

INFRARED IMAGING OF COTTON FIBERS USING A FOCAL-PLANE ARRAY DETECTOR

Michael Santiago Cintrón

Chanel Fortier

Doug J. Hinchliffe

James E. Rodgers

USDA-ARS-SRRC

New Orleans, LA

Abstract

Vibrational spectroscopy studies can be used to examine the quality and structure of cotton fibers. An emerging area of research relates to the imaging of cotton fibers. Herein, we report the use of a Fourier-transform infrared (FTIR) microscope to image developing cotton fibers. Studies were performed with an infrared microscope equipped with a detector with high spatial resolution, a Focal-Plane Array (FPA) detector. The experimental setup allowed for the examination of thousands of sample points simultaneously. Our preliminary results suggest that FTIR microscopy can be utilized as a tool to estimate important cotton fiber properties.

Introduction

Commercial advances in microspectroscopy have resulted in the availability of FTIR Microscopes with high spatial resolution. These high-resolution systems often contain a FPA detector that performs differently than the classic mercury-cadmium telluride (MCT) detectors (Lewis et al., 1995). A marked performance difference is observed, for example, when creating chemical maps of large sample areas. While FPA detectors allow for the simultaneous examination of thousands of sample points, systems with conventional MCT detectors require point-by-point examination. Thus, microspectroscopy systems equipped with a FPA detector could provide fast examinations of cotton fiber samples. This capability allows for the analysis of sample areas in a reduced amount of time and with high spatial resolution. Previous studies have shown that mid-infrared (Mid-IR) can be used to estimate important fiber properties and to monitor cell wall development (Abidi N et al., 2010; Liu Y et al., 2011). Hence the combination of spatial resolution of microspectroscopy combined with the analytical insight of vibrational spectroscopy could provide information on the chemical makeup and physical properties of cotton fibers. For this initial study, FTIR multi-point analysis of developing cotton fibers was performed. In addition, principal component analysis (PCA) scatter plots and high resolution chemical distribution maps are presented.

Material and Methods

The cotton fiber samples used were grown in 2009. The development of the line used, MD 90ne, and the cultivation of the fibers was previously described (Hinchliffe DJ et al., 2010). Samples were harvested between 18 and 40 days after flowering (DPA). Fully mature samples were also collected. Cotton fibers were examined with a Hyperion 3000 FTIR microscope (Bruker Optics, Billerica, MA) equipped with a focal-plane array (FPA) detector and video camera. The Hyperion system was connected to a Vertex 70 (Bruker) outfitted with a Mid-IR source. Fiber samples from each developmental stage were individually mounted on a metal plate with a small opening. FTIR microscope data was collected in the transmission mode. Scans were measured with a resolution of 8 cm^{-1} ($3800 - 900\text{ cm}^{-1}$), and the resulting spectra were corrected against an air background. Band assignments were taken from the Marechal and Chanzy FTIR study on cellulose I_{β} crystalline samples (2000).



Figure 1. Bruker Vertex 70 (left) and Bruker Hyperion 3000 spectrometer (right).

Results and Discussion

Multi-point FTIR microscopy

An FT-IR microscope equipped with a FPA mid-IR detector was used to examine multiple points of a fiber sample. Figure 2 shows the optical image of two cotton fibers (left) along with IR spectra extracted from various points along the fiber sample (right). All spectra were collected simultaneously, as such, the motorized stage where the samples are placed did not move during the examination. Automated spectra acquisition and FT transformation was performed by the OPUS interface program in less than 8 minutes for standard experimental conditions (32-64 scans, 8 cm^{-1} resolution). While only 7 points (spectra) are presented in Figure 2, the examination explored a total of 16,384 points in the area presented in Figure 2 (left). A similar effort with an FTIR microscope equipped with a MCT detector would require a significantly longer acquisition time since the motorized sample stage would have to move in the process of examining each single point. Major absorption bands in the extracted cotton fiber spectra resembled those acquired with a conventional FTIR spectrometer (Abidi N et al., 2010; Liu Y et al., 2011). However, some bands appeared deformed and with reduced intensity. For improved resolution cotton fibers were flattened with a roller pen.

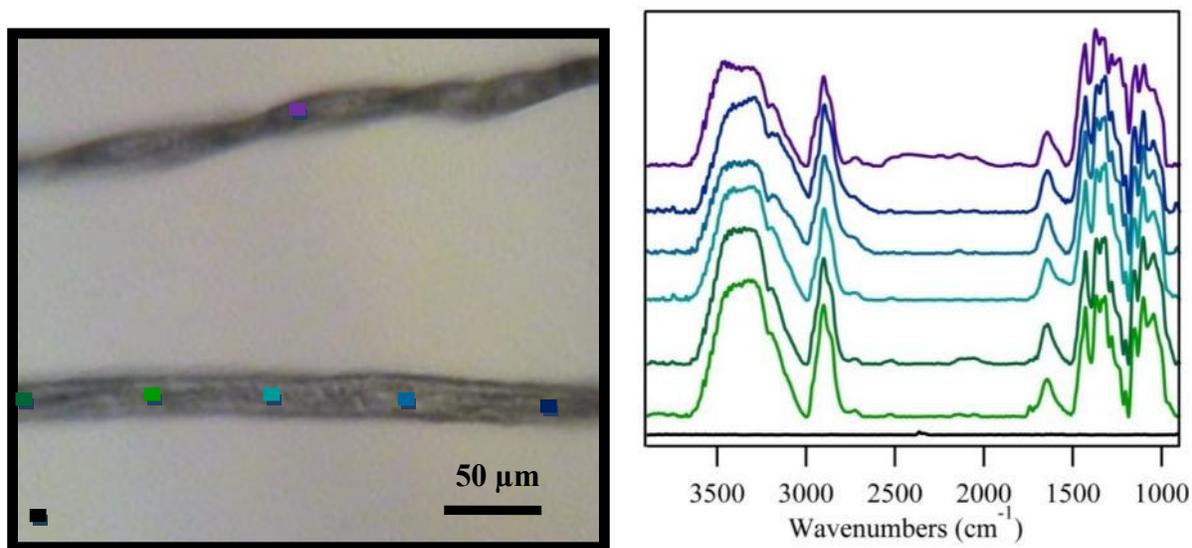


Figure 2. Multiple-point examination of mature cotton fibers with an FTIR microscope equipped with a FPA Mid-IR detector. Video image (20x objective) of two mature MD90 cotton fibers (left) and FTIR spectra of various sampling points (right).

Principal component Analysis of FTIR microscopy data

FTIR spectra of developing fibers were collected (not shown) and subjected to a two-component principal component analysis (PCA), Figure 3. Good separation of the spectra is observed, with spectra from each

developmental time point separating into a specific grouping (18, 24, 32 and 40 DPA). Most of this separation is observed along the principal component 1 axis (PC1; left to right), which suggests that this principal component is greatly influenced by the developmental progress of the samples. The PCA plot suggests that FTIR microspectroscopy spectra can be used to differentiate between developmental time points of cotton fibers.

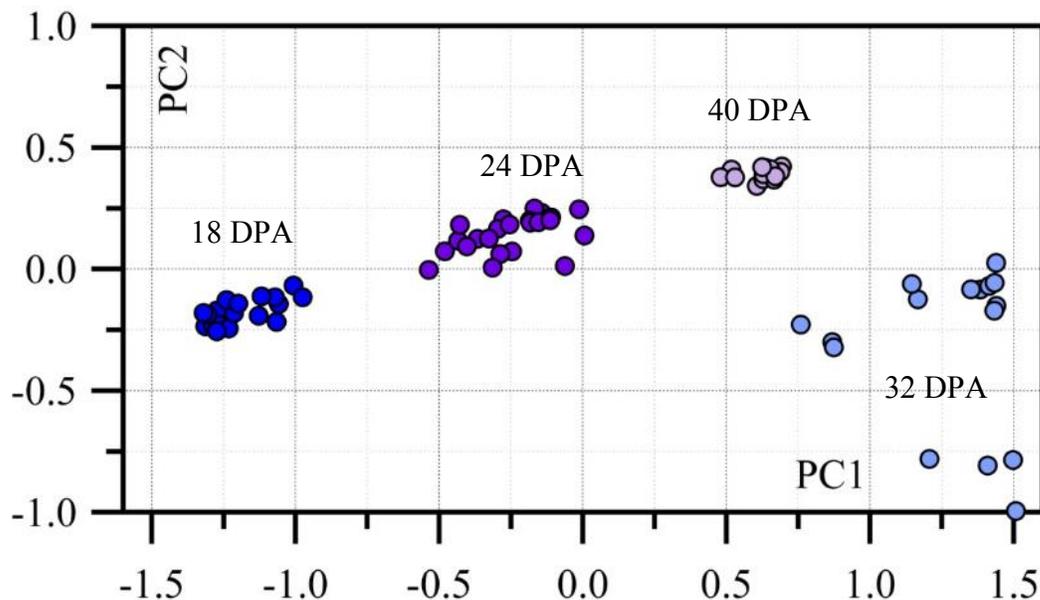


Figure 3. PCA scatter plots of FTIR spectra for cotton fibers harvested at the indicated developmental time points.

Chemical imaging using FTIR chemical distribution maps

Chemical distribution maps of selected spectral regions were calculated (Figure 4). These distributions map represent a form of chemical imaging. Figure 4 depicts an optical image of an immature cotton fiber (18 DPA) (left) and the distribution maps for two C-O peaks; a prominent C-O bending peak near 1054 cm^{-1} (ii), and a shoulder band near 1016 cm^{-1} . While the prominent peak is evenly intense throughout the fiber (as indicated by the red tone), the shoulder band is only moderately intense (yellow, soft red) in a few points along the fiber. While basic, similar distribution maps could be prepared to monitor cotton fiber development.

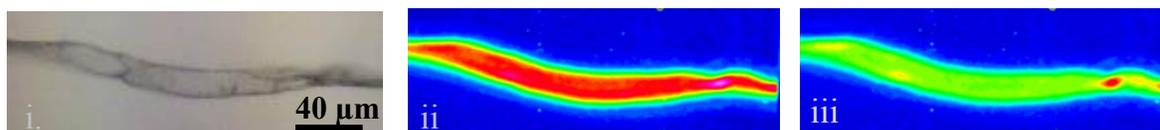


Figure 4. Chemical distribution maps for cotton fibers as determined with a FTIR microscope; developmental points shown are for a 18 DPA FIBER. The figure includes an optical image (i), while intensity maps correspond to the integration of a primary (ii) and a less prominent (iii) C-O peak ($\sim 1053\text{ cm}^{-1}$ and $\sim 1016\text{ cm}^{-1}$, respectively).

Summary

FTIR microspectroscopy along with PCA analysis can be used to assess general infrared spectral changes of developing cotton fibers. Measurements are fast and require little sample preparation. PCA scatter plots of FTIR spectra allows for the visual distinction of some of the developmental time points. Also, microspectroscopy with the FPA detector also allows for the chemical imaging of cotton fibers. Our results suggest that FTIR microspectroscopy with a FPA detector could be used to examine cotton fiber properties and cell wall development.

Disclaimer

The use of a company or product name is solely for the purpose of providing specific information and does not imply approval or recommendation by the United States Department of Agriculture to the exclusion of others.

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