

Infrared Spectroscopy

Author**P. Garside****Research Fellow in Conservation Science
Textile Conservation Centre
University of Southampton
Winchester Campus
Park Avenue
Winchester
SO23 8DL
UK**

Characterization of Textile Fibers Using the PerkinElmer Spectrum 100 with Universal ATR Polarization Accessory

Introduction

The ability to characterize textile fibers accurately is important in many fields, including forensic analysis, industrial production and in textile conservation where it may reveal information about the provenance and condition of an artifact which in turn can inform

its conservation and future interpretation. Conventional methods such as microscopy and spectroscopy can be used to investigate the morphology and chemistry of a sample, but reveal little about its microstructure. Combining infrared spectroscopy with a polarizer allows both the nature and orientation of chemical bonds within the sample to be explored, thus yielding vital microstructural information.

Method

Infrared spectra of fiber samples were collected with a PerkinElmer® Spectrum™ 100 FT-IR spectrometer, fitted with a UATR Polarization Accessory; spectra were recorded over the range 4000-625 cm^{-1} , with a resolution of 4 cm^{-1} and 32 accumulations. The polarizing filter was aligned such that the electric vector of the polarized radiation was oriented perpendicular to the plane of beam path; the fibers were positioned at a known angle on the ATR crystal, from a nominal origin of 0° in which the fibers were perpendicular to the beam path.

The PerkinElmer Frontier FT-IR supercedes the Spectrum 100.

Vegetable fibers

Fibers derived from plant sources are principally composed of cellulose ordered in various internal structures, from crystallites through fibrils to the cell wall; cellulose fibrils are wound around the axis of the fiber in the secondary cell wall, with a direction and angle of wind characteristic of the plant species. Hemp and flax fibers are particularly difficult to differentiate, especially when degraded, but can readily be distinguished using polarized ATR spectroscopy; spectra were recorded at 7.5° degree intervals (Figure 1), and a crystallinity index defined as $X = I_{1160}/I_{1060}$, which was then plotted against orientation (Figure 2). As can be seen, the difference between the hemp (cellulose aligned at 7.5° anticlockwise) and flax (7° clockwise) is obvious.¹ The orientation of cellulose in other plant fiber species can be investigated similarly.²

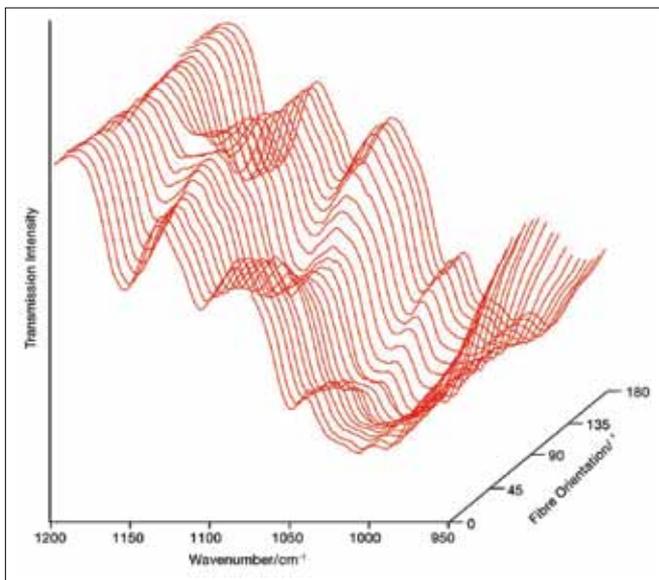


Figure 1. Polarized ATR spectra of flax (1200-950 cm^{-1}), recorded at 7.5° degree intervals, showing spectral variation with orientation.

Silk

Silk is a proteinaceous fiber, whose properties are dominated by the presence of extensive β -sheet crystallites aligned parallel to the fiber axis; as the protein degrades, the crystallites lose this strong alignment. If polarized spectra are recorded with the fibers aligned at 0° and 90°, and a crystallinity index defined as $X = I_{1615}/I_{1655}$, the alignment of the crystallites may be assessed by an orientational order parameter defined as $\Omega = X_{90^\circ}/X_{0^\circ}$. This value may be plotted against a physical parameter, such as breaking load (Figure 3), and it can be seen that as the microstructure of the fiber loses its alignment, so the fiber itself loses strength, providing a way of indirectly monitoring the condition of the material.³

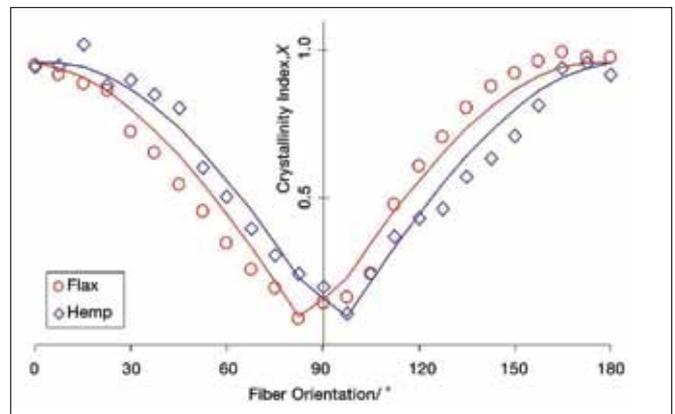


Figure 2. Fiber orientation against crystallinity index for flax and hemp (experimental points and theoretical values).

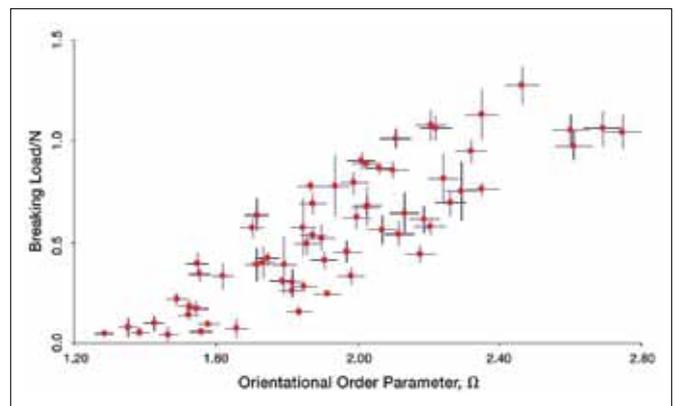


Figure 3. Orientational order parameter, Ω , against breaking load for silk samples in various states of deterioration.



Figure 4. Accurate characterization of historic textiles can help inform their future conservation.

Conclusion

Polarized ATR spectroscopy is a powerful tool which allows subtle microstructural features of a sample to be investigated. Problematic materials can be accurately characterized, and correlations can be drawn between changes in ordering at the molecular level and bulk physical properties, as has been carried out, for example, with archaeological Chinese silks.⁴ The technique can potentially be applied to any organic material which exhibits long-range ordering, and thus has applications in a wide range of fields.

References

1. P. Garside & P. Wyeth; *Studies in Conservation*; 51(3); 205; 2006.
2. P. Garside & P. Wyeth; *Applied Spectroscopy*; 61(5); 523; 2007.
3. P. Garside, S. Lahlil & P. Wyeth; *Applied Spectroscopy*; 59(10); 1242; 2005.
4. S. Greiff, et al.; *Scientific Analysis of Ancient and Historic Textiles* (R. Janaway & P. Wyeth, eds); 38; Archetype Publications; 2005.