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Analysis of Single Polymer Fibers by Fourier Transform Infrared Microscopy: The Results of Case Studies

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ABSTRACT: Infrared (IR) microscopic analysis was performed in tandem with traditional techniques of fiber analysis in case studies in order to determine the feasibility of IR microscopy as a routine technique for fiber analysis. All fibers which were comparable by visual, optical microscopy were also found to be comparable by IR microscopy. Several minor spectral differences between comparable fibers were encountered. The sources of these spectral differences fell into three major categories: (1) interference fringes, (2) contaminants, and (3) pressure effects due to sample preparation. The utility of a computerized spectral library of fibers for the identification of generic class and subclass was evaluated. Sheath and core bicomponent fibers were also examined along with a new type of poly(benzimidazole) (PBI), a high-performance fiber.

KEYWORDS: criminalistics, comparative analysis, fibers, microscopy, infrared microscopy, spectroscopic analysis, synthetic fibers

Analytical approaches for the analysis of textile fibers have been recently reviewed [1,2]. Of the many techniques discussed for the identification and comparison of fiber evidence, visual microscopy is the most frequently used in the forensic science laboratory. The identification of generic classes of fibers can usually be made by examination of the optical properties of fibers alone. In the FBI Laboratory, microscopical examination and determination of optical properties by polarized light microscopy are also supplemented by solubility testing, microspectrophotometric analysis, and examination of fluorescent properties when appropriate.

Among the remaining analytical techniques reviewed, infrared (IR) spectroscopy is the most well-established technique for polymer fiber analysis. Infrared analysis can provide more specific chemical information about polymer composition than optical

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microscopy, thus increasing the evidential value of the fiber match.³ In addition, The American Society for Testing and Materials (ASTM) now lists IR spectroscopy as the preferred method of analysis for identifying man-made fibers, stating "Where the data are consistent and the spectra obtained and interpreted by an experienced spectroscopist, the infrared procedure has no known bias" (ASTM D 276-87).⁴ Many papers [3-11] have addressed IR spectroscopy as a technique for the forensic characterization of textile fibers. Only the recent availability of Fourier transform (FT) techniques and microscope accessories, however, have made IR analysis of single fibers feasible on a routine basis in the high throughput setting of the forensic science laboratory.

We have recently described the development of an IR spectral library of single polymer fibers [12]. This database is based upon polymer composition and allows the identification of both the generic class and chemical subclass of fiber. The current listing of the library contents is given in Table 1. FT-IR microscopic analysis was performed in tandem with traditional techniques of fiber analysis in case studies at the FBI in order for the feasibility of FT-IR microscopy as a routine technique for forensic fiber analysis to be determined. The utility of the spectral library of polymer fibers was also evaluated.

³The term "match" is used in the sense that one fiber is the counterpart of the other in all the properties studied.

⁴See ASTM Method for Identification of Fibers in Textiles (D 276-86).

TABLE 1—*Contents of the IR spectral library of fibers.*^a

| Generic Class | Subgeneric Class |
|---------------|--|
| Acetate | acetate triacetate |
| Acrylic | (AN) (AN:MA) (AN:MA:MVP) (AN:VA) (AN:VA:MVP) (AN:MMA) |
| Aramid | Kevlar Nomex |
| Azlon | alginate casein ground nut |
| Modacrylic | (AN:VDC:MAA) (AN:VDC) (AN:VC) |
| Natural | cotton silk wool |
| Nylon | nylon 4 nylon 6 nylon 6,6 nylon 6,10 nylon 6,11 nylon 6,12 nylon 11 nylon 12 Qiana (Type 1) ^b Qiana (Type 2) |

TABLE 1—Continued.

| Generic Class | Subgeneric Class |
|---------------|--|
| PBI | Type 1 Type 2 |
| Polyester | (PET) (PCDT) (TA:PHBA:EG) (PHEB) (PBT) |
| Polyolefin | polyethylene polypropylene |
| Rayon | |
| Saran | Type 1 Type 2 Type 3 |
| Spandex | Type 1 Type 2 |
| Vinal | |
| Vinyon | (VC:VA) |

^aAcronym definitions:

- AN = acrylonitrile.
- EG = ethylene glycol.
- MA = methyl acrylate.
- MAA = methyl acrylamide.
- MMA = methyl methacrylate.
- MVP = methyl vinyl pyridine.
- PBT = poly(butylene terephthalate).
- PCDT = poly(cyclohexyl dimethylene terephthalate).
- PET = poly(ethylene terephthalate).
- PHBA = *p*-hydroxybenzoic acid.
- PHEB = poly(hydroxyethoxybenzoate).
- TA = terephthalic acid.
- VA = vinyl acetate.
- VC = vinyl chloride.
- VDC = vinylidene chloride.

^bThe variation of the polymer composition is currently unknown.

Experimental Procedure

Infrared spectra were obtained of the fibers encountered in 72 fiber matches made during routine casework at the FBI laboratory. Short sections of each fiber were flattened with a small metal roller (Part No. 0036-521, Spectra-Tech, Stamford, Connecticut), the surface of which had been roughened with silicon carbide sandpaper, on the glass stage of a stereomicroscope. The flattened fibers were then mounted across the 1.3-mm apertures of adhesive paper holders.⁵ The length of fiber used was about 2 mm, although shorter lengths could be accommodated by reducing the aperture diameter with small pieces of adhesive tape and mounting the fiber across the tape. A moderate pressure,

⁵Adhesive paper holders (Part No. 1000-071, Spectra-Tech, Stamford, Connecticut) are available by contacting Jeff D'Agostino, Spectra-Tech, Inc., 652 Glenbrook Rd., P.O. Box 2190-G, Stamford, CT 06906; phone 1-800/243-9186.

TABLE 2—Fiber types identified by IR spectra for the first 72 cases.

| Fiber Type | Number of Occurrences ^a |
|---------------------|------------------------------------|
| Acrylic | 24 |
| AN:MA | 9 |
| AN:MMA | 7 |
| AN:VA | 8 |
| Nylon | 19 |
| nylon 6 | 7 |
| nylon 6,6 | 12 |
| Polyester | 18 |
| PET | 18 |
| Polyolefin | 5 |
| polypropylene | 5 |
| Acetate | 4 |
| acetate | 2 |
| triacetate | 2 |
| Modacrylic | 3 |
| AN:VC | 3 |
| Rayon | 1 |
| Miscellaneous | 6 |
| cotton ^b | 5 |
| silk | 1 |

^aThe total number of occurrences is greater than 72 due to fiber blends and multiple fiber types per case.

^bCotton fibers were included in the study only when they were encountered as part of a fiber blend.

miniature diamond anvil cell (Part No. M-AII, High Pressure Diamond Optics, Tucson Arizona) was used where indicated.

A Nicolet 20SX FT-IR spectrometer (Madison, Wisconsin) equipped with a Spectra-Tech Research IR Plan microscope and a 0.25-mm narrow-band mercury cadmium telluride (MCT) detector was used. The image of each fiber was masked using both upper and lower rectangular apertures available on the microscope accessory in order to minimize diffraction effects.⁶ All spectra were acquired at a resolution of 4 cm⁻¹ with Happ-Ganzel apodization and 512 scans were co-added for each spectrum. Spectra were acquired from 4000 to 650 cm⁻¹.

Where necessary, residual carbon dioxide and water were removed from the spectra by subtraction, and baselines were normalized using the Nicolet correction routine. Fiber spectra from each match were compared and searched against the polymer fiber spectral library.

Results and Discussion

No fibers matched by the FBI Laboratory Hair and Fibers Unit were shown to be different by their IR spectra. The number of occurrences of each fiber type are given in Table 2. As expected, the most frequently encountered generic classes of synthetic fibers

⁶An excellent discussion of diffraction effects in IR microscopy is given in Ref 12.

were polyester, nylon, and acrylic. Note that cotton fibers were included in the study only when they occurred in a fiber blend and, therefore, the number of occurrences is not representative of the actual frequency of occurrence. All polyester fibers examined belonged to the subclass poly(ethylene terephthalate) (PET). This observation would greatly increase the significance of a match between polyester fibers which belong to a subclass other than PET because the data show that they are not frequently encountered. The collection of additional statistics from future casework would be required to support the significance of a match between fibers of an uncommon type. The casework statistics should differ from fiber production statistics due to three factors: (1) the degree to which the fiber is shed, (2) whether or not the fiber is readily transferred from the donor to recipient item, and (3) the degree to which the fiber will persist on the recipient item. All nylon fibers examined belonged to either the nylon 6 or nylon 6.6 subclasses. The acrylic fibers were all copolymer fibers consisting of either acrylonitrile:methyl acrylate (AN:MA), acrylonitrile:methyl methacrylate (AN:MMA), or acrylonitrile:vinyl acetate (AN:VA). The capability to identify the subclass of fiber within the generic class increases the evidential value of the fiber match by adding an additional point of comparison. As with polyester fibers, the occurrence of a fiber match involving nylon or acrylic fibers belonging to a subclass other than those encountered previously would also greatly enhance its evidential value.

Although all fiber matches were eventually confirmed by their IR spectra, several minor spectral differences were encountered during the course of the study. These differences were never so great as to obscure the identification of the fiber class and subclass, but they did initially prevent the two IR spectra from matching. Identification of the sources of these spectral differences is important so that they may be correctly interpreted and not erroneously attributed to chemical differences between the fibers in question. The sources of these spectral differences fell into three major categories: (1) interference fringes, (2) contaminants, and (3) pressure effects due to sample preparation.

Interference Fringes

The most frequently encountered spectral differences were the result of interference fringes. When the fiber is flattened it becomes, in effect, a thin film. Interference fringes arise due to internal reflection of the incident IR beam within the fiber. As a result, a sinusoidal wave is superimposed upon the spectral baseline, as can be noted in Fig. 1. Although the interference fringe is present over the entire wavelength range, it is most easily observed in the region above 1500 cm^{-1} . Interference fringes are most often encountered in the spectra of acrylics and modacrylics. These fringes can sometimes be reduced by roughening the surface of the fiber. Typically, the fiber is flattened on a microscope slide with a metal roller, the surface of which has been roughened by being scratched against silicon carbide sandpaper. Whether or not the interference fringes can be eliminated, it is important to remember that they are the result of sample thickness and surface smoothness, not of chemical composition. In short, the presence of interference fringes should not negate a match between two otherwise identical spectra.

Contaminants

Because the fibers were received in our laboratory after having been matched by routine methods, the most commonly encountered contaminant was a hydrocarbon-based mounting medium such as Permount. Figure 1 shows the IR spectra of acrylic (AN:MMA) fibers Q and K, which had been shown to match by visual microscopy. Note the increased intensity of the C-H stretching absorption bands near 2930 cm^{-1} and the C-H bending absorption bands near 1454 , 1384 , and 1366 cm^{-1} in the spectrum of fiber Q compared

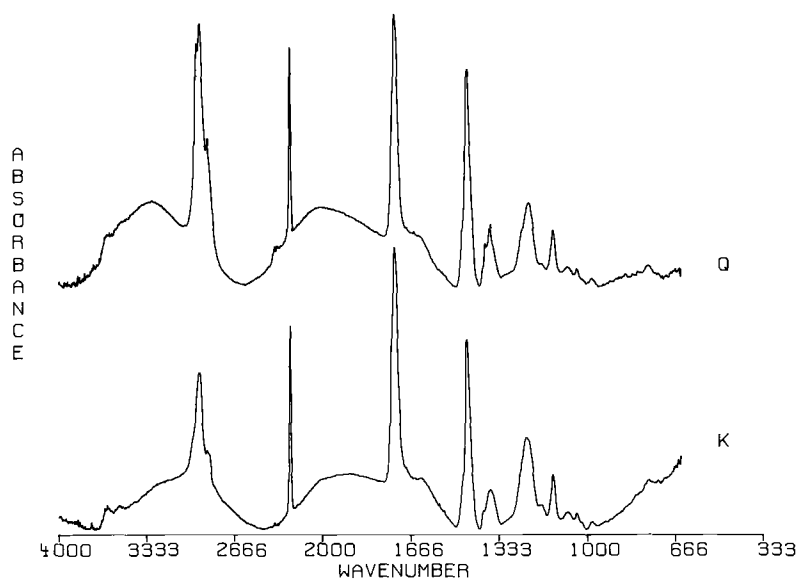


FIG. 1—IR spectra of acrylic (AN:MMA) fibers Q and K. Note the increased intensity of the C-H stretching absorption bands near 2930 cm^{-1} and the C-H bending absorption bands near 1454 , 1384 , and 1366 cm^{-1} in the spectrum of fiber Q. The irregular baselines are due to interference fringes, the result of sampling the flattened fiber which is, in effect, a thin film.

with that of fiber K. Originally, it was not known whether this difference originated from a difference in the polymer structure of fibers Q and K or from a contaminant on fiber Q.

Figure 2 shows the difference spectrum of the spectra in Fig. 1 compared with the spectrum of Permunt. The sinusoidal baseline in the difference spectrum is the result of the subtraction of the interference fringes in the spectra of fibers Q and K. The small

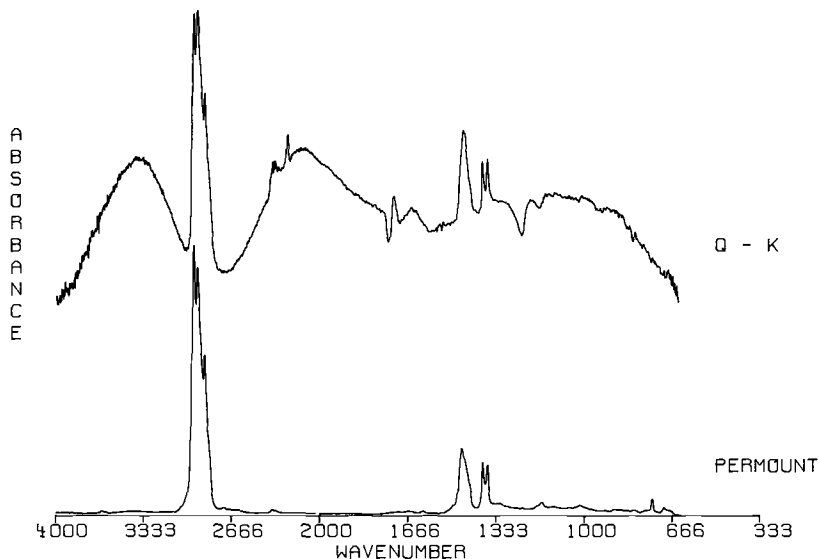


FIG. 2—Difference spectrum of the spectra in Fig. 1, obtained by subtracting the spectrum of fiber K from the spectrum of fiber Q, compared to the IR spectrum of Permunt.

residual bands in the spectra of the acrylic fibers arise from minor variations in intensities resulting from diffraction effects. These minor residual bands may be decreased by reducing the size of the aperture [13]. To confirm that the difference spectrum was indeed due to the presence of Permout on fiber Q, we washed the fiber with xylene and reacquired the IR spectrum. After washing, the spectra of fibers Q and K were shown to match.

Several fiber samples examined were contaminated with blood. Blood may be visible under an optical microscope; however, in one case the fiber itself was dark red, thus precluding a visual observation. Figure 3 [14] shows the spectra of acrylic (AN:MA) fibers Q1 and Q9, which were shown to match by visual microscopy. Note the additional peaks near 3305, 1652, and 1527 cm^{-1} in the spectrum of fiber Q9 compared with that of fiber Q1. Figure 4 shows the difference spectrum of the spectra in Fig. 3 compared with the spectrum of blood. Fiber Q9 was immersed in a saline solution (in which blood is soluble) in an ultrasonic bath for a few minutes to confirm the fiber match. The fiber was then air dried and its spectrum was recorded again. After washing, the additional peaks present in the spectrum of fiber Q9 were greatly reduced, and the spectra of fibers Q9 and Q1 could be matched.

The presence of a contaminant can, in certain instances, increase the evidential value of a fiber match when the contaminant is shown to be present on both the questioned and known fibers. Infrared spectra were obtained of fibers Q and K as shown in Fig. 5. Fibers Q and K were both identified as polypropylene; however, fiber K contained additional absorption peaks near 1730, 1651, 1017, 702, and 669 cm^{-1} . These differences initially precluded a match between fibers Q and K. Spectra shown in Fig. 6 were obtained after aperture size and sampling locations on the fibers were changed. An increase in aperture size resulted in the appearance of the additional bands in fiber Q, whereas a decrease in aperture size resulted in a reduction in intensity of the additional bands in fiber K. This fluctuation with aperture size revealed that the source of the additional bands was associated with the presence of a contaminant rather than a difference in

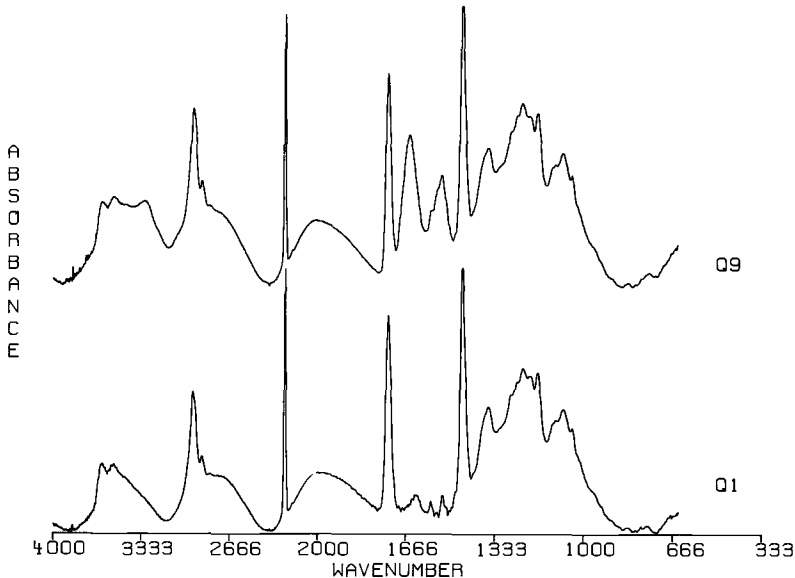


FIG. 3—IR spectra of acrylic (AN:MA) fibers Q1 and Q9. Note the additional absorption bands near 3305, 1652, and 1527 cm^{-1} in the spectrum of fiber Q9. Reproduced, with permission, from Ref 14.

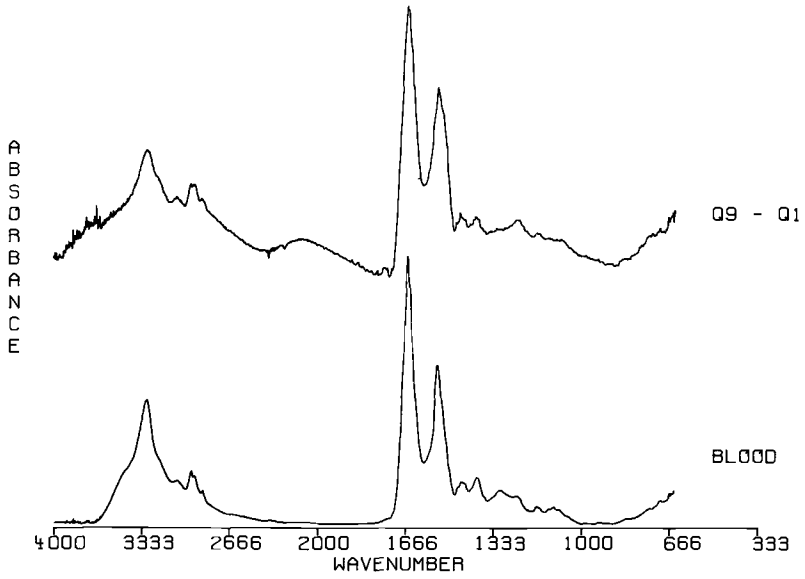


FIG. 4—Difference spectrum of the spectra in Fig. 3, obtained by subtracting the spectrum of fiber Q1 from the spectrum of fiber Q9, compared with the IR spectrum of blood. Reproduced, with permission, from Ref 14.

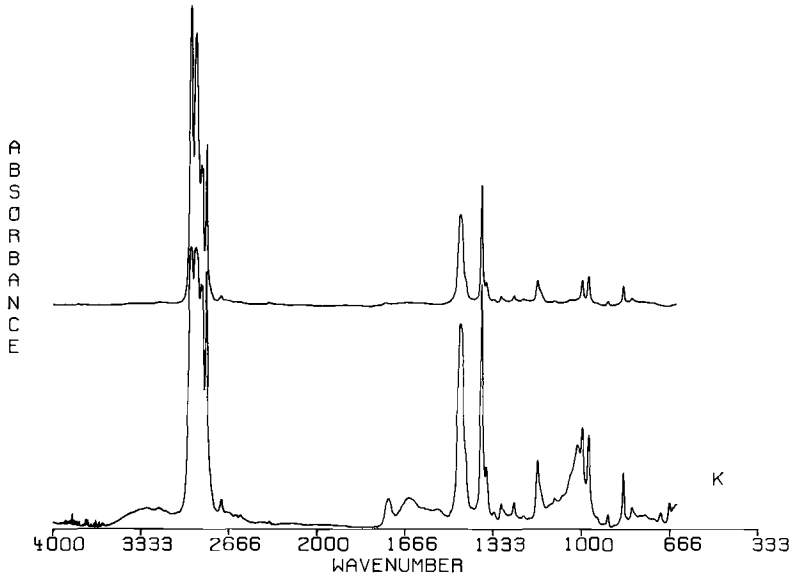


FIG. 5—IR spectra of polypropylene fibers Q and K. Note the additional absorption bands near 1730, 1651, 1017, 702, and 669 cm^{-1} in the spectrum of fiber K. Aperture sizes: Q = 60 by 92 μm ; K = 116 by 188 μm .

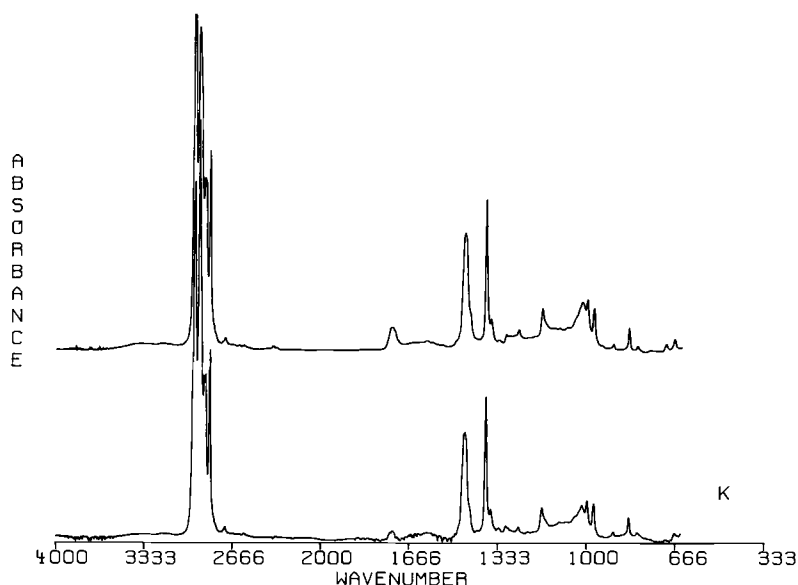


FIG. 6—Second set of IR spectra obtained of polypropylene fibers Q and K after sampling location and aperture size were changed. Aperture sizes: Q = 100 by 240 μm ; K = 24 by 48 μm . Note that an increase in the aperture size for Q allowed the inclusion of the contaminant in the sampling area, resulting in the presence of the additional peaks in the spectrum. Likewise, a decrease in the aperture size for K excluded much of the contaminant from the sampling area, resulting in a decrease in intensity of the additional peaks in the spectrum.

composition between fibers Q and K. Obviously, the increase in aperture size resulted in the inclusion of the contaminant in the sampling field, whereas the reverse process excluded most of the contaminant from the sampling field.

A miniature diamond anvil cell (DAC) was used to obtain a spectrum of a black particle found adhering to one of the fiber samples. Figure 7 compares this spectrum with the difference spectrum obtained by subtracting Q No. 1 from Q No. 2. From the spectral similarities in Fig. 7, the black particle was concluded to consist of the same material that produced the spectral differences between fibers Q and K shown in Figs. 5 and 6. The spectrum of the black particle is similar to a polyester resin, as shown in Fig. 8, with additional absorption bands at 1024 and 672 cm^{-1} . These peaks may be accounted for by the presence of talc (or other inorganic filler), as shown in Fig. 9. The contaminant, responsible for the spectral differences, was probably a glue used in the construction of the automobile trunk liner from which the fibers were subsequently found to have originated.

Pressure Effects Due to Sample Preparation

The third source of minor spectral differences between matched fibers was the variation in pressure applied during the sample preparation process (flattening). Infrared spectra were obtained for fibers Q and K as shown in Fig. 10. Although both fibers were identified as PET, minor spectral differences were observed in the 1450 to 1300- cm^{-1} region shown in the boxed portions of the spectra. The peak at 1340 cm^{-1} in the spectrum of fiber Q, Sample No. 1 was less intense than the corresponding peak in the spectrum of fiber K, Sample No. 1. Replicate samples were prepared and new spectra were obtained, as shown in Fig. 11. The relative intensity of the 1340 cm^{-1} band was approximately the same in

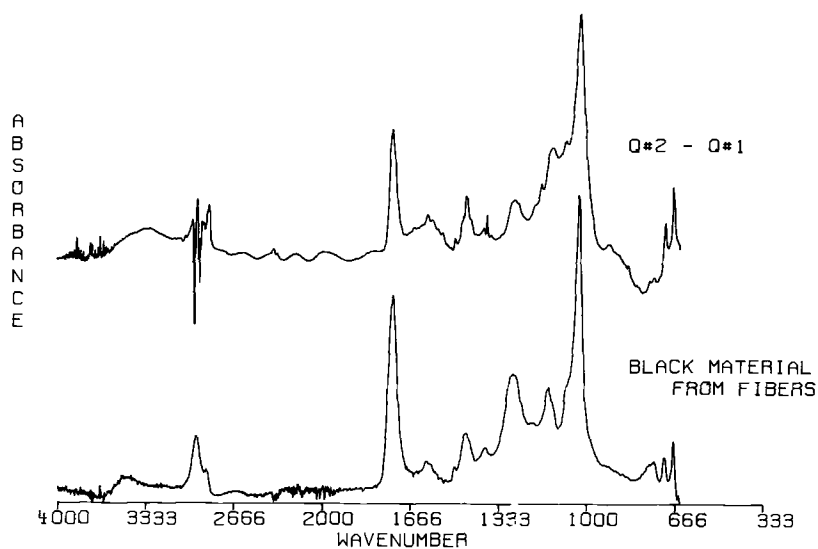


FIG. 7—Difference spectrum, obtained by subtracting the first spectrum of Q from the second, and the spectrum of a black particle found adhering to one of the fibers.

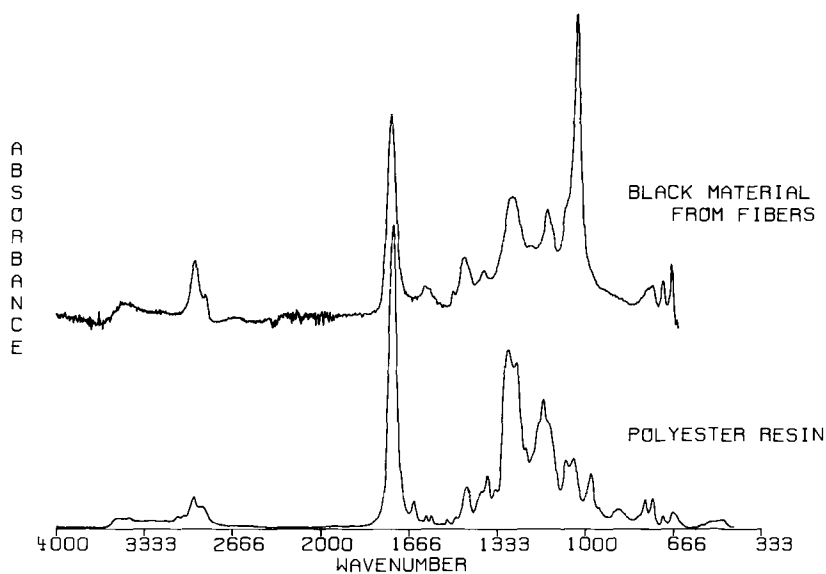


FIG. 8—IR spectrum of the black material found adhering to a fiber and the IR spectrum of an unsaturated polyester resin. Peaks near 1024 and 672 cm^{-1} are not accounted for by the resin.

both spectra, and the spectra were said to be consistent with fibers Q and K sharing the same polymer composition.

The difference in the initial spectra of Q and K may be attributed to differing degrees of crystallinity as a result of sample preparation [15]. The 1340 cm^{-1} band originates from a crystalline $-\text{CH}_2-$ deformation while the 1371 cm^{-1} band arises from an amorphous $-\text{CH}_2-$ [16]. A relative scale for the degree of crystallinity is obtained by ratioing the peak area of the crystalline band to that of the amorphous band. The larger the ratio,

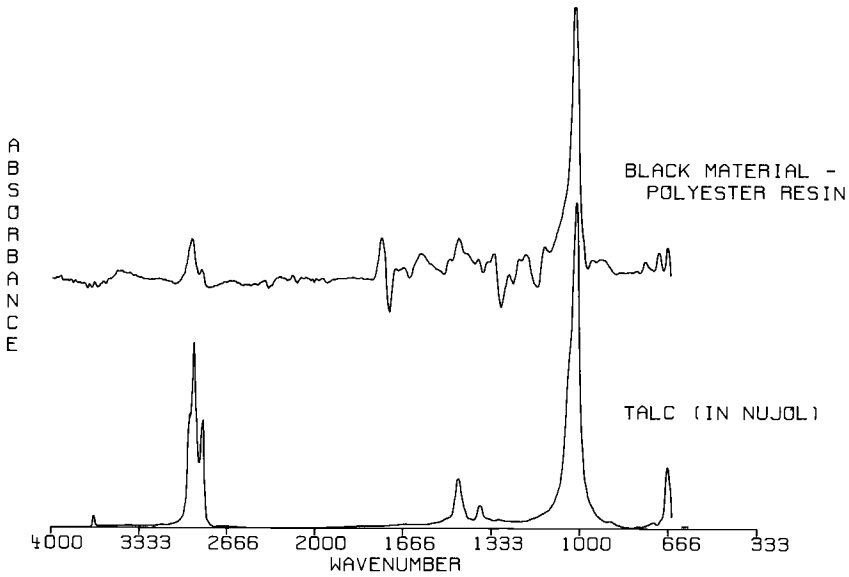


FIG. 9—Difference spectrum, obtained by subtracting the spectrum of polyester resin from the spectrum of the black material, and the spectrum of talc. The talc spectrum contains C-H absorption bands which originate from Nujol.

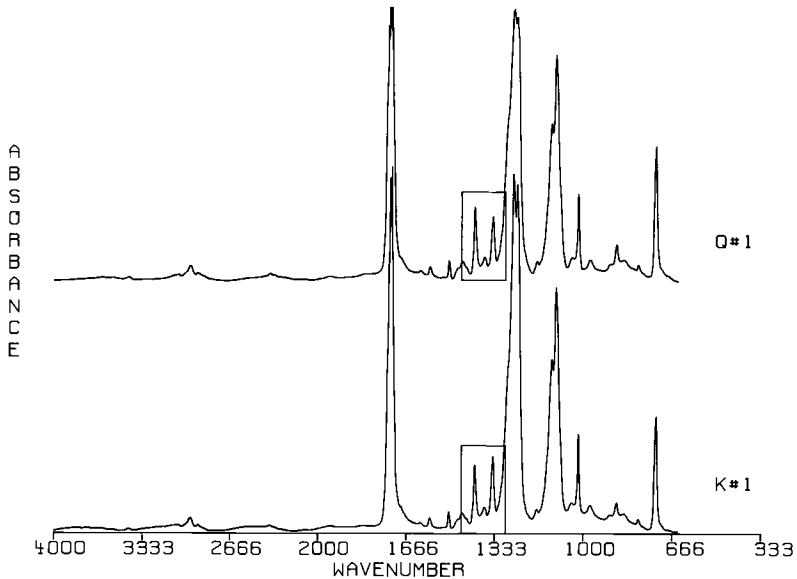


FIG. 10—IR spectra of the first sample preparations of PET fibers Q and K. Note the differing peak intensities between Q and K in the enclosed portion of the spectra.

the higher the degree of crystallinity of the PET. As given in Table 3, the degree of crystallinity varies with sample preparation. During flattening, the polymer is forced to flow as the result of shear forces, and the crystalline structure is altered.⁷ Thus, these differences in crystallinity may be attributed to variations in the applied pressure [17]

⁷Brasch, J. W., J. B. Labs, Columbus, OH, personal communication, 1989.

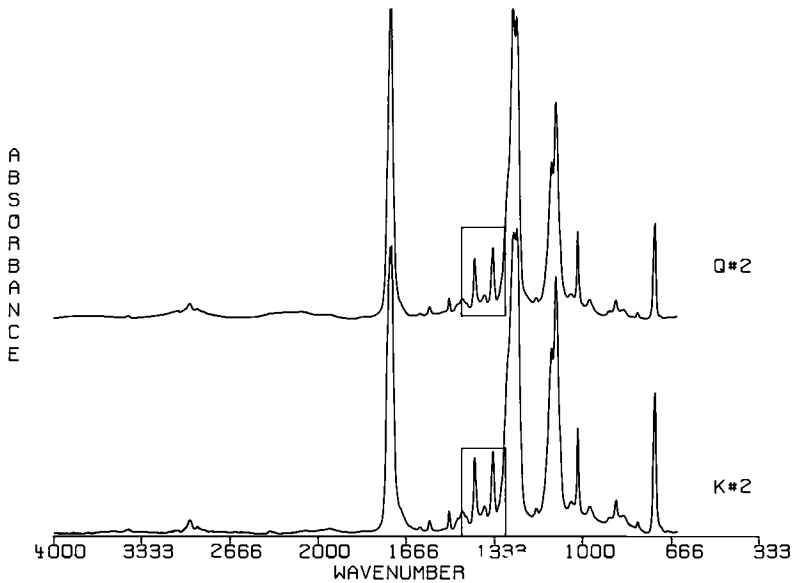


FIG. 11—IR spectra of the second sample preparations of PET fibers Q and K. Note the similarity between Q and K in the enclosed portion of the spectra.

TABLE 3—Crystalline/amorphous peak area ratios^a for two sample preparations of PET fibers Q and K.

| | Sample 1 | Sample 2 |
|---------|----------|----------|
| Fiber Q | 4.7 | 6.8 |
| Fiber K | 6.1 | 5.9 |

^a $\frac{\text{Crystalline peak (1340 cm}^{-1}\text{) area}}{\text{Amorphous peak (1371 cm}^{-1}\text{) area}}$

and shear forces during flattening and not to a structural difference between fibers Q and K. Similar variations in peak intensities may also be observed for other crystalline/amorphous sensitive bands, listed in Table 4 [16], in the spectrum of PET fibers. The effects of everyday usage on the crystalline/amorphous peak area ratios and batch-to-batch variations have not yet been explored.

Thinner samples, which would improve the quality of the spectra, may be prepared by exerting pressures higher than the gentle flattening of a fiber with a hand-held roller. Nylon fibers frequently require additional thinning to obtain a spectrum which is not overabsorbing. Thinner samples may be prepared by slicing off an edge from the flattened fiber with a scalpel followed by reflattening the section with the roller. These steps may be repeated many times to obtain a suitable spectrum.

Application of high pressures on fibers may result in changes in the IR spectra. This effect is shown in Fig. 12 for the spectrum of a nylon 6,6 fiber as the pressure exerted by a miniature DAC is gradually increased. As the sample is thinned due to higher pressures, smaller noise spikes are observed on the strongly absorbing peaks because the maximum absorbance is reduced [18]. Peak resolution, however, decreases as a result of changes in the crystalline structure of the polymer [17]. The fine details of the spectrum

TABLE 4—Crystalline/amorphous sensitive bands for PET.

| Band Frequency, cm^{-1} | Change with Increasing Crystallinity |
|----------------------------------|--------------------------------------|
| 1475 | + |
| 1456 | - |
| 1374 | - |
| 1344 | + |
| 1129 ^a | + |
| 1099 ^b | - |
| 1044 | - |
| 973 | + |
| 897 | - |
| 844 | + |

^aThe 1129 cm^{-1} band shifts to 1119 cm^{-1} in amorphous form.

^bThe 1099 cm^{-1} band shifts to 1109 cm^{-1} in crystalline form.

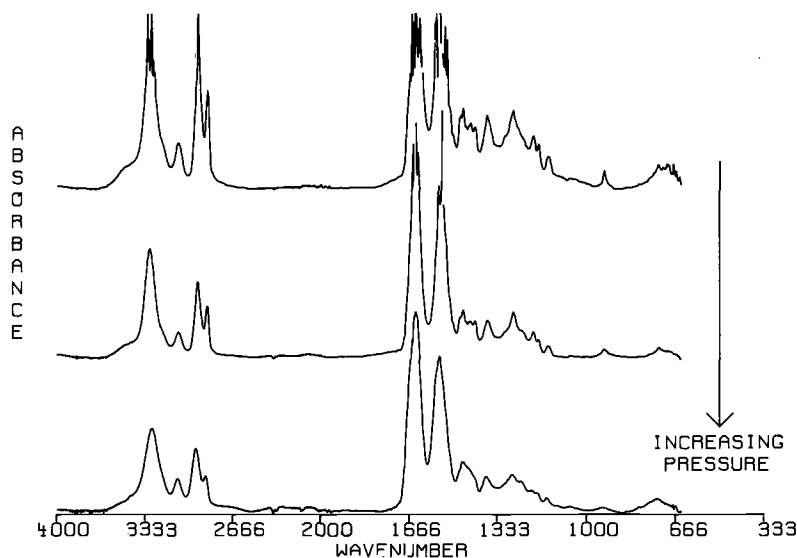


FIG. 12—The effect of increasing pressure on a nylon 6,6 fiber in a miniature diamond anvil cell. As the pressure is increased, the noise spikes on the strongly absorbing bands are reduced, but peak resolution decreases and the characteristic absorption band at 935 cm^{-1} becomes indistinguishable from the baseline.

below 1500 cm^{-1} are lost and the crystalline absorption band at 935 cm^{-1} , which distinguishes nylon 6,6 from nylon 6, becomes indistinguishable from the baseline.

To acquire maximum information on a nylon fiber, one may first obtain an initial spectrum by gently flattening the fiber with a roller to achieve good peak resolution in the region of the spectrum below 1500 cm^{-1} , similar to the procedure used for other fibers. Although intense noise may be noted on strongly absorbing bands, the weaker bands, such as those originating from crystalline interactions in the polymer, are more readily detected for identification purposes. Full-scale expansion of the spectral region below 1500 cm^{-1} may be useful for comparison of known and questioned fiber samples. Subsequent spectra may then be acquired, after further flattening, in order to obtain a thinner sample. With the noise spikes removed from the strongly absorbing bands, peak intensity ratios may then be compared. A similar approach may also be appropriate for

polyolefin fibers. For these fibers weak bands, originating from chemical modifications for dyeing processes [10] and other additives, become more apparent as thicker samples are used.

Bicomponent Fibers

The spectra of sheath and core bicomponent fibers were also examined. In order for us to characterize these fibers using optical microscopy, it was necessary to obtain cross sections of the fibers, an often tedious procedure. With IR microscopy, all that was required was to flatten the fiber. A spectrum was easily obtained of the sheath by sampling an area near the edge of the flattened fiber. The spectrum acquired near the center of the fiber displayed features representing predominately the core material along with weaker features of the already identified sheath material. Figure 13 shows IR spectra acquired from the sheath and core of a bicomponent fiber. The spectrum recorded from the edge of the flattened fiber identifies the sheath composition as polypropylene, while that acquired from the center of the fiber is easily identifiable as PET, although weak polypropylene bands are apparent, as expected. The spectrum of the sheath could easily be subtracted from that of the core in order to remove the polypropylene bands.

New Fiber Types

New types of fibers may be characterized by IR microscopy. As an exercise, a brown fiber received from Hoechst Celanese was examined. Efforts to identify the fiber by traditional methods, that is, optical microscopy, had failed. Figure 14 shows that the fiber spectrum is quite similar to that of a poly(benzimidazole) (PBI) fiber received earlier from a private collection. The spectrum of PBI was contained in the fiber spectral library already. The fiber was later confirmed to be a new variation of PBI, probably methylated at various points along the repeat unit of the polymer. Had the PBI fiber not been

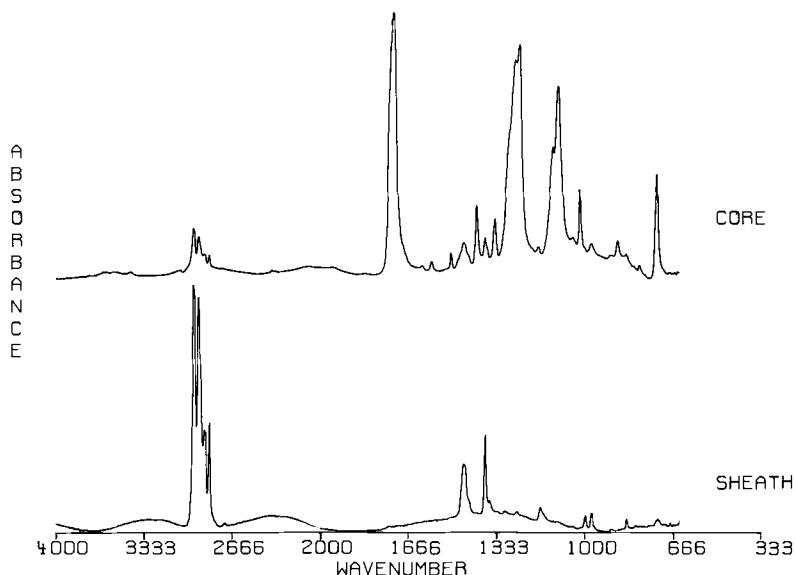


FIG. 13—IR spectra of the sheath and core of a flattened bicomponent fiber. The spectrum of the polypropylene sheath was obtained by sampling a location near the edge of the fiber, while the spectrum of the PET core was obtained by sampling the center of the fiber. Note the weak polypropylene absorption bands present in the PET spectrum.

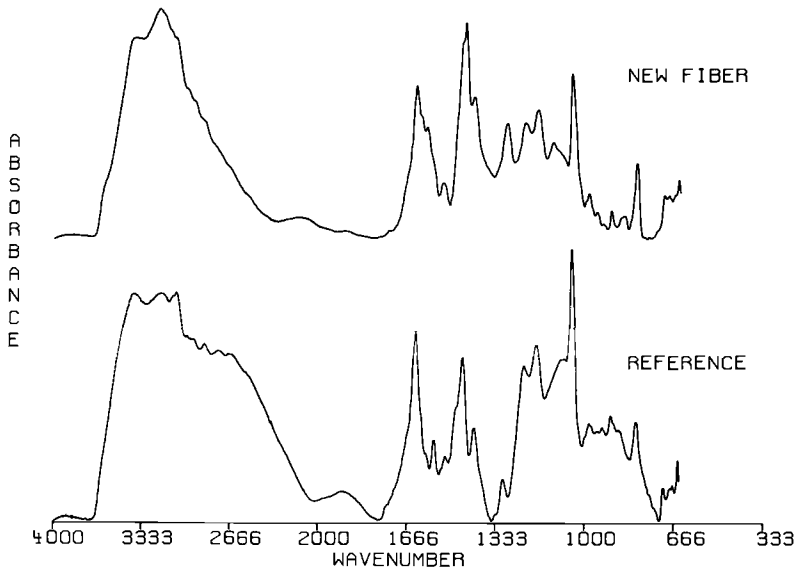


FIG. 14—Infrared IR spectra of a new fiber and a poly(benzimidazole) (PBI) fiber.

included in the fiber spectral library, a similar polymer could probably have been found in the commercial polymer libraries.

Spectral Library Search Results

Results of spectral library searches for the first 72 cases are given in Table 5. An absolute derivative difference searching algorithm [19] was used to compare the first derivatives of the unknown and reference spectra. This algorithm tends to minimize the contribution of irregular baselines while emphasizing peak positions. The correct first selection was made for the spectra of 48 acrylic, 12 acetate, 12 polyolefin, 10 modacrylic, and 2 rayon fibers searched against the fiber spectral library. The correct first selection was made for 39 of the 41 PET spectra searched. The first selection for the remaining 2 spectra was poly(terephthalic acid:p-hydroxybenzoic acid:ethylene glycol) (TA:PHBA:EG), with the second selection being PET. Infrared spectra of these 2 polymers are quite similar, but they may be distinguished by visual examination. This observation emphasizes the importance of visual examination of the first few selected reference spectra with the unknown spectrum prior to making an identification. The correct first selection was made for 16 of the 21 nylon 6 spectra searched. Results for nylon 6,6 were somewhat inferior, with the correct first selection being made for only 11 of the 23 spectra searched. For nylon 6,6, 22 of the 23 spectra, however, were listed in the top 2 selections. Thus, a visual comparison of the unknown spectrum with the first few selected reference spectra, as mentioned previously, would allow for an easy identification of the unknown fiber.

Conclusions

Infrared microscopy is a rapid, nonsubjective technique which can supplement visual optical microscopy in the examination of textile fibers. After two fibers have been matched via optical microscopy, identification of the generic class can quickly be confirmed by IR microscopy. In addition, the chemical subclass can also be identified, thus increasing the evidential value of the fiber match. Minor spectral differences may be observed which

TABLE 5—Results of spectral library searches for the first 72 cases.

| Fiber Type | Number of Spectra Searched ^a | Number and Position of Correct Selection | | | |
|---------------|---|--|--------|-------|---------|
| | | First | Second | Third | > Third |
| Acrylic | 48 | 48 | 0 | 0 | 0 |
| AN:MA | 17 | 17 | 0 | 0 | 0 |
| AN:MMA | 15 | 15 | 0 | 0 | 0 |
| AN:VA | 16 | 16 | 0 | 0 | 0 |
| Nylon | 44 | 27 | 11 | 2 | 4 |
| nylon 6 | 21 | 16 | 0 | 1 | 4 |
| nylon 6,6 | 23 | 11 | 11 | 1 | 0 |
| Polyester | 41 | 39 | 2 | 0 | 0 |
| PET | 41 | 39 | 2 | 0 | 0 |
| Acetate | 12 | 12 | 0 | 0 | 0 |
| acetate | 4 | 4 | 0 | 0 | 0 |
| triacetate | 8 | 8 | 0 | 0 | 0 |
| Polyolefin | 12 | 12 | 0 | 0 | 0 |
| polypropylene | 12 | 12 | 0 | 0 | 0 |
| Modacrylic | 10 | 10 | 0 | 0 | 0 |
| AN:VC | 10 | 10 | 0 | 0 | 0 |
| Rayon | 2 | 2 | 0 | 0 | 0 |

^aThe total number of spectra searched is greater than 72 due to multiple fibers per case.

arise from interference fringes, contaminants, or pressure effects due to sample preparation. Once the origins of these differences are clearly understood, their presence should not erroneously negate the fiber match. Infrared microscopy also allows easy analysis of bicomponent fibers and new fiber types not previously encountered in a forensic laboratory. The digitized spectral library can provide valuable assistance in the identification of both the generic class and chemical subclass of the fibers. For one to confirm the identification, however, visual examination of the first few selected reference spectra in the computer search with the unknown spectrum is essential.

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