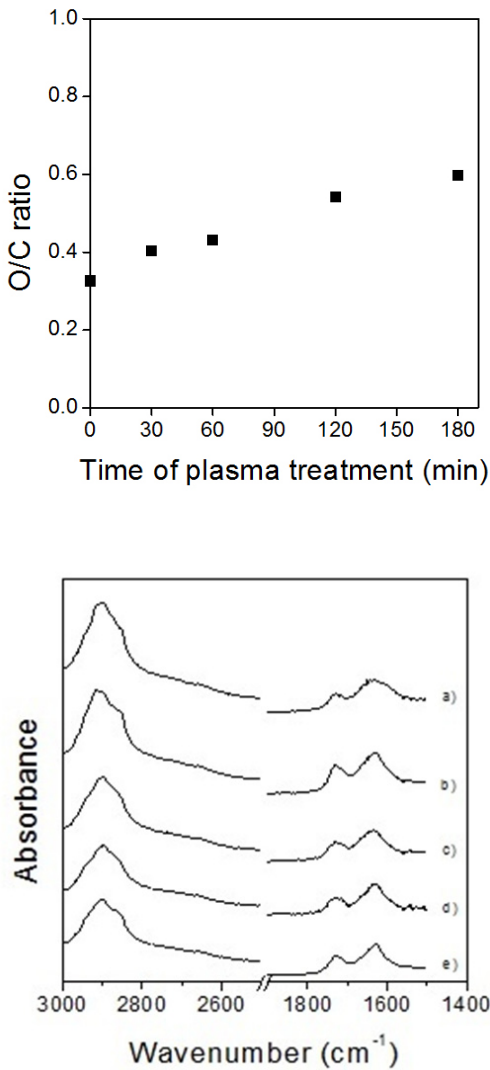
surface hydrophilicity of the plasma treated linen fabrics characterized by water wetting proved to be stable even two weeks, indicating the durability of the effects created by plasma treatment. A similar statement can be concluded from the contact angle. Difference in contact angle between the raw and plasma treated samples can be considered to be stable, even after two weeks.

### 3.2 Surface chemical composition

In order to support and explain more precisely the above data on plasma induced changes in physical properties of

the linen fabric, chemical composition of the fiber surface was characterized by XPS and FT-IR ATR methods (Fig. 4, upper and lower, respectively). XPS is a widely applied method to get information on the chemical composition of the surface of natural fibers and the differently bonded carbon and oxygen atoms as well. The O/C atomic ratio for the surface of raw and plasma treated linen samples as a function of time of plasma treatment is given in Fig. 4 (upper). The C 1s peaks for the raw and plasma treated (for 180 s) linen are shown in Fig. 5.

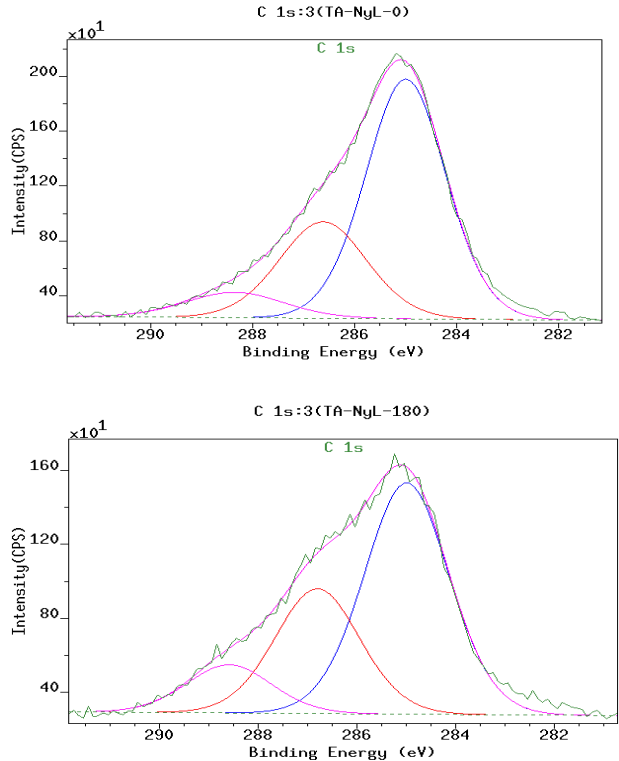
It is remarkable that the surface O/C atomic ratio (Fig. 4, upper) found for the untreated raw linen is 0.326. Data reported for the theoretical O/C ratios of cellulose (0.83), lignin (0.31 and 0.4), waxy materials (0.11) [18,19] and pectin (0.94, unpublished data) suggest that XPS most likely measures (in about 10 nm depth) the waxy outer layer of the linen fiber and the pectin present mainly in the middle lamella rather than lignin, since its amount is negligible in the outermost surface of linen. The O/C ratio increased steadily with the increase of duration of the plasma treatment from 0.326 to 0.597, indicating the etching and oxidative effects of air-plasma on the waxy surface layer and the polymers present. The changes induced by air-plasma treatment in the intensities of components of the C 1s peaks (Table 3, Fig. 5) corroborate a slight oxidation and the partial degradation of the waxy materials. Upon plasma treatment, the proportion of the C1 component (285.0 eV, pertaining to carbon C-C and C-H bonds) decreased especially at longer treatments (120 and 180 s), while those of C3 (288.3 eV, carbon in C=O and O-C-O) increased significantly and steadily with the time of the plasma treatment, suggesting the introduction of



**Figure 4:** Oxygen-to-carbon ratio from XPS survey spectra (upper) and FT-IR ATR spectra (lower) for the linen samples as a function of time of air-plasma treatment. For FT-IR ATR spectra: a) 0 s (raw), b) 30 s, c) 60 s, d) 120 s, e) 180 s.

**Table 3:** Results of decomposition of the C 1s peak for the surfaces of untreated and air-plasma treated linen as a function of time of plasma treatment.

Sample	C1 %	C2 %	C3 %
	285.0 eV C–C, C–H	286.7 eV C–O	288.5 eV C=O, O–C–O
Raw linen	67	27	6
Plasma treated - 30 s	67	25	8
Plasma treated - 60 s	67	24	9
Plasma treated - 120 s	65	24	10
Plasma treated - 180 s	54	28	17



**Figure 5:** Decomposition of the C 1s peak. Upper: raw, untreated linen; lower: plasma treated for 180 s.

some hydrophilic groups to the surface by plasma-aided oxidation. Plasma induced changes in the amount of carbon present as C2 (286.7 eV, carbon in C-OH and C-O-C) moiety are only negligible.

Observations based on the FT-IR ATR measurements are in agreement with the corresponding XPS statements. In Fig. 4, lower, the spectra of the raw (a) and plasma treated fabrics as a function of time (b-e) can be observed. The peak at 2850 cm<sup>-1</sup> is associated with the asymmetric and the symmetric stretching of methylene groups in long alkyl chains and indicates the presence of the waxy materials in the fiber surface [20]. The time dependence for the relative intensities of this peak shows a decrease upon air-plasma treatment (Table 4), indicating the partial removal of the waxy materials from the fiber surface.

Furthermore, the FT-IR ATR spectra also reveal significant differences in the ester region at ~1730 cm<sup>-1</sup> (Fig. 4, lower), which are related to the pectin molecules, since approximately 70% of carboxyl groups of galacturonic acid units in pectin are esterified with a methoxyl group [21]. Depending on the treatment time, the relative intensities of this peak increased from 0.34 to 0.53 (Table 4), which indicate that etching of the fiber surface by air-plasma can expose the pectin molecules



**Table 4:** Relative intensities for IR absorption bands (compared to 609 cm<sup>-1</sup>) of the plasma treated linen fabrics as a function of time of plasma treatment.

Wavenumber (cm <sup>-1</sup> )	Time of plasma treatment (s)				
	0	30	60	120	180
1730	0.34	0.47	0.38	0.38	0.53
2850	1.27	1.25	1.17	1.11	1.3

**Table 5:** Effect of plasma treatment on the color of the linen fabric. Whiteness and yellowness indices and color difference values as a function of time of plasma treatment.<sup>a</sup>

Time of plasma treatment (s)	WI <sub>Berger</sub>	YI <sub>E313</sub>	$\Delta H_{ab}^*$	$\Delta L_{ab}^*$	$\Delta C_{ab}^*$	$\Delta E_{ab}^*$
0	2.8	31.5	-	-	-	-
30	2.6	31.5	-0.4	-0.6	-0.1	0.7
60	2.1	32.1	-0.4	-0.5	0.1	0.6
120	1.8	32.5	-0.4	0.2	0.4	0.6
180	1.9	32.3	-0.4	0.3	0.3	0.6

<sup>a</sup>Calculation of the color difference values ( $\Delta H_{ab}^*$ ,  $\Delta L_{ab}^*$ ,  $\Delta C_{ab}^*$  and  $\Delta E_{ab}^*$ ) was based on the color of the untreated raw fabric.

**Table 6:** Ageing of the color created by plasma treatment of the linen fabric. CIELab color coordinates, whiteness and yellowness indices as a function of time elapsed from the plasma treatment.<sup>a</sup>

Time elapsed after plasma treatment (day)	L*	a*	b*	WI <sub>Berger</sub>	YI <sub>E313</sub>
0	64.1	2.3	11.5	2.6	31.7
1	63.9	2.4	11.7	1.8	32.5
7	64.3	2.4	11.9	1.7	32.6
14	64.2	2.3	11.7	2.2	32.2

<sup>a</sup>Time of plasma treatment: 180 s

(and probably the other polymer constituents, i.e. cellulose and hemicelluloses of the primary and secondary cell walls) and makes them more detectable on the fiber surface.

### 3.3 Changes in color

The effect of plasma treatment on color of the linen fabrics was also evaluated. Results in Table 5 reveal that air-plasma treatment slightly decreased the whiteness ( $\Delta WI_{\text{Berger}}$  from 2.8 to 1.9) and increased the yellowness ( $\Delta YI_{\text{E313}}$  from 31.5 to 32.3) of the linen

fabrics and brought about perceptible color differences ( $\Delta E_{ab}^* > 0.5$ ) between the untreated and plasma treated fabrics, even after the shortest plasma treatment (30 s). Analysis of the constituents of  $\Delta E_{ab}^*$  shows that plasma treatment affected the hue of the fabrics and the color had a slight reddish shade. Furthermore, the color of the plasma treated samples was more saturated (positive value for  $\Delta C_{ab}^*$ ) and for the longer treatments it was slightly lighter (positive value for  $\Delta L_{ab}^*$ ). At this point it should be noted that a more significant change in color of the linen fabric ( $\Delta E_{ab}^* = 1.6$ ) was observed after plasma treatment with a uniform glow discharge plasma system operating under atmospheric conditions [22].

Ageing of the color achieved by plasma treatment for 180 s was also evaluated. Results in Table 6 show the color coordinates (L\*, a\* and b\*) and the whiteness and yellowness indices measured after plasma treatment in a period of 1–14 days. All the data confirm that the slightly reddish shade of the color of the plasma treated fabric remained unchanged, indicating the stability of the plasma effect.

## 4 Conclusions

In this research, atmospheric pressure air-plasma treatment was applied on raw linen fabric in order to modify the surface of the fibers. All of the applied analytical methods revealed differences in properties of the raw and the plasma treated fabrics and also differences as a function of time of the plasma treatment. In general, air-plasma treatment leads to a significant increase in hydrophilicity, characterized by wicking and wetting properties and water contact angle, and to a slight but perceptible change in color of the fabrics. Furthermore, plasma treatment creates modified surfaces where the O/C ratio measured by XPS is significantly higher than that of the original raw fiber. The O/C ratio increased steadily with increasing the duration of the plasma treatment. This can be explained by mainly the destruction of the waxy surface layer and creation of polar groups by plasma-aided oxidation as well as exposure of pectin and other polymer components. Results obtained by FT-IR ATR technique indicated the exposure of pectin, which became more and more detectable on the surface with increasing the duration of the plasma treatment. Some of the properties such as wettability, wickability and color were tested to evaluate the ageing of the effects created by plasma treatment. Most of the results demonstrated the stability of the changes induced by plasma.

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