

TECHNICAL NOTE

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The Effect of Exposure to the Elements on the Forensic Characterization by Infrared Spectroscopy of Poly(ethylene terephthalate) Fibers*

ABSTRACT: Validation of a recently proposed differentiation method for single poly (ethylene terephthalate) (PET) fibers, based on integration of IR absorption bands, is presented. IR microscopy in the transmission mode, differential scanning calorimetry and optical microscopy were employed. The reliability of the technique has been extended to the cases in which the fibers being compared have experienced long exposure to the elements, as may happen if these items are recovered from open-air crime scenes. The effect of immersion in water and irradiation by the sun for a three-month period have been considered. The fibers remained morphologically and structurally unaltered. The significance of comparisons between fibers kept in different environments is thus enhanced, because the detrimental effect of the elements can be ruled out. This further validation of the proposed technique contributes to render the data presented by the forensic scientist less vulnerable to context effect and bias.

KEYWORDS: forensic science, fiber analysis, contact traces, polyethylene terephthalate, Fourier transform infrared spectroscopy

Textile fibers found at a crime scene can be very useful traces in the hands of investigators (1,2). They can provide information about contact between victim and suspect, or they can help in proving a connection between the crime scene and the persons implicated in the felony. Currently, forensic analysis of these items is mainly of a comparative kind: a sample of unknown origin is examined in order to determine if it could share a common origin with fibers from a known source. It is thus paramount that analyses yield significant results to be used in a court of justice. The scientist should take care that a complete and comprehensive protocol is performed, by which it can be possible to discriminate apparently similar fibers with a significant confidence. When characterizing a sample, the aim is to rigorously assess its rarity and evidential value, through a complete and specific description of the item. Textile fibers are usually characterized by observing their morphology with an optical microscope, by qualitatively identifying the polymer composition by Fourier Transform Infrared (FTIR) spectroscopy (3) and by a microspectrophotometric comparison of color (4), if the items are dyed. Other techniques such as thin layer chromatography, high performance liquid chromatography or capillary electrophoresis may complement the comparisons (5) but are applicable just if the fibers are colored. Colorless fibers are occasionally encountered in casework, though, and some other instrumental method (6) could be needed to replace microspectrophotometry.

With the advent of FTIR microscopy, it has become easier and faster to gather infrared (IR) data even on the very small fragments usually encountered in case investigation. The aim of forensic scientists, when characterizing a fiber, is to offer the most complete and specific description of the sample. This scope is quite easily accomplished when dealing, for example, with acrylics (7) that display a big variability in comonomer composition, even though the most common are the copolymers of acrylonitrile with, respectively, vinyl acetate, methylacrylate and methylmethacrylate (8), and thus can be efficiently sub-classified. Moreover, acrylics of the same composition can contain residues of different solvents used in the spinning process (7), therefore they can be further differentiated on the basis of this feature. Polyester fibers are, on the contrary, less variable in monomer composition (9). Although this class comprises poly (ethylene terephthalate) (PET), poly (cyclohexyl dimethylene terephthalate), poly (terephthalic acid: *p*-hydroxybenzoic acid: ethylene glycol), poly(hydroxyethoxybenzoate) and poly(butylene terephthalate) (PBT), PET is by far the most common subclass, PBT is rare, while the other polymers are practically never encountered in casework (8). Given this small variation in composition, the problem of classification of polyester fibers is one of relevant importance in the forensic science field.

Recently, a method has been proposed to differentiate morphologically similar colorless PET single fibers, using FTIR microspectroscopy (10). Forensic scientists often ignore colorless polyester fibers because they consider them too common to be significant. Actually, it has been reported in several frequency studies (11–14) that these items account for just about 1% of the general population of fibers found at random, making them not as ubiquitous as many analysts think. Even though choosing colored and infrequent target fibers is highly desirable, it may happen though to deal with

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casework in which colorless synthetic fibers should not be ignored. In this paper, a further validation of the proposed technique (10) is offered, by investigating how atmospheric elements influence the results of these analyses. It may in fact happen that fibers that remained on open-air crime scenes, exposed for long periods to the elements, must be compared to items coming, for example, from the well protected closet of a suspect. The effects of sunlight and water were monitored during a three-month period.

Materials and Methods

Samples

Fiber samples were obtained from the RIS (Reparto Investigazioni Scientifiche di Roma, Carabinieri, Italy) private collection. Ten colorless PET commercial samples were chosen. The different items were designated with a letter, representing the manufacturer, and a number, in case of more than one sample by the same producer (Table 1). Samples C1 through C3 were manufactured in the same location, C1 in a batch plant, C2 and C3 in a continuous plant, but with different fillers: a normal TiO₂ delustrant (C2) or a ceramic filler intended to strengthen the mechanical properties (C3). C4 was produced in a different industrial site with respect to the other C samples. F2 is different from F1 because it was treated to be flame retardant. The diameter of the fibers, which were all moderately delustered and all circular in cross-sectional shape, was $20 \pm 1 \mu\text{m}$ and the same for all the items. All the fibers matched after a comparison of the morphology by optical microscopy. The samples were subjected to conditions simulating exposure to sunlight and immersion in water. In the first experiment, a $220 \times 300 \text{ mm}$ cardboard box was internally lined with black bristol board, to favor the absorption of radiation. A short length of loosely bound tow (about 2 cm) was cut for each sample, in order to have several tens of fibers available for the measurements. The ten samples were attached by adhesive tape to the bottom of the box and were exposed to direct sunlight in Rome, from July 24th 2003 to September 24th 2003. Care was taken to orient the box in order to avoid the formation of shades on the samples. During all that period it never rained. Samples were left outside during the night, to make fibers experience a realistic alternation of high (around 45°C in the hottest days) and low temperatures (about 20°C in the coldest nights). Humidity hovered, in the period considered, around 75%.

In the second experiment, a number of fibers analogous to those exposed to sunlight were closed in sealed gas-chromatography vials

filled with water. The samples remained immersed in water from July 24th 2003 to September 24th 2003. Soaked fibers were adequately and thoroughly dried with paper towels before analyses.

Infrared Spectroscopy

IR absorption spectra were acquired on a Nexus FTIR spectrometer (Thermo Nicolet, Waltham, MA) equipped with a Continuum microscope (Thermo Nicolet, Waltham, MA). A MIR Globar source was used and the detector was of the MCT/A type. The spectral region spanned from 4000 to 650 cm⁻¹, with a resolution of 4 cm⁻¹. 256 acquisitions were gathered. Samples were suspended across slits in order to make measurements in the transmission mode. Both apertures were used, and aperture widths were kept as large as possible, taking care to exclude fiber edges, and were the same for all the samples. All fibers were oriented parallel to the y-axis on the microscope stage. No flattening was performed, not to alter the crystalline structure of the material. For each sample, the spectra of 10 different, randomly selected, single fibers were gathered, in order to observe intra-sample variation. Measurements were repeated 8 times on the same spot and along 8 different locations of the same single fiber to verify the repeatability of the data. Integration of the peaks was performed by making use of the same integration limits for all the samples. These limits were chosen to encompass about 95% of the total area, excluding the band edges (15). Nicolet Omnic software was used for the treatment of spectra.

Differential Scanning Calorimetry

All the measurements were carried out with a TA Instruments mod. 2920 (TA Instruments, New Castle, DE) calorimeter operating under N₂ atmosphere. Fiber samples weighing about 5 mg were closed in aluminum pans. A heating rate of 10°C/min was set, in order to observe the polymer melting peak. No erasure of thermal history was performed because it was of interest to evaluate the relative crystallinity of the samples as they were, taking thus into account every treatment they could have undergone since the synthesis of the polymer. Indium of high purity was used for calibrating the DSC temperature and enthalpy scales. The choice of the baseline in enthalpy evaluations has been standardized for all samples. Crystallinity (χ_c) was obtained using the expression $\chi_c = \Delta H / \Delta_m H^0$, where ΔH is the measured melting enthalpy and $\Delta_m H^0$ is the melting enthalpy of 100% crystalline PET, equal to 115 J/g (16).

TABLE 1—Absorbance ratios, with the corresponding relative standard deviations (indicated in parentheses), measured for the 10 PET samples untreated, immersed in water for three months (water) and irradiated by sunlight for three months (sunlight). The crystallinity of the untreated samples is also displayed, as obtained by DSC.

Sample	A ₃₄₄₀ /A ₈₇₄			A ₁₃₇₀ /A ₈₄₆			Crystallinity (%)
	Untreated	Water	Sunlight	Untreated	Water	Sunlight	
A	5.3 (6%)	5.4 (3%)	5.1 (8%)	0.4 (10%)	0.4 (7%)	0.4 (5%)	54.6
B	1.0 (10%)	1.0 (3%)	1.1 (5%)	0.3 (7%)	0.2 (6%)	0.3 (6%)	56.0
C1	0.7 (10%)	0.6 (7%)	0.7 (8%)	0.3 (10%)	0.3 (7%)	0.3 (8%)	54.6
C2	2.8 (4%)	2.8 (10%)	2.9 (5%)	0.5 (7%)	0.6 (5%)	0.5 (6%)	54.9
C3	2.3 (9%)	2.4 (6%)	2.4 (8%)	0.3 (4%)	0.2 (7%)	0.3 (3%)	54.2
C4	7.3 (7%)	7.2 (5%)	7.1 (5%)	0.3 (8%)	0.2 (10%)	0.3 (5%)	54.0
D	0.9 (9%)	0.8 (10%)	0.9 (7%)	0.5 (4%)	0.5 (7%)	0.6 (5%)	53.0
E	4.3 (2%)	4.4 (6%)	4.3 (4%)	0.3 (9%)	0.3 (5%)	0.2 (10%)	54.1
F1	0.9 (10%)	0.9 (7%)	1.0 (5%)	0.3 (8%)	0.3 (4%)	0.3 (3%)	55.0
F2	1.0 (4%)	1.0 (4%)	0.9 (5%)	0.3 (3%)	0.3 (2%)	0.3 (8%)	54.2

Optical Microscopy

For their morphological characterization, single fibers were mounted in XAM-neutral medium improved white (Gurr BDH, Poole, United Kingdom) on glass slides. Traditional methods of bright field transmission observation were adopted. The microscope used was an Axiotron (Zeiss, Oberkochen, Germany) equipped with PlanNeofluar 10 \times , 20 \times and 40 \times objectives. Comparisons were performed using a DM-R comparison microscope (Leica, Wetzlar, Germany).

Microphotographs were taken with a camera mounted on the Leica DM-R comparison microscope by a Leica WILD MPS 52, coupled with a Leica WILD MPS 46 exposition system. The film used was the 160 ASA Kodak.

Results and Discussion

A discrimination method for PET single fibers, based on unpolarized IR spectroscopy, has recently been proposed by this group (10). Four peaks in the IR spectrum, namely at 3440, 1370, 874 and 846 cm^{-1} , are integrated and two ratios of absorbancies are computed: that between the peaks at 3440 cm^{-1} and at 874 cm^{-1} (A_{3440}/A_{874}) and that between the signals at 1370 cm^{-1} and 846 cm^{-1} (A_{1370}/A_{846}) (Fig. 1). Ratioing of the signals was necessary to exclude the influence of the sample size on the intensity of the peaks.

The band at 3440 cm^{-1} is originated by the O-H stretching of diethylene glycol end-groups (17), while that at 874 cm^{-1} has been reported as a suitable normalization band relative to the aromatic backbone of the polymers (18–21). Thus, the A_{3440}/A_{874} ratio is proportional to the abundance of end groups in the chains, and consequently to the molecular weight of the polymer.

The second ratio considered for differentiation, A_{1370}/A_{846} , is related to the conformation adopted by the chains. In PET, the ethylene glycol linkages exist in two rotational conformations: a *trans* or extended form and a *gauche* or relaxed form. The passage from *trans* to *gauche* and vice versa is possible by rotation about C-C bonds. *Trans* conformers give rise to the bands at 846 cm^{-1} , while *gauche* ones origin the peaks at 1370 cm^{-1} , so A_{1370}/A_{846} represents the *gauche/trans* ratio. This quantity is, consequently, also related to the crystallinity of the fiber, because the *gauche* content is lower in the more crystalline samples (10,20,21). Crystallinity is a very process-dependent property of polymers, so it can be very useful in catching the subtle differences between fibers manufactured by different producers or even between batches by the same producer.

The representation of each sample on the plane formed by ratios A_{3440}/A_{874} and A_{1370}/A_{846} was very effective in differentiating the samples (10). Moreover, a categorization opportunity loomed, because the employed fibers appeared to be divided into three clusters (10). It was beyond the scope of the study to develop a method to categorize fibers according to the producer, because variations in the

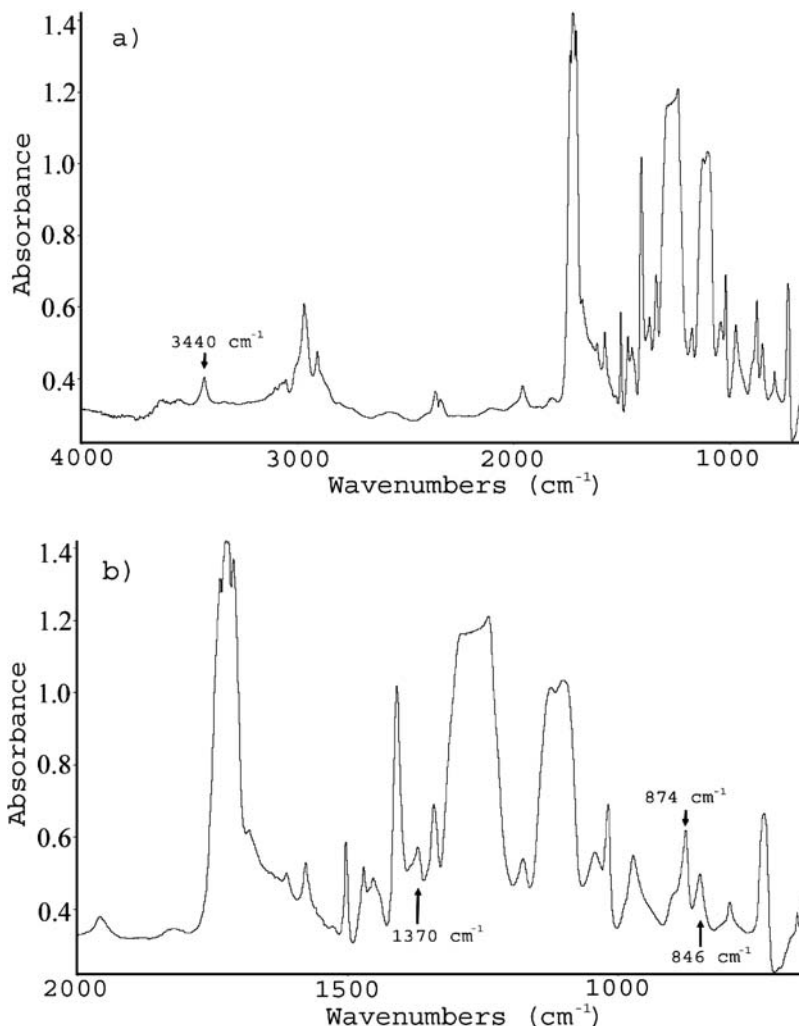


FIG. 1—IR spectrum of sample F2: (a) entire range and (b) 2000–650 cm^{-1} region. Arrows indicate the absorption bands chosen for quantification.

polymerization process by the same manufacturer over time may determine structural differences between different batches. In fact, varying the process parameters, producers can change the structure of the fibers and thus their mechanical properties. This way they can meet all different customers' requests, producing fibers of first or second choice, for different end-uses, starting from recycled raw materials, etc. The clustering into three groups is then not dependent on the producer, but rather on the end-use for which the fiber was intended. The usefulness of this method should be limited to comparisons between two items, to assess if they may share a common origin, and not to those cases in which, given a fiber coming from the crime scene, it is requested to describe the item of clothing that originated it and its producer.

It may be useful to note that some lab coats are cotton/polyester, and this may cause contamination problems. These may easily be overcome if, as it should be normal practice, in the examination of fiber-related items protective non-woven outer clothing were worn.

Although this procedure can be used in the comparison of PET fibers that experienced a similar treatment before being seized as items of evidence, it remained to show its validity when one of the samples being compared was left to the elements for a long time. Exposure to sunlight and immersion in water for three months were simulated. After this period the IR spectra were gathered on single fibers, as described in the Materials and Methods section, and integrated and the data were compared to those obtained on the same fiber that had not undergone the treatment.

The results are reported in Table 1. As can be seen, no significant difference in the intensity ratios can be detected, within the experimental error. If the measures were taken on the same spot or along different locations of the fiber, the relative standard deviation of the ratios was about 2 and 4%, respectively. As it can be seen in Table 1, results pertaining to different single fibers of the same sample are, in all cases, reproducible within 10%. This relatively high uncertainty lies in the acquisition mode selected. As already said, in fact, the spectra were gathered in the transmission mode without any flattening. This brings about very intense bands, with a loss in the accuracy and precision of absorbance data. It was chosen to minimize sample preparation, because any alteration to the crystalline structure of the polymer would have led to a variation in the *gauche/trans* content. One of the central aims of this study was to estimate how slightly different production processes of apparently

similar fibers influenced the structure of the end product. It is worth reminding that in casework fibers are routinely flattened before IR spectroscopy. Adoption of the proposed technique (10) would then imply gathering an IR spectrum of the unflattened item, and after that, if necessary, to proceed with the usual flattening. As already pointed out, in fact, estimating the absorbance ratios on flattened data would not produce reliable data. The addition of this further step in an examination protocol is justified by the possibility of obtaining quantitative data that may complement the comparison and make more objective the results.

Figure 2 shows the 3440 cm^{-1} band of PET for sample C1 before and after the treatments with water. It has been previously reported (22) that the diffusion of water into the polymer framework produces a shift towards higher wavenumbers and a great increase in the intensity of the peak at 3440 cm^{-1} . No such behavior has been noted: the 3440 cm^{-1} band does not show significant changes, within the experimental error, in position and intensity, whether the sample has been treated by immersion in water or not. Much more evident variations would have been expected in case of water absorption (22). Moreover, when optical microscopy was employed to compare the fibers before and after the weathering process, no swelling or morphological alteration was notable (Fig. 3). No change in the *gauche/trans* ratio was produced by immersion in water, even though it has been argued that water absorbance can induce some conformational modification (23,24).

The reason for this unexpected behavior could be in the structure of the fibers analyzed. The transport of water in polymers depends on a number of factors (22):

1. The chemical structure of the repeat units (acid, amide, ester, etc.);
2. The polymer micro-structure (molecular weight, morphology, crystallinity, glass transition temperature, etc.);
3. The chemical nature and levels of filler and other additives; and
4. The temperature.

All the analyzed samples had the same structure and were kept immersed in water at room temperature, but were different, even though slightly, for microstructure (especially crystallinity and, to a less extent, molecular weight) and probably filler content (this

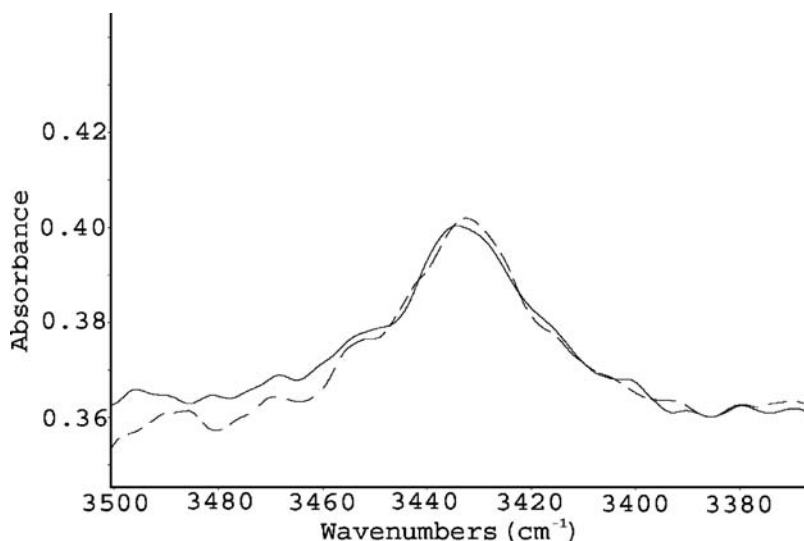


FIG. 2—Particular of the 3440 cm^{-1} IR band of sample C1 untreated (solid line) and kept in water for three months (dotted line).

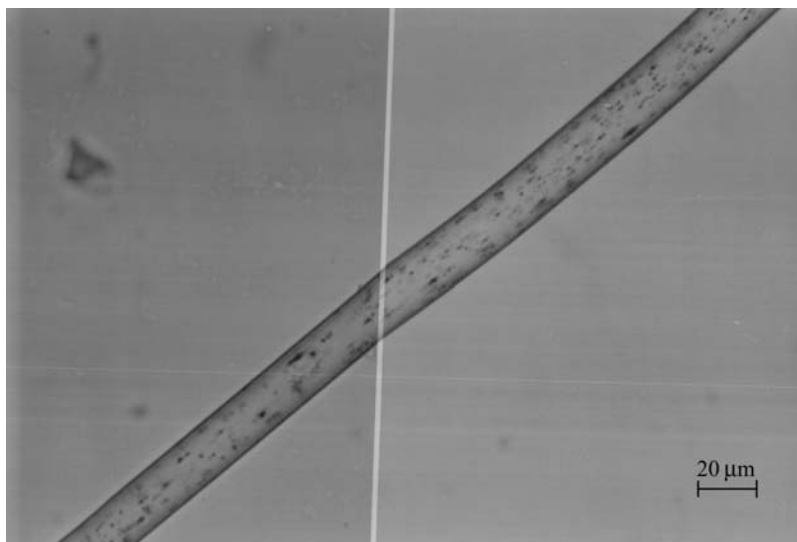


FIG. 3—Comparison of the morphology of sample C2 untreated (left) and after immersion in water for three months (right). The scale corresponds to 20 μm .

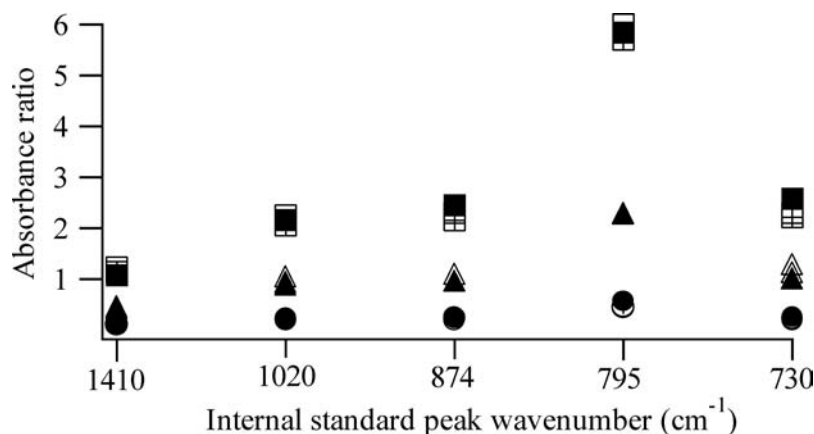


FIG. 4—Ratios of the IR bands at 3440 (circles), 2968 (triangles) and 1730 cm^{-1} (squares) with respect to five peaks used as internal standards: 1410, 1020, 874, 795 and 730 cm^{-1} . Closed (●, ▲, ■), open (○, △, □) and crossed (⊕, ⊔, ⊞) symbols represent untreated samples, fibers immersed in water, and samples irradiated by the sun, respectively.

commercially sensitive information was not available, but it is reasonable to assume that different producers use different additives). The diffusion coefficient has been found to vary with crystallinity according to a power law (25). A sharp decrease in the diffusion coefficient as a function of percent crystallinity has been reported, with a plateau beyond 25% (22). Crystallinity of the samples was evaluated by DSC and is shown in Table 1. As can be seen, commercial PET fibers are characterized by quite high crystallinity degrees. This very ordered framework of the polymeric chains prevents an appreciable absorption of water. The crystallinity was determined just for the untreated samples because it was of interest to assess the degree of order of the chains and thus to confirm why no absorption of water was observed. No DSC measurement was extended to treated samples because this technique requires unrealistic sample sizes for forensic purposes and it would be of no practical use in casework.

It is known, from studies of thermal analysis associated to IR spectroscopy (17), that PET, when brought to high temperatures (beyond 200°C), degrades giving a large quantity of products. The principal degradation compounds are carbon dioxide, acetaldehyde, vinyl benzoate, terephthalic acid and dimers. The hypothesis that we wanted to verify was if the heating, limited to a temperature

of about 45°C, but prolonged for very long periods of time and the action of ultraviolet irradiation by the sun didn't prejudice the validity of comparisons for forensic purposes. Annealing is in fact a well known process that brings about structural changes (26) and ultraviolet rays are quite detrimental (27) for polymers that are not adequately filled with UV-protecting agents.

From Table 1, it can be inferred that exposure to the sun for three months didn't produce any significant effect. To have a further insight, the bands at 3440, 2968 and 1730 cm^{-1} were monitored, all signals known to decrease if a degradation process occurs. The data for sample B are presented in Fig. 4. As can be seen, each signal was ratioed against five peaks, at 1410, 1020, 874, 795 and 730 cm^{-1} , commonly used as internal standards (18,20,28).

Again no alteration in the microstructure of the macromolecular chains can be detected. Finally, also after irradiation, the morphology remained unaltered (Fig. 5).

Conclusion

Further validation was proposed to back up a quantitative infrared spectroscopic method, intended for forensic comparisons of PET single fibers (10). It has been shown that the ratios between IR bands

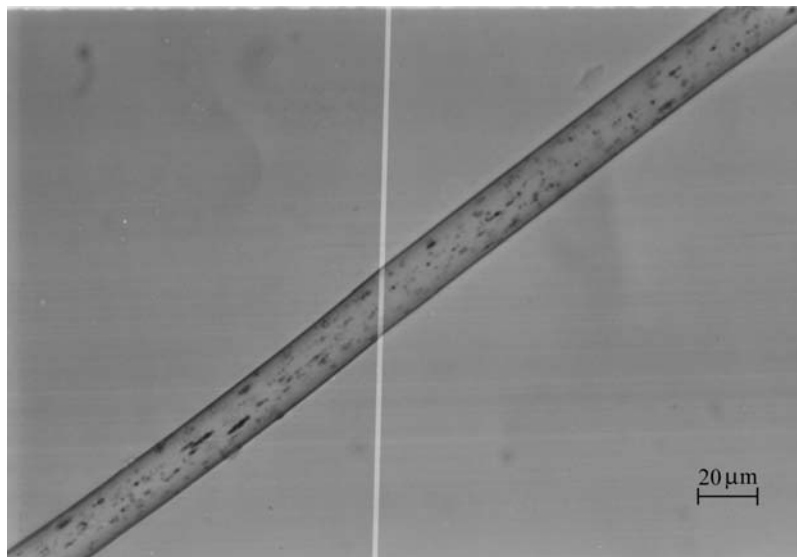


FIG. 5—Comparison of the morphology of sample F2 untreated (left) and after irradiation by the sun for three months (right). The scale corresponds to 20 μm .

at 3440 cm^{-1} and at 874 cm^{-1} (A_{3440}/A_{874}) and between signals at 1370 cm^{-1} and 846 cm^{-1} (A_{1370}/A_{846}) are unaffected by long exposures to strong sunlight or immersion in water. This result is of critical importance in enlarging the probative strength of the technique and, by reflection, of fibers. It may happen in casework, in fact, that fibers coming from a crime scene have been exposed to the elements for a long time, even for many weeks (1,2). It was thus necessary to confirm that a comparison can be significant, even if it is performed on samples that experienced very different conditions. It is very important that proposed characterization procedures take into account as many parameters as possible that could influence their reliability, so the Court can perceive the actual significance of the source of evidence (29–32). The more data, obtained by a validated technique, are available to the forensic scientist, the less vulnerable to context effect and bias he will be (33,34).

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