Effect of Applied Stress on the Alkaline Hydrolysis of Poly(ethylene terephthalate) at 40°C: Relevance to Medical Textiles

Mashiur Rahman,* **G. C. East**

Department of Textile Industries, University of Leeds, United Kingdom

ABSTRACT: Laboratory-accelerated degradation tests have been conducted to understand the stability of high tenacity poly(ethylene terephthalate) (PET) as a result of exposure to stressful chemical environments when used as an implantable prosthesis. The experiments were conducted at 40°C to mimic normal physiological conditions. At various time intervals, samples were collected and evaluated for weight loss (%), mechanical properties, and changes in the surface structure. The mechanical properties that were specifically observed include: breaking load, tenacity, breaking strain, work of rupture, and modulus. For all PET fibers loss of work of rupture was the highest. An empirical formula was developed to estimate degradation behavior over time, though such a predic-

tion cannot be verified without further research and observation. The transverse cracks formed on the stressed samples beyond the critical load in an alkaline environment at 40°C are similar to those found on the PET prosthetic grafts. The cracks in the experimental samples at 40°C and those recovered from human bodies both appear more 'corallite in structures' versus those at room temperature. Therefore, it is concluded that the arterial stress that the PET prosthetic graft encountered inside the human body was higher than the yield load of the fiber. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 102: 4814–4822, 2006

Key words: mechanical properties; electron microscopy; poly(ethylene terephthalate); degradation; prosthetic grafts

INTRODUCTION

Poly(ethylene terephthalate) or PET is being used for various medical applications as implantable materials such as artificial tendon, artificial ligament, vascular grafts, artificial kidney, aortofemoral grafts, and many extra-anatomic bypass grafts.[1,2] For various medical applications textile polymers must be durable in stressing conditions.[1,2] However, PET is susceptible to water or steam which causes hydrolysis of the chain. During alkaline hydrolysis, the hydroxide ions attack the electron-deficient carbonyl carbons of the polyesters to form an intermediate anion. Further reaction breaks the macromolecular chain and produces —COOH and O⁻—CH2—CH2—. The final products of the hydrolysis are hydroxyl groups and carboxylate anion groups.[3]

The use of PET in medical textiles is a relatively new development. As such the effects of blood and other surrounding fluids on the long term durability of PET is questionable, especially under stressing condition. Many deaths have been reported due to the failures of the PET prosthetic grafts.[2,4–7] In addition, there are

numerous other deaths which are suspected but remain unrecorded for various reasons, including lack of appropriate ongoing studies in patients, possible legal implications, and unperformed autopsy of deceased persons.[8] Some health professionals have already discarded the use of PET as a prosthetic grafts because of poor long term stability,[9–12] while others recommend that laboratory accelerated tests must be carried out to evaluate the long-term efficacy of PET prosthetic grafts.[13–14] Chakfe et al.[8] proposed a protocol for an *in vitro* investigation of physical and chemical properties changes of virgin polyester prostheses.

In a previous study, we showed the effect of applied stress on PET in the alkaline solution at room temperature, to investigate its long term durability in geotextile applications.[15] In the present study, we investigated the effect of applied stress on PET medical textiles at 40°C in the alkaline solution. The temperature, chemical environment, and applied stress were chosen to mimic the physiological condition of the human body. The pH of alkali used in the present study was much higher (pH ≫ 13) than the pH of the human blood (pH = 7.4),[16] and the pH of the extracellular fluid in the normal human body (pH = 7.4)[17]; however, this was necessary to accelerate the process. The results at 40°C confirm our room temperature data, and also provide proof of the formation of transverse cracks on the surface of the PET prosthetic grafts when the applied load exceeded the yield load. This

Correspondence to: M. Rahman (mashiur77@yahoo.ca).
Present address: Assistant Professor, Ahsanullah University of Science and Technology (AUST), Dhaka–1215, Bangladesh.

evidence also confirmed the findings of transverse cracks on PET prosthetic grafts removed from human bodies because of failures.[5,6]

EXPERIMENTAL

Materials

Three different types of high tenacity industrial grade polyesters were used as candidates for implants and meshes; they were, Trevira (HOECHST) type 730, 1100 dtex/200f coded as PET1; Tergel tech[®] (RHONE-POU-LENC), type 156, 1100dtex/192f coded as PET2, and standard high tenacity yarn from Far Eastern Textiles (Hong Kong), 1111dtex, 192f coded as PET3. The yarn samples were used without any prior treatment.

Methods

To subject the filament yarn to stress a method was developed using a hollow stainless steel tube 16.0 cm long. The outer diameter of the tube was 10.0 mm and the inner diameter was 9.0 mm. The tube contained 40 holes of 1.0 mm diameter in four horizontal lines and there were two 2.0 mm diameter holes at both ends of the tube. One end of the yarn was knotted with the 2.0 mm hole of the tube, while the other end was tied with the specific load. The filament yarn was wound manually on the tube without any overlapping and keeping the speed of the winding constant. After completion of the winding of a specific length of yarn, the end of the yarn was then wrapped around a screw fitted in the 2.0-mm hole at the other end of the tube and the screw was tightened to maintain the applied stress.

All of the experiments were carried out in a rotary dyeing machine (ROTADYER) and the yarn was placed in the plastic dyeing tubes with 100 mL of chemical and treated for a specific length of time at a prescribed temperature. After completing each experiment, the alkaline samples were treated in water for 5 min and then the samples were immersed in 100 mL of 0.1% HCl to neutralization. A final wash was given with distilled water. The washed samples were then dried at room temperature for 48 h.

All the chemicals used were AR grade and obtained from BDH (UK) and Vickers (UK). The solution of the NaOH was standardized with potassium hydrogen phthalate using phenolphthalein as an indicator, a procedure described by Vogel.[18]

Weight loss and mechanical properties

Weight loss of samples was obtained by taking the weight of the samples before and after treatment (dried samples) using an electronic balance. The mechanical properties of the individual filament taken from the treated yarns were determined on an INSTRON-1026 universal tester with a series-IX test method. The crosshead speed was 50 mm/min, the load cell was 500 g, and full scale load was 100 g. The fiber samples were first glued on cardboard squares with an inner dimension of 20 mm and clamped in the tensile tester at a 20-mm gauge length. Before testing, all fibers were conditioned in a standard atmosphere of 20°C ± 2°C and 65% ± 2% relative humidity for at least 48 h. At least 20 specimens of each sample were tested and their averages and standard deviations were compared.

Scanning electron microscopy

To obtain the SEM microphotograph, a Cambridge Stereoscan S360 was used, operating at an accelerating voltage of 10 kV and photographs were taken by a camera directly attached to the instrument. The fiber samples were coated with thin gold (20 nm) using a Polaron E500 sputter coating unit to a pressure of 13.3 Pa (0.1 Torr). The samples were kept under this condition for 6 min with air current flow of 18 mA.

RESULTS AND DISCUSSION

Weight loss measurement

The weight loss (%) data, together with the rate of degradation (R_D) h^{-1} of PET1 and PET2, are shown in Table I. This table tracks the results of up to 96-h treatment period, in which relaxed and different stressing

TABLE I
Weight Loss (%) of PET1 (NaOH–9.81%), PET2 (NaOH–10.06%), and PET3 (NaOH-10%) at 40°C
for Different Stress Level

| Load on the fiber, g (MPa) | Treatment time (h) | | | | | | | | | Rate of degradation (RD) h^{-1} | | |
| | 24 | | | 48 | | | 96 | | | | | |
	PET1	PET2	PET3	PET1	PET2	PET3	PET1	PET2	PET3	PET1	PET2	PET3
'0'	3.4	5.6	4.2	8.0	11.0	7.8	15.3	18.5	15.2	0.16 ± 0.01	0.22 ± 0.02	0.17 ± 0.01
100 (12.3)	–	–	–	–	–	–	18.7	–	–	–	–	–
500 (61.5)	3.5	5.4	3.8	8.2	10.8	16.2	16.5	17.6	16.2	0.1 ± 0.01	0.21 ± 0.02	0.19 ± 0.01
1000 (123)	3.4	4.8	4.1	8.3	11.6	17.5	18.9	20.6	17.5	0.17 ± 0.03	0.22 ± 0.02	0.18 ± 0.01
2000 (246)	4.5	5.4	4.2	8.5	10.5	18.7	20.2	25.2	18.7	0.19 ± 0.02	0.24 ± 0.02	0.19 ± 0.01

TABLE II
Breaking Load Data of PET1 at 40°C for Different Stress Levels, 9.81% NaOH

Load on the fiber, g (kPa)	Treatment time (h)					
	24		48		96	
	Breaking load (cN)	Breaking load loss (%)	Breaking load (cN)	Breaking load loss (%)	Breaking load (cN)	Breaking load loss (%)
Control filament	47.8 ± 2.5	–	47.8 ± 2.5	–	47.8 ± 2.5	–
0 (0)	44.8 ± 2.8	6.4	40.1 ± 4.3	16.0	36.8 ± 1.3	23.0
100 (12.3)	–	–	–	–	34.1 ± 2.9	28.7
500 (61.5)	40.7 ± 2.7	14.9	38.8 ± 3.1	19.0	34.0 ± 3.0	28.9
1000 (123)	39.1 ± 4.0	18.2	36.1 ± 1.9	24.6	31.3 ± 2.9	34.6
2000 (246)	39.0 ± 2.2	18.4	32.5 ± 2.9	32.1	19.8 ± 4.8	58.6

conditions were observed at 40°C. The weight loss (%) was approximately linear with time at a rate of 0.16% per hour for PET1 and 0.22% for PET2 for the relaxed ('0' and lower loaded yarns (12.3–123 kPa). However, after 96 h of treatment, weight loss was significantly higher for the highest loaded samples (246 kPa) than the relaxed and lower loaded samples.

The weight losses of PET2 samples were higher than the weight losses of the equivalent PET1 samples, even though PET1 is a slightly finer fiber (5.5 dtex) compared to PET2 (5.7 dtex). It must be realized that part of the weight loss for both samples is due to finish dissolving, although PET1 has the higher amount of finish (1.3%) than PET2 (1.0%). Very similar weight loss (%) result was obtained for the PET3 fiber.

The reason for the increased weight loss (%) due to highest applied stress is unknown. However, one suggestion is that the weight loss of the polyesters is related to the surface area of the fibers.[19,20] We have calculated the increased surface area of the PET1 fibers which was found to be only 2.0%, by taking the extension of 5.0% due to the 2000 g (246 kPa) applied load from the load-elongation curve. Therefore, the slight increase in surface area for PET1 (2.0%) does not account for the greater discrepancy in the amount of weight loss for the relaxed and highest loaded samples of PET1 and PET2. Another factor that might have contributed to the higher weight losses was the fact that there were broken filaments in the hydrolysis bath of the highest loaded samples.

After obtaining the very similar weight loss (%) results for all three PET, we have decided to discuss only the results of PET1 and PET2 in detail.

Mechanical properties

For both PET1 and PET2 samples, the breaking load decreased with an increase in treatment time as well as with increase in stressing on the fibers (Tables II and III). The breaking load loss of the stressed samples is higher than that of the relaxed samples and the most significant losses occurred at the highest loading conditions. For example, after 96 h of treatment, the PET1 samples which were under 246 kPa load, lost 58.6% of breaking load compared to only 23% for relaxed samples, under the same experimental conditions; PET2 samples lost 73 and 22.6%, respectively. It should also be noted that very similar loss in tenacity was observed for both PET samples.

Alkaline hydrolysis takes place only at the surface of the fibers resulting in thinner fiber; hence the percentage weight loss should be proportional to the percentage breaking load. However, a nonlinear relationship between weight loss and breaking load loss was reported due to the presence of solvents in the alkaline bath, titanium dioxide in the fiber, and crimp in the fiber.[21–23] The highest loaded samples (246 MPa) of both PET1 and PET2, in all treatment conditions, showed percentage loss in breaking load approximately three to four times than the percentage loss in

TABLE III
Breaking Load Data of PET2 at 40°C for Different Stress Levels, 10.06% NaOH

Load on the fiber, g (kPa)	Treatment time (h)					
	24		48		96	
	Breaking load (cN)	Breaking load loss (%)	Breaking load (cN)	Breaking load loss (%)	Breaking load (cN)	Breaking load loss (%)
Control filament	52.4 ± 4.3	–	52.4 ± 4.3	–	52.4 ± 4.3	–
0 (0)	50.3 ± 1.6	4.0	46.7 ± 1.3	10.9	40.5 ± 1.6	22.7
500 (61.5)	48.2 ± 1.4	8.1	43.1 ± 1.7	17.9	38.8 ± 3.9	26.1
1000 (123)	47.1 ± 2.5	10.2	42.5 ± 2.6	19.0	35.3 ± 3.4	32.6
2000 (246)	40.5 ± 5.5	22.8	29.2 ± 6.8	44.4	14.1 ± 6.3	73.1

TABLE IV
Breaking Strain Data of PET1 at 40°C for Different Stress Levels, 9.81% NaOH

Load on the fiber, g (kPa)	Treatment time (h)					
	24		48		96	
	Breaking strain (%)	Loss in breaking strain (%)	Breaking strain (%)	Loss in breaking strain (%)	Breaking strain (%)	Loss in breaking strain (%)
Control filament	20.1 ± 0.6	–	20.1 ± 0.6	–	20.1 ± 0.6	–
0 (0)	19.1 ± 1.7	5.0	18.7 ± 0.9	7.5	17.6 ± 1.0	12.9
100 (12.3)	–	–	–	–	16.1 ± 1.2	20.0
500 (61.5)	16.2 ± 1.0	19.9	15.5 ± 1.2	23.2	15.0 ± 1.0	25.6
1000 (123)	15.8 ± 1.3	21.6	15.7 ± 1.3	22.3	14.6 ± 1.6	27.4
2000 (246)	15.0 ± 1.0	25.5	12.8 ± 1.3	36.6	9.8 ± 2.1	51.5

weight. It is reported that in relaxed condition, the loss in breaking load for filament yarn can be between one and two times the percentage loss in weight,[23–25] which our study also confirms. For the highest loaded samples (246 kPa) the loss in breaking load and the loss in tenacity are very similar, therefore, the hydrolysis under applied loading conditions is no longer restricted to the surface. It is hypothesized that under higher loading conditions, the hydroxyl ions are able to penetrate deeper inside the PET fiber and causes internal hydrolysis. The scanning electron micrographs, shown later, display the formation of the deeper surface cracks under the heaviest loads.

The breaking strain (Tables IV and V) and work of rupture (Tables VI and VII) decrease with the increasing treatment time and increasing loading condition, and the loss was most significant at the highest loading condition. For both PET1 and PET2, the modulus of the fibers decreased (∼20%) significantly after 96 h of treatment. However, the decrease in modulus was not significant between the samples of relaxed condition and lower loading conditions. For all PET fibers, loss of mechanical properties during alkaline hydrolysis, both in relaxed and loading conditions decreased in the following order: modulus < breaking strain < breaking load < work of rupture; with work of rupture being the highest losing property. The data shown in the present study were obtained at 10% NaOH concentration which is unrealistic for medical applications, however, we have established that simi-lar results could be found for more diluted concentrations of NaOH under stressing conditions.[15]

Since the properties of the fibers decreased significantly when exposed to alkali and load, the PET samples were treated with water alone under loading conditions to understand the hydrolysis mechanism, and whether the loss of properties under such conditions was partly due to water hydrolysis under load. At room temperature, there were no significant changes in properties; however at 40°C, with the highest load of 246 kPa, PET1 samples shows a slight decrease (10%) in breaking load and tenacity, and a slight increase in modulus. A more significant decrease was observed in the breaking strain (20%) and work of rupture (30%). PET3 samples, under the same conditions, show a slight (10%) decrease in breaking load and tenacity, and a slight increase in the modulus. However, once again more significant decrease was observed for the breaking strain (20%) and work of rupture (30%) under highest loading conditions.

When treated with water and loading conditions, the physical appearances of the fibers did not change. The fibers retained their original luster after treatment in water for all loading conditions and at all temperatures. The handling properties of both relaxed and loaded samples were the same as the original fibers, whereas, alkaline-treated samples with higher loads tended to have a stiffer handle which will be discussed in the results of SEM microphotography section.

TABLE V
Breaking Strain Data of PET2 at 40°C for Different Stress Levels, 10.06% NaOH

Load on the fiber, g (kPa)	Treatment time (h)					
	24		48		96	
	Breaking strain (%)	Loss in breaking strain (%)	Breaking strain (%)	Loss in breaking strain (%)	Breaking strain (%)	Loss in breaking strain (%)
Control filament	17.5 ± 1.1	–	17.5 ± 1.1	–	17.5 ± 1.1	–
0 (0)	16.8 ± 1.2	4.3	16.7 ± 1.1	4.5	15.4 ± 1.1	12.0
500 (61.5)	15.2 ± 1.0	12.9	14.2 ± 1.1	19.1	14.1 ± 1.4	19.6
1000 (123)	14.3 ± 1.2	18.2	13.3 ± 1.0	23.9	12.4 ± 1.3	29.3
2000 (246)	12.2 ± 1.6	30.5	9.7 ± 2.0	44.3	6.0 ± 2.5	65.6

TABLE VI
Work of Rupture Data of PET1 at 40°C for Different Stress Levels, 9.81% NaOH

| Load on the fiber, g (kPa) | Treatment time (h) | | | | | |
| | 24 | | 48 | | 96 | |
	Work of rupture (N cm)	Loss in work of rupture (%)	Work of rupture (N cm)	Loss in work of rupture (%)	Work of rupture (N cm)	Loss in work of rupture (%)
Control filament	10.1 ± 0.9	–	10.1 ± 0.9	–	10.1 ± 0.9	–
0 (0)	7.0 ± 1.2	30.6	7.3 ± 1.4	27.9	6.5 ± 0.6	35.8
100 (12.3)	–	–	–	–	5.4 ± 0.9	46.9
500 (61.5)	6.5 ± 1.0	36.2	5.8 ± 0.9)	42.2	4.7 ± 0.7	53.8
1000 (123)	6.2 ± 1.1	39.2	5.7 ± 0.8	43.6	4.5 ± 1.1	56.0
2000 (246)	5.7 ± 0.9	43.6	3.8 ± 0.8	62.7	1.8 ± 0.7	82.0

Scanning electron microscopy

A remarkable phenomenon was observed when polyester fibers were hydrolyzed in alkali under stressing conditions. After treatment with loads of 1.5 kg (184 kPa) and 2.0 kg (246 kPa), it was noticed that the fibers had lost their brightness, an effect obvious even to the naked eye. The delustred appearance was noticed at the loads of 184 and 246 kPa and appeared to increase with increasing reaction time. Such a change in appearance was not observed when the samples were treated with alkalis alone, in either relaxed condition or lower level of loads (≤123 kPa), nor for the samples which were treated with the highest load of 2.0 kg in water alone. This delustring effect in the alkaline-treated samples is thought to be due to the presence of voids which are generated within the fibers, caused by exposure to both alkali and load.

The SEM micrographs of original PET1, PET2, and PET3 are cylindrical in shape and the surface of the fibers is smooth. Minor spots were seen on all PET samples which could be due to the particulate impurities during manufacturing of the fibers. When treated in relaxed condition with the alkali, the fiber surface remains quite smooth with a few defects detectable along the length of the fiber. These defects appear to be short, elongated pitting, parallel to the fiber axis. The presence of TiO2 is responsible for this type of pitting on the surface as reported in the literature.[22,26,27]

Figures 1–3 show the SEM microphotographs of PET1 treated with aqueous NaOH under 246 kPa

stressing conditions at 40°C for different treatment times. The surface of these samples shows deeply penetrating and acutely elongated cracks which increased with treatment time. The fiber edge seen in the samples treated for 48 h (Fig. 1) is smooth and unpitted. The same fiber at 72 h (Fig. 2) shows more pitting. After 96 h of treatment, the sample had a weight loss of 20.2% and corresponding loss in work of rupture of 82.0%, and has also lost smooth the linear edge of the fiber (Fig. 3). The surface showed deep cracking and irregularities. Similar cracks and surface irregularities were observed in the sample treated with alkaline with a load of 184 kPa. By contrast, only shallow pitting on the surface of the fibers was observed with the lower loaded samples (≤123 kPa). SEM microphotographs of PET2 samples which were treated for 48 h (Fig. 4) and 96 h (Fig. 5) respectively, with 246 kPa load also showed the same pattern of increased cracking and loss of smooth linear edge over time. The appearance of cracks on the room temperature treated samples is quite different than the appearance of cracks generated at 40°C. The cracks are rather 'dull' and 'unpronounced' at room temperature[15]; whereas, at 40°C, the heavily degraded samples of PET1 and PET2, the cracks are deeper and more acute, resembling a jagged 'corallite structures' (Figs. 3 and 5).

Both PET1 and PET2 samples show the formation of large cracks of varying length and width across the surface of the fiber. Yet there are differences in the formation of cracks between PET1 and PET2 samples. In

TABLE VII
Work of Rupture Data of PET2 at 40°C for Different Stress Levels, 10.06% NaOH

| Load on the fiber, g (kPa) | Treatment time (h) | | | | | |
| | 24 | | 48 | | 96 | |
	Work of rupture (N cm)	Loss in work of rupture (%)	Work of rupture (N cm)	Loss in work of rupture (%)	Work of rupture (N cm)	Loss in work of rupture (%)
Control filament	9.6 ± 1.1	–	9.6 ± 1.1	–	9.6 ± 1.1	–
0 (0)	8.6 ± 1.2	10.4	7.8 ± 0.7	18.0	6.0 ± 0.7	32.3
500 (61.5)	7.1 ± 0.7	26.2	6.0 ± 0.8	37.6	5.2 ± 1.2	45.8
1000 (123)	6.6 ± 1.0	30.7	5.5 ± 0.8	42.5	4.2 ± 0.8	56.3
2000 (246)	4.6 ± 1.2	51.5	2.6 ± 1.0	72.5	1.3 ± 0.7	86.7
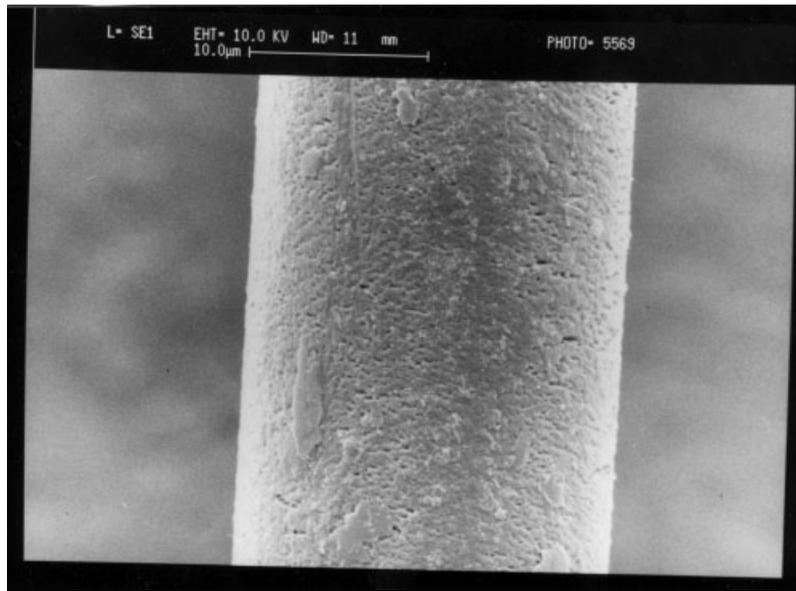
Figure 1 SEM micrograph of PET1 sample, treated with 9.81% NaOH/48 h/40°C temperature/2 kg load.

PET1 samples cracks are produced in two directions (Fig. 3), longitudinal and transverse, with most of the cracks being transverse, at right angle to the direction of applied load. In the PET2 fibers the cracks are transverse only, at right angles to the applied load. The cracks in the PET2 samples were also wider and longer. For example, the longest transverse crack in the PET1 sample was found to be 6.7 7uuml;m (Fig. 3), while in the PET2 it was 7.5 üm (Fig. 5). It is likely that the greater length of the cracks in the PET2 samples is responsible for the larger loss in mechanical properties. The differences between these two PET samples may reflect differences in processing history and in particular differences in the draw ratio and thermal history.

As the surface cracks formed only in the most heavily loaded samples, it was suspected that the load alone might have been responsible for the formation of cracks. However, SEM microphotographs of PET1 and PET2 samples which were treated with only water and 246 kPa load at 40°C temperature for 96 h show no cracks, nor any longitudinal marks such as are seen in alkaline-treated samples treated under relaxed conditions. PET sample treated with only water and highest load are very similar to the original PET sample.

The implications of the present study become significant and applicable when compared to PET prosthetic grafts removed from the deceased human bodies. In
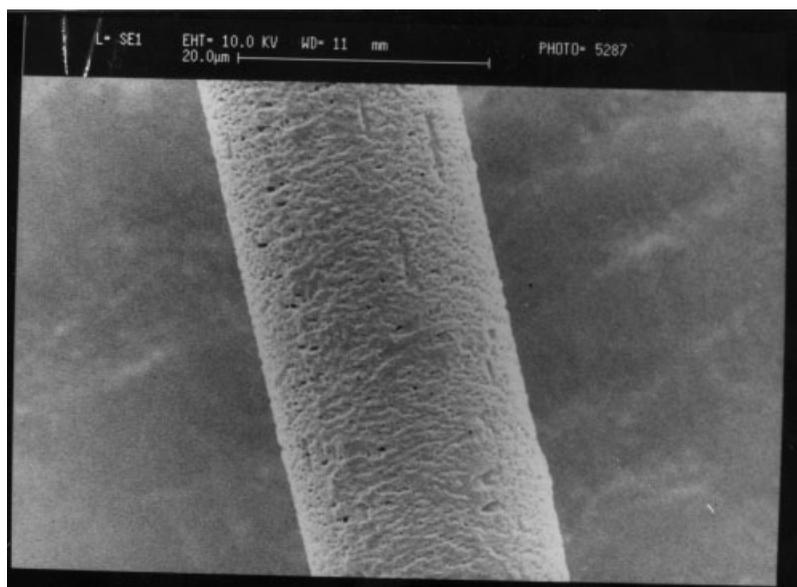
Figure 2 SEM micrograph of PET1 sample, treated with 9.81% NaOH/72 h/40°C temperature/2 kg load.
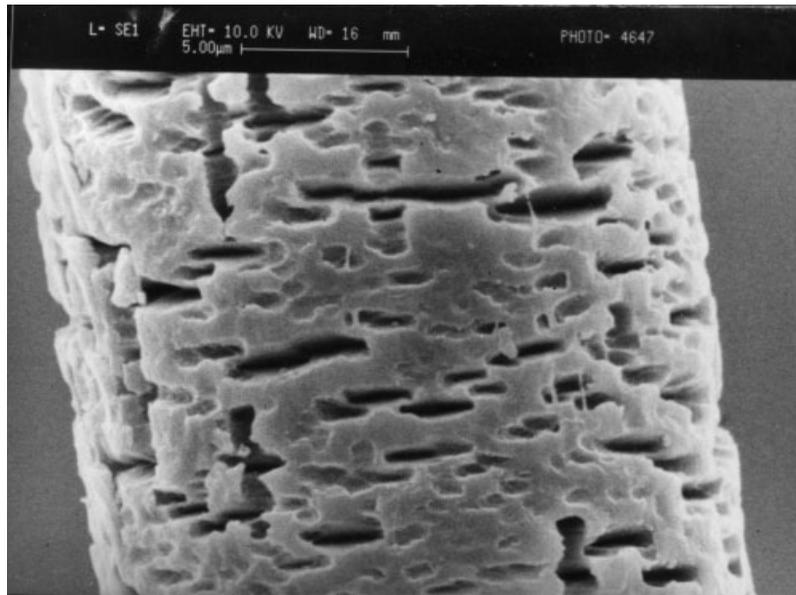
Figure 3 SEM micrograph of PET1 sample, treated with 9.81% NaOH/96 h/40°C temperature/2 kg load.

these cases, where PET failures resulted in death,[5,6] similar transverse cracks and irregularities in fiber edge were observed. We believe that the present study is first to confirm that transverse cracks formed on the PET prosthetic grafts occurs when the applied load exceeded the yield load in an alkaline environment. The yield load was measured using 'The Slope Threshold Method,'[28] which was found to be 1.22 ± 0.05 kg for PET1 and 1.24 ± 0.08 kg for PET2 which is lower than the applied load of 1.5 kg (184 kPa) and 2.0 kg (246 kPa). In certain parts of the human body, the PET prosthetic grafts remain under stressing conditions which is greater than critical level of load.[5] Where the

environment is similar to the alkaline condition, deep transverse cracks formed, which eventually cause failure in the PET prosthetic grafts, resulting in death. We speculate that the higher temperature (40°C) is responsible for an increased mobility of the OH⁻ ions, thus producing deeper penetration in PET fibers through the highly stressed ester bonds and resulting in the 'corallite structures' observed.

STATISTICAL ANALYSIS

The sodium hydroxide hydrolysis data obtained in the laboratory accelerated tests revealed a small effect of
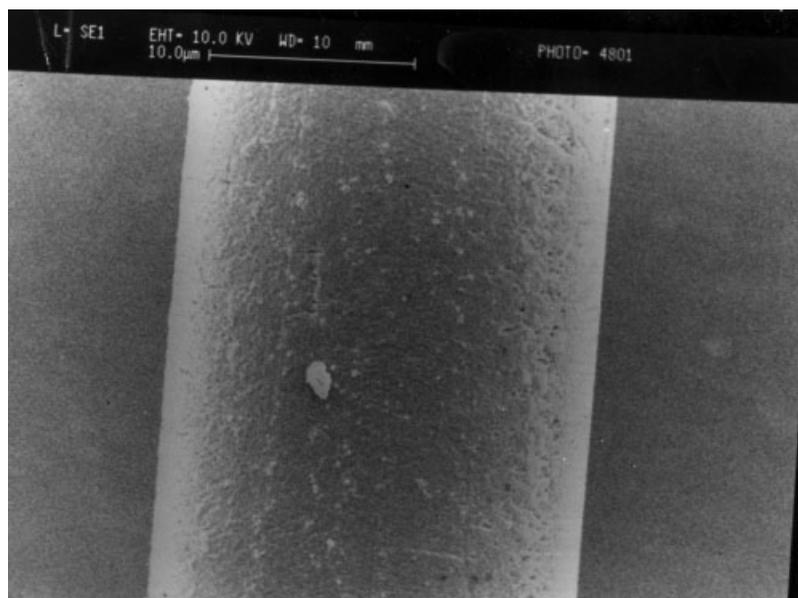
Figure 4 SEM micrograph of PET2 sample, treated with 10.06% NaOH/48 h/40°C temperature/2 kg load.
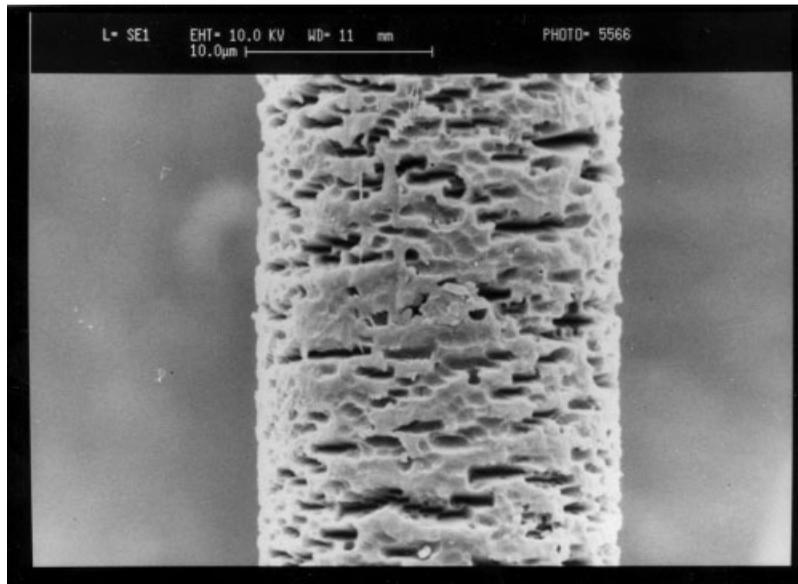
Figure 5 SEM micrograph of PET2 sample, treated with 10.06% NaOH/96 h/40°C temperature/2 kg load.

lower level of load. However, marked decreases in properties were obtained with the higher loads. Empirical relationships were examined using Statistical Package for Social Science (SPSS), a computer program used to analyze the data.[29] It was found that for a given concentration of sodium hydroxide and temperature of treatment, the breaking load can best be described by the following equation:

$$Y = \beta_0 - \beta_1 T - \beta_2 L^2 \qquad (1)$$

where Y = filament breaking load (cN); T = treatment time (days); L = applied load (g); 95% confidence limit for a prediction of $Y = Y^2 \pm t_{0.025}S$, [S^2 is the variance about regression]

From eq (1) it can be seen that the loss of breaking load in aqueous sodium hydroxide solution is linear with the treatment time and with the square of applied load over the range of values considered. The following equations were obtained for two different types of high tenacity PET at 40°C temperature:

For PET1, at 40°C temperature and 9.81% NaOH:

$$Y = 47.1 - 3.44T - 2.26 * 10^{-6}L^2 \qquad (2)$$

($R^2 = 0.85$, $S = 2.88$, 95% confidence limit = ±6.27).

For PET2, at 40°C temperature and 10.06% NaOH:

$$Y = 56.4 - 4.79T - 4.36 * 10^{-6}L^2 \qquad (3)$$

($R^2 = 0.90$, $S = 3.56$, 95% confidence limit = ±7.77).

The predicted values from the empirical equations are shown in Table VIII. The differences (errors of prediction) are all within the 95% confidence limits as quoted above. The form of these equations helps to explain why the effect of load is only slight at low loads but very serious at higher loads. The equations for PET1 and PET2 have similar constants and that the equations seem to fit the room temperature results[15] rather better than the data at 40°C. These equations are, of course, only empirical and have been obtained for very high pH of alkali, obviously similar equations could be obtained for lower, perhaps more realistic pH of alkali which is close to the alkalinity of physiological condition.

We should also mention that the data given in the present study is presented in a table format, so that it may be readily accessed for use in other applicable medical situations. For example, we can easily analyze the work of rupture data for PET fibers, which may be a relevant property when considering patients who are subject to hypertensiveness.

CONCLUSIONS

The direct comparison of weight loss and mechanical properties data with published results is difficult due

TABLE VIII
Predicted Breaking Load of PET1 and PET2 from eqs. (2) and (3) Respectively

Load on the fiber, g (kPa)	Treatment time (h)					
	1 (24 h)		2 (48 h)		4 (96 h)	
	PET1	PET2	PET1	PET2	PET1	PET2
'0'	43.7	51.7	40.2	47.0	33.3	37.6
100 (12.3)	–	–	–	–	33.2	–
500 (61.5)	43.1	50.6	39.6	45.9	32.7	36.5
1000 (123)	41.4	47.3	37.9	42.6	31.0	33.3
2000 (246)	34.7	34.2	31.2	29.5	24.2	20.1

to the lack of information regarding PET materials. Most of the literature provides only registered or commercial names of the products, such as Dacron®,[6,7] Dacron Weavenit,[30] Dacron Milliknit,[31] Cooley Double Velor,[32] and so forth. Only one work could be found that provided the full specification of PET prosthetic grafts as ''semi dull, round cross section, textured multifilament, Type 56T Dacron polyester yarn.''[33] Unfortunately, this particular type of polyester is an apparel grade PET, hence direct comparison cannot be made. Another work was found which did simulate PET as a prosthetic graft, and reported that deep notches occurred when PET ruptured at a stress of 60 N/cm.[14] However, the authors failed to mention whether or not this was the yield load.

A few studies have actually attempted to confront the issue of degradation in PET (Dacron®, Stentor, Vanguard, Aneur-X, and Talent) as a prosthetic graft,[8,13,14] however, the experimental methods used and the data collected made no specific conclusions as to why this textile failed in the human body. Contrastingly, our present study does propose a specific cause of PET failure, as seen in the comparison of SEM micrographs of PET samples exposed to applied load and alkaline environment at 40°C, and those of PET recovered from deceased human bodies. It is believed that the present study offers the first answers to the possible causes of PET prosthetic graft failures which have resulted in death. The present study also answers the question of how to test the biomechanical properties of PET[8] prior to being used as prosthetic grafts.

We want to emphasize that the present study leads us to believe that high tenacity PET can be a useful material as a prosthetic graft so long as it is not used in stressing conditions above its yield load. In general, the implications of the present study supports the view that, because of the stress and chemical environments inside the human body, regular follow-ups of patients with implantable materials made from high tenacity polyester are required for a possible replacement, as the long term durability has not yet been conclusively determined.

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