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Using Small-Angle Light Scattering to Discriminate Among Single Fibers Subjected to Consumer-Like Uses

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ABSTRACT: Small-angle light scattering (SALS) patterns and scanning electron photomicrographs (SEM) of single fibers were recorded for five different types of polyester fibers when new as well as after being laundered by either of two different methods or after being exposed to outdoor weather. SALS was a sensitive tool for discriminating among the fiber types when new as well as after each of the three treatments. In addition, SALS was able to discriminate among fibers from the same yarn bobbin in each of the four different conditions examined. SALS was more sensitive in detecting some types of physical changes than was SEM.

KEYWORDS: criminalistics, synthetic fibers, lasers

In a wide variety of criminal investigations, fiber analysis often is critical. Analyzing fiber evidence, however, is difficult largely because of limitations in sample size. That is, a short segment of a single fiber typically has a mass of only a few micrograms, and insufficient sample size precludes the use of many methods of analysis. In addition, it is desirable to use analytical methods that leave samples completely unaltered for further analysis or presentation as evidence.

Most methods of fiber analysis depend only on fiber identification (usually meaning assignment of generic group, such as polyester, or trade name, such as Dacron®) and often possess limited evidential value. But methods that allow discrimination among fiber types within a generic group, such as between Dacron Type 56 polyester and Dacron Type 242 polyester, hold potential for greater evidential power. Similarly, methods that allow discrimination among fibers produced identically but used by different consumers, such as between Dacron 56 laundered in hot water and Dacron 56 laundered in cold water, hold promise of yielding even greater evidential power.

Our society is characterized by widespread consumption of a relatively small number of fiber generic groups produced by a relatively small number of manufacturers so that relatively limited evidential power results from generic group or trade-name identification. For example, even though fibers from a crime scene and a suspect's clothing both are identified as polyester, evidential power might be limited since nearly everyone in our society commonly wears fabrics containing polyester. On the other hand, if fibers from both the crime scene and the suspect's clothing are characterized 20 different ways and

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are found to be very similar in each way, then the fibers hold greater evidential power, regardless of their being identified as polyester or not. Small-angle light scattering (SALS) is a simple, quick, and economical method of characterizing single fibers a variety of different ways.

Characterizing polymeric materials by SALS has proved to be useful in many areas of basic research [*1*]. Efforts were begun recently to determine the usefulness of SALS when applied to fiber problems encountered in forensic science [*2,3*]. The first paper of this series [*2*] described the method's ability to discriminate among single fibers of different generic groups, such as polyester and nylon. The second paper of the series [*3*] reported that SALS was able to discriminate among different fiber types within one generic group, such as Acrilan® Type B16 acrylic, Acrilan Type S82 acrylic, and Creslan® Type 68 acrylic. The method also was found to be useful in quantitatively characterizing surface topography, opacity, and fiber cross-sectional shape without the necessity of preparing a cross-sectional slice as in microscopy. SALS produces absolutely no fiber structural changes, whereas many methods of fiber analysis including some methods of microscopy induce chemical or physical changes in fiber structure. Thus, in the short time SALS has been investigated as a tool for forensic science examination of fibers, it has proved to be useful in many ways.

The fibers examined in the studies cited were supplied directly by the fiber producers and were not used by consumers. The practicing forensic scientist, however, rarely examines new fibers but instead is asked to analyze fibers subjected to consumer use. Therefore, it is more realistic to evaluate the utility of SALS for forensic science fiber analysis by examining the method's ability to discriminate among fibers having identical manufacturing origin but subjected to different consumer use, that is, its ability to go beyond discrimination based on generic group or fiber type differences and characterize fibers for comparison on the basis of consumer use. In this paper we report preliminary results of such an examination.

Five types of polyester fibers were chosen for the study. Three simulated consumer practices were applied to the fibers before the SALS analysis. The practices chosen were outdoor weather exposure and the laundry techniques of mild, low-temperature hand washes and vigorous, high-temperature machine washes.

While meaningful differences among various fibers have been demonstrated by SALS, the question still remains as to whether meaningful similarities can be demonstrated among fibers from a single source, such as fibers within a consumer's shirt. Scattering patterns collected during all three studies undertaken thus far have shown consistent and reproducible patterns for fibers in each sample studied. However, as with most methods of analysis, multiple measurements on an actual consumer-used textile must be made to provide a quantitative measure of the method's actual usefulness and limitations. Consequently, a fourth paper in this series will follow that provides a quantitative assessment of differences among different fiber samples and similarities among fibers within one sample.

Apparatus and Experimental Procedure

The SALS apparatus and methods of use have been described in detail elsewhere [*2,3*]. All SALS measurements in the study reported here were made by using a Hughes 5-mW polarized laser with each fiber sample suspended vertically in air. The polarization direction of the analyzer was horizontal and that of the incident beam was vertical (Hv). Sample-to-film distance was held constant for each fiber at 483 mm. Exposure times ranged from 4 to 6 s. Because the fibers were heavily crimped, we used special procedures, detailed elsewhere [*3*], to enhance reproducibility of measurements.

Scanning electron microscopy (SEM) was performed by using standard procedures.

The polyester fibers examined included one type of Encron®, three of Dacron, and one of Fortrel® as listed here: Encron knit-de-knit textured by Burlington Madison Yarn Co.; Dacron 56 false-twist stretch, textured by Spray Textured Yarns, Inc.; Dacron 242 false-twist stretch, textured by Frank IX and Sons, Inc.; and Fortrel 660 false-twist stretch and stabilize, textured by Macfield Texturizing, Inc. Samples from each of the five yarn packages were subjected to treatments representing 50 low-temperature hand washes following Test IA of American Association of Textile Chemists and Colorists (AATCC) Test Method 61-1975; other samples were subjected to treatments representing 30 vigorous high-temperature machine washes following Test IIIA of AATCC Test Method 61-1975 [*4*]. In addition, other samples were subjected to outdoor weather continuously for one month following exposure conditions as dictated in AATCC Test Method IIIB-1978 [*5*]. Consequently, 20 fiber groups were examined by SALS and SEM.

Results and Discussion

To conserve space, SALS patterns and SEM photomicrographs are provided for only one fiber series, Dacron 242. The SALS patterns in Fig. 1 and the SEM photomicrographs in Fig. 2 represent Dacron 242 in the new, low-temperature laundered, high-temperature laundered, and outdoor-exposed states. Examination of SALS patterns of all fibers studied showed that each fiber when new produced a distinctive, reproducible scattering pattern, which allowed it to be distinguished from the other new fibers. In addition, expected information [*2,3*] concerning size, cross-sectional shape, and surface topography of each fiber type when new was obtained by SALS and confirmed by SEM.

The sensitivity of SALS to physical changes accompanying laundering and outdoor exposure can be appreciated by examining the SALS patterns and SEM photomicrographs of Dacron 242, provided in Figs. 1 and 2. The scattering pattern of a fiber changes when laundered by either method or exposed to outdoor weather. The changes allow new fibers to be distinguished from those subjected to consumer use. In addition, it can be seen that changes in the scattering patterns of the fibers differ as a result of either of the two laundering methods or outdoor exposure. Consequently, SALS offers a method for discrimination among identical Dacron 242 fibers used differently by consumers. Analogous changes in scattering were observed for each of the fiber types examined and hence conclusions drawn for Dacron 242 may be assumed to be general. In addition, the ability of SALS to discriminate among all fiber types examined in this study remained through both laundering methods and outdoor exposure since each fiber type laundered and weathered distinctively.

It is helpful to characterize physical changes observed in the fibers used in this study as they were being subjected to laundering or outdoor exposure. Three kinds of physical changes were observed: surface deposition, surface pitting, and fibrillation.

Exposing all fiber types to outdoor weather increased the amount of material deposited on the fiber surfaces, and the amount of scattering, as observed by the SALS, increased as surface deposition increased. An increase in surface deposition can be seen for Dacron 242 by comparing Figs. 2a and 2d. Its effect on scattering can be seen by comparing Figs. 1a and 1d. Though all fiber types showed increases in surface deposition, the amount of deposition varied for each fiber type. In addition to surface deposition, one fiber, Encron, developed surface pitting, which produced an increase in scattering similar to that of surface deposition.

The two laundering methods affected the fibers differently. All fibers showed some increase in surface deposition and hence light scattering, although deposition for some fibers was small. Some fibers showed greater deposition when laundered at the high temperature than when laundered at the low temperature, yet other fibers showed the reverse trend. Dacron 242, illustrated in Figs. 2a, 2b, and 2c, showed only a slight increase in sur-

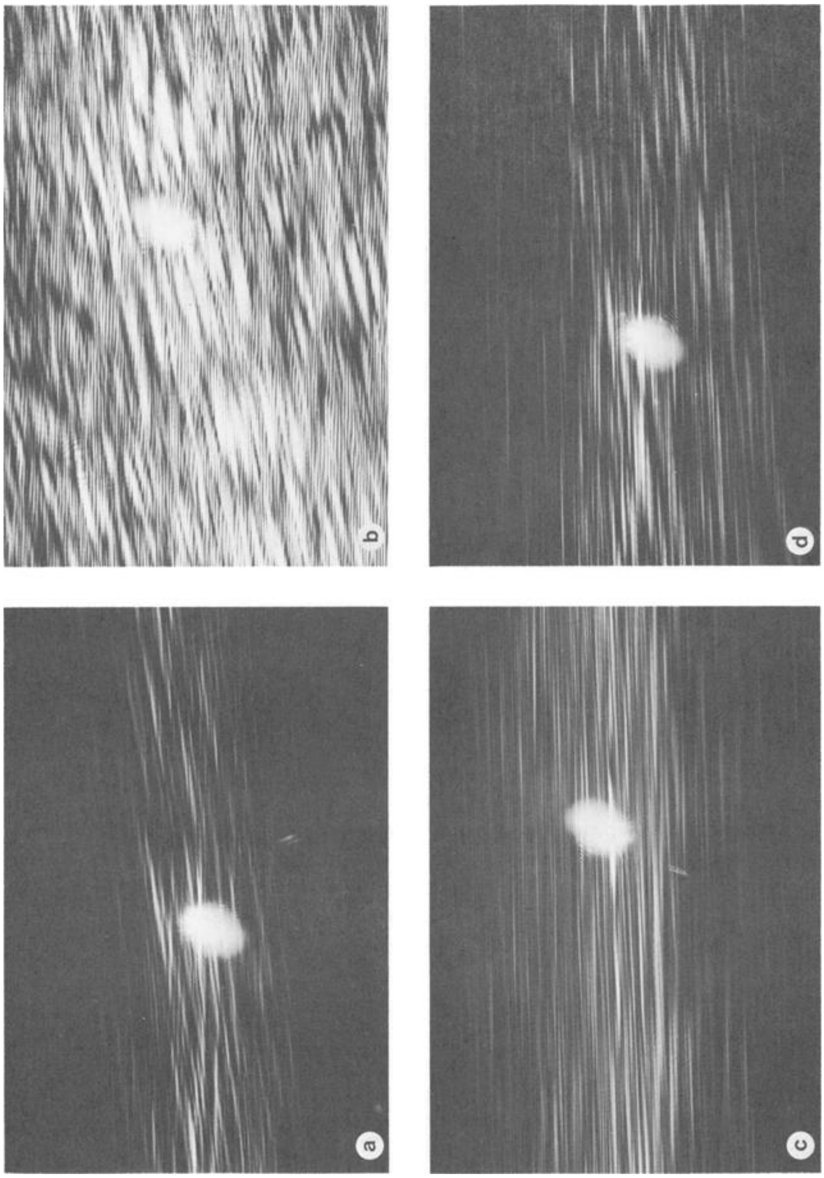

FIG. 1—*The SALS Hv patterns of Dacron 242 fibers from* (a) *a new yarn,* (b) *yarns subjected to treatments representing 50 low-temperature hand washes,* (c) *30 vigorous, high-temperature machine washes, and* (d) *continuous outdoor exposure for one month. All patterns were recorded with the fiber axes suspended vertically in air.*

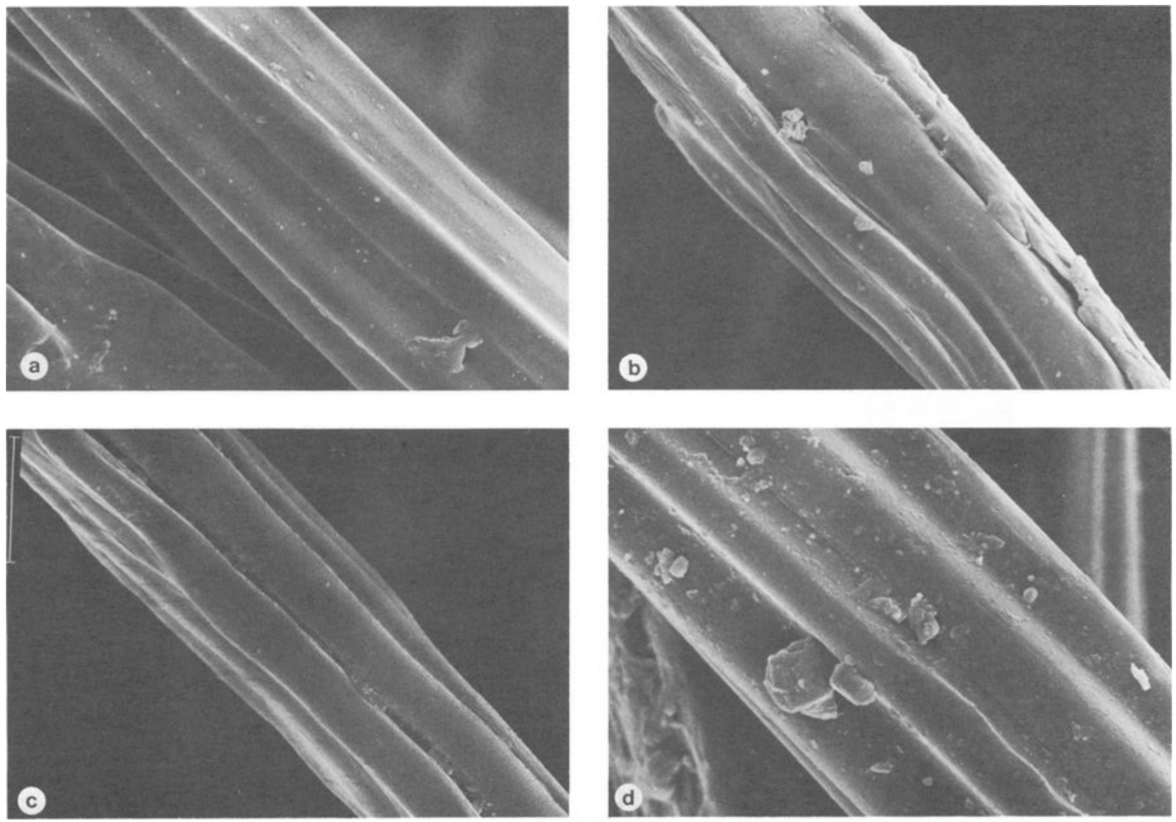

FIG. 2—*SEM photomicrographs of Dacron 242 fibers from* (a) *a new yarn,* (b) *yarns subjected to treatments representing 50 low-temperature hand washers,* (c) *30 vigorous, high-temperature machine washes, and* (d) *continuous outdoor exposure for one month.*

face deposit, with no distinction as to laundering method. Corresponding SALS patterns for Dacron 242 would be expected to show small increases in scattering for the laundered samples compared with that of a new fiber. An examination of Figs. 1a, 1b, and 1c does not reveal such small changes because they are masked by large increases in scatter resulting from fibrillation attributed to abrasion during laundering. Interestingly, mild, low-temperature laundering of Dacron 242 resulted in more severe fibrillation than did vigorous, high-temperature laundering. That finding is illustrated in the SEM photomicrographs of Figs. 2b and 2c and is reflected in the SALS patterns of Figs. 1b and 1c.

It is important to note that SALS is more sensitive in detecting induced fibrillation than is microscopy, as can be appreciated by comparing the differences in light scattering between Figs. 1a and 1b with the differences in fibrillation between Figs. 2a and 2b observed by SEM. That sensitivity results from the fact that the SALS pattern reflects sampling over the entire fiber surface, whereas examination by SEM shows only one surface at any given time.

We attribute the greater fibrillation that results from the low-temperature laundering method than from the high-temperature method to temperature effects rather than to the greater number of launderings by the low-temperature method. The glass-rubber transition temperature of the fibers subjected to laundering probably lies between the high and low temperatures used in laundering these samples. As a result, the fibers would behave as a soft rubber during high-temperature laundering and as a brittle plastic during low-temperature laundering. The brittle plastic would be expected to fibrillate more readily than the soft rubber. We tested this hypothesis by laundering one additional sample of Dacron 242 30 times at a low temperature (40°C) and another sample 30 times under identical conditions except at a high temperature (71°C). The SALS patterns revealed much more fibrillation in the sample laundered at low temperature compared to that in the sample laundered at high temperature. Consequently, it appears that temperature effects are responsible for differences in fibrillation between the samples. In any case, different laundering practices result in different physical changes in fiber structure and the changes can be readily detected by SALS. Furthermore, SALS is more sensitive in detecting the changes than is SEM.

Summary

Five types of polyester yarn were subjected to outdoor exposure and two common laundering procedures. Physical changes in the fibers resulting from these treatments were recorded by SALS and documented by SEM. It was shown that SALS provides a means for discriminating among different commercial polyester fiber types when new as well as after being subjected to multiple launderings or prolonged outdoor exposure. In addition, SALS was capable of discriminating among fibers of identical type and commercial origin but subjected to different laundering practices or exposure to outdoor weather. SALS was more sensitive in detecting some types of physical changes resulting from laundering than was SEM.

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