

Production Scale Plasma Modification of Polypropylene Baselayer for Improved Water Management Properties

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Gulnara Fauland undertook analytical characterization and material testing, Floriana Constantin undertook data interpretation and article revision, Hossain Gaffar undertook the full scale plasma processing and Thomas Bechtold planned the experimental design, contributed to data interpretation, and article preparation.

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ABSTRACT: Through its hydrophobic properties, polypropylene (PP) offers unique potential as a functional fiber for a wide range of applications, for example, in nonwovens for hygiene applications or as a baselayer in sports textiles. Current work is focused on the modification of PP presently used in baselayers for sports textiles to increase the hydrophilicity by use of a production scale plant for low pressure plasma treatment. Attention was directed toward an increase in hydrophilicity and time stability of the achieved modification during storage. Changes in the fabric were characterized by sorption of the cationic dye (methylene blue), water retention value, water transport properties, Fourier transform infrared spectroscopy and color measurement. The obtained results indicate an improved wettability and wicking. The extent of modification decreased with storage time and parallel yellowing of treated samples was observed. This indicates chemical rearrangement of the products initially formed on the fiber surface. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2015**, *132*, 41294.

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INTRODUCTION

Lightweight fabrics that form polypropylene (PP) hold a specific position in the design of functional sports clothing. Favorable transport and cooling properties can be achieved when the hydrophobic PP is used as a baselayer and the fabric is in direct contact with the skin. Good mechanical and chemical properties such as low specific weight (0.91 g cm^{-3}), high fiber strength ($42\text{--}53 \text{ cN tex}^{-1}$) and good resistance to acids and alkali make the material well suited for textile production. However, the low surface energy ($28\text{--}30 \text{ mN m}^{-1}$) of the hydrocarbon polymer results in poor wettability (0.05% at 20°C) and difficult wet processing in textile dyeing and finishing.¹ The low surface energy forms the physical basis for the application of PP as a functional fiber; With low binding capacity for sweat, PP demonstrated its almost unique ability to spread sweat between the skin and the baselayer PP-fabric. Thus, PP based materials are widely used as functional baselayers, which exhibit high cooling capacity and rapid drying properties.

Improved, spontaneous distribution of sweat between the skin and fabric from the modification of the surface properties of the PP-fabric to increase surface energy would be desirable. Scientific models for sweat transportation and drying of PP baselayer fabrics show that modification of the fiber toward more hydrophilic behavior could lead to further improvement in functionality.

As a result of the low polarity of PP-fibers and limited temperature stability options for wet textile processing in aqueous media is rather limited, for example, technical dyeing or chemical modification of PP in aqueous media still require improved solutions. Thus, plasma processing of PP-based materials has been studied extensively, as this dry process can be used to achieve a wide range of surface modifications.^{2,3} The influence of different process gases in the plasma atmosphere on the hydrophilization of PP-nonwovens has been reported in the literature. Highest impact has been observed, when oxygen or air

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Table I. Characteristics of PP Fabric

Parameter		Value	Units
Material		99% PP 1% elastin	-
Mass/area	m_a	145	g m^{-2}
Thickness	d	1.36 (at 1.25 Pa)	mm
		1.12 (at 2.5 Pa)	mm
Fabric density	ρ_F	0.107 ^a	g cm^{-3}
Porosity	p	0.893 ^a	-
Fiber density	ρ_f	0.91	g cm^{-3}

^acalculated for thickness at 1.25 Pa

were used as process gases. After longer treatment with high radiation energy, significant deterioration of the fiber surface was displayed in SEM micrographs.^{4,5}

PP-nonwovens could be modified by atmospheric plasma to achieve a more homogenous coating with Fe_2O_3 nanoparticles without use of surfactants, which explained the high wettability of the web after plasma treatment.⁶ The plasma modification of the PP-surface can also be used to increase the adhesion strength of PP-nonwoven fabrics, which is of interest for the production of layered nonwoven laminates.^{7,8}

Low temperature plasma treatment of woven PP-fabric was used in an approach to increase the dyeability for acid dyes, cationic dyes, and vat dyes.^{1,9} The effect of plasma treatment on the PP-polymer could be observed by an increase in the characteristic Fourier transform infrared spectroscopy (FTIR) absorptions for C=O and C—O bonds. The distribution of different functional groups on the modified surface of the polymer is dependent on the physical conditions applied and the composition of plasma gas atmosphere used.¹⁰ Atmospheric plasma activation of PP-nonwovens has also been reported to support following grafting reactions of acrylates as a pretreatment for biocidal finishing and for increased dyestuff uptake.^{11–14} Plasma modification of fiber morphology was also reported to modify the thermal and mechanical properties of treated PP-fabric^{15–17} and has been proposed as a pretreatment before metal coating to achieve antibacterial properties.¹⁸

Low pressure plasma modification of PP-surfaces can be used to introduce polar groups which serve as anchors for functionalization with polyhedral oligomeric silsesquioxanes to achieve higher water contact angles or as electrostatic sorbent sites for cationically modified silica nanoparticles.^{19,20} The formation of a surface coating by grafting also can be initiated by plasma treatment thus resulting in a combined plasma modification and polymer grafting process.²¹ Modification of PP-fibers by grafting and use of γ -irradiation also have been described as processes to modify and optimize PP-composites.^{22–25}

The wide use of PP-fibers as transfer layer in functional nonwovens has led to intensive investigation of low pressure and atmospheric plasma processes for modification of wettability and dynamic water adsorption.^{26–30} Considerable increase in hydrophilicity could be obtained by plasma modification, the limited time stability of the achieved modifications requires

attention as a considerable loss of hydrophilicity can occur during several days of storage.^{3,31} Thus, when plasma activation of PP-fabric is intended as pretreatment to achieve a stronger binding of finishing components from aqueous solution, the following wet treatment is performed shortly after the activation as the effect of activation tends to decrease with storage time.^{11,32} Improvement of time stability can be achieved by a combination of plasma treatment with following grafting which, however, reduces the favorable environmental profile of the plasma step and requires additional processing.³³

The use of hexamethyl-disiloxane (HMDSO) fragmentation under plasma conditions has been studied extensively to achieve polymer layer depositions on various substrates. As an example, the successful modification of PP-fabrics could be achieved by HMDSO containing plasma and an increase in water wettability can be achieved.³⁴

As a wide range of scientific results on lab-scale is already available as literature, a transfer in a full scale process was investigated in this study. A highly porous knitted PP-fabric which is already in use as a baselayer for sports textiles was treated with low-pressure plasma in a production scale installation. A mixture of HMDSO and oxygen was used as a process gas. Major interest was directed towards the results of the achieved effects and the time dependency of the measured properties. The plasma treated fabric was characterized by methylene blue sorption, water retention value (WRV), wicking and water spreading properties, FTIR, and color measurement. The stability of the achieved modification was studied as function of time of up to 4 months to investigate the long term stability of the properties.

EXPERIMENTAL

Materials and Chemicals

The material used for the tests was a knitted PP-fabric (99% PP, 1% Elastin, kindly provided by Skinfit, Maeder, Austria). Technical details are given in Table I. Average fabric density and porosity was obtained by division of the mass per area with the fabric thickness. According to eqs. (1) and (2),

$$\rho_F = \frac{m_a}{d} \cdot 10^{-6} \quad (1)$$

$$p = 1 - \rho_F \quad (2)$$

With ρ_F = fabric density (g cm^{-3}), m_a = fabric mass per area (g m^{-2}), d = thickness (m), p = porosity.

Purity of the chemicals used: NaOH (analytical grade, Roth, Karlsruhe, Germany), methylene blue (microscopy grade, Merck, Darmstadt, Germany), boric acid (analytical grade, Zeller, Hohenems, Austria). A technical grade alkylethoxylate was used as nonionic surfactant.

Plasma Treatment

Technical scale plasma treatment was performed in a semicontinuous low pressure plasma reactor (Plasmabionic GmbH, at Grabher AG, Lustenau, Austria).³⁵ HMDSO was added with a flow of $1000 \text{ sccm min}^{-1}$ and O_2 was added with a flow of $1000 \text{ sccm min}^{-1}$ (gas flow rate in standard cubic centimeters, 20°C , atmospheric pressure). The treatment was performed for 2 min at a pressure of 0.2 mbar and with 9 kW power.

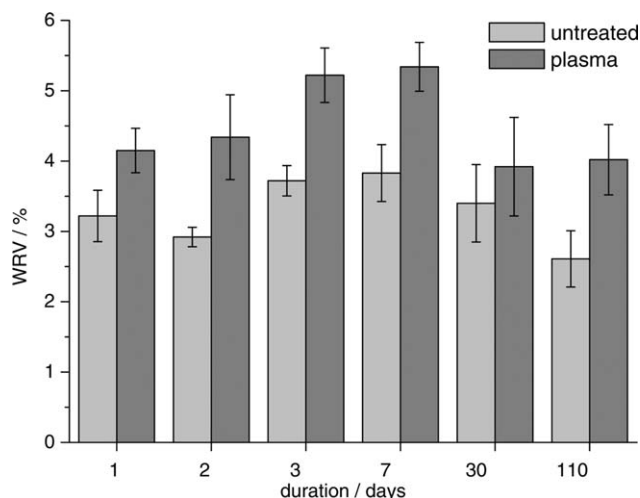


Figure 1. Changes in the water retention property of untreated and plasma treated fabric.

(A schematic presentation of the plasma chamber is given in the Supporting Information Figure S1)

Color Measurement and FTIR Spectroscopy

Diffuse reflectance was used to determine the color coordinates and whiteness index (Konica Minolta Spectrophotometer CM 3610d and Spectra Magic Software). Diffuse reflectance of the fabrics was measured from 360 to 750 nm with and without UV-light (25.4 mm sample diameter). To calculate whiteness the formula according Berger was used.³⁶

Fourier Transform Infrared spectra (FTIR) were measured in transmission and with an attenuated total reflectance (ATR) stage (Bruker Vector 22, Bruker, Wien, Austria). The ATR stage was equipped with a diamond crystal. Transmission fibers were embedded in KBr pellets. The resolution was 4 cm^{-1} with 128 scans.

Determination of Moisture Content and Carboxyl Group Content

The sorption of the cationic dye methylene blue was used to identify carboxyl groups on the fiber surface according to the method for determination of the carboxyl group content in cellulose samples.^{37–39} Thus, for convenience herein we use the wording “carboxyl group content” as term for the total sorption of methylene blue, which was analyzed by photometry.

A sample with known weight was put into a 50 mL solution which contained 25 mL methylene blue solution (300 mg L^{-1}) and 25 mL buffer solution (0.5M boric acid solution adjusted to pH 8.5 by addition of NaOH). After 24 h of shaking the sorption, equilibrium was reached and the concentration of methylene blue in the residual solution was determined by photometry at 664.5 nm using a Hitachi U-2000 double-beam Spectrophotometer (Hitachi, Inula, Wien, Austria; path length of cuvette 1 cm). From the observed change in methylene blue concentration the sorbed amount of dye was calculated as mmol/kg PP-fabric.

Five repetitions were performed and the mean value and the standard deviation were calculated.

Water Retention Value

An exact mass of 0.5 g of fabric was placed in 40 mL of deionized water for 2 h. After immersion, fibers were centrifuged at $4000 \times g$ for 10 min using 50 mL centrifuge tubes with a filter inlay to remove capillary water (Multifuge 1L, D-37520 Osterode, Germany). The samples were weighed and dried at 105° for 4 h, then placed in a dessicator to cool and then weighed again. WRV was calculated according to eq. (3).³⁹

$$\text{WRV} = \frac{m_1 - m_2}{m_2} \cdot 100 \quad (3)$$

where m_1 and m_2 are wet mass (g) and mass after drying (g), respectively. Three independent measurements were calculated and values are given as a mean and standard deviation.

The WRV represents a measure for the amount of water molecules preferentially bound through sorption and fiber swelling, while capillary water is removed through the centrifugation step.

Water Transport

To describe the water wicking properties a laboratory test was designed for the visualization of the wicking properties. A volume of $100\ \mu\text{L}$ of water containing methylene blue as a marker dye was placed beneath the fabric layered on the convex glass surface of a beaker. The liquid migrates into the fabric by capillary forces only. Photographs were taken to compare samples.

Liquid spreading test: A specific approach was designed to evaluate the ability of the PP-fabric to spread liquid between a ceramic surface and the PP-fabric. On a model surface (ceramic tray) a volume of $20\ \mu\text{L}$ of colored test liquid (0.2% methylene blue and 0.05% nonionic surfactant in deionized water) was deposited. Then, test fabric (diameter 8.5 cm) was placed on top of the test surface and rotated slowly within a 10.5 cm ring (30 cycles: three repetitions each; five turns clockwise and five turns anticlockwise). Through surface tension and mechanical action, liquid is deposited on the test surface. At the end of the test, the fabric was removed and the area stained by the colored test liquid was determined by scanning and image analysis (Adobe Photoshop).

RESULTS AND DISCUSSION

Water Retention Value

Through its hydrocarbon character, PP-fibers do not exhibit significant water uptake due to swelling. Water is kept in the fabric structure due to capillary effects, and the major part can be removed by centrifugation. An increase in WRV after centrifugation thus can be used as an indicator of an achieved surface modification of the PP-fiber. The results of WRV determination before and after plasma treatment as function of storage time are shown in Figure 1.

WRVs of untreated fibers range from 2.6 to 3.8% w/w. One day after treatment WRVs of the treated fabric were statistically higher compared to the untreated fabric. The highest increase was determined 7 days after plasma treatment, when values up to 5.3% w/w were recorded. The increase in the WRV value by approximately 1%, was found to be statistically significant at a confidence level of higher than 99.9% S' for a one-sided test

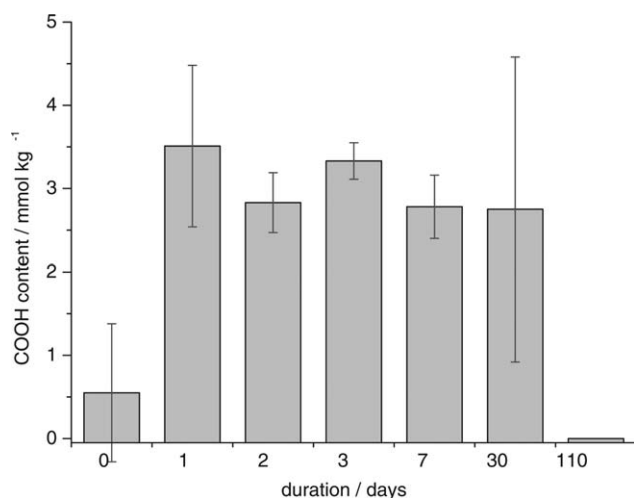


Figure 2. Changes in carboxyl group content as a function of time (0 = untreated sample).

(Student t-Test, paired samples). Through the plasma treatment polar groups were formed on the surface of the nonpolar PP-fabric, which then led to an increase in hydrophilicity and in parallel, an increase in WRV of the treated fabric was observed.

Carboxyl Group Determination

Oxygen plasma treatment leads to the formation of oxidized groups (carboxyl-, carbonyl, peroxide-, and hydroxyl functionalities). Thus, an increase in carboxyl group content on the surface of PP-fabric is expected as a result of the plasma activation. Additionally in the presence of a HMDSO atmosphere silicon containing organic compounds and silicon-oxides will form part of the surface layer. This will result in an increase in adsorption of methylene blue from an aqueous solution.

Accessible carboxyl groups present on the fiber surface, as function of time, were analyzed by the sorption of the cationic dye methylene blue from an aqueous solution. In characterization of cellulose fibers the accessible carboxylic groups are analyzed by selective sorption of the cationic dye methylene blue.^{37–39} At neutral pH conditions the carboxyl groups dissociate to negative carboxylate groups, which remain bound to the polymer fiber. Methylene blue is a cationic dye which binds to the carboxyl groups through ionic interactions. As a stoichiometric reaction between a single carboxyl group and one methylene blue molecule occurs, the sorption of methylene blue can be used to determine the carboxylic group content. Photometry is used to determine the methylene blue concentration of the treatment solution before and after a sorption experiment. The pH of the buffered solution measured 8.5, thus full dissociation of all accessible carboxylic groups was achieved. The cationic dye then sorbs selectively at the negatively charged carboxylates, and total sorption capacity is used as measure for carboxylic groups.

As expected, for the untreated fiber a low value of 0.7 mmol carboxyl groups per 1 kg PP-fiber was determined. Only a few end-groups of the polymer will bear carboxyl groups and the accessibility of the nonpolar fiber for the aqueous test solution is low. Only carboxylic groups which are accessible on the surface of the fibers will be determined. The carboxyl group content of the untreated PP-fiber is near to the detection limit of the method, which can be seen at the error bar given.

Immediately after the plasma treatment the amount of charged groups was found to reach 3.5 mmol kg⁻¹ PP-fibers, which is almost five times the value of the untreated fabric. During the storage, reorganization and follow-up reactions lead to a decrease in carboxyl group content with time, for example, 30 days after the treatment, the number of the carboxyl groups had decreased almost by 50%. The carboxyl group content in

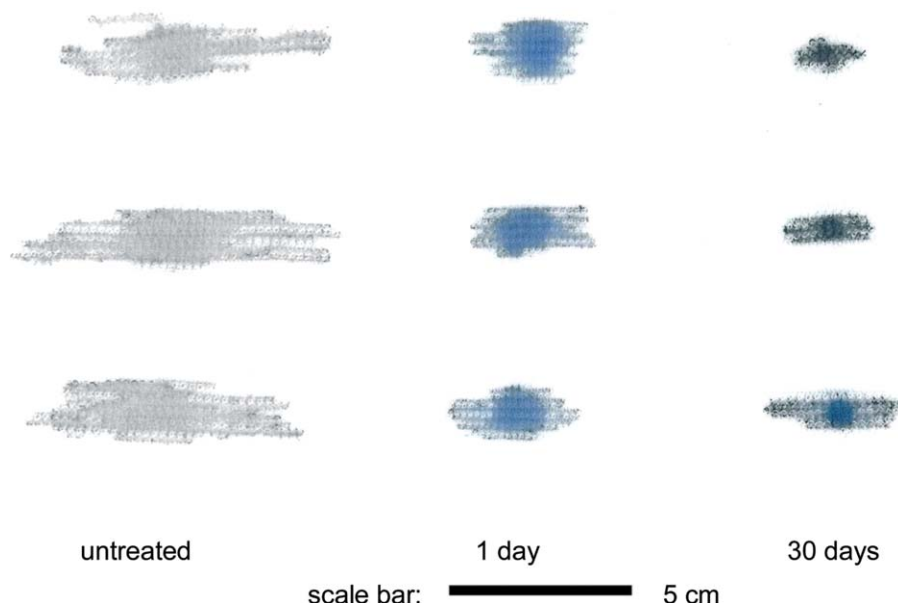


Figure 3. Spontaneous wicking of a water based test solution in untreated and plasma treated fabrics (1 day and 30 days after treatment). Scale bar represents 5 cm length. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

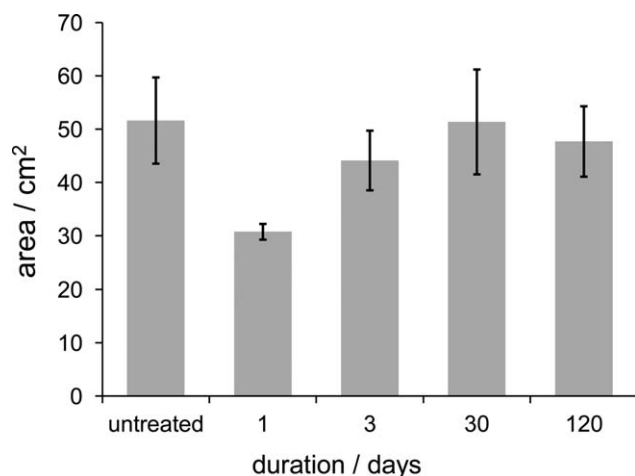


Figure 4. Comparison of the stained area in a dynamic spreading test for untreated and plasma treated fabric as a function of time after plasma activation.

plasma treated fabric determined 110 days after the treatment was no longer different from zero (shown as zero in Figure 2).

Statistical comparison of the average values for the carboxyl group content indicates an increase of the carboxyl group content with a confidence level above 90% for the results after 1, 2, and 7 days (*F*-Test, 90 % confidence level, one sided). Carboxyl group content after 3 days is increased on a 95% confidence level, while the results after 30 are indifferent due to one outlier showing high carboxyl group content.

Also loss in accessibility of carboxyl groups through polymer chain mobility in amorphous parts of the fibers could cause a decrease in methylene blue sorption.

Wicking/Spreading Test

To visualize the spontaneous wicking of water into the fabric structure, horizontal wicking tests were performed with a defined volume of colored test liquid. The plasma treatment led to marked differences in wicking behavior. In wicking tests with untreated material, the drop of liquid mainly enters the hydrophobic material along the elastin filaments, which exhibit higher polarity than PP. Through the hydrophobic properties of the untreated PP the water preferentially spreads along the elastin filaments in the knitted fabric, which leads to a larger area of stained fabric. One day after plasma treatment the colored liquid marks distinct areas in the fabric structure, which indicates more localized wicking into the structure of the PP-fabric. Through a water uptake into the fabric structure a smaller but darker circular area is marked by the dye, as a higher local concentration is achieved in the fabric. Thirty days after the initial plasma activation, the stained area decreased and only a small area was stained by the marker dye (Figure 3). Through progressive loss of hydrophilicity after 110 days the colored drop remained on the fabric surface and was dried there without wetting.

For application of PP-textiles, the observed increase in wicking properties is valuable to support the maximum cooling effect and rapid drying. From practical experience, it is known that

Table II. Diffuse Reflectance of Untreated and Plasma Treated Samples at 425 nm in % of Standard White (100%) as a Function of Storage Time (Measurement with UV-Light and Without UV-light) and b^* -Coordinate (Measured Without UV-Light)

Sample	Time	<i>I</i> (including UV) (%)	<i>I</i> (without UV) (%)	b^*
Plasma	1 day	134.5	101.2	-3.08
	1 month	132.6	100.1	-2.55
	4 months	131.6	99.6	-2.52
Untreated	1 day	136.4	102.7	-3.63
	1 month	136.3	102.7	-3.69
	4 months	134.5	101.7	-3.43

direct contact and slight movement between skin and a base-layer is relevant to achieve optimum spreading of sweat and maximum cooling on the skins surface.

To simulate this behavior, a dynamic spreading test was used to assess and compare the behavior of the plasma modified materials. A colored liquid was placed between a test surface (skin model) and a fabric sample. After defined movement of the textile sample, the area of colored test liquid spread on the test surface was determined (Figure 4). Hydrophobic fabric samples will hold the test liquid between test surface and fabric surface. Thus, during the movement of the sample the colored liquid stains the larger area. Hydrophilic samples will absorb the test liquid into the fabric structure and part of the liquid is held inside the fabric. Thus, higher hydrophilicity is indicated by a smaller area of stain measured on the test surface as the test liquid adsorbed/wicked more into the fabric.

One day after the plasma treatment the surface of the fabric exhibited increased hydrophilicity. The mean area of colored liquid on the model surface was reduced to 30.8 cm² compared to the 46.7 cm² of untreated fabric. During storage (3–120 days) the initially observed hydrophilicity reduced and

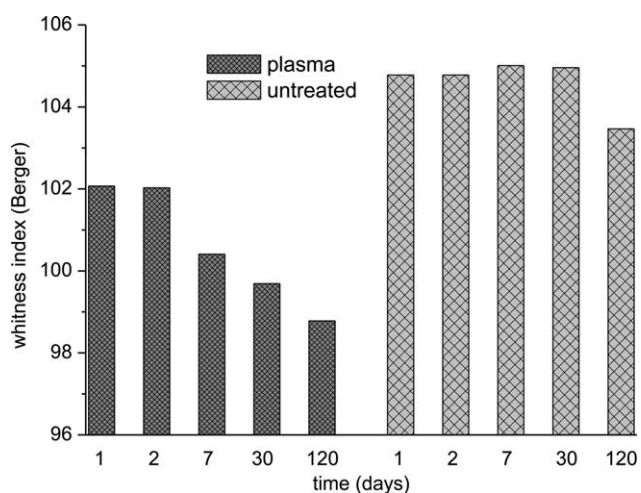


Figure 5. Whiteness index as a function of time for plasma treated and untreated PP.

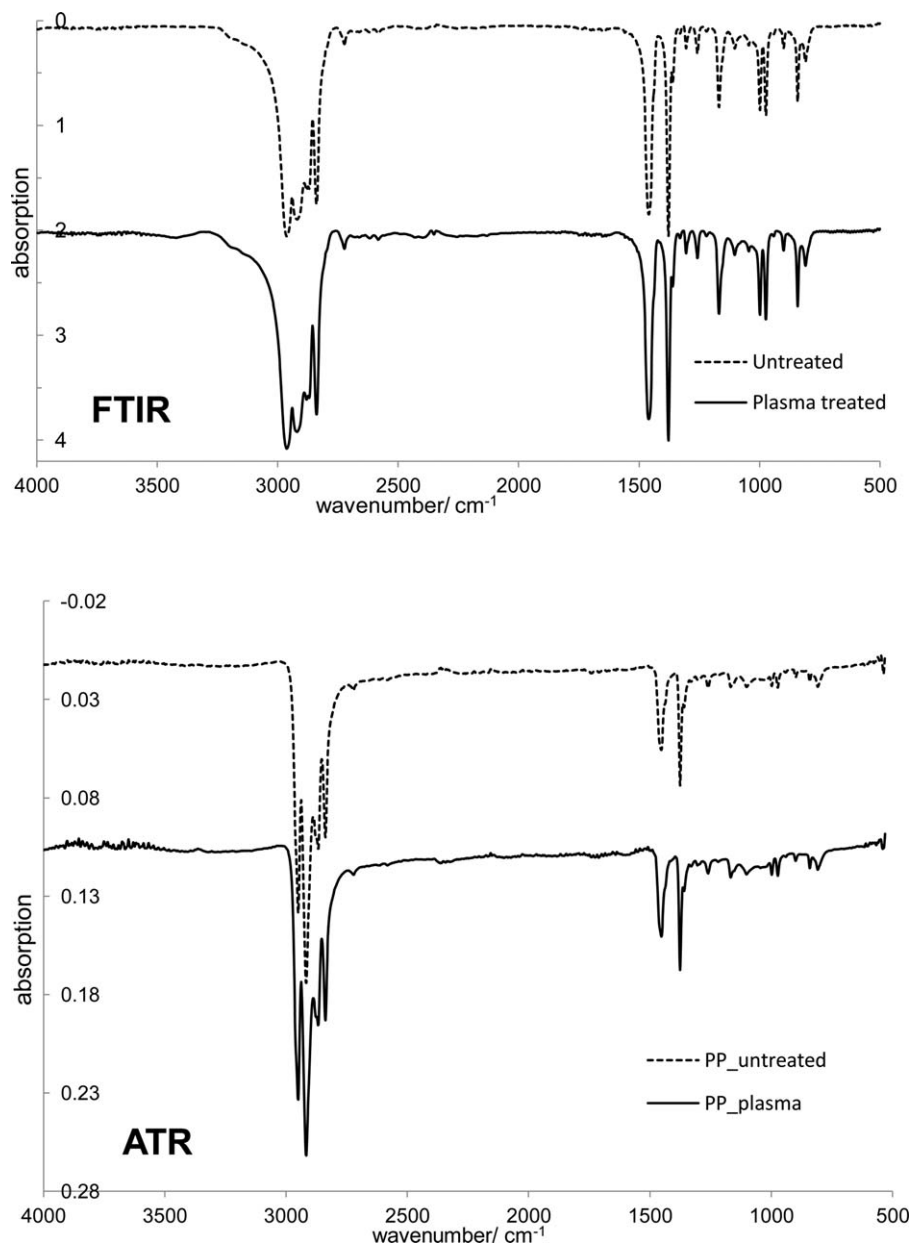


Figure 6. FTIR (KBr-pellet) and ATR-FTIR (fabric) spectra of (---) untreated and (—) plasma treated PP fabrics.

differences observed with samples tested later were no longer significant.

Color Assessment

In this study, a white fabric was used which allowed observation of slight color changes, in particular yellowing. Immediately after the plasma treatment, the samples did not exhibit any visual color change; however, a distinct fabric yellowing occurred 30 days following the treatment. Through plasma treatment, reactive species are formed on the surface. Through slow chemical follow up reactions, colored by-products can be formed which lead to undesired yellowing of the material and changes in the appearance of colored textiles. Fabric yellowing on plasma treatment is a widespread problem known to occur

when oxidation of products form on the surface after modification takes place.

Plasma treated fabrics and untreated samples were analyzed in the wavelength region between 360 and 740 nm. Minor differences were observed in the wavelength region between 430 and 480 nm. Selected values for diffuse reflectance at 425 nm related to standard white are given in Table II. The measurements with UV-light and without UV-irradiation indicate that the function of the optical brightener was not affected by the plasma. As this additive is embedded in the PP-matrix, the risk of chemical damage during plasma treatment is low.

From practical experiences, the accuracy of the method to measure the diffuse reflectance can be assessed with $\pm 2\%$. Thus, the

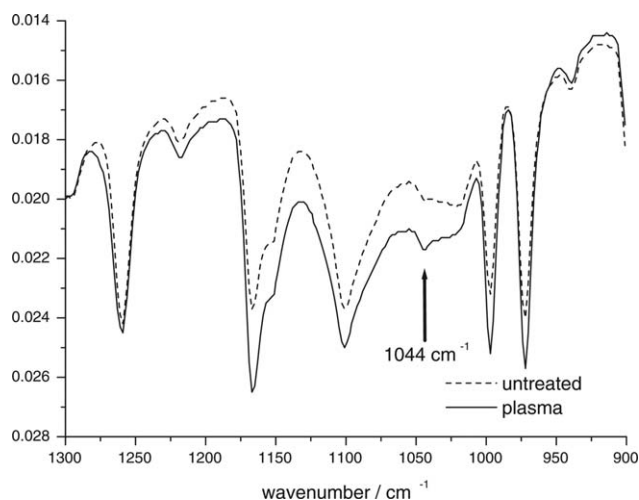


Figure 7. Magnified part of FTIR-ATR spectra of (.....) untreated and (—) plasma treated samples

changes observed after 1 and 4 month are significant. The b^* values given in Table II also support this finding as in practice changes of a single color CIELab-coordinate near to unity will be accepted as significant and visible difference.

Whiteness index, according to Berger, was calculated from diffuse reflectance spectra to quantify the yellowing of the samples as function of time (Figure 5). During storage, the untreated samples maintained a whiteness index around 104, while the plasma treated samples exhibited a time dependent decrease in whiteness index. This indicates slow follow-up reactions on the surface of the plasma treated samples, which led to formation of yellow-brown colored products.

FTIR/ATR Measurements

FTIR spectra of untreated samples and plasma treated samples are given in Figure 6. The spectra were recorded both in transmission and as ATR spectra. While the transmission spectra include the full cross section of the fiber, the ATR spectra considered sections located near to the fiber surface. The plasma treatment was expected to cause chemical reactions on the fiber surface, thus changes should be detectable preferentially in the ATR spectra. Formation of oxidized products, for example, carboxyl or carbonyl groups, should be detected in the IR spectra by their characteristic wavenumbers around 1700 cm^{-1} . No changes between treated and untreated samples could be observed both in transmission and ATR techniques in the entire range from $500\text{ to }4000\text{ cm}^{-1}$. Most probably the achieved molecular changes on the surface of polymer remained below the detection limit of the FTIR technique, which usually requires presence of 1–2% w/w of a substance for detection. For changes in wetting characteristics, the modification of a thin surface layer would be sufficient, thus properties can be modified, while no distinct indication is found in the FTIR spectra.

The Si—O—Si bond of siloxanes is expected to show a strong absorbance in the wavenumber region between $1000\text{ and }1130\text{ cm}^{-1}$, thus a more detailed analysis was undertaken in

this part of the FTIR spectra. In Figure 7(a), magnified representation of the FTIR-ATR spectra (Figure 6) is given for the wavenumber interval between $1300\text{ and }900\text{ cm}^{-1}$. At 1044 cm^{-1} , a small peak can be detected in the treated samples which could indicate the formation of Si—O bond containing products on the surface of the polymer. This could be due to deposition of HMDSO follow-up products formed during reactions of HMDSO in the plasma gas.

CONCLUSIONS

PP is used in functional sports clothing as a baselayer due to its unique ability to distribute sweat over a large area of the skin. The low surface energy of PP of $28\text{--}30\text{ mN m}^{-1}$ hinders spontaneous wicking of pure water (72.8 mN m^{-1}). Sweat exhibits a reduced surface energy in the magnitude of 32 mN m^{-1} , which is similar to surface energy of skin ($>28\text{ mN m}^{-1}$).⁴⁰ A slight increase in surface energy of the PP-fabric could lead to a wider spreading of sweat which thereby increases evaporation and intensifies overall cooling effect.

Production scale treatment of commercially used PP-fabric by low pressure plasma treatment increased hydrophilicity on the materials surface. The treated fabric exhibited improved wicking properties, which support spreading of sweat between the skin and a PP baselayer. Marked changes were observed in water uptake determined by a WRV, which increased by approximately 25% from values of 3–4% (w/w) to 4–5% (w/w). An increase in accessible carboxylic group content was indicated by the methylene blue sorption. During storage, the initially observed values for WRV and methylene blue sorption decreased, which indicates slow chemical rearrangement and further changes on the surface structure which occur during storage.

Yellowing was observed during storage, which is due to a slow formation of colored follow-up products.³⁴ This behavior has to be considered when white or light colored material is treated.

FTIR/ATR analysis could not prove formation of polar groups, as their concentration remained below the detection limit of the analytical technique.

The time dependent changes in material properties after plasma modification are of particular importance for the assessment of the durability of the effects obtained. Loss in hydrophilicity during storage time has to be considered. The use of a more intensive surface etching with plasma and stabilization of the achieved modification through chemical after treatment should be studied as strategies to achieve a more time stable modification of PP. In addition, the influence of wash and wear processes should also be considered in future work to provide a full assessment of durability of the modification during consumer use.

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