Moisture Sorption on the Wetting Behavior of Poly(*p*-Phenylene Terephthalamide) Fibers in Water and Epoxy Resin

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SYNOPSIS

The moisture sorption of poly(*p*-phenylene terephthalamide) (PPTA) fibers and the effects of moisture on the wetting behavior of these fibers in water and in an epoxy resin were studied. The moisture regains in the Kevlar 149 fibers followed a third order polynomial dependency on increasing relative humidity at 23°C. When preconditioned at 0% relative humidity (R.H.), water wettability of Kevlar 49 fibers was superior to that of Kevlar 149 fibers. Resin wettability of the dried Kevlar 49 fibers, on the other hand, was lower than that of Kevlar 149 fibers. Wettability in water and resin of these two fiber types was affected differently by moisture. Exposure to 97% R.H. moisture level significantly lowered water wettability of Kevlar 49 fibers but did not affect the wettability of Kevlar 149 fibers in water. Resin wettability of Kevlar 49 fibers was improved upon exposure to moisture, but the opposite was observed on Kevlar 149 fibers.

INTRODUCTION

The amide linkages in the poly(*p*-phenylene terephthalamide) PPTA structure enhance the strength of the fibers by inter- and intra-molecular hydrogen bonds. These functional linkages are also responsible for moisture sorption in the fibers. The moisture content of Kevlar 49 fibers was found to vary from lot to lot and has been reported ranging from 1.69 to 3.50% when exposed to 50% relative humidity at 23°.[1-3] The moisture content was associated with the Na_2SO_4 content in the fibers.[4] The moisture sorption data on Kevlar 149 is not available in the current literature.

Concerns have been raised over the effects of moisture absorption and diffusion in PPTA fibers on the performance of composites containing PPTA fibers. Moisture exposure was found not to affect the tensile properties of Kevlar fibers[4] or the longitudinal tensile properties of the fiber/epoxy composites.[5,6] However, the flexural strength and stiffness,[7] transverse stiffness, and strength and elongation[8] of the Kevlar fiber composites were reported to be significantly lowered upon exposure to moisture. To date, the published work related to the moisture effects on the PPTA fibers has mainly focused on the mechanical properties of the already-formed fiber composites. How moisture exposure to fibers affects the fiber wetting or the fiber/resin interfacial properties is not understood.

The wetting of fibers governs how the resin interacts and penetrates into the fibrous structures and is crucial to the processing, fibers/resin adhesion, and performance of the composites. In our earlier investigation, the wetting and adhesion properties of Kevlar 49 single fibers were studied with the Wilhelmy principle and a micro-debonding method, respectively.[9] The wettability of the fibers in an epoxy resin and the fiber/resin adhesion were both lowered by argon glow discharge, suggesting a possible correlation between wettability and adhesive bonding of fibers in the resin.

This study was performed with the intention of understanding two aspects of moisture effects on PPTA fibers. One aspect was the moisture sorption behavior and the other was the effect of moisture sorption on the wetting behavior of the PPTA fibers.

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EXPERIMENTAL

Materials

The poly(p-phenylene terephthalamide) (PPTA) fibers used in this study were Kevlar 49 and 149 filaments from Du Pont. The fibers were immersed in an ultrasonic carbon tetrachloride bath for 5 min, followed by rinsing in fresh carbon tetrachloride. Unless otherwise specified, all cleaned fibers were stored over desiccant in vacuum before use.

Liquids

Wetting properties of the Kevlar fibers were evaluated in two liquids, that is, doubly deionized and distilled water and an epoxy resin previously described.[9] Hexadecane was used as the total wetting liquid for fiber size derivation. The surface tensions (γ_{LV}) of these liquids were measured by the torsion ring method (ASTM D-971 and D-1331).

Moisture and Water Sorption

The moisture absorption in Kevlar 149 fibers was measured by weight gains after exposing fibers to a range of moisture levels in enclosed chambers. The water was driven out of the fibers by heating under vacuum at 105°C. The fiber weight was monitored at 30-min intervals after 2 h of drying until no weight loss was detected. The equilibrium dry weight (W_d) of Kevlar 149 fibers was obtained after a total drying time of 3 h. The dried fibers were then exposed to different moisture levels at 23°C. Moisture levels at 22.2, 32.7, 39.2, 52.8, 80.2, 84.2, and 97.2% relative humidity (R.H.) were provided by saturated salt solutions of $CH_3COOK.5H_2O$, $MgCl_2.6H_2O$, $NaI.2H_2O$, $Mg(NO_3)_2.6H_2O$, $(NH_4)_2SO_4$, KCl, and $CuSO_4.5H_2O$, respectively.[10] The fibers were weighed every 4 h, after 48 h of moisture exposure, until no further weight gains were detected. Depending upon the moisture levels, the equilibrium fiber weights were reached anywhere from 48 to 96 h from the initial exposure to moisture. Therefore, the equilibrium fiber weight obtained after 96 h of moisture exposure was used as the moist weight (W_m). Five to eight fiber samples were measured at each humidity level. Water vapor sorption was expressed as moisture regain (%):

$$\text{Moisture regain (\%)} = [(W_m - W_d)/W_d] \times 100 \quad (1)$$

Liquid water retention was measured after the fibers were immersed in water for 48 h. Capillary water was removed by centrifuging wetted fibers under a 900 gram force. The equilibrium weight (W_w) was obtained after 25 min of centrifugation. Seven fiber samples were used to obtain the liquid water retention value (%):

$$\text{Water retention (\%)} = [(W_w - W_d)/W_d] \times 100 \quad (2)$$

Wetting Force Measurement

The experimental procedures and fiber mounting method, utilizing the Wilhelmy principle, was described earlier.[9] Briefly, the wetting force (F_w) is the vertical attraction force exerted on the fiber surface as the fiber is immersed into a liquid:

$$F_w = \gamma_{LV} P \cos\theta \quad (3)$$

where γ_{LV} = surface tension of liquid, dyne/cm; P = perimeter of the fiber, cm; θ = contact angle between liquid and solid interface.

Unless described otherwise, the wetting force measurements on each fiber were taken approximately 300 to 1500 μm from the fiber tip. Five steady state wetting measurements, about 300 to 400 μm apart, were made during advancing and one steady state run was taken during receding on each fiber. The steady state measurement is defined as one that is not affected by the rate of liquid movement. Generally, the steady state wetting force can be measured when the liquid rate was sufficiently low. In this study, the rates of water movement less than 5 μm/s and resin movement rates less than 1.5 μm/s allowed the steady state measurements. Dynamic wetting was also used to study the wetting behavior during consecutive wetting cycles. The advancing and receding rates in water were maintained between 1.0 and 1.5 μm/s, whereas those in the epoxy resin were less than 1.0 μm/s.

The fibers were kept under either 0% or 97% R.H. for at least four days before testing. The 0% R.H. environment was provided by using a vacuum desiccantor, whereas a desiccantor with saturated CuSO4 solution in place of the desiccant was used to obtain the 97% R.H. Wetting force measurements were conducted under a constant 20°C temperature and 65% R.H. Each fiber sample was brought to this environment immediately prior to the measurement, which typically lasted 15 to 30 min, depending upon the rate and depth of immersion. Though moisture sorption (or desorption) could occur when fibers were moved from an atmosphere with a lower (or higher) moisture level, the effect was assumed to be small due to the short duration of exposure.

Fiber Size Determination

When a liquid wets a solid completely, the liquid–solid contact angle θ becomes zero and cosine θ is 1. Equation 3 is thus reduced to $F_w = \gamma_{LV} P$. The perimeter (P) and diameter (d) of cylindrical fibers can then be derived from the wetting force along the fiber axis:

$$P = F_w/\gamma_{LV} \quad (4)$$

$$d = P/\pi \quad (5)$$

Hexadecane with a measured surface tension of 26.7 dyne/cm was used as the total wetting liquid. Fiber size was derived from dynamic measurements along a 500 mm section on each fiber. Since fiber sizes were found to vary, for more accurate derivation of the wetting characteristics, the size of each fiber was determined. For fibers previously wetted in water, the fiber size measurements, which were found to be indifferent with or without drying, were conducted shortly after the wetting experiments. For those wetted in resin, the size measurements were made two days after the wetting experiment. These measurements were performed on sections of the same fibers, which were not previously exposed to the resin.

Fiber–Liquid Interface Characteristics

The cosine of the contact angle (θ), the work of adhesion (W_{ad}), and the contact angle of a liquid–fiber interface can be calculated from the wetting force in the liquid as:

$$\cos\theta = F_w/P\gamma_{LV} \quad (6)$$

$$W_{ad} = \gamma_{LV}(1 + \cos\theta) \quad (7)$$

$$\theta = \cos^{-1}(F_w/P\gamma_{LV}) \quad (8)$$

RESULTS AND DISCUSSION

Fiber Size

In our previous study,[9] Kevlar 49 fibers were measured to have an average perimeter of 38.8 (±4.5) μm. The average diameter was calculated to be 12.4 (±1.4) μm, assuming perfect cylindrical configuration. The average perimeter and diameter of the untreated Kevlar 149 fibers were 40.7 mm (±4.2) and 12.9 μm (±1.4), respectively. Though Kevlar 149 fibers seemed to be slightly larger than Kevlar 49 fibers, the size difference between these two fibers was not significant at a 0.05 significance level. However, the fiber-to-fiber size variations for both PPTA fibers were relatively large to warrant individual fiber size determination for deriving wetting, adhesion, and surface energy data.

Moisture Sorption

The moisture regain values measured on the Kevlar 149 fibers were 0.91 (±0.26), 1.29 (±0.16), 1.270 (±0.06), 1.53 (±0.20), 2.172 (±0.09), 2.06 (±0.05), and 3.15 (±0.47)% at the seven relative humidity levels described earlier. The moisture regains of Kevlar 149 fibers appeared to follow a third order polynomial dependence on relative humidity at 23°C (Fig. 1).

Liquid water retention in Kevlar 149 fibers ranged from 2.51 to 9.92 wt % among five samples, or an average of 5.84 (±2.57) wt %. The water retention in Kevlar 149 fibers from exposure to liquid water was not significantly different from exposure to moisture at 97% R.H. This indicates that the amount of water in Kevlar 149 fibers was similar whether the exposure was either in the liquid or vapor form.

Equilibrium moisture content in Kevlar 49 fibers was reported to be almost linearly proportional to the relative humidity at 23°C.[4] This nearly linear moisture sorption isotherm was attributed to the existence of microvoids and the lot-to-lot variation was associated with the Na_2SO_4 contents in the fibers. It was believed that moisture diffusion was aided by the interfibrillar microvoids and that the absorption to both the hygroscopic Na_2SO_4 impurities[2] and the hydrophilic PPTA molecules occurred.[3]

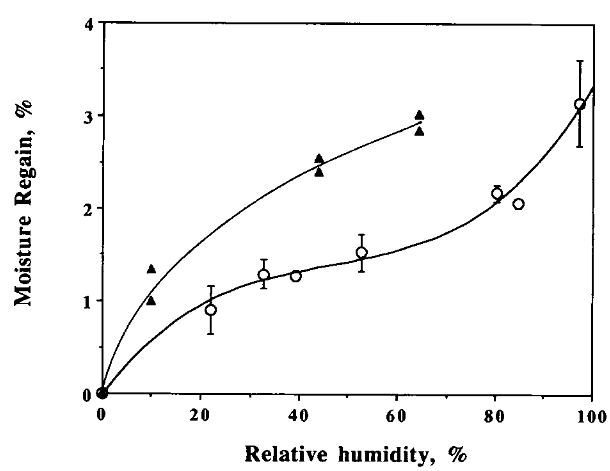

Figure 1 Equilibrium moisture sorption isotherm of PPTA fibers at 23°C: (O) Kevlar 149 fibers, (▲) Kevlar 49 fibers (data extracted from Ref. 4).

Table I Wetting Characteristics[a] of Kevlar 49 Fibers in Water: Preconditioned at 0% and 97% Relative Humidity

Relative Humidity (%)	Advancing		Receding	
	Cos θ_a	θ_a Degree	Cos θ_r	θ_r Degree
0%[b]	0.429 (±0.034)	64.6 (±1.9)	0.843 (±0.151)	32.6 (±18.4)
97%[c]	0.253 (±0.044)	75.3 (±2.6)	0.745 (±0.116)	41.3 (±9.7)

[a] Number in ( ) denotes standard deviation.
[b] Average from eight fibers with ten steady state measurements on each fiber.[1]
[c] Five steady state measurements were performed on each of the three fibers.

Table II Wetting Characteristics[a] of Kevlar 149 Fibers in Water: Preconditioned at 0% and 97% Relative Humidity

Relative Humidity (%)	Advancing		Receding	
	Cos θ_a	θ_a Degree	Cos θ_r	θ_r Degree
0% R.H.[b]	0.221 (±0.041)	77.2 (±2.4)	0.668 (0.077)	47.0 (±5.9)
97% R.H.[c]	0.267 (0.060)	75.5 (±4.41)	0.835 (0.011)	33.0 (±1.10)

[a] Number in ( ) denotes standard deviation.
[b] Five measurements were made on each of the 7 fiber specimens.
[c] Five measurements were made on each of the 3 fiber specimens.

The nonlinear moisture sorption isotherm of Kevlar 149 fibers is similar to what has been observed for hydrophilic polymers in general. At 50% R.H., the moisture regain for Kevlar 149 fibers (1.52 ± 0.20%) is lower than the reported range (1.69 to 3.50%) for Kevlar 49 fibers. Assuming both PPTA fibers are identical in their chemical structures and the extent of sodium salts, the lower moisture absorption indicates lower moisture accessibility of the Kevlar 149 fibers and possible morphological differences between the two fibers.

Wetting in Water

Kevlar 49 Fibers

The advancing and receding water wetting contact angles of Kevlar 49 fibers, preconditioned under 0% R.H., were 64.6° (±1.9°) and 32.6° (±18.4°), respectively.[9] The large standard deviation in the receding contact angle might be indication of the increased heterogeneity of the wetted fiber surfaces. Exposure to 97% R.H. significantly increased the advancing water contact angle of Kevlar 49 fibers to 75.3° (±2.6°), whereas the receding water contact angle (41.3° ± 9.7°) was not significantly changed (Table I). Moisture exposure lowered the water wetting behavior of Kevlar 49 fibers significantly during advancing, but did not affect their wettability during receding. It was also noted that the wetting force of the moisture exposed fibers was not as variable as that of the dried fibers during receding.

Kevlar 149 Fibers

The wetting characteristics of Kevlar 149 fibers conditioned under 0% and 97% R.H. were also measured in water (Table II). The advancing and receding water contact angles for the 0% R.H. preconditioned Kevlar 149 fibers were 77.2° (±2.4°) and 47.0° (±5.9°), respectively. After the exposure to 97% relative humidity, the advancing and receding contact angles were 75.5° (±4.4°) and 33.3° (±1.1°), respectively. The precondition humidity levels did not significantly affect the wetting behavior during advancing, but the fibers preconditioned at higher relative humidity level were more water wettable during receding (at a 0.05 level).

In addition to the different moisture sorption behavior in the two PPTA fibers, the effects of moisture sorption on water wettability also differ. Water wettability, as expressed by the work of adhesion values, is listed in Table III for these PPTA fibers preconditioned at the 0% and 97% R.H. levels. Under the dry state (0% R.H.), Kevlar 149 fibers were less water wettable than Kevlar 49 fibers. Moisture sorption through exposure to 97% R.H. decreased water adhesion to Kevlar 49 fibers, but it did not affect the adhesion of water to Kevlar 149 fibers. The resulting water wettability of fibers precondi-

Table III Work of Adhesion (dyne/cm) of Kevlar 49 and 149 Fibers in Water

	Kevlar 49	Kevlar 149
Preconditioned at 0% R.H.		
Advancing	103.7	88.7
Receding	133.8	122.9
Preconditioned at 97% R.H.		
Advancing	91.0	90.8
Receding	127.1	133.3

tioned at 97% R.H. was similar for the two PPTA fibers.

Wetting Cycles

The dynamic wetting behavior during two consecutive wetting cycles of Kevlar 149 fibers, preconditioned at 0% R.H. in water, was recorded (Fig. 2). The wetting hysteresis in water was indicated by the difference between the advancing and receding scans of the primary wetting cycle. During the second wetting cycle, the advancing signals fluctuated greatly. However, the wettability in water was significantly improved as indicated by the higher steady state and receding forces. The steady-state advancing contact angle was calculated to be 18.7° and the receding contact angle was 0°. This difference in behavior during the second wetting cycle may be explained by the absorbed water on the fiber surfaces. The water absorbed on the fiber surfaces can cause viscous drag, resulting in the observed force reduction and fluctuation. In addition, the surface water may also reduce the fiber–water contact angle during receding, which is detected as an increase in the attraction force.

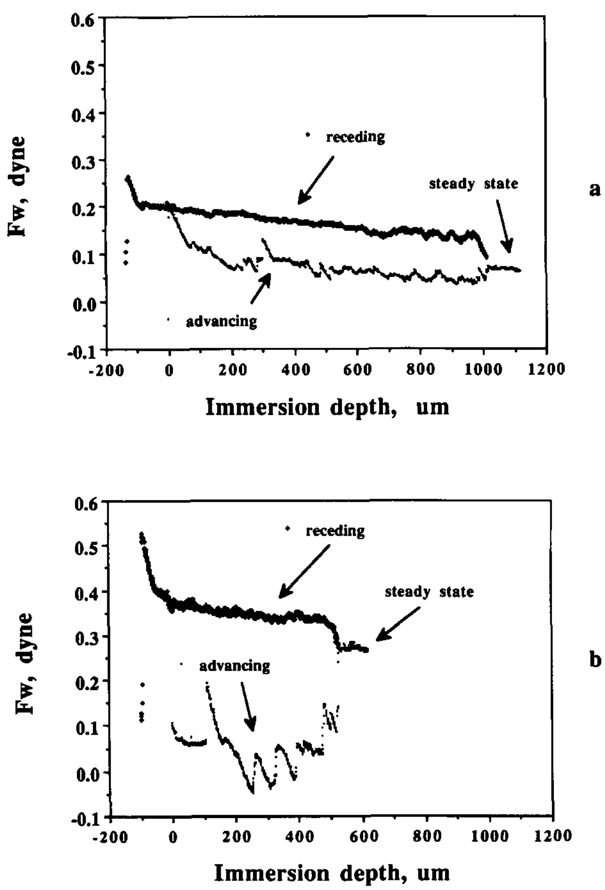

Figure 2 Dynamic wetting behavior of Kevlar 149 fibers in water: (a) first wetting cycle, (b) second wetting cycle.

Table IV Wetting Characteristics[a] of Kevlar 49 Fibers in Epoxy Resin: Preconditioned at 0% and 97% Relative Humidity

	Advancing		Receding	
Relative Humidity	Cos θ_a	θ_a Degree	Cos θ_r	θ_r Degree
0%[b]	0.858 (±0.055)	30.9 (±3.7)	0.874 (±0.050)	29.1 (±4.1)
97%[c]	0.981 (±0.018)	9.2 (±8.1)	0.986 (±0.025)	5.6 (±2.4)

[a] Number in ( ) denotes standard deviation.
[b] Average from eight fibers with ten steady state measurements on each fiber.[1]
[c] Five steady state measurements were performed on each of the three fibers.

It should be noted that exposure to moisture at 97% R.H. did not affect the water wettability of Kevlar 149 fibers, but surface absorbed water (during consecutive wetting in water) significantly improved water wettability. This observation suggests that moisture absorbed from the exposure to 97% R.H. resides mainly in the bulk of the fibers but not on the fiber surface, thus moisture does not affect water wettability.

Wetting in Epoxy Resin

Kevlar 49 Fibers

For Kevlar 49 fibers preconditioned at 0% R.H., the advancing and receding contact angles were 30.9° (±3.7°) and 29.1° (±4.1°), respectively (Table IV). As the preconditioned moisture content increased from 0 to 97% R.H., these contact angles were lowered to 9.2° (±8.1°) and 5.6° (±2.4°), respectively. The resin wettability of Kevlar 49 fibers appeared to be better preconditioned at the higher moisture level. At each moisture level, the advancing and receding wetting forces in epoxy resin were similar.

Kevlar 149 Fibers

Table V lists the wetting properties of Kevlar 149 fibers at the two preconditioned moisture levels. The advancing and receding contact angles were increased from 9.3° (±4.3°) and 2.9° (±4.2°) to 32.2° (±7.0) and 26.8° (±1.5°), respectively, when the precondition moisture level increased from 0 to 97% R.H. Again, resin wettability was not significantly

Table V Wetting Characteristics[a] of Kevlar 149 Fibers in Epoxy Resin: Preconditioned at 0% and 97% Relative Humidity

Relative Humidity (%)	Advancing		Receding	
	Cos θ_a	θ_a Degree	Cos θ_a	θ_r Degree
0% R.H.:[b]	0.986 (±0.012)	9.3 (±4.3)	0.997 (±0.004)	2.9 (±4.2)
97% R.H.:[c]	0.842 (±0.062)	32.2 (±7.0)	0.892 (±0.012)	26.8 (±1.5)

[a] Number in ( ) denotes standard deviation.
[b] Five steady state measurements were performed on each of the two fibers.
[c] Five steady state measurements were performed on each of the three fibers.

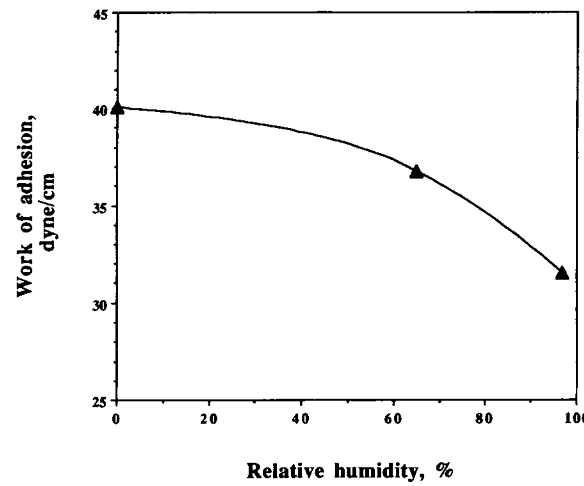

Figure 3 Relationship between work of adhesion and preconditioning moisture level of Kevlar 149 fibers.

different between advancing and receding at either moisture level.

To examine more closely the moisture effects on resin wettability, the wetting behavior of two Kevlar 149 fibers, preconditioned at 0% and 65% R.H., was measured (Table VI). For the 65% R.H. condition, the fiber was immersed in resin twice consecutively. The 65% R.H. exposure lowered the resin wettability. The second wetting cycle lowered the wettability even further and the resulting wettability was similar to the fibers preconditioned at the higher moisture level of 97% R.H. The negative effect of moisture exposure on resin wettability of Kevlar 149 fibers is clearly indicated by the relationship between the work of adhesion in resin and the preconditioning moisture level (Fig. 3).

Since the water wettability was different on the fibers previously wetted in water than those exposed to 97% R.H., the resin wettability of prewetted Kevlar 149 fibers was also examined. Figure 4 shows the wetting force measurements of a Kevlar 149 fiber in two consecutive water and epoxy resin cycles. The dynamic wetting behavior in the epoxy resin [Fig. 4(b)], following the initial wetting in water [Fig. 4(a)], was similar to that of the fibers preconditioned under 97% R.H. Hysteresis was not observed and the advancing and receding wetting contact angles in resin were 32.6° and 33.0°. The above observation indicates that surface absorbed water, either from vapor phase exposure or from direct contact with liquid water, lowers the resin wettability of Kevlar 149 fibers in a similar manner.

Table VI Wetting Characteristics of One Kevlar 149 Fiber in Epoxy Resin: Varying Relative Humidity and Immersions

Condition	Advancing		Receding	
	Cos θ_a	θ_a	Cos θ_r	θ_r
0% R.H.:	0.993	6.8	0.995	5.7
65% R.H.: First Immersion	0.927	22.0	—	—
65% R.H.: Second Immersion	0.845	32.3	—	—

The 40.63 dyne/cm surface tension of the epoxy resin is much lower than that of water (72.6 dynes/cm). The better wetting ability of the epoxy resin explains the much lower noise level observed in the wetting measurements in resin as well as the similarity in force measurements between advancing and receding and between consecutive wetting cycles.

After prolonged preconditioning at 0% R.H., little or no water is presumed to be present in or on the surfaces of the fibers. In comparison to Kevlar 49 fibers, the lower water wettability of Kevlar 149 fibers indicates lower polar interaction between water and the fiber surfaces. If the extent of polar interaction is an indication of surface polarity, then the Kevlar 149 fibers are less polar than Kevlar 49 fibers at 0% R.H. The fact that the more polar Kevlar 49 fibers are less resin wettable, and the less polar Kevlar 149 fibers are more resin wettable, indicates a possible inverse relationship between the resin wettability and the surface polarity of these PPTA fibers.

The observation made on Kevlar 49 fibers support this inverse relationship between surface polarity and resin wettability. Moisture exposure to Kevlar 49 fibers decreased their water wettability (Table III) but increased their resin wettability (Table VII). The reduced water wettability and the increased resin wettability can also be explained by the reduced surface polarity, which is consistent with the above hypothesis. However, moisture exposure on the less polar Kevlar 149 fibers significantly lowered the wettability of the fibers in the epoxy resin without affecting their water wettability. If moisture exposure did not affect the polar interaction of Kevlar 149 fibers, their decreased resin wettability has to be explained by factors other than the surface polarity of these fibers. The results of this study suggest that additional information on the surface characteristics of the PPTA fibers, such as surface energetics, may help to explain the differences in fiber wettability and the effects of moisture exposure on their wetting behavior.

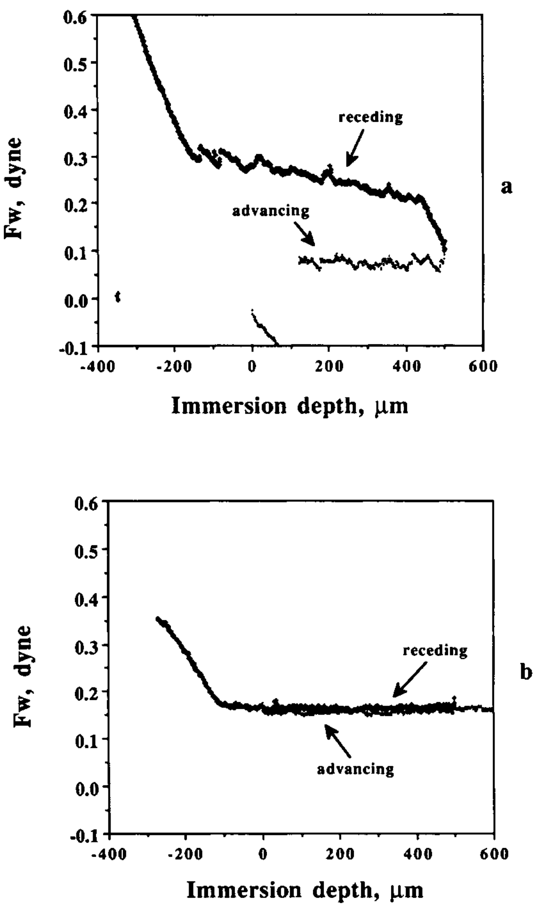

Figure 4 Consecutive wetting cycles of Kevlar 149 fibers: (a) first cycle in water, (b) second cycle in epoxy resin.

Table VII Work of Adhesion (dyne/cm) of Kevlar 49 and 149 Fibers in Epoxy Resin

	Kevlar 49	Kevlar 149
Preconditioned at 0% R.H.		
Advancing	75.4	80.7
Receding	75.9	81.2
Preconditioned at 97% R.H.		
Advancing	81.1	75.0
Receding	81.1	77.2

CONCLUSIONS

The wetting properties of Kevlar 49 fibers, preconditioned at 0% R.H., were reported earlier.[1] This article focused on the wetting behavior of the Kevlar 149 fibers preconditioned at 0 and 97% R.H. and that of the Kevlar 49 fibers preconditioned at 97% R.H. A third order polynomial dependency of moisture regain on increasing relative humidity was observed for Kevlar 149 fibers at 23°C. At 50% R.H., the moisture regain for Kevlar 149 fibers (1.52 ± 0.20%) is lower than the reported range (1.69 to 3.50%) for Kevlar 49 fibers. Assuming both PPTA fibers are identical in their chemical structures and the extent of sodium salts, the lower moisture absorption indicates lower moisture accessibility of the Kevlar 149 fibers. Moisture sorption is a diffusion process and can be highly dependent upon fiber morphology. The very different moisture sorption behavior of Kevlar 49 fibers and 149 fibers suggests possible differences in their morphology.

Kevlar 49 and 149 fibers, preconditioned at 0% R.H., had distinctly different wetting behavior in water and in the epoxy resin. The water wettability of Kevlar 49 fibers were much higher than that of Kevlar 149 fibers, whereas the resin wettability of Kevlar 149 fibers was better than that of Kevlar 49 fibers. Opposite effects of moisture exposure on the wetting behavior of Kevlar 49 and 149 fibers in these liquids were observed. Moisture exposure lowered water wettability of Kevlar 49 fibers but did not affect wetting behavior of Kevlar 149 fibers in water. The preconditioning moisture exposure, on the other hand, improved the resin wettability of Kevlar 49 fibers, but reduced the wettability of Kevlar 149 fibers in the resin. Reverse resin wettability for the two Kevlar fibers resulted from moisture exposure. Exposure to either 97% R.H. or water lowered resin wettability similarly for Kevlar 149 fibers.

The results of this study showed that the moisture sorption properties and the moisture effects on the wetting behavior of the two PPTA fibers were distinctly different. Additional research is underway to investigate the structural as well as surface energetic causes for the observed differences in these two PPTA fibers.

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