TOPOGRAPHICAL EFFECTS OF O_2- AND NH_3-PLASMA TREATMENT ON WOVEN PLAIN POLYESTER FABRIC IN ADJUSTING HYDROPHILICITY

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Abstract:

Hydrophilisation of polyester textile materials has been investigated over the last twenty years using low-pressure and atmospheric plasmas. According to these studies, wettability and capillarity of fabrics can be significantly improved depending on the process gas used. In the present study, the effects of low pressure O_2- and NH_3-plasma on the morphology and topometry of fabrics on four different length scales, as well as the influence of the topographical changes of textile structures on the resulting water spreading and absorption rates were investigated. The results of the topographic characterisation using two non-contact optical methods and wettability measurements indicate that the modification of filament nano-topography cannot satisfactorily explain the drastic changes observed in wettability. Dimensional changes (relaxation and shrinkage) as well as changes in warp morphology and inter-yarn spaces are more decisive for inducing hydrophilicity in polyester woven plain fabrics than an increase in the surface nano-roughness of their filaments.

Key words:

Polyester textiles, plasma, roughness, topography, wetting.

Introduction

Polyester fibre is one of the most popular fabrics, second to cotton fibre, as measured by production tonnage in recent years. The technical merits and commercial versatility of the polyester fibre production system have led to successful product development and applications.

Polyester has some excellent properties, such as high tenacity (0.35 to 0.45 N tex^{-1}), high lustre, resistance to chemicals (acids, alkalis, bleaches), abrasion and creasing, and fair resistance to sunlight and soiling, etc. One hundred percent polyester staple yarns are used for high strength applicable aspects. It has some remarkable drawbacks such as non-absorbency, hydrophobicity, flammability etc. For these reasons, staple fibres are used primarily in blends with other fibres, especially wool, cotton, viscose and modal. The blending level depends on the end use and the other properties of the fibre. Chemical modification is currently combined with physical modification, which results in the development of various fibre variants exhibiting improved processing and performance characteristics.

These physical, mechanical and chemical attributes make polyester an excellent candidate not only for apparel and textile products, but also for industrial and composite applications. However, polyester fibres retain little moisture and do not transport aqueous fluids. The hydrophobic character of polyester fibres makes them difficult for hygienic use, difficult to dye and to finish in aqueous media.

In recent years, to overcome these limitations of polyester fabric, excellent progress has been made by means of fibres modification in order to functionalise their required properties. Surface morphology and surface chemistry can be modified by several surface-sensitive techniques such as coating, hydrolysis, plasma discharge, photochemical treatment, etc.

The hydrophilisation of PET textile fabrics was investigated in [1-3] using low-pressure plasmas. According to these studies, the wettability and capillarity of fabrics can be significantly improved depending on the process gas used. Hossain et al. [1,3] demonstrated through the evaluation of a capillary rise test and using modified forms of Washburn's equation, both static and dynamic contact angles within a textile structure. Ferrero [2] proposed processing of capillary rise data that allowed for characterisation of fabric wettability, improved by plasma treatment in different gases. In his study, the capillary diffusion coefficients derived through Washburn's equation were influenced in most cases by the nature of the gas, plasma power, exposure time and ageing. Additionally, Hossain et al. [3] suggest that both the weave construction and yarn type, are two important parameters in addition to pressure, power and time, which have to be considered during the surface activation of textiles, as the resulting wettability can be quite significantly varied.

In the present study, the effects of exposure time of low pressure O_2- and NH_3-plasma on the morphology and topometry of fabrics on four different length scales, as well as the influence of these topographical changes on the resulting water spreading and absorption rates were studied.

Table 1. Specifications of the polyester fabric used.

Parameter	Value
Fineness (warp)	295 dtex
Fineness (weft)	295 dtex
Yarn density (warp)	270 +/- 5 yarn / dm
Yarn density (weft)	270 +/- 5 yarn / dm
Width	80 cm
Fabric weight	170 g/m^2
Weave	1 / 1 plain

Materials

A polyester test fabric was used as the substrate for this study. The fabric was supplied by wfk-Testgewebe (Krefeld, Germany). The material was used as received without any pretreatment. Different parameters of the test fabric are given in Table 1.

Experimental

Plasma modification of PET fabrics

The plasma treatment was done in a computer-controlled customised MicroSys apparatus (Roth&Rau, Germany) with a cylindrical vacuum chamber, made of stainless steel, with a diameter of 350 mm and a height of 350 mm. The base pressure obtained with a turbomolecular pump is $<10^{-7}$ mbar. A Micropole mass spectrometer (Ferran, US) was used to monitor the residual gas. On the top of the chamber, a 2.46 GHz electron cyclotron resonance (ECR) plasma source RR 160 (Roth&Rau) with a diameter of 160 mm and a maximum power of 800 W was mounted. The process gas was introduced into the active volume of the plasma source via a gas flow control system. When the plasma source was on, the pressure was measured by a capacitive vacuum gauge. The samples were introduced by a load-lock system and placed on a grounded aluminium holder near the centre of the chamber. The distance between the sample and the excitation volume of the plasma source was about 200 mm. For the plasma treatments, the following parameters were applied: i) process gas NH_3 (99.999%, Messer Griesheim, Germany), flow 15 standard cubic centimetres per minute, pressure 3.6 10-3 mbar, effective microwave power 600 W and ii) process gas O_2 (99.95 %, Messer Griesheim, Germany), flow 15 standard cubic centimetres per minute, pressure 3.6 10^{-3} mbar, effective microwave power 100 W.

Topographic characterisation

Two non-contact optical methods using white light were applied to measure the surface topography of polyester fabric surfaces: scanning chromatic confocal imaging and high-resolution scandisk confocal microscopy (SDCM).

The scanning chromatic instrument used for the topographical analysis was a MicroGlider device (Fries Research & Technology, Germany). Unlike a conventional microscope, which simultaneously images all the points in the field of view, a chromatic confocal microscope images only one object point at a time. The field is reconstructed by (x,y) scanning. This novel optoelectronic setup, based on a quasi confocal, z-axis extended field, was developed for high resolution non-contact 3D surface metrology, including roughness characterisation and surface flaw detection. The instrument uses a chromatic white-light sensor (CWL), which is based on the principle of chromatic aberration of light. White light is focused on the surface by a measuring head with a strongly wavelength-dependent focal length (chromatic aberration). The spectrum of the light scattered on the surface generates a peak in the spectrometer. The wavelength of this peak along with a calibration table reveals the distance from sensor to sample. The sensor works on transparent, highly reflective or even matte black surfaces [4-5]. It is extremely fast and has virtually no edge effects.

A confocal microscope µsurf (Nanofocus, Germany) was also used to measure the topography of the surfaces studied. Scandisk confocal microscopy is an optical imaging technique used to increase micrograph contrast and/or to reconstruct 3D images by using a spatial pinhole to eliminate out-of-focus light or flare in specimens that are thicker than the focal plane [6]. The measuring principle is that the light of a LED is focused by a movable objective on an object's surface, then reflected and finally captured by a detector. If the object is out of focus, its illumination and image on the detector are unsharp, resulting in a very low output signal. Once the object's surface is in the focus of the objective and the detector, a maximum signal is received. Very precise information on height can be obtained by gradually moving the focal point (lens) in the z-direction. This punctiform principle is extended into a field of view method by the use of a rotating Nipkow disc which distributes the incident light beam line by line over the object's surface to measure 3D topography and 2D profiles.

Cut-off length (L_m), defined as the length of one side of the square sampling area, and resolution (distance between measured points Δ_x, assuming that $\Delta_x = \Delta_y$) are the most important sampling parameters, which apart from particular instrumental dependent parameters, such as light intensity, measuring frequency, etc., have to be calibrated before characterising topography.

In [7-9], an innovative method for a topographic characterisation of textile materials using different length scales was presented, which makes it possible to consider and separately analyse specific morphologies caused by weave, yarn and filament/fibres, and to investigate the influence of topography on wettability by modification processes, e.g. construction parameters, thermofixing, impregnation with soil release polymers and the mechanical effect of wash-dry cycles. In these studies, a cut-off length of 1 mm and a lateral resolution of 1 µm were considered as optimal sampling conditions to characterise the topography of a woven plain polyester fabric with 380 warps/cm and 400 wefts/cm. In order to ensure statistical reliability, a cut-off length of 2.6 mm and a lateral resolution of 1 µm were used in the present study.

Characterisation of fabric wettability

The static contact angle measurement used to characterise interactions between a liquid and a solid surface is no longer thought to be adequate in all cases. In many practical applications, the wetting phenomena of interest are 'dynamic' in nature, involving a moving wetting line at which equilibrium is never attained. The contact angle of a moving wetting line is generally called dynamic contact angle [10]. An image sequence of the initial contact angle and dynamic changes of contact angle of a drop, its base, height and volume over time can be captured to monitor the interaction of the droplet spreading across or penetrating into the solid surface.

Dynamic wetting measurements were carried out with a dynamic spreading, absorption and contact angle tester FibroDAT 1122 HighSpeed (Fibro System, Sweden) according to the sessile drop method to estimate the degree of hydrophobicity. The device is equipped with a high speed video camera which collects up to a 1000 images per second. Some advantages of this equipment over other contact angle measuring systems, as well as the measuring procedure are detailed in [11]. Water droplets of a 10 µL volume were applied to the fabric surface under investigation by a short stroke from an electromagnet. The strength of the stroke was minimised to avoid oscillation effects. After deposition, the droplet was stabilised on the surface and reliable data were collected

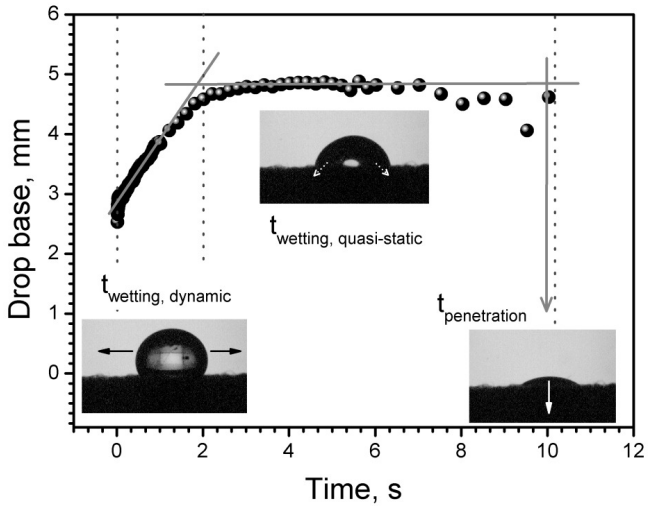

Figure 1. Three different wetting regimes of a textile surface.

thereafter for 20 s. The instrument was located in a temperature-controlled laboratory maintained at 23±1°C. Relative humidity of 50±4% was kept constant. For a water-fabric system, an average contact angle of 20 single measurements was obtained.

On the basis of macroscopic water drop base changes, the wetting behaviour of the water drop can be divided into three different regimes (Figure 1): i) dynamic wetting, defined as growing of the drop diameter depending on time (also known as spreading), the quasi-static wetting, ii) where the drop diameter remains approximately constant and iii) penetration, which is marked by liquid drop absorption into fabrics, again depending on time.

For a theoretical treatment of capillary flow in fabrics, the fibrous assemblies are usually considered to consist of a number of parallel capillaries [12]. The movement of a liquid in a non-homogeneous capillary system, such as fabrics, however, is discontinuous: the wetting front advances into the capillary system in small jumps, because the irregular capillary spaces have various dimensions. Therefore, wicking, the spontaneous flow of a substrate driven by capillary forces [13], is affected by the morphology of the fibre surface as well as by the structure of the corresponding capillary system depending on construction parameters such as fineness of filaments and yarn, warp and weft density and the type of weave. While a textile surface consists of parallel horizontal und vertical capillaries, the surface yarns controls, according to experimental results [7], the wetting behaviour of a liquid drop.

Results and discussion

Textile macro-morphology: bi-dimensional changes

Changes in distances between yarns are considered in this study as the only macro-morphological effect of plasma treatment on the fabric structure, according to the methodology of textile topographical characterisation on different length-scales presented recently in [7-9]. Figures 2 and 3 show measurable changes in distances between warps and wefts caused by modification with O_2- and NH_3-plasma treatments, respectively. In both cases, the first fabric response is to close warps and wefts. However, under more radiation, they tend to open again proportionally to the duration of treatment. This effect can be seen more easily by comparing the surface area change in percent, as shown in Figure 4, which was calculated on the basis of the bi-dimensional changes of warps and wefts.

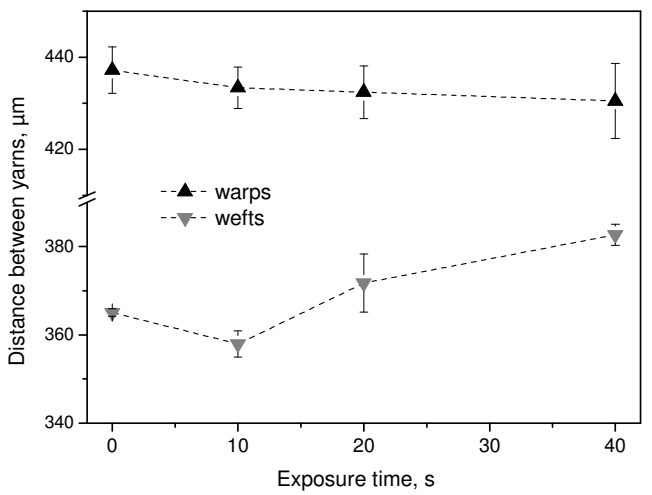

Figure 2. Effect of exposure time of O_2-plasma on distance between yarns.

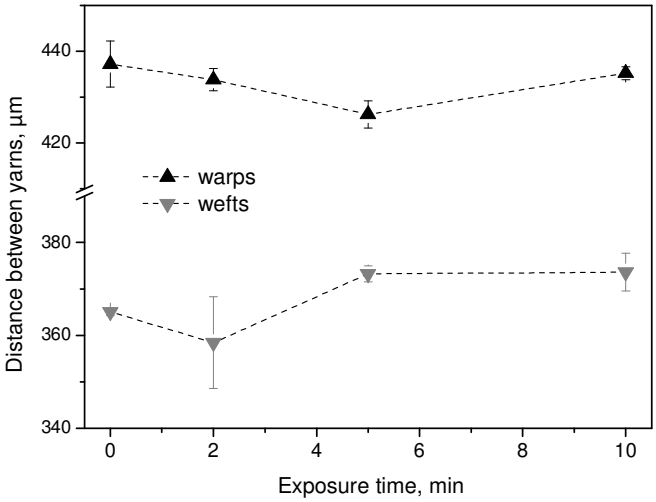

Figure 3. Effect of exposure time of NH_3-plasma on distance between yarns.

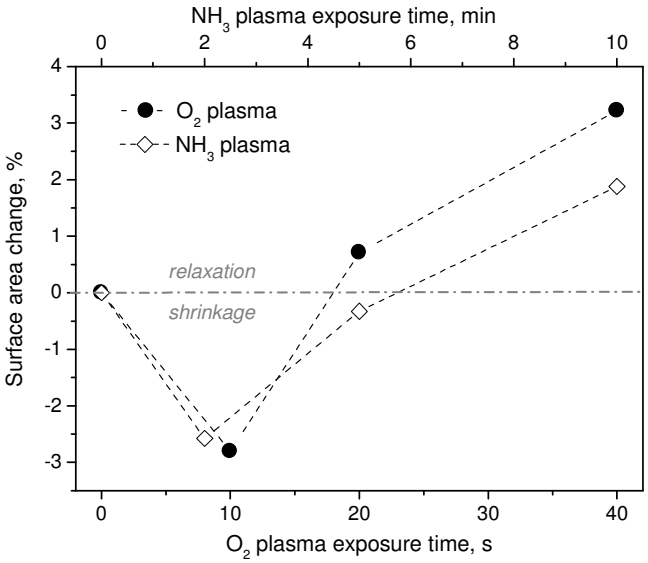

Figure 4. Effect of exposure times of both plasma treatments on bi-dimensional changes of fabric surface area.

It is important to note that the distances between yarns correspond to the average of distances between yarns centres. Changes of yarns densities are not considered on the macro-morphological scale; however, the effect of plasma treatments

on the spaces between yarns and small changes in distances between filaments will be considered in the next sections as changes in the inter- and intra-yarn porosities, respectively.

Textile meso-topography: inter-yarn volumetry

In a woven structure, warps and wefts have sinusoidal traces [7-9]. The meso-scale of textile materials describes the surface topography produced by the amplitudes of these traces and the spaces between yarns, without attending to macro-morphology and the details corresponding to fibres or filaments. To isolate this length scale, smooth filtering with a factor of 40% was applied to the topographical data using the software FRT Mark III V.3 8.10 developed by Fries Research and Technology (Germany). Figure 5 graphically shows the effect of this filtering method on the original topographical data by removing the roughness due to filament irregularities. In this graph, higher waves correspond to warps, while smaller ones correspond to wefts. To characterise the meso-scale, the maximal waviness height Wmax, defined in DIN EN ISO 4287 and ASME B46.1, was used to quantify the amplitude of the highest warps for each fabric.

According to these results, different plasma treatments sequentially increase warp waviness. If we now consider a sequential increase of weft distance during treatment, reported in section *Textile macro-morphology: bi-dimensional changes*, there are three possibilities to explain the waviness behaviour shown in Figure 6: (i) an increase of warp diameter, i.e. more separation between filaments, (ii) the longitudinal relaxation of warp filaments or (iii) both effects together.

Inter-yarn spaces, the void volume between yarns, also called fabric meso-porosity, was quantified using the 'lubricant filled profile valley area' established for 2D-profiles in DIN EN ISO 13565 and DIN 4776, but in the present study extended to 3D-topographies. The mathematical process was applied to the topographical data after smooth filtering (Figure 7). The sequential separation between yarns, especially by wefts, reported in section *Textile macro-morphology: bi-dimensional changes*, correlates with the increase of inter-yarn spaces shown in Figure 8.

Textile micro-topography: intra-yarn volumetry

Unlike the macro- and meso scales, the micro length scale reveals the influence of filaments on the resulting topography. A selection of the optimal cut-off length in this case not longer depends on some statistical of mathematical criteria as seen in the macro- and meso-length scales, rather on the size and location of the set of filaments. To study the micro-topography of the fabrics, warps and wefts were zoomed using a cut-off length of 232 µm for both yarns (Figure 9). Intra-yarn porosity was also quantified using the method mentioned in section *Textile meso-topography: inter-yarn volumetry*, previous removing of micro-waviness, a consequence of yarn profile and fabric meso-topography. Intra-yarn porosity decreased proportionally to O_2-plasma treatment, as can be seen in Figure 10. However, NH_3-plasma treatment had no specific influence on this parameter, as can be seen in Figure 11.

According to these results, in the case of O_2-plasma treatment, the increase of warp amplitude (W_{max}) observed by characterising the meso-topography (Figure 6) was a consequence of warp filament elongation and not their separation.

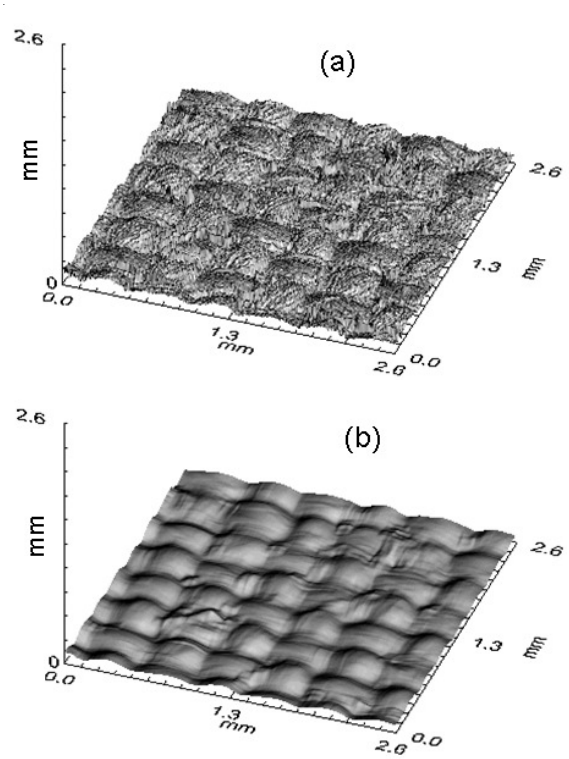

Figure 5. Smooth filtering using a factor of 40%: (a) original fabric surface, (b) the same surface after applying a mathematical filter.

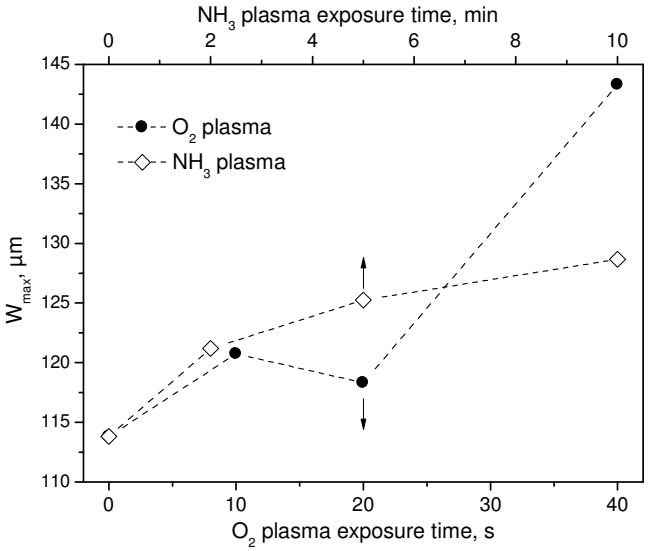

Figure 6. Meso-topographical effect of plasma treatments, characterised as changes in the warp amplitudes using the maximal waviness height W_{max}.

Textile nano-topography: filaments profile roughness

High resolution scandisk confocal microscopy was used to measure the arithmetic mean roughness of filaments using length profiles between 120 and 160 µm by horizontal and vertical resolutions of 500 nm and 3 nm, respectively. Ten measurements were performed on each fabric. The results are presented in Figure 12.

Both plasma treatments had contrasting effects on filaments nano-roughness. Under O_2-plasma, filament roughness increased from 40 nm to 44 nm during the first 10 seconds. After 40 seconds of treatment, filament roughness decreased up to 34 nm.

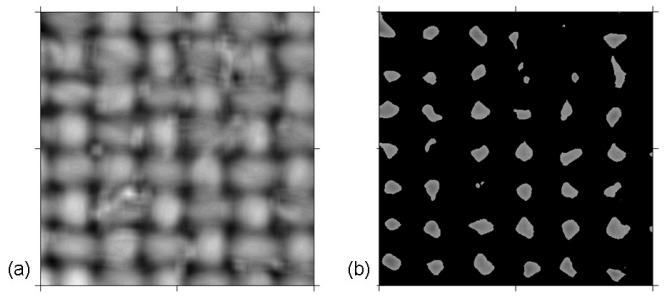

Figure 7. Inter-yarn porosity was calculated using the parameter 'lubricant filled profile valley area' extended to 3D-topography. (a) topographical contrast map, (b) the same map after isolating the inter-yarn pores.

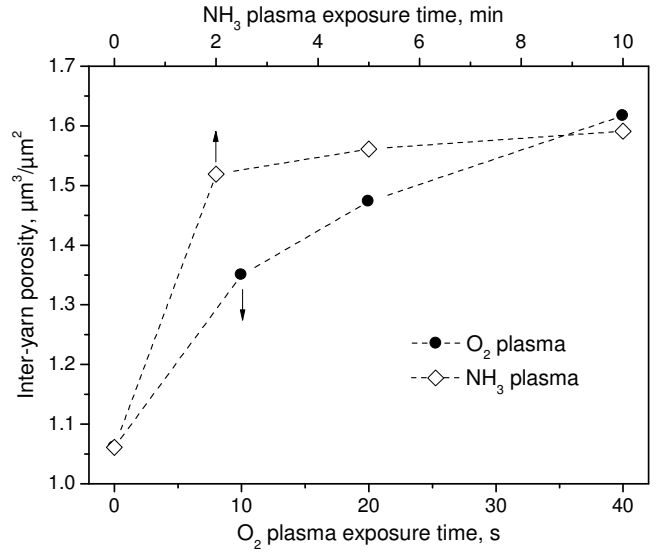

Figure 8. Effect of plasma exposure times on the increase of inter-yarn porosity.

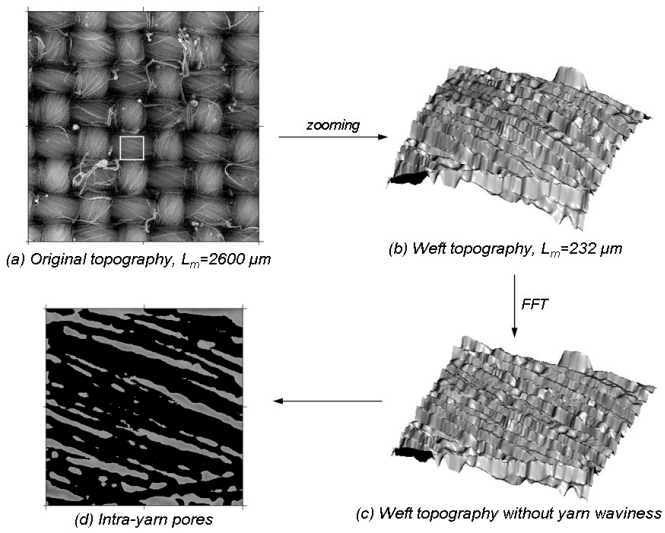

Figure 9. Quantification of intra-yarn porosity: (a) yarns must be zoomed out from the original topographic map, (b) yarn topography includes the curvature due to yarn profile, (c) this curvature is removed using Fast Fourier Transformations, (d) the intra-yarn porosity can be quantified using the algorithm 'lubricant filled profile valley area'.

NH_3-plasma led to an initial decrease of filament roughness, from 40 nm to 30 nm. After 10 minutes of exposure, filament roughness increased again up to 36 nm.

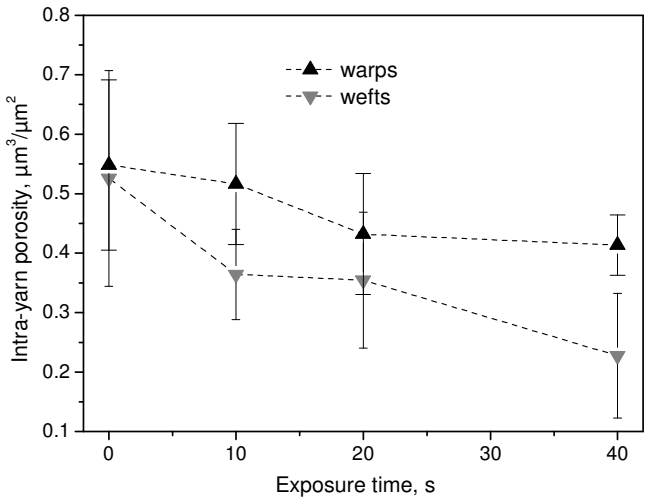

Figure 10. Effect of treatment with O_2-plasma on intra-yarn porosity.

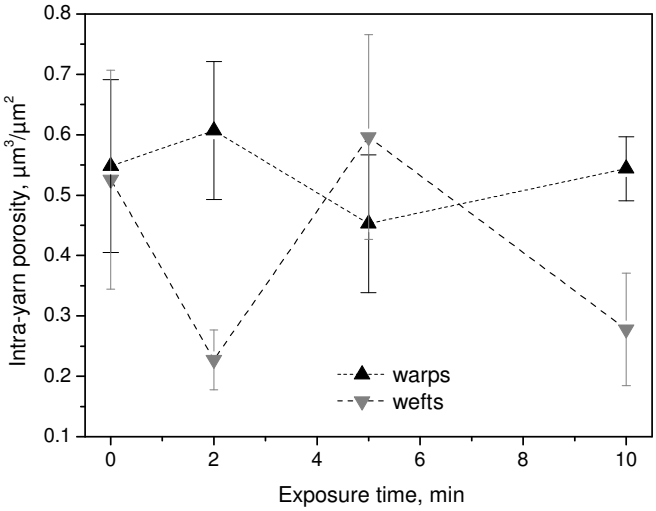

Figure 11. Effect of treatment with NH_3-plasma on intra-yarn porosity.

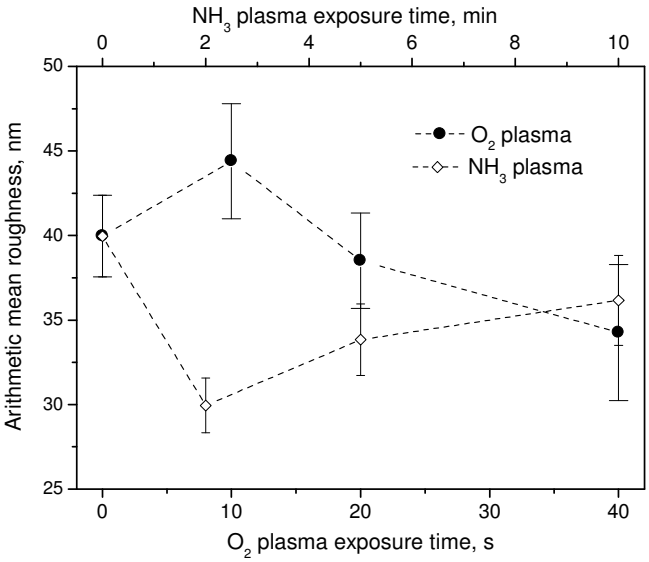

Figure 12. Effect of both plasma treatments on nano-roughness of filaments.

Textile Wetting

Spreading and total water absorption rates were measured using the method described in section *Characterisation of*

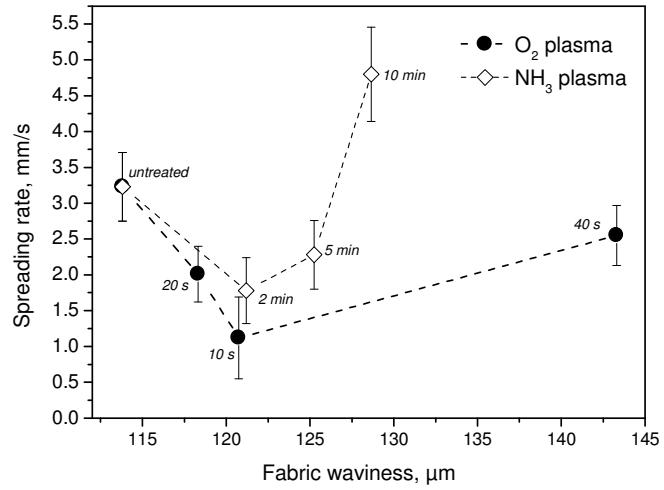

Figure 13. The lowest spreading rate corresponds to minimal fabric waviness for both plasma treatments.

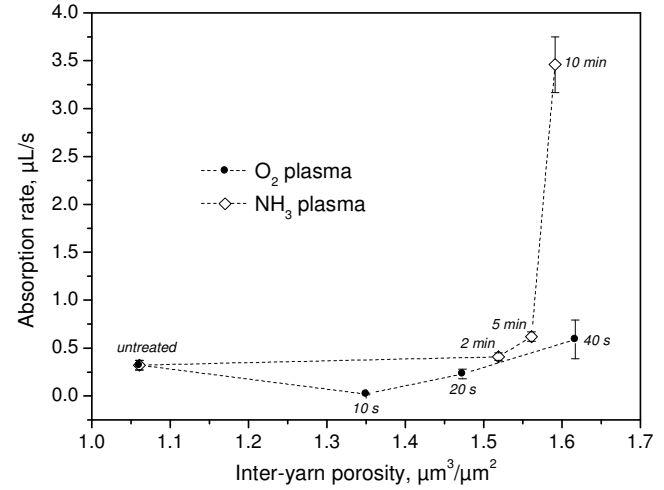

Figure 14. Effect of inter-yarn porosity on the absorption rate of 10µL water drops.

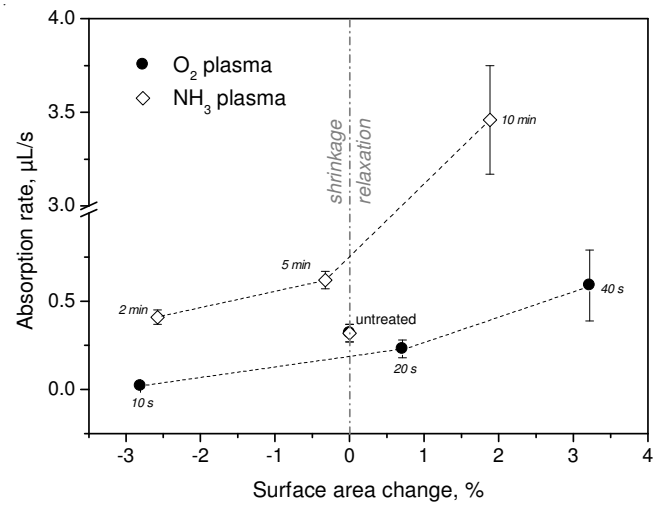

Figure 15. Both plasma treatments lead to an initial shrinkage and a posterior relaxation and hydrophilisation.

fabric wettability. The results, listed in Table 2, show that both treatments led to initial fabric hydrophobisation, followed by a sequential hydrophilisation.

This was assessed in [7-9], using the waviness to characterise the meso length scale, since the meso-topography of woven polyester fabrics controls the spreading rate of a water drop.

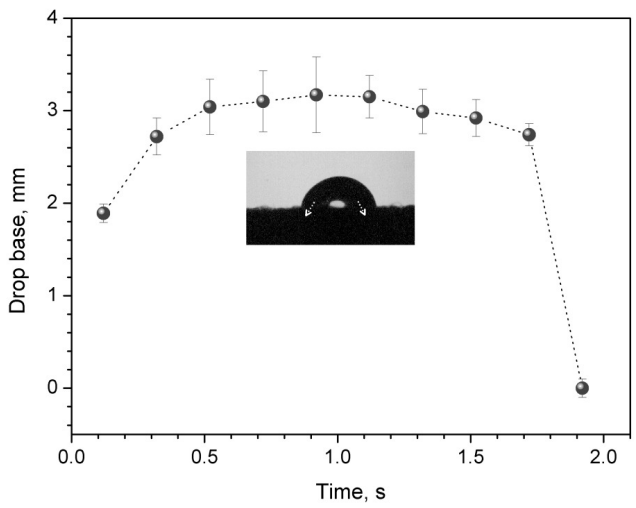

Figure 16. Increase of a water drop base on the untreated fabric. Spreading and quasi-static wetting occurs almost simultaneously on the studied fabrics.

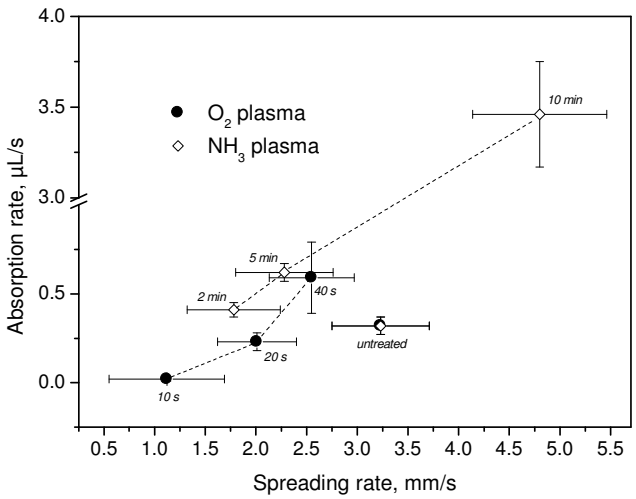

Figure 17. Correlation between spreading and absorption is the result of the partial simultaneity of both regimes.

Table 2. Wetting parameters and their standard deviations of untreated and plasma-treated fabrics.

Plasma Treatment	Exposure time	Spreading rate		Absorption rate	
		mean	σ	mean	σ
		mm/s		*µL/s*	
untreated	-	3.23	0.48	0.32	0.05
O_2	10 s	1.12	0.57	0.02	0.01
	20 s	2.01	0.39	0.23	0.05
	40 s	2.55	0.42	0.59	0.20
NH_3	2 min	1.78	0.46	0.41	0.04
	5 min	2.28	0.48	0.62	0.05
	10 min	4.80	0.66	3.46	0.29

Figure 13 shows that the spreading rate reaches a minimum value at a waviness of 121 µm, by both treatments: 10 seconds of O_2-plasma and 2 minutes of NH_3-plasma. This similarity between the two different plasma treatments suggests that waviness effectively controls dynamic wetting.

Inter-yarn porosity, i.e. the free spaces between yarns, was increased sequentially by both treatments, as shown previously in Figure 8. However, according to Figure 14, the volumetric

effect of O_2-plasma treatment on spaces between yarns had practically no influence on water absorption rate. In the case of NH_3-plasma, the water absorption rate dramatically increased from 5 minutes to 10 minutes of exposure to radiation, while the increase in inter-yarn porosity was negligible in this interval. As mentioned before, dimensional changes (relaxation/ shrinkage) of fabrics at the macro scale are related to the meso- and micro-topography by the modification of repetitive unit size and, therefore, yarn amplitude, spaces between yarns and spaces between filaments. Figure 15 shows the influence of dimensional surface changes on the water absorption rate. The initial effect of both plasma treatments was fabric shrinkage, and hydrophobisation in the case of O_2-plasma. Then, the sequential plasma irradiation led, in both cases, to fabric relaxation and hydrophilisation.

Wetting behaviour of a water drop is divided into three different regimes, according to Figure 1. Dynamic wetting or spreading, defined as growth of the drop diameter depending on time, occurs during the first seconds or in some cases micro seconds of wetting followed by quasi-static wetting, i.e. absorption into intra-yarn spaces. However, both regimes are strong interconnected, and can occur almost simultaneously. In these cases, rapid spreading allows the liquid to be in contact with more surface in a shorter time, which can accelerate liquid absorption in the intra-yarn spaces. Figure 16 shows the wetting curves for untreated and O_2-plasma treated surfaces. The relationship between water spreading and absorption due to partial simultaneity is shown in Figure 17. Leroux et al. [14] investigated the effect of atmospheric air-plasma treatments on woven and non-woven polyester textile structures and concluded that hydrophilisation depends on nano-topographical changes of filaments and on the level of surface oxidation. Additionally, they found that plasma treatments generate polymer chain-scissions of the weakest bonds of the polyester, which not only depends on plasma treatment parameters but also on textile porosity and hence on the fabric structure. However, the woven and non-woven textiles used by these authors were supplied by two different manufacturers and therefore both materials had different polyester filaments and hence different degrees of polymerisation and crystallinity, which makes it difficult to compare the chemical effect of plasma between both textiles used.

Conclusion

In the present study, the effects of exposure time of low pressure O_2- and NH_3-plasma on the morphology and topometry of fabrics on four different length scales, as well as the influence of these topographical changes on the resulting water spreading and absorption rates were investigated.

Plasma treatment on polyester fabrics led to surface oxidation and polymer chain-scissions [14] as well as an increase in filament nano-roughness. However, according to our results, the measured dimensional changes to the fabrics, as well as changes in warp amplitude and inter-yarn spaces can more clearly explain the increase of water spreading and absorption rate, i.e. fabric hydrophilicity, of polyester woven plain fabrics treated with O_2- and NH_3-plasma.

The study carried out here enables us to better understand the topographical effect of low plasma treatment on polyester fabrics on different length scales in enhancing hydrophilicity.

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