

RAMAN SPECTROSCOPY FOR FIBER ANALYSIS

APPLICATION NOTE RAMAN-023 (A4)

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Abstract

This application note documents the use of a TSI ChemLogix™ ProRaman-L instrument with a microscope to examine a variety of single textile fibers.

Motivation

Law agencies around the world utilize fiber analysis to help place suspects at the scene of the crime. Transfer of fibers can happen during close contact between a victim and a suspect. Additionally, fiber transfers are useful evidence when trying to establish the identity of a suspect in a break-in. Although fibers are not as conclusive as DNA evidence, it is useful in lead establishment and in narrowing fields of potential suspects.^[1]

Fibers are important trace evidence because they can be transferred when two objects come into contact. They can be transferred from one individual to another (primary transfer), but fibers can also be transferred from items belonging to the perpetrator to the victim (secondary transfer). At crime scenes, fibers are collected with gloved hands or tweezers and then sent to a laboratory for analysis. Fibers are classified and compared by origin (natural or synthetic), color and physical characteristics such as crimp, pliability and resilience.^[1,2]

When fibers are present in sufficient quantities, composition can be partially determined through observing the material burn. Plastics, for example, melt into globs and wool smells like burning hair. More sophisticated analysis is available with scanning electron microscopy (SEM). SEM will readily distinguish natural from man-made fibers and can perform elemental analysis. However, SEM is expensive and requires a highly trained operator. Additionally, this analytical technique is not very good at identifying fibers sources in more detail.

Raman spectroscopy has been used recently to identify individual textile fibers by origin. Raman has a unique set of advantages for this type of forensic analysis in that it is able to identify individual fiber types through recognition of molecular bonds and that the technique is non-destructive. It can also be utilized performed through a microscope, enabling molecular analysis of single fibers.^[3,4]



Samples

Several very small samples of fabrics were taken from clothing belonging to TSI application scientists. They included threads from a cotton/poly t-shirt, a poly/cotton lab coat, a shoe string of unknown composition, a silk scarf, a wool sweater, a linen shirt, a piece of garden twine and some fibers from a laboratory-grade cotton swab.

These fibers were presented to the measurement system on a small aluminum block. The block was slightly moistened so that the fibers would lie flat on it. The fibers, in general, did not lift from the block, but the cotton material from the swab had fibers that would not stick to the substrate and were analyzed at some distance from the surface.

The only sample that was analyzed whole was the silk scarf. The other samples were small pieces of material cut from the seams of the garments and shredded to expose individual threads.

All the samples used in this comparison were white in color. A future applications note will document the use of Raman spectroscopy on colored textile materials.

Measurements and Results

All samples were analyzed with a TSI ChemLogix ProRaman-L coupled to an Olympus CX31 microscope. This instrument has a 785 nm excitation laser, with full power used through the microscope to generate spectra. The samples were introduced and roughly positioned using the 10x objective. Once the sample threads were seen on the Microviewer, the 40x objective was rotated into use. The individual fibers were focused, the location of the laser beam was confirmed and the analysis begun. Photos of most of the single fiber samples were also acquired. Two have been presented here, one of a shoe lace thread illuminated from the top with an LED (Figure 1) and the same thread with the laser position shown (Figure 2). The figure with the location of the laser beam shown was taken with very low laser power, as when the laser power is sufficient to perform an analysis, the camera is saturated.

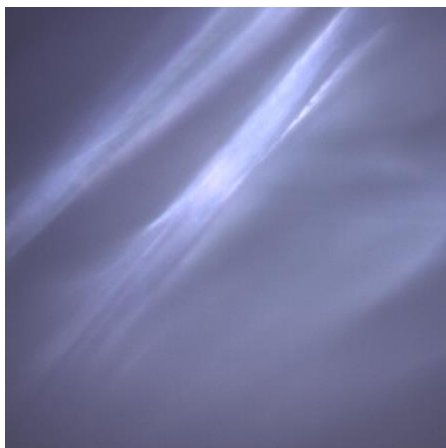


Figure 1. Thread from shoe lace, 40x, illuminated from top.

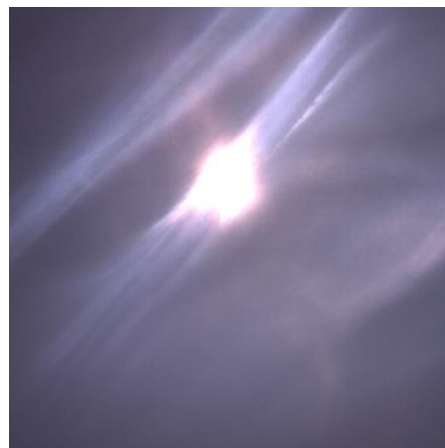


Figure 2. Thread from shoe lace showing location of laser interaction. This photo was acquired under very low laser power to prevent camera saturation.

Most of the threads were of similar dimensions, except the thread from the garden twine sample, which was much thicker than any of the others.

Maximum laser power was used on all these samples, without any sign of damage. Figure 3 shows all the spectra of fibers that are plant-based. In general, the Raman features from the base materials are similar.

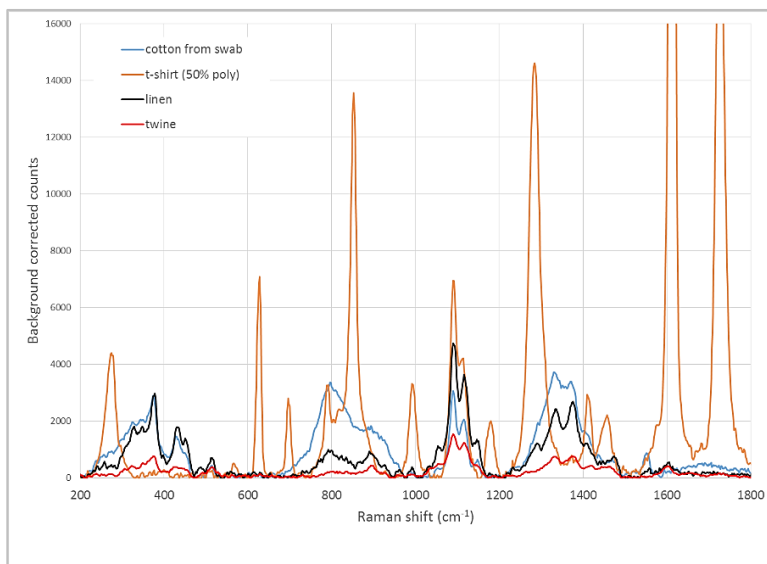


Figure 3. Raman spectra of all plant-based textile material samples.

The most obvious spectral difference in this series is the presence of the very prominent polyester (polyethylene terephthalate) in the spectrum of the thread from the t-shirt. These features include 702 cm^{-1} (ring C-C stretch), 795 cm^{-1} (ring torsion and C=O stretch), 858 cm^{-1} (ring C-C, ester (CO-C)), 998 cm^{-1} (O-CH₂), 1095 cm^{-1} C-O and C-C stretch. C-O-C bend), 1180 cm^{-1} (ring C-C stretch), 1289 cm^{-1} (CO-O stretch), 1310 cm^{-1} (ring C-C stretch), 1613 cm^{-1} (ring C1-C4 stretch) and 1725 cm^{-1} (C=O stretch). Only one of these features overlaps with those observed in the samples without polyester, 1095 cm^{-1} .

The contribution to this feature can be seen by looking at this portion of the spectrum slightly closer in (Figure 4), where it appears on top of a set of lines clearly belonging to a structure in cotton, linen and twine. The one animal-based fiber (silk) that was examined does not have any intensity in this region.

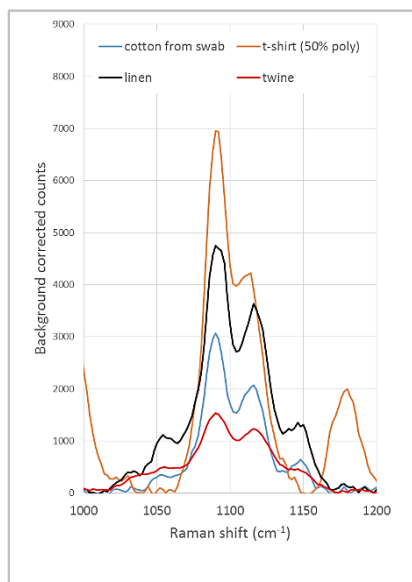


Figure 4. Close-up of spectrum in Figure 3, showing a set of lines clearly originating in plant materials.

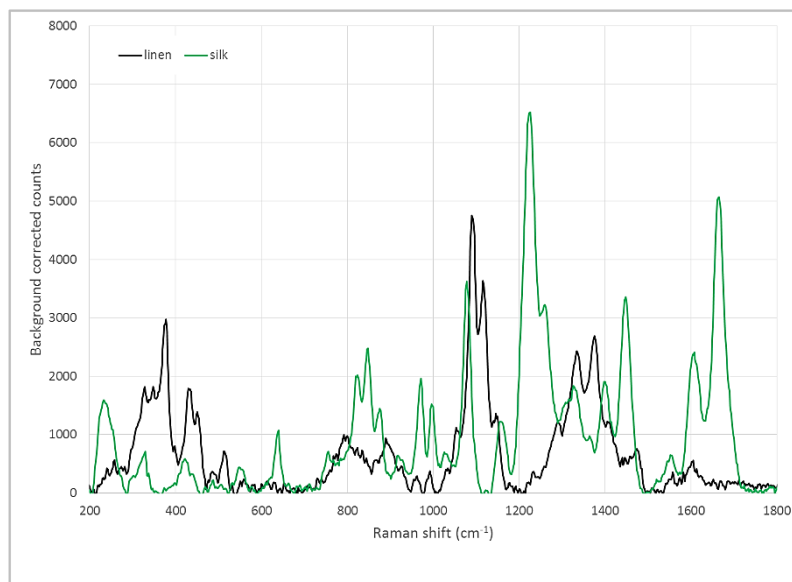


Figure 5. Raman spectra of silk and linen, demonstrating the differences between animal and plant fibers.

Silk is a material that has been well-studied with Raman spectroscopy. Several peer-reviewed publications discuss both dispersive and polarized results of analysis of silk from both silkworms and spiders. Raman spectroscopy is used principally in these analyses as an interrogator of molecular structure, probing the differences between worm silk and spider silk. These molecular differences explain the surprising differences in physical properties. In comparison to the spectra in reference^[5], it is clear the silk scarf sample used in this application note was produced from silk worms, rather than spiders.

Summary

This short application note has focused on the identification of single fibers with Raman spectroscopy. The differences are so clear that a small database of these materials could easily be generated and used to identify unknown samples. This data was all acquired on white materials; colored textile samples will be analyzed for an upcoming application note.

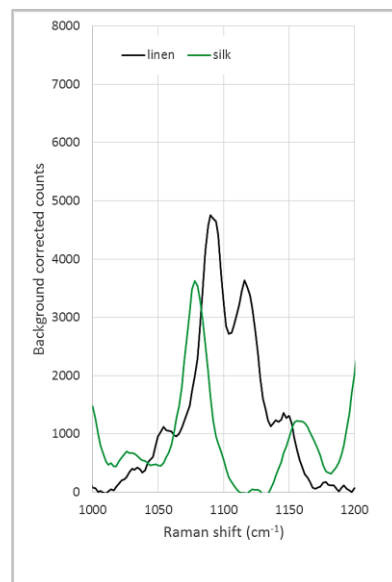


Figure 6. Close-up of spectra of linen and silk showing differences in the region around 1100 cm⁻¹.

References

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