

An Analysis of the Moisture-Related Properties of Hydrolyzed Polyester

ERIN MURPHY SANDERS and S. HAIG ZERONIAN*, *Division of Textiles and Clothing, University of California, Davis, California 95616*

Synopsis

Alkaline hydrolysis causes pitting of the surface of polyester (PET) fibers and films and improves their wettability, as indicated by contact angle measurements. The enhanced wettability is due to an increase in either the number or the accessibility of polymer hydrophilic groups to water and/or an increase in the roughness of sample surfaces. The increase of void space in the PET yarn and fabric structure, induced by treatment in aqueous NaOH together with the increased wettability of the fibers, was effective in improving the moisture transport properties of the materials. The NaOH-treated PET fabrics transported the water further than isolated corresponding yarns, possibly because, in the fabrics, the spaces between the yarns acted as an additional reservoir that permitted further wicking to occur. It is apparent from immersion and equilibrium wicking capacity tests that a hydrophilic topical finish, as well as a change in the yarn/fabric structure and the hydrophilicity of their surfaces can increase the water holding capacity of PET fabric. The moisture regain and water retention values of the samples were determined, and it was found that such tests are not sufficiently sensitive to distinguish between the hydrophilicity of nontreated PET fabrics and that of PET fabrics modified either by application of a topical finish or by NaOH treatment.

INTRODUCTION

A clear understanding of the absorption and transport of water in textile assemblies is of importance for many textile processes such as dyeing and finishing or for elucidation of apparel comfort. A variety of empirical test methods have been developed to measure the moisture-related properties of fibers, yarns, and fabrics. Comparisons of the performance of different fiber and fabric assemblies in several moisture tests have been done. To date, however, no study has compared the difference in sensitivity of a variety of moisture tests to physical and chemical changes in a single fiber type and fabric structure.

Alkaline hydrolysis was chosen as the medium for the chemical and physical alteration of polyester (PET) fibers because it has previously been found to be a progressive surface reaction¹⁻³ which increases the hydrophilicity of the fiber surfaces. A PET sample treated with a hydrophilic finish was also included in the study for comparison, since the route preferred by the textile industry to modify fabric properties is by means of the application of topical finishes.⁴

The Washburn equation [eq. (1)],⁵ which expresses the distance traveled in a given time by a liquid flowing under capillary pressure, was used to measure changes in the capillary radius r and the water transport constant k_s , due to hydrolysis:

$$L = (r\gamma t \cos\theta)^{1/2}/2\eta = k_s t^{1/2} \quad (1)$$

where L is the distance traveled, γ is the surface tension of the water, t is the time, η is the viscosity of the water, and θ is the advancing contact angle.

* To whom correspondence should be addressed.

Others have been successful in the application of such equations to yarns of varying structure and fiber content.⁶⁻⁸ A comparison of the water transport rates for fabrics of different fiber contents and their individual yarns indicated that water transport within a fabric may be almost entirely due to the water transport in the yarns.⁷ In the present study, it was of interest to find if changes in the yarn capillary radii and water transport rates could be correlated with increased treatment time in aqueous NaOH.

The water holding and transport properties of the PET fabrics were measured with the following tests: drop absorbency, vertical wicking, moisture regain, water retention, immersion test, equilibrium wicking, and advancing contact angle. A cotton fabric and PET film were also tested to provide comparative data.

Materials

A heat set fabric with 100% PET fiber content (Dacron type 54) was obtained from Testfabrics Inc., Middlesex, NJ. Zelcon, a nonionic, polymeric, hydrophile (NPH) finish, was applied to a portion of this fabric by the Technical Laboratory of the Specialty Chemicals and Products Division of E. I. DuPont de Nemours Co. to give 4% on weight of fabric based on pickup. Mylar D film (2 mil thick) was obtained from the Polymer Products Department of E. I. DuPont de Nemours and Co., Circleville, OH. One surface of the film was pure PET, while the other was modified in an unspecified manner by the manufacturer.⁹ Cotton fabric was obtained from the Wade Manufacturing Co., Wadesboro, N.C. Fabric characteristics are given in Table I. All chemicals used in the treatment and analysis of the fabric were of analytical reagent grade. Doubly distilled deionized (DDD), type-I water obtained from a Millipore MilliQ system was used throughout.

TABLE I
Characterization of Fabrics

Fabric treatment	Weight (g/m ²)	Thickness ^a (cm)	Yarns per cm ^b		Yarn tex ^c	Air permeability
			(warp)	(weft)	(warp)	(m ³ /s-m ²)
<i>Polyester</i>						
None	127.2	0.010	24.5	21.2	33.9	1.43
H ₂ O, 60°C, 6 h	126.6	0.011	24.5	21.2	33.5	1.38
NaOH, 60°C, 2 h	109.4	0.010	24.5	21.2	29.3	1.62
NaOH, 60°C, 4.5 h	95.9	0.010	24.5	21.2	26.8	1.77
NaOH, 60°C, 6 h	88.2	0.009	24.5	21.2	22.9	2.08
Topical finish ^d	131.8	0.012	24.5	21.2	34.2	—
<i>Cotton^b</i>						
None	89.2	0.038	32.3	20.9	17.3	—

^a Measured at a pressure of 35 g/cm² for polyester samples and 1.4 g/cm² for cotton.

^b Polyester and cotton data taken from Refs. 20 and 16, respectively.

^c Yarn tex (weft) values for untreated polyester and cotton yarns are 20.1 and 13.1, respectively.

^d A nonionic polymeric hydrophile (NPH).

Methods of Treatment

PET fabric samples (20 × 20 cm) were treated under nitrogen with 10% aqueous sodium hydroxide solution at 60°C. To terminate the reaction, samples were rinsed several times with deionized water before steeping in 1% aqueous HCl for 3 min. They were then washed several more times with deionized water until the rinse water was acid free as indicated by litmus paper.

PET film samples (21.59 × 12.7 cm) were formed into cylinders and then treated in the same manner as the PET fabric samples.

Characterization of Products

Physical Properties

ASTM procedures were used to determine fabric weight,¹⁰ fabric thickness,¹¹ fabric air permeability,¹² and cotton yarn number.¹³

The PET yarn tensile properties, specific stress, % strain, rupture energy, and initial modulus were measured on a table model Instron Universal Testing Machine equipped with an integrator. The yarns were tested using a gauge length of 7.6 cm at a constant rate of elongation of 2.54 cm/min. Fiber and yarn diameters were measured for each treatment time with an Imanco Quantimet 720 Image Analyzer. The procedure used for scanning electron microscopy was essentially that reported earlier.¹⁴

Moisture-Related Properties

Drop absorbency measurements were based on AATCC Test Method 79-1972.¹⁵ The procedure followed for the vertical wicking test was essentially that of Weirick¹⁶ and was performed on both fabric strips (four tests per direction) and yarns (four tests per direction). For the yarn samples, the lower edge was weighted down to prevent floating. Water retention values were obtained on yarn samples centrifuged at 900 g for 30 min in an International Clinical Centrifuge Model CL in the manner described previously.¹⁶ Absorption moisture regains at 59% and 100% RH and 21°C were measured by a previously described desiccator/weighing bottle procedure¹⁶ on samples ground in a Wiley mill to pass through a 20-mesh screen. The method followed for the immersion value was essentially AATCC Test Method 21-1975.¹⁵ The equilibrium wicking test is a modified version of the vertical wicking test. A line of dye was drawn at a point close to the average height of rise as determined previously by the vertical wicking test. The dye was used to indicate whether the water had wicked to that height. The samples were left suspended as in the wicking test for 24 h. The section of fabric in between the water-fabric interface and the line of dye was cut away and weighed wet and then dried for 3 h at 110°C in an air oven to obtain the equilibrium wicking value as the amount of water held (g) per 100 g of dry sample. Contact angle measurements on film, fiber, and yarn samples were taken with a Kernco Contact Meter within 1 min of drop application. The measurements were made at 65% RH and 21°C.

TABLE II
Percent Weight Loss of Treated Polyester Fabric and Film

Treatment	Weight loss (%)
<i>Fabric</i>	
None	—
H ₂ O, 60°C, 6 h	0.14
NaOH, 60°C, 2 h	14.5
NaOH, 60°C, 4.5 h	25.3
NaOH, 60°C, 6 h	30.9
<i>Film</i>	
None	—
H ₂ O, 60°C, 6 h	0.05
NaOH, 60°C, 2 h	4.8
NaOH, 60°C, 4.5 h	11.0
NaOH, 60°C, 6 h	13.5

RESULTS AND DISCUSSION

Physical Characteristics

The increase in the percent weight loss (Table II) with time of hydrolysis and the consequent small decrease in the tenacity and elongation of the PET fabric (Table III) upon treatment in aqueous NaOH agrees with previous findings.^{2,17-19} It had been found previously that after 2 h of treatment in a 10% solution of NaOH at 60°C, with the same concentration and treatment temperature used in the present study, the density of the PET sample did not decrease.¹⁹ The intrinsic viscosity¹⁹ and the number of carboxyl end groups²⁰ did not change appreciably either when compared to the respective values of the nontreated PET. It is feasible that the aqueous NaOH attacks at the ends of the polymer chains and, thus, the number of hydrophilic end groups do not increase as much as if attack occurs in the middle of the chain. Since the described analytical data was obtained on entire samples rather than material taken from fiber peripheries, we are unable to distinguish the location of the attack. However, the fact that the density of the PET samples did not decrease after 2 h of treatment indicates that the surface was homogeneously "peeled" away and that regions of high accessibility were not solely removed. The latter regions would have lower density than the overall density of the semicrystalline fiber. If only low order regions of polymer were removed, the fiber density would have increased after the NaOH treatment.

TABLE III
Tensile Properties of Warp Yarn Taken from Polyester Fabrics*

Fabric treatment	Tenacity (gf/tex)	Specific rupture energy (gf/tex)	Breaking elongation (%)	Initial modulus (gf/tex)
None	25.32 ± 1.03	4.09 ± 0.27	30 ± 1.13	156.8 ± 5.2
H ₂ O, 60°C, 6 h	26.32 ± 0.86	4.16 ± 0.22	29 ± 0.66	161.0 ± 9.3
NaOH, 60°C, 2 h	23.87 ± 0.58	3.48 ± 0.11	27 ± 0.39	165.0 ± 7.7
NaOH, 60°C, 4.5 h	21.40 ± 0.80	2.91 ± 0.15	25 ± 0.45	139.3 ± 6.5
NaOH, 60°C, 6 h	23.55 ± 0.87	3.15 ± 0.17	24 ± 0.59	164.8 ± 7.1

* Mean values and standard errors of 10 tests calculated.

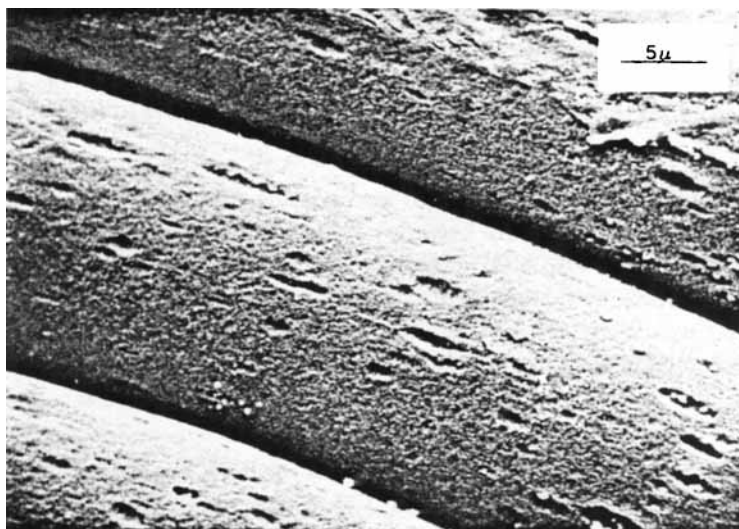


Fig. 1. Scanning electron micrograph of polyester fibers treated for 4.5 h with aqueous NaOH at 60°C.

With increased treatment time in NaOH, the PET films progressively lost weight (Table II) and in addition became opaque, which could be due to increased light scattering from the pitted surface. The smooth surface of the PET fibers and of the PET film not treated with aqueous NaOH became increasingly pitted as the aqueous NaOH treatment proceeded. The pits tended to be elongated and oriented along the longitudinal axis of the fibers (Fig. 1). In contrast the pits on the films were rounder (Fig. 2). The pits on the fiber and film may form in regions highly accessible to the alkali. Possibly the drawing process used in fiber manufacture causes the pits to be elliptical on the fibers rather than round as on the films.

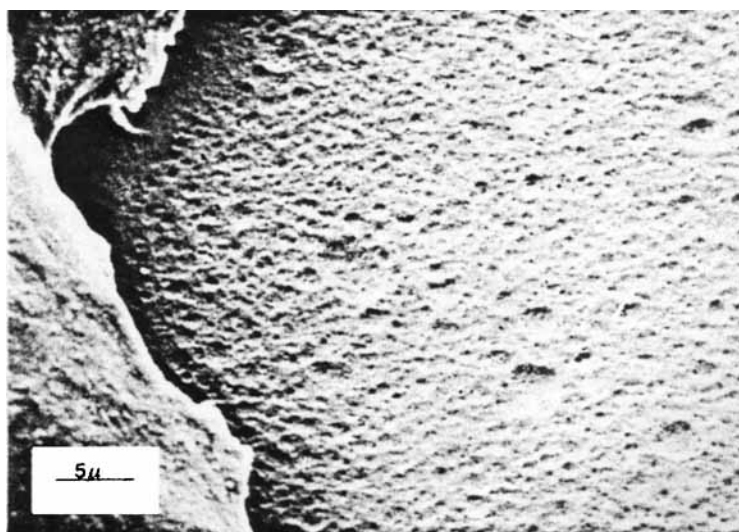


Fig. 2. Scanning electron micrograph of polyester film treated for 4.5 h with aqueous NaOH at 60°C.

The NaOH treatments progressively decreased the fiber diameters and the warp and weft yarn diameters (Table IV) as the reaction time was increased. Two different fiber sizes were found to be present in the yarns with a ratio of about 50:50. Each of these diameters is listed. The fiber linear density (tex) was calculated using the average fiber diameter and the following equation:

$$\text{tex} = d^2\pi\rho/4 \times 10^{-5} \quad (2)$$

where d is the fiber diameter (cm) and ρ is the fiber density (1.403 g/cm³).

The PET fabric structure became more porous due to the weight loss brought about by the aqueous NaOH treatment (Table I). The progressive increase in air permeability with treatment time can be represented by a linear relation $A = 0.106 t + 1.39$, where A = air permeability (m³/s·m²) and t = treatment time (h) (correlation coefficient = 0.990).

Contact Angles

Contact angles were significantly reduced for PET film, fiber, and yarn samples treated with aqueous NaOH (Table V). A previously found contact angle²¹ of 71.0° for a PET–water system is in excellent agreement with the untreated PET film value reported here. For fiber–water systems, Hollies and co-workers⁶ determined a contact angle of 79.0° and Miller²² reported a value of 75.0°. The value obtained in the present study is closer to that obtained by Hollies.

NPH-treated PET fibers had a contact angle of 72.2°, which was significantly lower than that of the NaOH-treated fibers. A contact angle considerably below those of the NaOH-treated PET fibers was also found for the cotton fibers.

Yarn (warp and weft) contact angle measurements were taken for the untreated and NaOH-treated PET samples. There is a clear decrease in contact angle due to treatment with aqueous NaOH. There does not appear to be a significant difference in contact angle between the NaOH-treated samples. The reason for the difference in contact angle between the untreated PET and the PET treated in water at 60°C is not known. The NPH-treated warp yarn again had a lower contact angle than the NaOH-treated samples. The contact angle of 30° for the cotton warp yarn is intermediate between that of the NaOH-treated PET yarn and that of the NPH-treated yarns. Hollies and co-workers⁶ reported a contact angle of 49.0° for a PET yarn. This value is much lower than the contact angle for the untreated PET yarn reported here, and is probably due to a difference in yarn structure. Chandler and Zeronian¹⁶ found a contact angle of 30.5° by the vertical rod method for the same cotton yarn as used in this study. Fisher and co-workers²⁰ found a contact angle of 26.0° for the NPH-treated PET yarn, using the vertical rod method. Both results are in good agreement with those obtained in the present study.

Contact angle measurements on yarn samples involve more than simple surface attraction. The spaces between the fibers can act as capillaries, allowing water movement within them. If the water can enter these spaces, it would seem reasonable that the yarn contact angles would be lower than that of the fiber or film, and, indeed, others have found this to be true.⁶ This effect would be greater the more hydrophilic the material is. Thus the untreated PET film and warp yarn have similar contact angles, as does the untreated PET fiber and warp yarn. In contrast, NaOH-treated PET yarns have lower contact angles than the NaOH-treated fibers and film. Yarn structure has previously been found to be

TABLE IV
Polyester Yarn and Fiber Diameters and Average Fiber Tex

Fabric treatment	Yarn diameters* (μ)		Fiber diameters ^b (μ)	Fiber tex ^c
	Warp	Weft		
None	241.0 \pm 5.9	169.2 \pm 4.0	13.0 \pm 0.19	18.2 \pm 0.12
H ₂ O, 60°C, 6 h	232.8 \pm 8.7	169.7 \pm 3.9	13.4 \pm 0.58	18.3 \pm 0.15
NaOH, 60°C, 2 h	226.2 \pm 5.1	152.2 \pm 3.8	11.5 \pm 0.16	16.5 \pm 0.14
NaOH, 60°C, 4.5 h	226.2 \pm 6.6	140.4 \pm 3.7	11.3 \pm 0.24	15.8 \pm 0.19
NaOH, 60°C, 6 h	217.5 \pm 3.8	135.6 \pm 3.1	11.2 \pm 0.42	16.0 \pm 0.18

* Mean values and standard error of 44 readings.

^b Mean values and standard error of 30 readings.

^c Calculated from $d^2 \pi \rho / 4 \times 10^{-5}$, where d = average fiber diam (cm) and ρ = density (1.403 g/cm³).

TABLE V
 Contact Angles (Degrees)^a of Water on Films, Fibers, and Yarns

Treatment	Film	Fiber	Yarn	
			Warp	Weft
<i>Polyester</i>				
None	71.1 ± 0.31	81.4 ± 0.83	70.8 ± 1.71	84.3 ± 3.36
H ₂ O, 60°C, 6 h	69.9 ± 0.35	82.6 ± 0.79	72.5 ± 4.00	63.0 ± 3.54
NaOH, 60°C, 2 h	65.0 ± 0.35	80.5 ± 1.12	34.0 ± 1.86	45.0 ± 3.00
NaOH, 60°C, 4.5 h	59.8 ± 0.55	75.9 ± 1.30	33.3 ± 1.63	38.4 ± 1.35
NaOH, 60°C, 6 h	61.5 ± 0.70	77.6 ± 1.11	34.4 ± 2.88	32.9 ± 1.13
Topical finish ^b	—	72.2 ± 1.35	26.6 ± 0.87	—
None ^c	71.0 ^d	79.0 ^e	49.0 ^e	—
		75.0 ^f		
Topical finish ^{b,c}	—		26.0 ^f	—
<i>Cotton</i>				
None	—	66.0 ± 1.45	30.7 ± 0.74	—
None ^c	—	—	30.5 ^g	—
			18.0 ^g	

^a Average and standard error of 50, 100, and 40 readings for film, fiber, and yarn respectively.

^b Nonionic polymeric hydrophile (NPH).

^c Determinations by other workers.

^d Reference 21.

^e Reference 6.

^f Reference 20.

^g Reference 16.

an important factor in yarn contact angle measurements,⁶ and so it was anticipated and found to be true that the warp and weft yarns would have different contact angles.

Possible reasons for the lowering of the contact angles for PET film, fibers, and yarns by NaOH treatment are as follows:

(a) The hydrolysis may have increased the accessibility of hydrophilic groups present on the surface of the fiber and film. It has been suggested that the polar groups are submerged inward in PET fibers,⁴ but hydrolysis may have changed this. (b) If chain scission occurred at random along the polymer chains, then the number of hydrophilic groups on the fiber and film surfaces would increase. (c) The increased roughness of the surface of the sample may be affecting the contact angle. It has been proposed that if the contact angle for a liquid–solid system is less than 90°, and the surface is subsequently roughened, the contact angle will decrease and the wettability will increase.²³ The lower contact angles and greater attraction for water observed on the NPH-treated PET and on the cotton fibers and yarns can be explained by the presence of hydrophilic groups on their surfaces. In the yarn samples, the water would be attracted to the hydrophilic surface of the fibers and so would be able to more easily enter the capillaries, thus producing a smaller contact angle. Despite its greater hydrophilicity, the cotton warp yarn had a slightly higher contact angle than the NPH-treated PET warp yarn, although the reverse is observed for the fiber contact angles (Table V). This might be due to yarn structure effects caused by fiber swelling in the case of the cotton fibers. The large drop in contact angles observed for the NaOH-treated PET warp and weft yarns may be due to a change in yarn structure (i.e., capillary size) and to an increase in the wettability of the fiber surface.

Water Transport Properties

The NaOH-treated yarns wicked water further than the untreated yarns (Table VI). In both the treated and untreated yarns, the water was wicked greater than half of the total distance traveled within the first minute. For a given treatment, there was no significant difference between the wicking ability of the warp and weft yarns after the first minute.

A significant difference in distance wicked was found between the NaOH-treated and untreated PET fabrics (Table VII). The NPH-treated PET fabric wicked the water the farthest after 10 min. The cotton fabric transported the water farther than the 2-h-NaOH-treated PET sample, but less than the 4.5-h-treated sample after 10 min. In the weft direction, both the polyester and cotton fabrics transported the water a shorter distance than in the warp direction.

When the 10-min wicking measurements are compared for the PET yarns and fabrics, it is interesting to note that the fabrics wicked much farther than the yarns. The fabric data also indicates a greater difference between the untreated and NaOH-treated PET samples. We suggest that, as water is wicked by the fabric from the tray of water, it moves from the spaces between the yarns (larger capillaries) to the spaces between the fibers (smaller capillaries). Thus two reservoirs are available to the fabrics, the spaces between the yarns and the tray of water. Single yarns, though, only draw water directly from the tray. The observation that the height of rise for the yarn samples leveled off within 5 min, while the fabric samples continued to wick, lends some support to this suggestion. The interlacing yarns (parallel to the water surface) draw water away from the vertical yarns, but may actually help in the transfer of water. The superior ability of the NPH topical finish to transport water is apparent from the fabric wicking data. Despite its hydrophilicity, the cotton fabric did not transport water the farthest, possibly due to fiber swelling.

The yarn capillary radii were calculated using the Washburn equation (Table VIII). An increase in capillary radii was found after the aqueous NaOH treatments.

The NaOH-treated yarns had larger water transport constants k_s than the untreated PET yarn (Table VIII). 1-min yarn wicking data was used in calculating the yarn k_s values, since it was observed that the Washburn equation did not hold after that time. The 10-min wicking data was used in calculating the fabric k_s values. The NaOH- and NPH-treated PET fabrics had a higher k_s than the untreated sample. The cotton fabric had a k_s that was intermediate between the 2-h- and 4.5-h-NaOH-treated samples (Table VIII).

The NaOH treatments significantly increased the ability of the PET fabric to transport water from the fabric surface into the interior of the fabric as measured by the drop absorption test (Table IX). The increased porosity of the NaOH-treated PET fabrics (Table I) was probably responsible for this observation. The larger spacing between the yarns and fibers would allow the drop to enter the fabric structure more easily. The increased wettability of the fiber surface was probably also a factor. The NPH-treated fabric absorbed water drops faster than either the PET or cotton samples because the hydrophilic functional groups attracted the water into the fabric capillaries. The cotton fabric had a slower drop absorption rate possibly because the water was preferentially absorbed into the interior of the fibers causing the fibers to swell, thus reducing the size of the capillary spaces.

TABLE VI
Polyester Yarn Wicking (cm) at Different Time Intervals

Fabric treatment	1 min		2 min		4 min		5 min		10 min	
	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft
None	0.9	1.8	1.2	1.4	1.4	1.6	1.6	1.8	1.6	1.8
H ₂ O, 60°C, 6 h	1.5	2.3	1.7	1.7	1.7	2.5	2.5	2.4	2.3	2.4
NaOH, 60°C, 2 h	2.4	2.8	2.6	2.6	2.6	3.3	3.3	2.9	3.3	2.9
NaOH, 60°C, 4.5 h	2.4	3.0	2.6	2.6	2.6	3.1	3.1	3.0	3.0	3.0
NaOH, 60°C, 6 h	2.9	2.8	2.9	2.9	2.9	3.1	3.1	2.9	3.1	2.9

TABLE VII
Fabric Wicking (cm) at Different Time Intervals

Fabric treatment	1 min		5 min		10 min	
	Warp	Weft	Warp	Weft	Warp	Weft
<i>Polyester</i>						
None	1.3	1.0	2.2	1.4	2.6	1.5
H ₂ O, 60°C, 6 h	1.4	0.9	2.4	1.3	2.9	1.6
NaOH, 60°C, 2 h	2.9	2.1	5.2	3.5	6.2	4.1
NaOH, 60°C, 4.5 h	3.8	2.7	7.0	4.7	8.8	5.8
NaOH, 60°C, 6 h	3.7	2.9	7.0	5.1	8.7	6.2
Topical finish ^{a,b}	—	—	—	—	10.5	8.6
<i>Cotton^c</i>						
None	—	—	—	—	7.2	5.4

^a A nonionic polymeric hydrophile (NPH).

^b Ref. 20.

^c Ref. 16.

Water Holding Properties

Treatment in aqueous NaOH appeared to improve the water holding capacity of the PET fabric as measured by the immersion test (Table IX). The immersion value of the NPH-treated fabric was larger than that of the untreated PET sample but less than that of the NaOH-treated PET materials. The immersion value of the cotton fabric was higher than that of all the PET samples.

The differences found between the NaOH-treated and untreated PET samples in the immersion test are probably due to a combination of effects such as the difference in the amount of void space in the fabric structures that can be filled with water, as well as the increased wettability of the sample surface. The percent weight loss data (Table II) and capillary radii calculations (Table VIII) would suggest that the increase in void space is an important factor. The data obtained with the topical finish indicates that increasing the surface hydrophilicity of the fibers is insufficient of itself to increase the water holding capacity of PET fabrics and that increased porosity is also required. The cotton fabric had a much larger immersion value than the PET samples, simply because it was able to absorb water internally, as well as hold it within its fabric structure.

In order to distinguish water held internally by the cotton fibers from that held

TABLE VIII
Warp Yarn Capillary Radii and Water Transport Constants for Warp Yarn Wicking and Fabric Wicking (in Warp Direction) Calculated from the Washburn Equation

Fabric treatment	Capillary radii ^a (μ)	Yarn wicking k_s (cm ² /s)	Fabric wicking k_s (cm ² /s)
<i>Polyester</i>			
None	0.08	0.004	0.011
H ₂ O, 60°C, 6 h	0.19	—	0.014
NaOH, 60°C, 2 h	0.30	0.018	0.064
NaOH, 60°C, 4.5 h	0.17	0.015	0.129
NaOH, 60°C, 6 h	0.21	0.016	0.126
Topical finish ^a	—	—	0.180
<i>Cotton</i>			
None	—	—	0.086

^a A nonionic polymeric hydrophile (NPH).

TABLE IX
Moisture-Related Properties of Polyester and Cotton Fabrics

Fabric treatment	Moisture regain (%) at 59% RH, 21°C	Water retention (%)	Immersion value (%)	Drop absorption, (min)
<i>Polyester</i>				
None	0.43	10.0	13.1	17.6
H ₂ O, 60°C, 6 h	0.44	12.0	11.7	20.0
NaOH, 60°C, 2 h	0.42	11.0	21.4	2.5
NaOH, 60°C, 4.5 h	0.42	10.3	23.6	0.5
NaOH, 60°C, 6 h	0.44	10.0	25.4	0.6
Topical finish ^a	0.47 ^b	10.6 ^b	17.8	0.6
<i>Cotton</i>				
None	6.30 ^c	47.1 ^c	69.8	0.34 ^c

^a A nonionic polymeric hydrophile (NPH).

^b Ref. 20.

^c Ref. 16.

by the fabric structure, the fiber saturation regain of cotton was subtracted from its immersion value. The water retention value of the cotton sample (Table IX) was taken as the equivalent of the fiber saturation value of cotton as suggested by Scallan and Carles.²⁴ The amount of water held in interfiber and interyarn voids then was 22.7%, which is quite similar to the immersion values of the NaOH-treated PET samples where only insignificant amounts of water can be held internally by the fibers (Table IX).

The weight of water held per unit area by the PET and cotton fabric samples in the immersion test was calculated (Table X). It was found that the water holding capacity of the PET samples increased slightly with treatment in aqueous NaOH. The NPH-treated sample had approximately the same water holding ability as the NaOH-treated samples. The weight of water held by the cotton fabric per unit area was much greater than that obtained for the PET samples. It is of interest to note that the data from immersion tests discriminates between the three NaOH-treated samples when presented as immersion values (Table IX) and yet, when calculated as weight of water held per unit area, it does not (Table X). This is because the immersion value is based on the original dry

TABLE X
Weight of Water Held per Unit Area in Immersion Test and Water Absorbed by Wicking at 65% RH and 21°C after 24 h

Fabric treatment	Immersion test (mg/cm ²)	Water absorbed by wicking (g/100 g dry sample)
<i>Polyester</i>		
None	1.7	19.5
H ₂ O, 60°C, 6 h	1.5	19.9
NaOH, 60°C, 2 h	2.4	41.8
NaOH, 60°C, 4.5 h	2.3	32.5
NaOH, 60°C, 6 h	2.4	38.9
Topical finish ^a	2.4	60.2
<i>Cotton</i>		
None	6.1	111.7

^a A nonionic polymeric hydrophile.

weight of fabric which differs for each sample due to the difference in percent weight loss between the samples, though the sample areas do not change.

The samples were soaked in water for both the water retention and immersion tests. However, the excess water was removed from the sample by centrifugation in the former and by passing through a wringer in the latter. Little difference was observed between the water retention and immersion values for the untreated and 60°C water-treated PET samples (Table IX). However, the immersion value became significantly larger than the water retention value for the NaOH and NPH-treated PET samples and for the cotton fabric. This would seem to indicate that the changes in void space and/or increased fiber wettability are more effective in increasing the water holding capacity of the fabrics than that of the individual yarns. It also appears that the method of water removal only matters if the sample is wettable.

The percent water transported and/or absorbed by wicking after 24 h in a standard atmosphere (65% RH, 21°C) (equilibrium wicking test) was determined (Table X). The percent water transported by wicking increased in the PET samples upon treatment in aqueous NaOH. The NaOH-treated samples were probably able to transport more water due to the change in the size of their capillary spaces and to the increased wettability of fiber surfaces as discussed previously. The NPH finish definitely increased the ability of the PET fabric to transport water. The cotton fabric held more water in its structure due to its good absorption and water transport characteristics. To separate water held within the cotton fibers from that held within the cotton fabric structure, the water retention value was used for the fiber saturation value of the cotton fibers and subtracted from the total amount held after 24 h. A value of 64.6% was calculated for water held in interfiber and interyarn spaces of the cotton fabrics. This is similar to that held by the NPH-treated fabric, where insignificant amounts of water can be absorbed internally by the PET fibers.

Both the moisture regain obtained at 59% RH and water retention values of the PET samples did not appear to be affected by either the NaOH treatments or the application of the topical finish, NPH (Table IX). Thus these techniques were not sensitive enough to detect the changes in the hydrophilicity of the PET fabrics caused by the NaOH and NPH treatments. As expected, the moisture regain and water retention of the cotton fibers were much greater than that of the PET samples.

Moisture regain measurements on the treated and untreated PET samples were made at 100% RH and 21°C over a period of 26 days. Little difference was found between the abilities of the different samples to absorb moisture. The values after 26 days ranged from 1.7% for the 6-h-treated sample to 2.5% for the untreated sample. The moisture regains were plotted against time of exposure to a relative humidity of 100%. The relations were linear.²⁵ The instantaneous regain (intercept of the slope with the ordinate axis from a plot of moisture regain vs. time) also indicates that there is little difference between the untreated and treated PET samples. These values ranged from 0.75% to 1.0%. Condensation of water on the weighing bottles occurred over time. The time at which the condensation occurred varied for each bottle and did not seem to affect the reproducibility of the data. Despite the inadequacy of the desiccator method due to condensation of water on the bottles, the values obtained were of the same order of magnitude as those reported in the literature. For example, reported

regains of 0.6–0.8%²⁶ and 1.1%²⁷ agree well with the instantaneous value found in this study while a value of around 3% for PET yarn²⁸ is slightly higher than the value reported here for extended exposure to water vapor. This data indicates that, in contrast to cotton, the water retention value (Table IX) is not an accurate measure of moisture regain at 100% RH for PET yarns.

CONCLUSIONS

There are at least three factors which may contribute to the hydrophilicity of NaOH-treated polyester: (a) increased surface roughness; (b) possible increase in the number of hydrophilic groups on the fiber surface caused by chain scission; (c) increased accessibility of the available hydrophilic groups on the fiber surfaces due to hydrolysis.

Moisture regain determinations at 59% RH and 21°C and water retention values were not sensitive enough to detect changes in the hydrophilicity of the PET yarns and fabrics due to hydrolysis or to application of a topical finish. The application of NPH, a hydrophilic topical finish, to the PET sample provided it with better water transport properties than a cotton fabric, as indicated by wicking and drop absorption tests. This may be due to the swelling of cotton fibers upon immersion in water. However, additional studies with fabrics of different weight and construction are necessary before this finding can be generalized. Cotton fabric can hold more water than PET treated with NPH since water is held within the cotton fibers as well as in the pores of the fabric and yarns. For PET rendered hydrophilic by NaOH treatment, the distance water was wicked in 10 min by individual yarns was less than that wicked by fabric composed of the same yarns.

References

1. C. G. Govindan Namboori and Malcolm S. Haith, *J. Appl. Polym. Sci.*, **12**, 1999 (1968).
2. A. De Maria, *Am. Dyestuff Rep.*, **68**, 30 (1979).
3. M. R. Padhye and A. N. Nadaf, *Ind. J. Text. Res.*, **4**, 99 (1979).
4. B. M. Latta, "Comfort Finishing of Synthetic Fabrics," in *Clothing Comfort—Interaction of Thermal, Ventilation, Construction and Assessment Factors* N. R. S. Hollies and R. F. Goldman, Eds., Ann Arbor Science Publishers, Ann Arbor, Mich., 1977 pp. 33–53.
5. E. W. Washburn, *Phys. Rev., Second Ser.*, **17**, 273 (1921).
6. N. R. S. Hollies, M. M. Kaessinger, and H. Bogaty, *Text. Res. J.*, **26**, 829 (1956).
7. N. R. S. Hollies, M. M. Kaessinger, B. S. Watson, and H. Bogaty, *Text. Res. J.*, **27**, 8 (1957).
8. F. W. Minor, A. M. Schwartz, E. A. Wulkow, and L. C. Buckles, *Text. Res. J.*, **29**, 931 (1959).
9. J. Heacock, private communication.
10. ASTM D1910-64, *Annual Book of ASTM Standards, Part 24, Textile Materials*, American Society of Testing and Materials, Philadelphia, 1972.
11. ASTM D1777-64, Ref. 10.
12. ASTM D737-69, *Annual Book of ASTM Standards, Part 30, Textile Materials*, American Society of Testing and Materials, Philadelphia, 1974.
13. ASTM D2260-64, Ref. 10.
14. M. K. Tomioka and S. H. Zeronian, *Text. Res. J.*, **44**, 1 (1974).
15. American Association of Textile Chemists and Colorists, *AATCC Technical Manual*, Howe, New York, 1973, Vol. 52.
16. J. Chandler and S. H. Zeronian, *Text. Chem. Colorists*, **11**, 20 (1979).
17. C. G. G. Namboori, *Text. Chem. Colorist*, **1**, 24 (1969).
18. J. D'Arcy, H. Hall, B. P. Ridge, and J. Whinfield, U.S. Pat. 2,590,402 (1952).

19. M. S. Ellison, L. D. Fisher, K. W. Alger, and S. H. Zeronian, *J. Appl. Polym. Sci.*, **27**, 247 (1982).
20. L. D. Fisher, K. W. Alger, and S. H. Zeronian, unpublished data.
21. A. El-Shimi and E. D. Goodard, *J. Colloid Interface Sci.*, **48**, 242 (1974).
22. B. Miller, "The Wetting of Fibers," in *Surface Characteristics of Fibers and Textiles—Part II*, M. J. Schick, Ed., Marcel Dekker, New York, 1977.
23. B. W. Cherry, *Polymer Surfaces*, Cambridge University Press, Cambridge, 1981.
24. A. M. Scallan, and J. E. Carles, *Svensk Papperstidn.*, **75**, 699 (1972).
25. E. M. Sanders, M.S. thesis, University of California, Davis, 1981.
26. R. W. Moncrieff, *Man-Made Fibres*, 5th ed., Wiley, New York, 1970.
27. I. Marshall and J. R. Whinfield, "Polyester Fibres," in *Fibers from Synthetic Polymers*, R. Hill, Ed., Elsevier, New York, 1953.
28. R. Jeffries, *J. Text. Inst.*, **51**, T399 (1960).

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