

The Use of FTIR Microspectroscopy for the Identification of Thermally Changed Fibers

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ABSTRACT: The analysis of thermally changed, e.g., melted, decomposed, burned, or incinerated textile fibers by FTIR spectroscopy is described. This method allows us to observe the pathway of fiber thermal degradation and to find the chemical (organic and inorganic) compounds in degraded fibers. The results of these examinations can be applied in the forensic analysis for the identification and differentiation between thermally changed fibers.

KEYWORDS: forensic science, criminalistics, fibers, thermal degradation, organic and inorganic compounds, fourier transform infrared microanalysis, fiber identification

Identification of thermally changed fibers is one of paramount importance in analysis carried out for court purposes. Thermally changed fibers (melted, burned, carbonized, and incinerated, etc.) can be found in cases of arson, conflagration, and road accidents.

The knowledge of changes that occur in fibers under high temperature treatment could, on the basis of analysis of already changed material, provide information about the conditions in which the change occurred and become the key to successful identification of thermally changed fibers.

When a fiber is heated, its morphology, physical properties, and chemical composition may change. The nature of these changes can be related to the range of applied temperature, the time, rate of heating, the pressure, and the composition of the surrounding atmosphere. Of course, the process is different for various types of fibers and is dependent on the kind of polymer of which the fiber is made.

Polymers may be divided into thermoplastics and thermosets (1). The polymers of the first group deform plastically and flow while being heated (e.g., polyamides, polyolefins, and polyesters). The second group of polymers is infusible because of cross-link between the molecules, which is often done only after some manufacturing steps (e.g., acrylics). Thermosets can still be deformed after cross-link but their response is largely elastic so that they tend to regain their shape after the removal of the stress.

The examination discussed in this paper was carried out to obtain more information concerning the chemical structure of fibers that had undergone the process of thermal degradation and to

investigate the possibility of identification of fibers changed in such a way.

Chemical composition may be determined by infrared spectroscopy. Infrared spectral measurements are widely used in forensic laboratories for the identification and comparison of fibers (2). The infrared microspectroscopical method enables the analysis of small amounts of material, and by using the latest measuring devices (e.g., microscope, diamond anvil optical cell), the material under analysis is not destroyed.

The scope of the research included the analysis of 11 types of thermally changed textile fibers. Those fibers most commonly used on an everyday basis were selected for these examinations.

Materials

The following types of fibers were changed thermally:

- (a) Natural fibers
 - sheep wool: Polish merinos, English Cheviot, German merinos.
 - cotton: Sudanese, Pakistani, Egyptian, and
 - flax: Polish.
- (b) Synthetic fibers
 - nylon 6: Polana^(R) (ZWCH Stilon),
 - polyester: Elana^(R) (ZWCH Elana),
 - acrylics: Anilana^(R) (ZWCH Anilana), and
 - polypropylene.

The following procedures of thermal treatment were used:

- (a) *The Procedure for Fiber Burning*—A lap of fibers was introduced into the flame of a gas burner. At the moment of ignition, it was removed from the flame and left in the air until it was extinguished by itself. This procedure resembles the authentic cases of arsons or conflagrations because a direct contact between fibers and flame occurs.
- (b) *The Procedure for Fiber Melting (Decomposition)*—The samples of the fibers were placed in round dishes of about 1-mm diameter and introduced into the heating cell of SGV 500 PLUS (HWS Labortechnik). The fibers were heated with a rate of per 10° min, in the temperature range from 150 to 300°C. The process of melting and decomposition of fibers was observed on the screen of the device.
- (c) *The Procedure for Fiber Incineration*—The fiber samples were placed in platinum evaporating dishes of 7-cm diameter and placed in a muffle furnace. The process of incineration was carried out starting from room temperature until the inside of the furnace reached 800°C. The fibers were kept at this temperature for about an hour.

¹Institute of Forensic Research, Westerplatte 9, 31–033 Krakow, Poland.

²Duisburg University-Northwestern German Textile Research Center, Frankenring 2, D-47798 Krefeld, Germany.

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Method

Spectroscopic measurement in the infrared were carried out in the spectral range from 4000 to 800 cm^{-1} (except for some incinerated fibers, the range 4000 to 500 cm^{-1} was used) on the Bio-Rad Digilab Division UMA 300A microscope connected with spectrometer.

Samples of all original fibers, decomposed, and burned natural thermosets fibers (i.e., wool, cotton, and flax) were analyzed using Miniature Diamond Anvil Optical Cell (Type IIA diamonds) manufactured by High Pressure Diamond Optics, Inc. The cell consisted of two triangular plates with optically finished diamonds. Samples were placed on the lower diamond anvil, and then the top plate was positioned to bring the two halves together. The three screws were used to keep the two halves of the cell and to squash the fibers sample between them.

Samples of melted (decomposed) and burned synthetic fibers (mainly thermoplastic) (e.g., nylon 6, polyester, polypropylene, and acrylic), and all incinerated fibers were crushed with potassium bromide in an agate mortar and then pressed into a disk of 5-mm diameter and about 0.5-mm thick.

In many of the infrared spectra sloping baselines were observed. This is believed to result from different imperfections of thermally changed fibers, mainly connected with particle sizes. Signal treatment was done by instrument software to artificially flatten the baseline.

The analysis of absorption band intensities was performed without determination of their actual values (which is the basis of quantitative analysis) and were only used by comparisons. The band intensities are closely related to the thickness of analyzed samples. Because of difficulties in flattening many of the thermally changed fibers and the use of different sample preparation methods (e.g., pressing between diamonds, crushing with KBr), no absolute comparison of intensities values between different specimens was attempted.

Intensities of bands are classified as very weak, weak, medium, or strong; a strong intensity is attributed to the highest bands of particular spectrum. In all the figures given, Y-axis corresponds to absorbance values registered. The values of absorption frequencies were assigned by the instruments computer and a single frequency occurs within $\pm 2 \text{ cm}^{-1}$ deviation of the quoted value.

Results and Discussion

The results of examination and their discussion are arranged by the type of fibers analyzed.

Wool Fibers

The FTIR spectra of original and burned, decomposed, and incinerated Polish merinos wool fibers are shown in Fig. 1. The FTIR spectrum of the burned Polish wool (Fig. 1b) shows very little change from the original wool spectrum (Fig. 1a). An exception is the absorption at 1172 cm^{-1} , 1080 cm^{-1} (S=O stretching), and a relatively higher intensity of bands at 1243 cm^{-1} and in the 1460 to 1300 cm^{-1} region (CH_2 , Amide III), which is related to the known decomposition process of wool fibers (3-6).

The degradation process of wool is connected with the reactions of the protein main chain and the side-chains of the 18 different types of amino acid residue of which wool is composed. The studies of the wool oxidation (3-6) indicate the complete oxidation of the disulfide cystine linkages in the protein chain to give cysteic acid residues as shown in (Scheme 1):

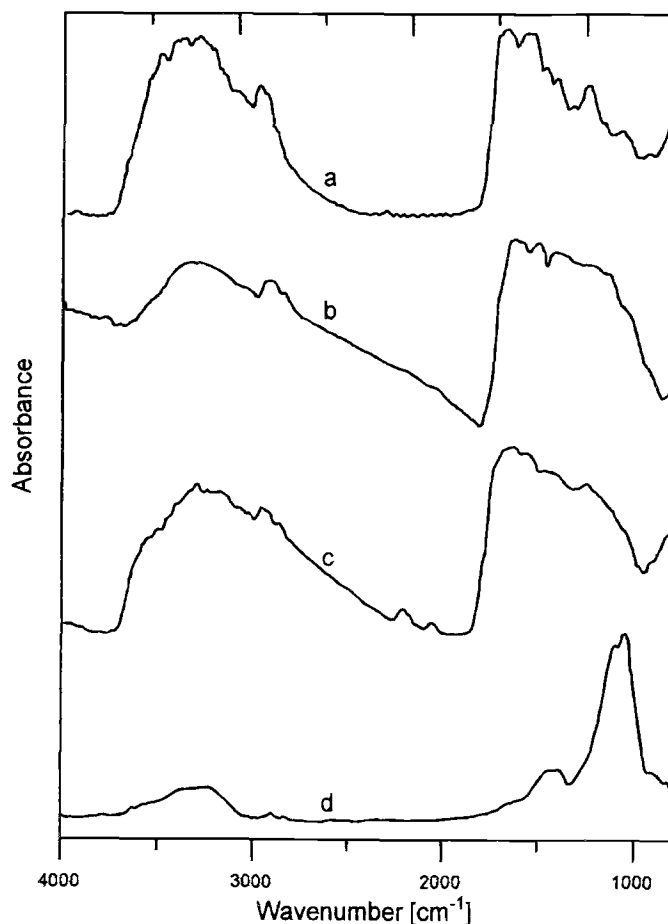
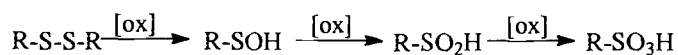


FIG. 1—FTIR spectra of Polish wool fibers: a) original, b) burned, c) decomposed, and d) incinerated.



The end product of such a reaction sequence, a sulphonic acid exhibits two strong absorptions, one in the region 1260 to 1150 cm^{-1} and another in the region 1080 to 1010 cm^{-1} (7). The analysis of burned Polish wool spectrum shows the presence of sulfonic acid in these materials.

The spectrum of decomposed Polish wool (Fig. 1c) resembles the original wool spectrum (Fig. 1a) in the location of the characteristic bands. Relatively stronger absorption bands exist in the region 1470 to 1300 cm^{-1} and at 1237 cm^{-1} (CH_2 , Amide III vibrations), the appearance of weak bands at 2213 cm^{-1} , and 2059 cm^{-1} can also be observed.

The absorption bands in the region 2300 to 2000 cm^{-1} observed in the spectrum of decomposed Polish wool (Fig. 1c) can be attributed to $\text{C}\equiv\text{N}$, $\text{C}=\text{NH}^+$ and NH^+ vibrations. The presence of these groups can be attributed to formation of lysinoalanine and other groups derived from different amino acid residues resulting from the wool thermal degradation (3).

A comparison between the frequencies of the most evident absorption bands in the spectra of incinerated: Polish sheep wool, English Cheviot wool, and German merinos wool indicate that the chemical composition of these incinerated wool fibers (i.e., inorganic minerals) is not the same, each sample has its own specific composition, which can be summarized as follows:

• Incinerated Polish Sheep Wool—The dominant compound is a sulfate mineral (CaSO_4 is suggested) in the hydrated form. In addition, the phosphates and carbonate minerals are also found.

• Incinerated English Cheviot Wool—A phosphate salt in the hydrated form is the dominant compound. A considerable amount of sulfate, silicate, and carbonate compounds are also found.

• Incinerated German Merinos Wool—The dominant compound is phosphate salt in the hydrated form. Silicate, sulfate, and carbonate compounds are also found.

The occurrence of sulfate salts in the incinerated wool fibers could be determined by a kind of amino acids which was presented in the protein side chains. From the most common amino acids, only cysteine, cystine, and methionine contain sulfur atoms in their structure (4). X-ray diffraction, electron diffraction, and dark-field electron microscopy were used by Blakey (5) for observation of silicon and phosphorus in animal fibers. Apatite, hydroxyapatite, and other calcium salts were also identified in different kinds of animal fibers (5).

Cotton Fibers

The spectra of original and burned, decomposed, incinerated Sudanese cotton fibers are given in Fig. 2. Each sample of cotton fibers after burning exhibited three destructive residues of different colors (brown, gray, and black). The spectra of each from these particular parts are also given in Fig. 2.

The spectrum of the brown part from burned Sudanese cotton (Fig. 2b) shows very little change compared with the original cotton spectrum (Fig. 2a). However new absorption bands in the region about 1700 cm^{-1} ($\text{C}=\text{O}$ vibrations) were found. The presence of these bands in the spectrum of burned cotton (Fig. 2b) can be explained by alcohol groups in cellulose being oxidized to carboxyl or aldehyde groups (3,8,9). Additionally, compared with the original cotton spectrum, the relative intensity of the bands in the region 3500 to 3200 cm^{-1} is increased, possibly because of the formation of hydrates, hemiacetals, or hemialdol groups (3,8).

The characteristic cellulose bands were observed also in the decomposed Sudanese cotton spectrum (Fig. 2c); however, bands at about 1700 cm^{-1} become weaker compared with the spectrum brown residue from burned cotton (Fig. 2b). The spectra of the black (Fig. 2d) and gray (Fig. 2e) parts from burned Sudanese cotton do not indicate the presence of characteristic cellulose bands. The absorption bands at 1585 cm^{-1} ($\text{C}=\text{C}$ vibrations), in the region of 1470 to 1400 cm^{-1} (CO_3 vibrations), in the region 1240 to 1055 cm^{-1} ($\text{C}-\text{H}$ deformation and benzene ring substitution), and at 880 cm^{-1} ($\text{C}-\text{H}$ deformation and benzene ring substitution and CO_3 vibrations) are observed.

Hofman et al. (8) report that when cellulose is heated above 300°C , its structure is gradually replaced by an aromatic condensed system and eventually by a charred material. Cobb (8) studied the X-ray diffractograms of cellulose residues and observed that at 300°C , a breakdown of the cellulose structure occurred, at 400°C , carbon repatterning was in evidence, and at 500°C , the formation of a hexagonal network of carbon atoms was found. The complete carbonization of cellulose was observed at 1500°C . The carbonization of cellulose according to Cobb (8) is shown in the following (Scheme 2):

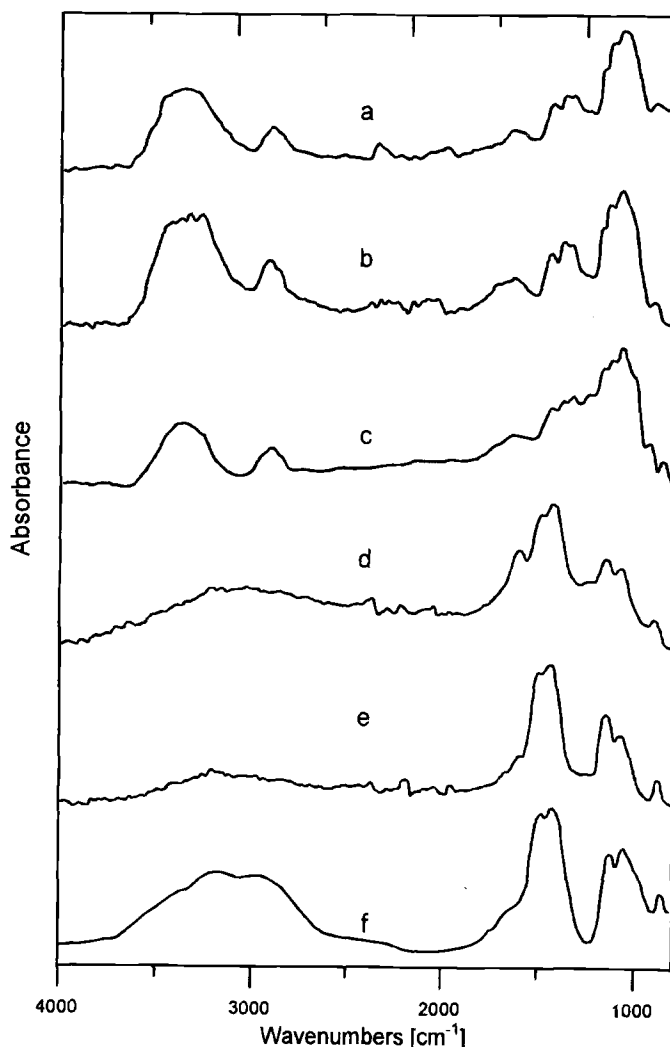
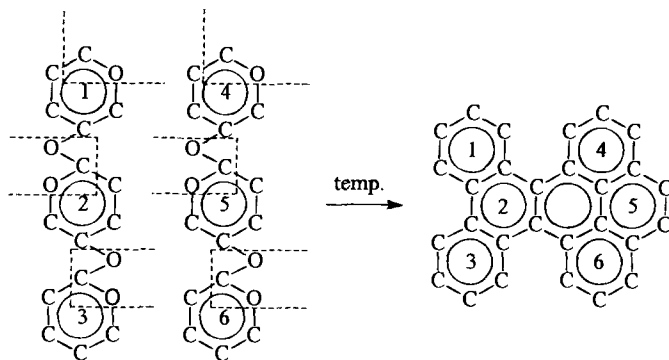


FIG. 2.—FTIR spectra of Sudanese cotton fibers: a) original, b) burned-brown part, c) decomposed, d) burned-black part, e) burned-gray part, and f) incinerated.



The results of IR examinations of the burned, black, and gray part of the cotton fibers confirm that the cotton structure after burning can be replaced by an aromatic condensed system of degraded cellulose and by the inorganic mineral compounds of cotton (Fig. 2d, e). The frequencies, relative intensities, and suggested assignments (7) of bands existing in the spectra of incinerated Sudanese, Pakistani, and Egyptian cotton fibers reveal their

mineral composition. The result of these examinations can be summarized as follows:

- Incinerated Sudanese Cotton—The dominant compound is the carbonate salt in the hydrated form. The presence of silicates and sulfates are also observed.
- Incinerated Pakistani Cotton—Silicates in the hydrated form are the dominant compounds. A small amount of sulfates, phosphates, and carbonates is found also.
- Incinerated Egyptian Cotton—The dominant compounds are silicates in the hydrated form. Carbonates, sulfates, and phosphates exist also as additional minerals.

The presence of silicates (mainly SiO_2) in ashen cotton fibers was confirmed by X-ray fluorescence analysis (10).

Flax Fibers

The spectra of original, burned, decomposed, and incinerated Polish flax fibers are given in Fig. 3. The spectra of burned flax

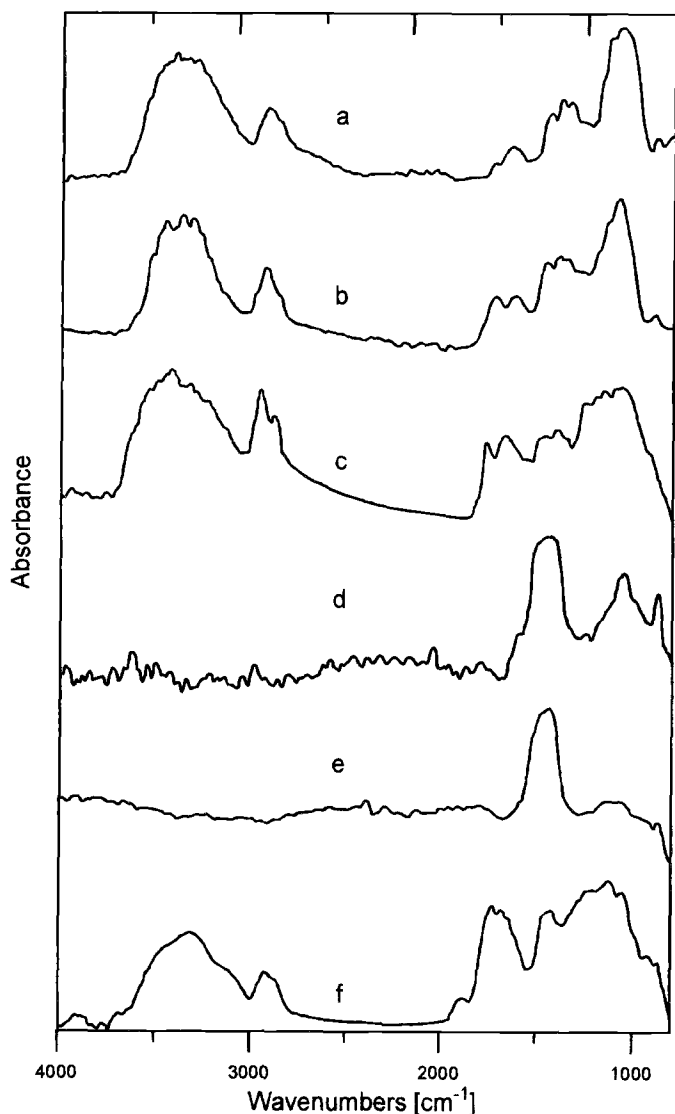


FIG. 3—FTIR spectra of Polish flax fibers: a) original, b) burned-black part, c) decomposed, d) burned-brown part, e) burned-gray part, and f) incinerated.

are from the brown, gray, and black parts of fiber residues obtained after burning.

The spectrum from the black part of burned flax (Fig. 3b) shows very little change compared with the original flax fibers spectrum (Fig. 3a), only the intensity of bands in the region 1730 to 1600 cm^{-1} ($\text{C}=\text{O}$ vibrations) is increased. This result indicates the oxidation of alcohol groups in cellulose to carboxyl and aldehyde groups. A similar result was obtained for brown part of burned cotton fibers (Fig. 2b).

The spectrum of decomposed flax fibers (Fig. 3c) is similar to the original flax spectrum (Fig. 3a); however, the intensities of the bands in the regions 2950 to 2800 cm^{-1} (CH_3 , CH_2 vibrations), 1735 to 1600 cm^{-1} ($\text{C}=\text{O}$ vibrations), and 1220 to 1100 cm^{-1} ($\text{C}-\text{H}$, benzene ring substitution) are increased.

The spectra from brown (Fig. 3d) and gray (Fig. 3e) burned parts of flax fibers show only a few distinct absorption bands: at about 1582 cm^{-1} ($\text{C}=\text{C}$ vibrations), 1440 cm^{-1} (CO_3 vibrations), in the region 1250 to 1000 cm^{-1} ($\text{C}-\text{H}$, benzene ring substitution), and about 880 cm^{-1} (CO_3 , $\text{C}-\text{H}$, benzene ring substitution). The result of these examinations—which are similar to those obtained in the case of black and gray burned parts of cotton—indicate the presence of aromatic condensed system as the form of degraded cellulose and inorganic mineral compounds in brown and gray parts of the burned flax fibers. Hydrated silicates and carbonates' salts are the main inorganic constituents of incinerated flax fibers.

Polyamide Fibers

IR spectra of original, burned, melted, and incinerated Polana fibers are shown on Fig. 4. The spectra obtained for burned (Fig. 4b) and melted (Fig. 4c) Polana resemble the original Polana spectrum (Fig. 4a) in the location of the characteristic bands. Only the relative intensity of absorption bands is different. When the spectrum obtained for burned Polana fibers (Fig. 4b) is analyzed, a weak band at 2251 cm^{-1} , attributed to $\text{C}\equiv\text{N}$ vibrations and absent in original Polana spectrum (Fig. 4a) is noticed.

This result can be interpreted as indicating the presence of nitriles in burned Polana fibers. Studies for the thermal degradation process of amides (3,11,12) confirm that nitriles could be a final product of thermo-oxidative decomposition of nylon 6. The other products of thermo-oxidative decomposition of nylon 6 are water, carbon monoxide, carbon dioxide, acetaldehyde, formaldehyde, methanol, carboxylic acids, amines, and cyclopentanone (3).

In the case of melted Polana (Fig. 4c), the absorption bands in region 3350 to 2850 cm^{-1} (HCH_2 , NH , and OH vibrations) have relatively higher intensity than the absorption bands at 1642 , 1537 cm^{-1} (Amide I, Amide II vibrations), the strongest in original Polana spectrum (Fig. 4a). TiO_2 is the dominant inorganic compound of incinerated Polana fibers (Fig. 4d). The additional mineral compounds are silicates. During some manufacturing process's, titanium dioxide is incorporated in the polyamide-polymer to improve the texture of the fibers. The opacity of nylon fibers is controlled by an amount of titanium dioxide added with fully dull yarns containing about 2% TiO_2 (13).

Polyester Fibers

The spectra of original, burned, melted, and incinerated Elana fibers are shown in Fig. 5. The analysis of burned (Fig. 5b) and melted (Fig. 5c) Elana spectra do not indicate differences from original Elana spectrum (Fig. 5a) in the number and the location of absorption bands. However, differences in the relative intensity

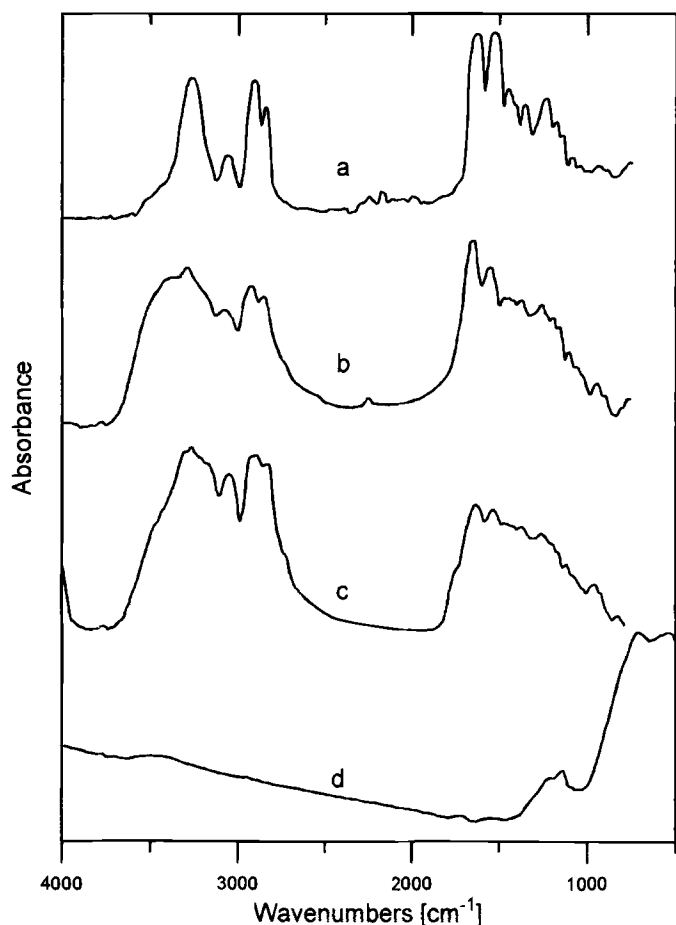


FIG. 4—FTIR spectra of Polana fibers: a) original, b) burned, c) melted, and d) incinerated.

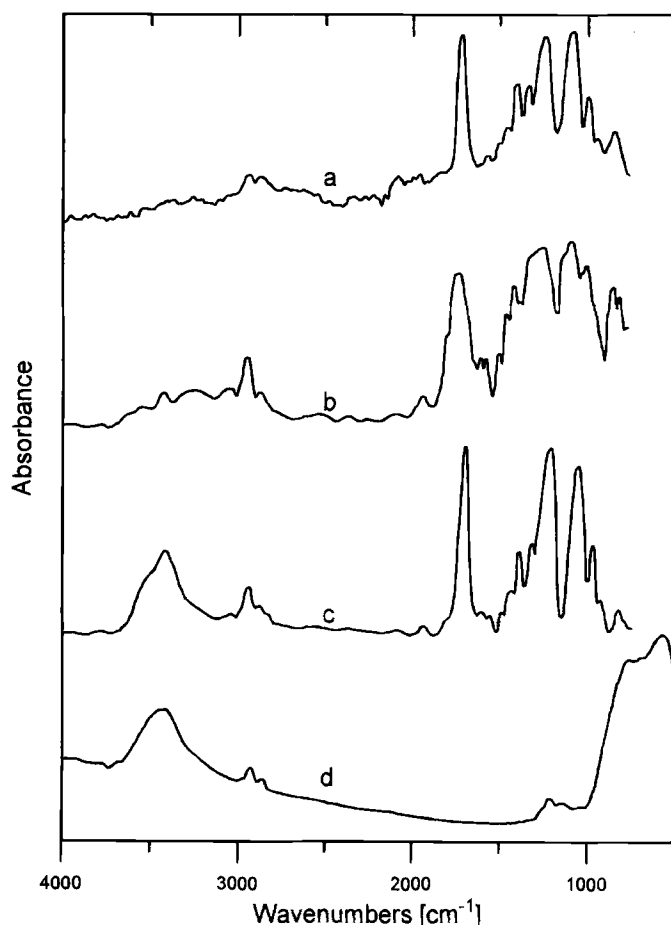
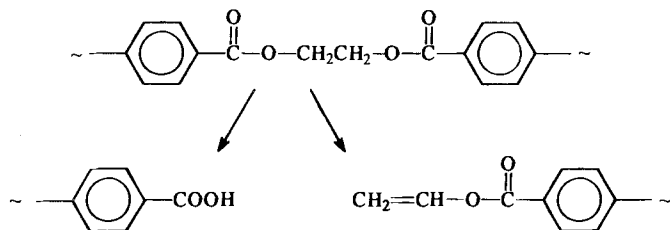


FIG. 5—FTIR spectra of Elana fibers: a) original, b) burned, c) melted, and d) incinerated.

of bands exist, particularly in the region 3430 to 2850 cm^{-1} (OH vibrations) and at 1645 cm^{-1} (C=C vibrations), where the intensity of bands in the burned (Fig. 5b) and melted (Fig. 5c) Elana spectra is increased compared with original Elana spectrum (Fig. 5a). During the thermal degradation of polyester carboxyl and vinyl ester groups are formed by the β -elimination process (3) as shown in Scheme 3:



The presence of C=C band in the spectra of burned (Fig. 5b) and melted (Fig. 5c) Elana can be detected as a result of this change. The spectrum obtained for incinerated Elana fibers (Fig. 5d) is similar to a spectrum of TiO_2 . Consequently, TiO_2 is a dominant inorganic compound of incinerated Elana fibers. Silicates can also be observed as additional compounds in incinerated Elana fibers. Titanium dioxide is incorporated in the polyester-polymer during some manufacturing processes for the same reason as it is added to polyamides.

Acrylic Fibers

Figure 6 presents the original Anilana fiber spectrum and the spectra of burned, decomposed, and incinerated fibers. In the spectrum of burned Anilana (Fig. 6b), the appearance of bands of relatively higher intensity than in the original Anilana spectrum (Fig. 7a) at 3468 , 3368 cm^{-1} (assigned to N-H vibrations), at 2187 cm^{-1} (C≡N vibrations) and in the region 1688 to 1498 cm^{-1} (C=N vibrations) are observed. Simultaneously, a band at 1172 cm^{-1} (assigned to C-N and N-H vibrations) and bands at 1802 , 1775 cm^{-1} (C=O vibrations) are decreased in intensity.

The same relations between the spectra of original (Fig. 6a) and decomposed (Fig. 6c) Anilana fibers are observed. However, in the spectrum of decomposed fibers, the intensity of bands in the region 3600 to 3150 cm^{-1} (N-H vibrations) increases, and the intensity of band at 2245 cm^{-1} (C≡N vibration) decreases.

FTIR studies of the thermal degradation of polyacrylonitrile (PAN) performed at 200°C in air and under reduced pressure are presented by Coleman and Petcavich (14). These authors suggest also the mechanism of PAN thermal degradation consistent with the results obtained for burned and decomposed Anilana fibers. Their most important observance, for the identification of thermally changed acrylics fibers by FTIR, was that at fixed temperature, the nitrile absorption band at about 2240 cm^{-1} shows a decrease in intensity as a function of degradation time. The analysis of incinerated Anilana fibers (Fig. 6d) reveals that the charry material is similar in structure to an aromatic condensed material.

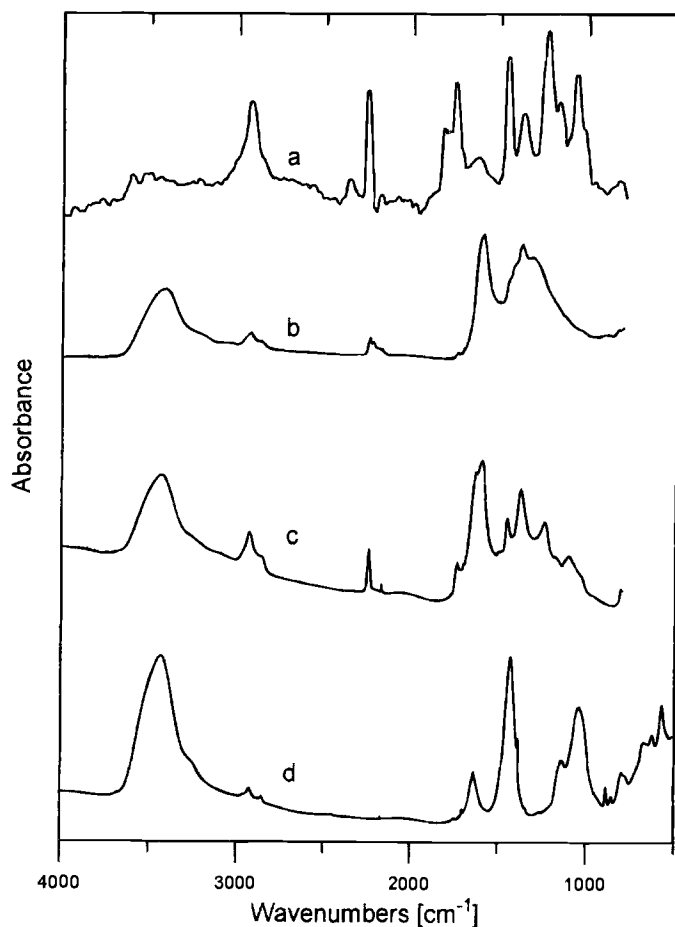


FIG. 6—FTIR spectra of Anilana fibers: a) original, b) burned, c) decomposed, and d) incinerated.

Polypropylene Fibers

The FTIR spectra of original, burned, and melted polypropylene fibers are shown in Fig. 7. The spectra of burned (Fig. 7b) and melted (Fig. 7c) polypropylene fibers resemble the original polypropylene spectrum (Fig. 7a) in the location of the characteristic bands; however, differences in the relative intensity of bands exist. In the case of melted polypropylene (Fig. 7c) the relatively higher as in original spectrum (Fig. 7a) intensity of bands in the region 3200 to 3140 cm^{-1} , at 2720 cm^{-1} , at 1160 cm^{-1} (C=C vibrations), at 1370 cm^{-1} (C-H deformation vibrations), at 1160 cm^{-1} (C-C skeletal vibrations), in the region 1000 to 970 cm^{-1} , at 897 cm^{-1} , and 840 cm^{-1} (C-H, C-C skeletal vibrations) are observed. A similar result was noticed when the relative intensities of bands in the spectra of original (Fig. 7a) and burned (Fig. 7b) polypropylene fibers were compared. In addition, in the spectrum of burned polypropylene fibers (Fig. 7b), the intensity of the absorption bands at about 1640 cm^{-1} (C=C vibrations) increases. It was not possible to obtain incinerated polypropylene fibers in the same way that has been used for getting other kinds of incinerated fibers.

Conclusion

The comparison between FTIR spectra of burned, decomposed, and incinerated fibers reveals an important information concerning the pathway of their thermal degradation. The different attachments, e.g., diamond anvil optical cell can be applied in the FTIR

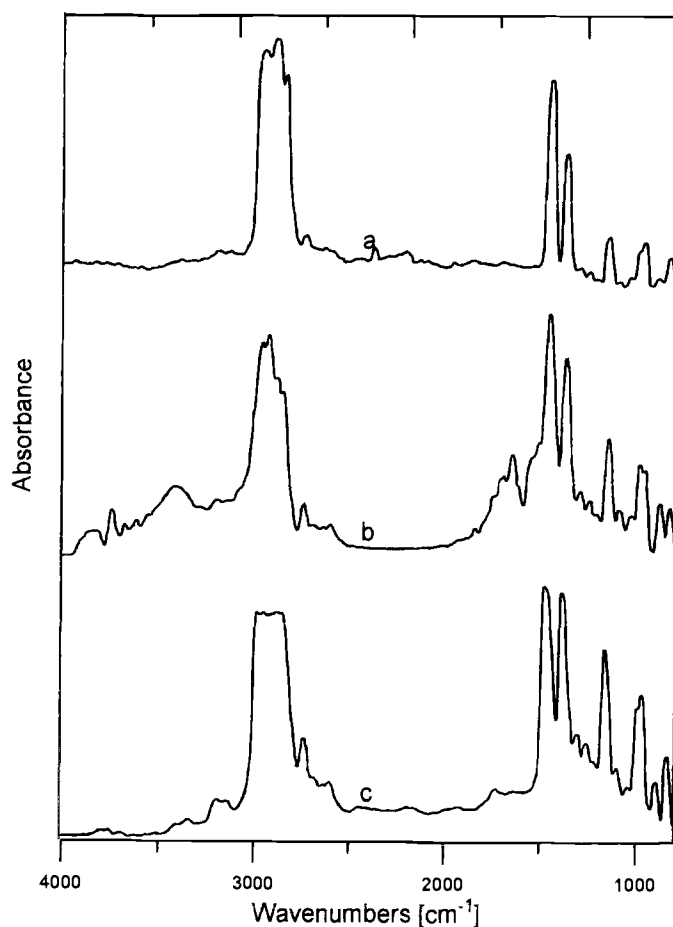


FIG. 7—FTIR spectra of polypropylene fibers: a) original, b) burned, and c) melted.

microspectroscopical examination of thermally changed fibers. The small field of view of the microscope and the small thickness of fibers pressed between two diamonds are helpful to obtain good quality spectra in spite of different imperfections existed in thermally changed fibers and their small amount attainable in forensic examination.

The identification of burned, melted, and decomposed fibers is often possible because of presence in their IR spectra many of characteristic for original spectrum absorption bands. In the case of incinerated fibers, the identification of their mineral compounds is possible by FTIR. This is indicated, because for this limited population studies, each sample has its own specific inorganic mineral composition and the differentiation between cotton and wool fiber origins could be achieved.

However, one should emphasize that the subject of this research was undyed fibers, rarely used on an everyday basis. The presence of various kinds of dyestuff, pigments, and especially flame-retardant compounds may influence the results of these examinations and further studies are required.

During the fiber burning procedure, the conditions of treating-time and temperature were rather uncontrolled, consequently, the obtained results will not be exactly repeatable in all respects, especially in absorption bands intensities. However, data for fiber identification regarding criminalistic work are possible using the FTIR microscope techniques described.

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Address requests for reprints or additional information to
 Jolanta Was, Ph.D.
 Institute of Forensic Research
 Westerplatte 9, 31-033 Krakow
 Poland