

# Contact Angle Determination on Plasma-Treated Poly(ethylene terephthalate) Fabrics and Foils

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Received 28 November 2005; accepted 14 February 2006

DOI 10.1002/app.24308

Published online in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** The surfaces of polyester (PET) fabrics and foils were modified by low-pressure RF plasmas with air, CO<sub>2</sub>, water vapor as well as Ar/O<sub>2</sub> and He/O<sub>2</sub> mixtures. To increase the wettability of the fabrics, the plasma processing parameters were optimized by means of a suction test with water. It was found that low pressure (10–16 Pa) and medium power (10–16 W) yielded a good penetration of plasma species in the textile structure for all oxygen-containing gases and gaseous mixtures used. While the wettability of the PET fabric was increased in all cases, the Ar/O<sub>2</sub> plasma revealed the best hydrophilization effect with re-

spect to water suction and aging. The hydrophilization of PET fabrics was closely related to the surface oxidation and was characterized by XPS analysis. Static and advancing contact angles were determined from the capillary rise with water. Both wetting and aging demonstrated a good comparability between plasma-treated PET fabrics and foils, thus indicating a uniform treatment. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 1452–1458, 2006

**Key words:** PET fabric; RF plasma; wettability; capillary rise; suction test

## INTRODUCTION

The plasma treatment of fabrics has gained in importance over the past years as a textile finishing process for technical and medical textiles as well as for composite materials to improve their surface properties like wettability and adhesion.<sup>1–8</sup> Compared to conventional wet-chemical textile finishing, plasma technology is also of increasing interest because of its environmental sustainability.<sup>3,9</sup>

Plasma processing, a developing field of applied physics and chemistry,<sup>10</sup> modifies the outermost layers of the fabric surface in the nanometer range, while the bulk properties as well as the quality of the material remain unaffected.<sup>11–14</sup> Poly(ethylene terephthalate) (PET) fabrics made of manmade-fiber reveal a moderate hydrophilic character due to the lack of polar groups. Moreover, hydrophobic sizes are generally used during the fabric production.<sup>15</sup> To obtain a good wettability, i.e., a high surface energy, some polar groups such as hydroxyl (—OH) or carboxyl (—COOH) should thus be formed at the fabric surface.<sup>6,16</sup> For the hydrophilization of fabrics, a low pressure and low temperature plasma treatment with ox-

xygen-containing gases is expected to enable a good penetration of the textile structure because of long-living oxygen radicals.<sup>17</sup> Poll et al. considered the working pressure as the most important parameter to obtain a mean free path in the gas phase, which should be lower than textile distances.<sup>11</sup> Therefore, we performed RF plasma discharges with air, CO<sub>2</sub>, water vapor, He/O<sub>2</sub>, and Ar/O<sub>2</sub> mixtures by varying pressure, power, and treatment time.

Contact angles describing the liquid–vapor and liquid–solid interfaces are widely used for the study of the wetting/nonwetting phenomena on a solid material. Interfacial tension can be derived on flat, homogeneous surfaces by using liquids with different surface tensions.<sup>18</sup> For heterogeneous structures such as textile fabrics, however, the contact angle is not only affected by interfacial tension, but also by other phenomena such as surface roughness, chemical heterogeneity, polar groups, sorption layers, suction, porosity, swelling, molecular orientation, yarn tension etc. which complicate the direct measurement.<sup>19,20</sup> Contact angle measurements on single fibers are described in numerous reviews and articles mainly based on the wetting force measurement by the Wilhelmy principle.<sup>21–24</sup> However, this process is not suitable for woven fabrics because of liquid uptake in the pore structure by capillary forces.<sup>24</sup>

In this study, the contact angle on a plasma-treated PET fabric was determined by observing the capillary rise as a function of time on vertically attached fabric

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Contract grant sponsor: The Commission of Technology and Innovation (CTI), Bern, Switzerland.

strips with the lower end being immersed in the wetting liquid. According to Poiseuille's law, the theoretical basis of this capillary rise test was described by Washburn.<sup>25</sup> The rate of liquid penetration into a porous body is given by:

$$\frac{dH}{dt} = \frac{R_D^2}{8\eta H} \left( \frac{2\gamma \cos\theta_A}{R_S} - \rho g H \right) \quad (1)$$

where  $H$  is the height reached by the liquid at time  $t$ ,  $R_D$  the mean hydrodynamic radius of pores,  $\eta$  the viscosity of the liquid,  $\gamma$  and  $\rho$  the surface tension and density of the liquid on the solid, respectively,  $\theta_A$  the advancing contact angle of the liquid on the solid, which is normally larger than the static one, and  $g$  the acceleration due to gravity.  $R_S$  represents the mean static radius of pores which is a constant equal to the geometrical radius of pores. The hydrodynamic radius  $R_D$ , on the other hand, also depends on the tortuosity of the pores,<sup>26</sup> which is not directly related to geometrical considerations.<sup>27</sup>

At first, hydrostatic pressure might be neglected and the integration of eq. (1) yields the modified Washburn equation:

$$H^2 = \frac{R\gamma \cos\theta_A}{2\eta} t \quad (2)$$

where  $R = \frac{R_D^2}{R_S}$ . Thus, a plot of  $H^2$  versus  $t$  should be linear giving the wicking coefficient or so-called capillary diffusion coefficient  $D_C = \frac{R\gamma \cos\theta_A}{2\eta}$  that is related to the porosity of the fabric and properties of the liquid  $\rho$  and  $\eta$ .<sup>28</sup>

At equilibrium, when capillary and hydrodynamic forces are equal, the maximum height is reached by the liquid at:

$$H_{Eq} = \frac{2\gamma \cos\theta_{Eq}}{R_S \rho g} \quad (3)$$

If the hydrostatic pressure can not be neglected, eq. (1) can be written as

$$\frac{R_D^2 \rho g}{8\eta} dt = \left( \frac{H}{H_{Eq} - H} \right) dH \quad (4a)$$

by substitution of  $\cos\theta_{Eq}$  using eq. (3), which gives

$$\frac{R_D^2 \rho g}{8\eta} t = H_{Eq} \cdot \ln \frac{H_{Eq}}{H_{Eq} - H} - H \quad (4b)$$

or simply,  $Ct = H_1$ .

$R_D$  can be calculated from the slope of the straight line, where  $C$  is a coefficient depending only on the size of the capillaries and the nature of the liquid.<sup>29</sup>

Considering that a liquid with a low surface tension such as hexane, decahydronaphthalene (decaline), or octane yields a complete wetting of the hydrophilized fabric,<sup>23,27</sup> it can be assumed that the static contact angle  $\theta_{Eq}$  equals zero, i.e.,  $\cos\theta_{Eq}$  is one, and

$$R_S = \frac{2\gamma}{H_{Eq} \rho g} \quad (5)$$

Thus, the mean static radius of pores can be determined through the capillary rise of a completely wetting liquid. Consequently, water contact angles, both static [using eqs. (3) and (5)] and advancing [using eqs. (2), (4 (b), and (5))], on plasma-treated PET fabrics can be calculated to evaluate the degree of hydrophilicity using some modified forms of Washburn's equation derived from Poiseuille's law, as discussed above.

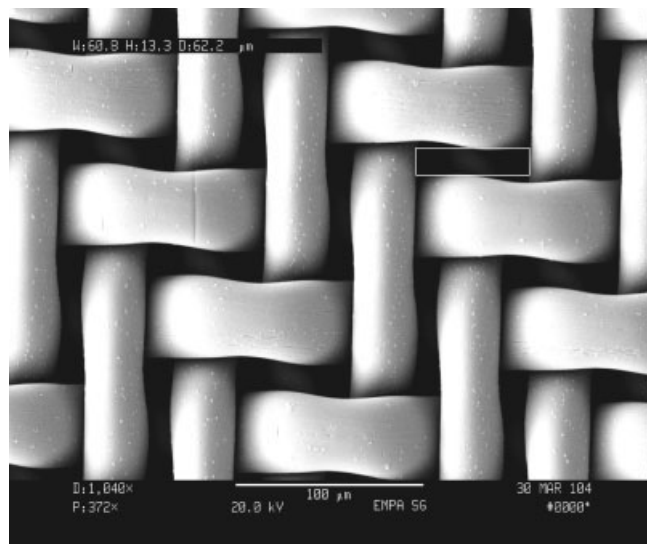
At first, plasma process parameters were systematically optimized in this study by means of a suction test. Second, the capillary rise test was used to compare the apparent contact angles within the textile fabric to PET foils treated by using the same plasma parameters. An aging study for both fabrics and foils was carried out with the aim of evaluating the penetration of the fabric structure by the plasma species. The chemical composition at the fabric surface was investigated by means of XPS.

## EXPERIMENTAL

### Plasma treatment

The single layer, tightly woven PET fabric (EPI 76, PPI 76, GSM 43.5) used in this study was supplied by Sefar Inc., Switzerland (Fig. 1). The fabrics and foils (50  $\mu\text{m}$  in thickness) were cut into pieces of 10  $\times$  15  $\text{cm}^2$  for plasma processing. The untreated foils showed a static contact angle of  $\sim 70^\circ$ .

To enhance the penetration of reactive plasma species into the textile structure in the micrometer range,<sup>11</sup> plasma treatments were carried out at low pressure to hydrophilize the fabric and foils. A capacitively coupled RF batch reactor (13.56 MHz) was used as schematically shown in Figure 2. A defined geometry and power coupling allowed reliable plasma treatments at varying conditions (power, pressure, treatment time, gases). Fabrics and foils were processed at the internal, driven electrode (10  $\times$  15  $\text{cm}^2$ ), while the cylindrical recipient acted as grounded electrode. A shower head above the electrode enabled homogeneous treatments over the entire electrode area. The vacuum system composed of a rotary pump and a roots pump generated a base pressure of  $10^{-2}$  Pa. The working pressure under gas flow was mea-



**Figure 1** Scanning electron micrograph of the used PET fabric. The mesh opening is  $13.3 \mu\text{m}$  wide.

sured by a Baratron pressure gauge (MKS Instruments, Germany) which was adjusted by a throttling valve.  $V/I$  probe measurements (ENI model 1640) indicated an absorbed power of 70% and a ratio of (negative) bias to excitation voltage of 0.8 due to the asymmetric setup.

To optimize the plasma treatments, an extensive parametric study was performed by varying important plasma parameters such as pressure (1.5–60 Pa), power (5–40 W), treatment time (0.5–5 min), and process gas (air,  $\text{CO}_2$ , water vapor,  $\text{Ar}/\text{O}_2$ , and  $\text{He}/\text{O}_2$  as well as pure Ar). The plasma-treated samples were then kept in a conditioned room ( $20 \pm 2^\circ\text{C}$ ,  $65 \pm 2\%$  RH) for further experiments.

### Suction test

When the contact angle of water on a plasma-treated PET fabric is below a certain level, the water droplet is sucked in, which was the case after all plasma treatments performed. To investigate different plasma conditions, a simple way to measure wetted area was used in our laboratory and combined with a camera and a computer-controlled program (MatLab). A normal syringe was used to drop a defined quantity of water ( $50 \mu\text{L}$ ) onto the tensioned sample, and pictures of the spreading droplet were taken as a function of time to derive the wetted area.

### Capillary rise test

For the capillary rise test, a Camag chromatogram immersion device III (Germany) composed of a tank filled with the particular liquid, a micrometer scale, and an adjustable carrier was used. The textile sam-

ples were cut into  $15 \times 2 \text{ cm}^2$  strips parallel to the warp direction. The strips were mounted in parallel to the scale, where its zero point maintained contact with the liquid surface in the tank, and were partly immersed into the liquid (at a depth of 1.5 cm). A light weight, which should not affect the geometrical structure of the sample, was placed at the end of the strip to keep it in a vertical position. The samples were conditioned before being tested. Bi-distilled water and decaline (Merck, Germany) were used for the capillary flow. The capillary rise was observed visually and the capillary heights were recorded every 5 s in the first minute, every 10 s in the following 6 min, and after 1.5–2 h, the equilibrium height readings were also recorded both for decaline and water. It is noted that the decaline equilibrium heights were the same for every experiment, thus verifying the assumption of a complete wetting. Whenever the liquid front did not rise evenly, an average value was taken.

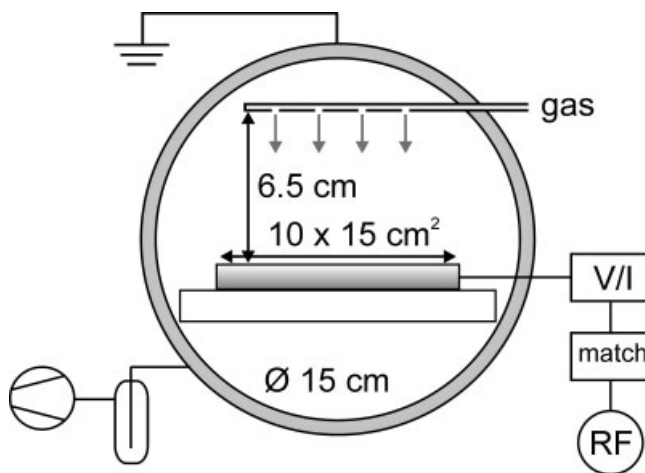
### Material characterization

The characteristics of the fabric surface were examined by scanning electron microscopy (SEM) using AM-RAY 3200-C-Eco-SEM and electron spectroscopy for chemical analysis (ESCA, PHI 5600).

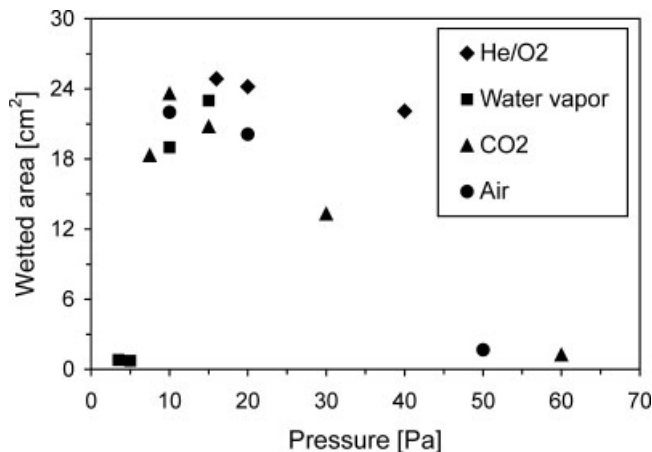
## RESULTS AND DISCUSSION

### Plasma treatment

To optimize the process parameters and to obtain a suitable gas or gaseous mixture, treatments were carried out at varying pressure, power, and exposure times. It is obvious that due to physical and chemical changes on fabric surfaces, plasma treatments have a significant effect on surface tension which contributes to the fabric wettability. Most of all, pressure has a strong influence on gas discharge, mainly its temper-



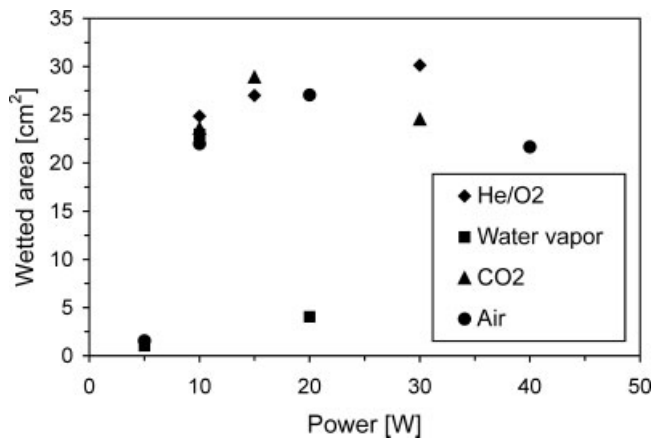
**Figure 2** Schematic drawing of the plasma reactor used.



**Figure 3** Effect of gas pressure on the suction of a water droplet into plasma-treated PET fabrics using different gases (1.5 min, 10 W).

ature, activation rate, flow rate, and plasma-induced molecular fragmentation due to collisions resulting in excitation and recombination processes.<sup>30</sup> The variation in pressure at a fixed exposure time (1.5 min) and power (15 W) is shown in Figure 3. The wettability of the fabric is mostly improved at low pressure (10–16 Pa) because of the more intense gas-phase molecular fragmentation. Conversely for a higher pressure, wettability is reduced significantly because of a low activation rate, which leads to a reduction in molecular fragmentation within the gas phase. It should be mentioned that oxygen-containing gases were used, because long-living atomic oxygen radicals are able to penetrate into the textile fabric, and the mean free path length of about 400  $\mu\text{m}$  was long compared to the textile distances of the fabric (<15  $\mu\text{m}$ ).

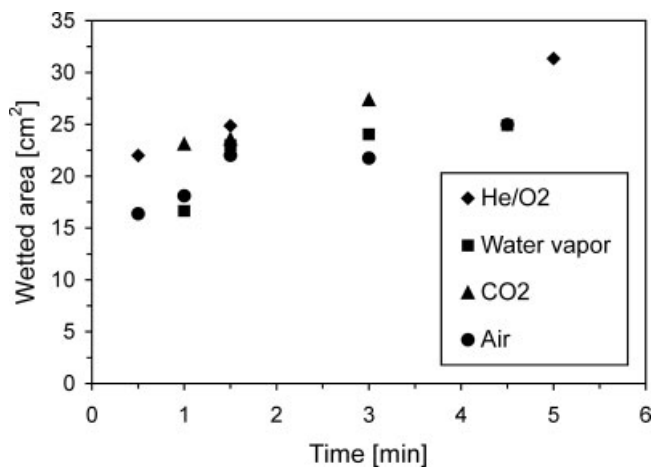
The variation of power revealed that a power input of 10–15 W already yields a maximum wetted area, as can be seen from Figure 4. At a lower power of 5 W, the formation of polar groups was slow at short exposure times, resulting in low wettability.<sup>13</sup> At a fixed power (10–15 W) and pressure (10–16 Pa), as shown in Figure 5, the fabric wettability gradually increased with an increase in treatment time. However, a good hydrophilization effect was already obtained after a short plasma exposure. Moreover, from Figures 3–5, it was found that a He/O<sub>2</sub> mixture performed best compared to CO<sub>2</sub>, air, and water vapor with respect to the wetted area using the suction test. Ar/O<sub>2</sub> mixtures were found to be comparable to He/O<sub>2</sub> at the derived optimum parameters of 5-min treatment time, 16 Pa pressure, and 15 W power input for the hydrophilization of the used PET fabric. Addition of oxygen to Ar or He yields more excited species in the plasma zone, such as long-living He\* and Ar\* metastables and long-living O atoms, which are able to penetrate into textile structures.



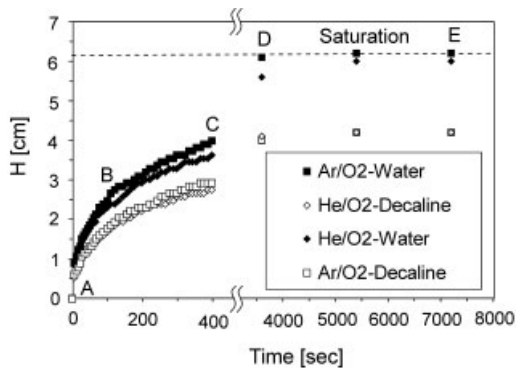
**Figure 4** Effect of power input on the wetted area of plasma-treated PET fabrics using different gases (1.5 min, 10–16 Pa).

### Capillary rise analysis

Capillary rise within a textile is influenced by a number of factors, especially fabric structure (yarn count, fabric density, weave design, porosity, fiber content etc.), which will be investigated in greater detail in a further study to be published elsewhere.<sup>31</sup> Here, the spontaneous liquid wicking of He/O<sub>2</sub> or Ar/O<sub>2</sub> plasma-treated PET fabrics is reported as a function of time. It is evident from the capillary rise  $H$  over time  $t$ , as shown in Figure 6, that in all cases after plasma treatment, the capillary rise with water is remarkably improved because of surface oxidation, whereas without treatment, no rise can be obtained. In region A–B, the liquid wicks spontaneously into the fabric because of the capillary pressure, where acceleration due to gravity in Washburn's equation (eq. (2)) is neglected. The B–C region of the curves illustrates the subsequent capillary rise, which in-



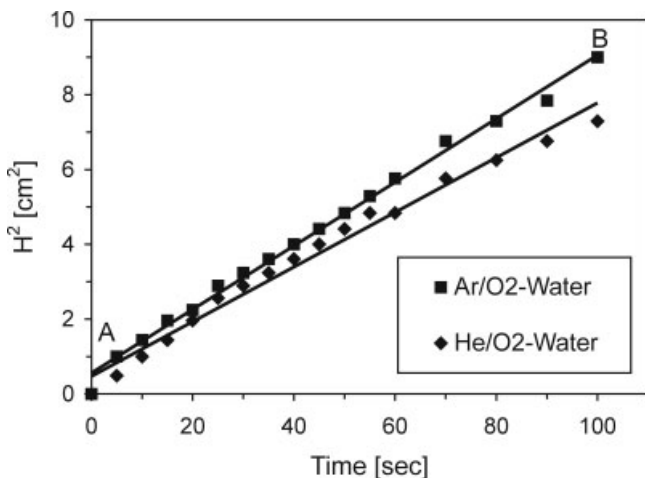
**Figure 5** Water-wetted area of plasma-treated PET fabrics using different gases depending on treatment time (10 W, 10–16 Pa).



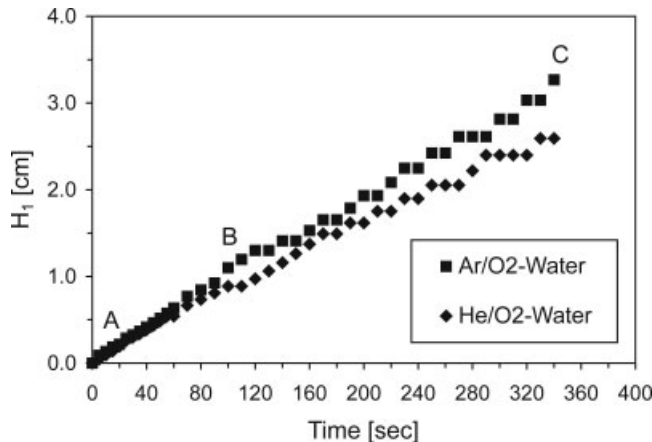
**Figure 6** Capillary rise obtained on plasma-treated PET fabrics using water and decaline as liquid.

creases very slowly before reaching the equilibrium state (D–E). At point D, the liquid front reaches its maximum height. When capillary and hydrodynamic forces are equal (D–E), no further rise is observed.

It was found that the capillary rise and equilibrium height of decaline was the same for all experiments performed because of its excellent wetting properties. Thus, the cosine of  $\theta_{Eq}$  (static contact angle) is one and the static radius of pores can be easily determined from eq. (5). In the spontaneous region of wicking (A–B), the derived curve from the capillary rise data is shown in Figure 7, showing that the square root of capillary rise  $H^2$  is proportional to time  $t$ . This linear curve is valid for short times according to the Washburn equation [eq. (2)]. Under conditions of prolonged rising (region B–D), this equation becomes inadequate and hydrostatic pressure has to be taken into account. This behavior is well described by Zhmud et al.<sup>32</sup> Evidently, the analysis of the linear fit ( $H^2 - t$  curve) is valid in the experimental time range of up to 100 s



**Figure 7** Square root of capillary rise on plasma-treated PET fabrics depending on time of rise.

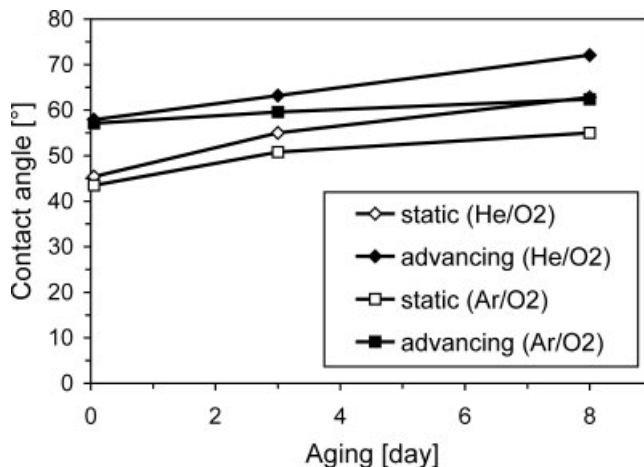


**Figure 8** Linear dependence of  $H_1$  values [see eq. (4b)] on time calculated from capillary rise data on plasma-treated PET fabrics.

(A–B) for the examined fabric. If all data and equilibrium height are taken into account, straight lines are also obtained for  $H_1$  versus  $t$  (Fig. 8), proving the validity of the presented evaluation, and the constant C can be determined from the slope of this curve as described by eq. [4(b)]. Thus, hydrodynamic and static radii can be calculated yielding  $\theta_A$  and  $\theta_{Eq}$ .

**Contact angle**

As described earlier, contact angles, both static and advancing, can be obtained from the capillary rise test. It is clearly shown in Figure 9 that static contact angles are smaller than the corresponding advancing ones (as it is expected). Both contact angles increased gradually with storage time at ambient temperature, indicating aging of the plasma-treated fabrics. The average static and advancing contact angles on fabrics obtained 2 h

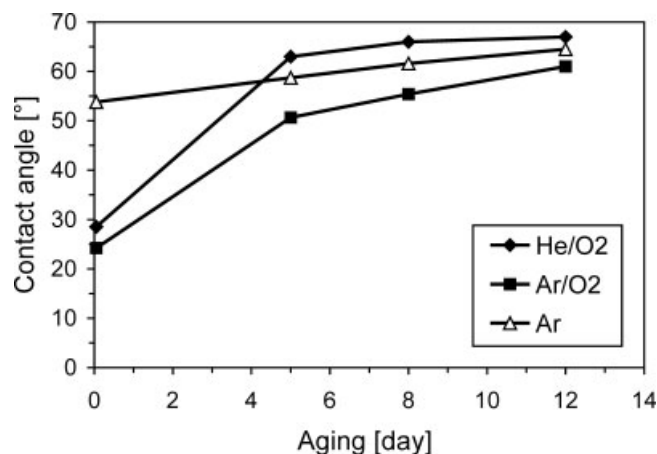


**Figure 9** Increase of water contact angle (static and advancing as obtained from the capillary rise test) with aging for different plasma-treated PET fabrics (5 min, 16 Pa, 15 W).

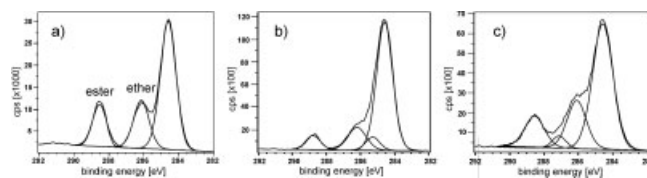
after the plasma treatment were  $\sim 44.0^\circ$  and  $\sim 57.5^\circ$  respectively, whereas with a storage time of 8 days, these values were attained within the range of  $55\text{--}63^\circ$  and  $62\text{--}72^\circ$ , respectively, where Ar/O<sub>2</sub> treatments exhibit a slightly better aging behavior. The static contact angle ratio between 2 h and 8 days aging is 1.3, which is similar to that found for the advancing one (1.2), i.e., the trend in changing contact angles versus aging is proportional. For longer periods (after 10 days), the capillary rise test can no longer be evaluated consistently because of a bad suction of water into the fabric at a water contact angle above  $65^\circ$ . Aging relies on reorganization processes on modified polymeric surfaces. For the improved performance of Ar/O<sub>2</sub> plasmas compared to He/O<sub>2</sub> plasmas, a more crosslinked polymer surface might be assumed yielding a reduced reorientation of polar groups toward the textile bulk.<sup>8</sup>

For the comparison of the textile treatments, absolute (static) contact angles on foils were also measured after plasma treatment at the same conditions and the aging behavior was observed (Fig. 10). Comparing the static contact angles on fabrics and foils (Figs. 9 and 10), it can be seen that similar wetting conditions are obtained, indicating a good penetration into the textile structure using Ar/O<sub>2</sub> or He/O<sub>2</sub> plasmas. Especially, the aging behavior is very similar.

A pure Ar plasma, on the other hand, which results in a noticeable hydrophilization of PET foils (Fig. 10), yields no pronounced treatment of the PET fabric, i.e., a capillary rise can not be detected. Hence, it can be inferred that oxygen radicals play an important role in the penetration of the textile structures at the pressure range used (10–16 Pa), whereas reactive Ar particles (ions and neutrals) show a too long mean free path and a too low reactivity to effectively activate the covered fiber areas of the PET fabric hindering the capillary rise.



**Figure 10** Increase of water contact angle (static) with aging for different plasma-treated PET foils (5 min, 16 Pa, 15 W).



**Figure 11** ESCA C 1s spectrum of a reference PET sample (a), the untreated PET fabric (b), and the plasma-treated PET fabric (c). Ar/O<sub>2</sub> plasma at 5 min, 16 Pa, and 15 W was used.

### Surface characterization

While SEM pictures show no remarkable change of the rather smooth fiber surfaces after plasma treatment, ESCA reveals an increase in oxygen-containing functionalities and a cleaning of the fiber surfaces (Fig. 11) which is responsible for the obtained hydrophilization by carboxyl, carbonyl, and hydroxyl groups. Compared to a reference spectrum of PET, the fabric surface reveals a nanometer thick layer (regarding the information depth of ESCA of  $\sim 5$  nm) of hydrocarbons, probably mineral oils, used as sizing agents during fabric production.

### CONCLUSIONS

The hydrophilization of a PET textile fabric was investigated using low-pressure RF plasmas. Depending on the process gas used, the wettability and capillarity of PET fabrics can be significantly improved, while the results are strongly correlated with the plasma parameters (especially pressure and power) which were optimized using a suction test. Various plasma treatments with air, CO<sub>2</sub>, water vapor, He/O<sub>2</sub>, and Ar/O<sub>2</sub> as well as different treatment conditions were examined. The evaluation of a capillary rise test by using modified forms of Washburn's equation enables the obtaining of both static and advancing contact angles within the textile structure. Thus, the treatment on fabrics can be compared to the treatment on flat substrates like foils and the penetration of reactive plasma species into textile structures can be investigated. It was found that oxygen-containing plasmas (mixtures of Ar or He with O<sub>2</sub>) revealed a similar wetting and aging behavior achieved within the PET fabric compared to those of a PET foil under the same processing conditions (5 min, 16 Pa, 15 W). A pure Ar plasma, on the other hand, yielded a worse hydrophilization of the fabric, indicating the importance of oxygen radicals for the penetration into tightly woven fabrics. Reactive Ar species have a more vertical directed interaction on the fabric surface where the interyarn surface and overlapping zone (the place where the warp and weft yarn are in close contact) remain untreated and thus decay wettability and capillarity.

Therefore, the gas selection took priority over other parameters regarding the plasma penetration into the heterogeneous textile structure.

However, the modified polar surface is not stable, since aging due to reorganization processes was obtained. Nevertheless, capillary rise and suction of the plasma-treated PET fabric could be observed over a period of at least 8 days, which is of importance for subsequent processing steps such as coating, lamination, coloration, or other wet-chemical processes.<sup>33,34</sup> Hence, plasma activation using nonpolymerizable gases is appropriate for cleaning and activation of textiles to improve subsequent process steps such as lamination or coating. To obtain more permanent hydrophilic treatments on textiles, the deposition of nanoscaled functional plasma coatings is required, which we have demonstrated recently.<sup>35</sup>

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