INFRARED IMAGING OF COTTON FIBER BUNDLES

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Abstract

The infrared imaging of cotton fibers bundles is presented. Chemical images of cotton bundles were obtained with an infrared (IR) attenuated reflectance unit (ATR) and a macro sampling chamber equipped with a Focal-Plane Array (FPA) detector. Vibrational spectra and chemical images were acquired by grouping data points in the FPA. Grouped data presented reduced spectral noise and enhanced chemical images. The established method was used to examine cell wall development in cotton bundles. Imaging studies were quick and provided a distribution of cotton bundle composition and cell wall development. Future studies will seek to measure cotton fiber properties using this infrared imaging method.

Introduction

Infrared spectroscopy has been previously used for cotton fiber quality determinations (Liu 2013). Recent studies that rely on IR reflection accessories (e.g., ATR) have shown the potential for determining cotton fiber maturity, crystallinity and degree of cell wall development (Abidi N et al., 2010; Liu Y et al., 2011; Liu Y et al., 2014). ATR-based techniques are of particular interest since they allow for quick, reproducible examinations that require small amount of fiber samples. There is additional interest in developing imaging techniques that examine cotton fiber properties. Chemical imaging, the process in which visual images that represent the spatial composition of a sample, has been recently used for the identification of fiber trash contaminants and for monitoring cell wall development (Mustafic A et al., 2016; Santiago Cintrón M et al., 2016). Still, progress on IR imaging of cotton fiber bundles has been limited. This study presents the first use of infrared imaging of cotton fiber bundles. In order to quickly access cell wall development on fiber bundle samples, an infrared micro sampling chamber equipped with a FPA detector was used. FPA detectors allow for the simultaneous examination of thousands of points along a sample. Chemical images were acquired by the grouping of multiple pixels in the detector array. The ATR accessory was employed to examine cotton fibers undergoing cell wall development, and to visualize the distribution of this developmental process in a cotton fiber bundle.

Material and Methods

Cotton fiber samples were grown in 2015 in Stoneville, MS. Cotton fibers bundles were examined with a IMAC macro sampling chamber (Bruker Optics, Billerica, MA) equipped with a focal-plane array (FPA) detector and FastIR single reflection ATR unit (Harrick Scientific Products, Pleasantville, NY, USA). The IMAC system was connected to a Vertex 70 (Bruker) outfitted with a Mid-IR source and a DTGS detector (Figure 1). Fiber bundles samples were placed atop the ATR crystal and secured with a metal clamp in a manner that assured consistent pressure for all samples. FTIR microscope FPA data was collected in the reflection mode by acquiring 128 scans at a resolution of 8 cm^{-1}. Spectra were corrected for atmospheric gases and for their baseline using the OPUS spectroscopy software (Bruker; version 6.5). Chemical images were calculated using the 3D function of OPUS. Spectral bands were compared to cotton and contamination standards. Cotton assignments were taken from the Marechal and Chanzy FTIR study on cellulose (2000).

Figure 1. Bruker IMAC Sampling chamber equipped with FPA detector (right).

Results and Discussion

Cotton Fiber Examination and Data Grouping

A single reflectance ATR unit was used along with a Focal Plane Array detector to create chemical images of cotton fiber bundles. While early work resulted in chemical images and spectra with high spectral noise, grouping of data into discrete area groups provided spectra with refined features. Figure 2 shows the progression of data grouping for a cotton fiber bundle. All spectra show C-O bands typically associated with cotton and cellulose-based products. However, the lower the groping order, the higher the level of distortion is observed in other cotton IR bands. Data grouping is a simple procedure that reduces the number of pixels examined by the detector. While this process reduces the spatial resolution capability of the chemical image, a significant number of pixels are retained even at the highest levels of data grouping.

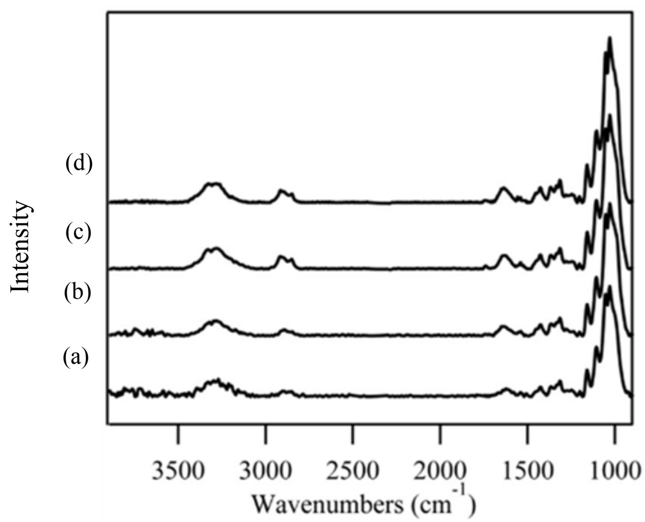

Figure 2. FTIR spectra of a cotton fiber bundle as determined with a single reflection ATR accessory and a FPA Mid-IR detector. Spectra were produced by grouping data into defined areas of (a) 2x2, (b) 4x4, (c) 8x8, and (d) 16x16 pixels. Spectra are the average of five sampling points, and are shown shifted along the y–axis (normalized absorbance).

Cotton Fiber Bundle Imaging

A typical set of cotton bundles chemical images with various levels of data grouping are shown in Figure 3. These chemical images illustrate the integration of the cotton C-O bands (1185-950 cm^{-1}) in each pixel set without normalization. Unfortunately, the chemical image with the 2X2 data grouping shows great variability and noise near the sampling area corners. As the data groups grew larger, the quality of the spectral data improved. Further grouping of pixels into 8x8, and 16x16 data sets improved the appearance of the chemical images (Figure 3c & d) and their uniformity. Few areas in both images result in areas with significant noise or artifacts. These chemical images provide spectral examination of 64 distinct points that were simultaneously examined, in contrast, a common benchtop FTIR unit with an ATR attachment can only examine one sample area at a time.

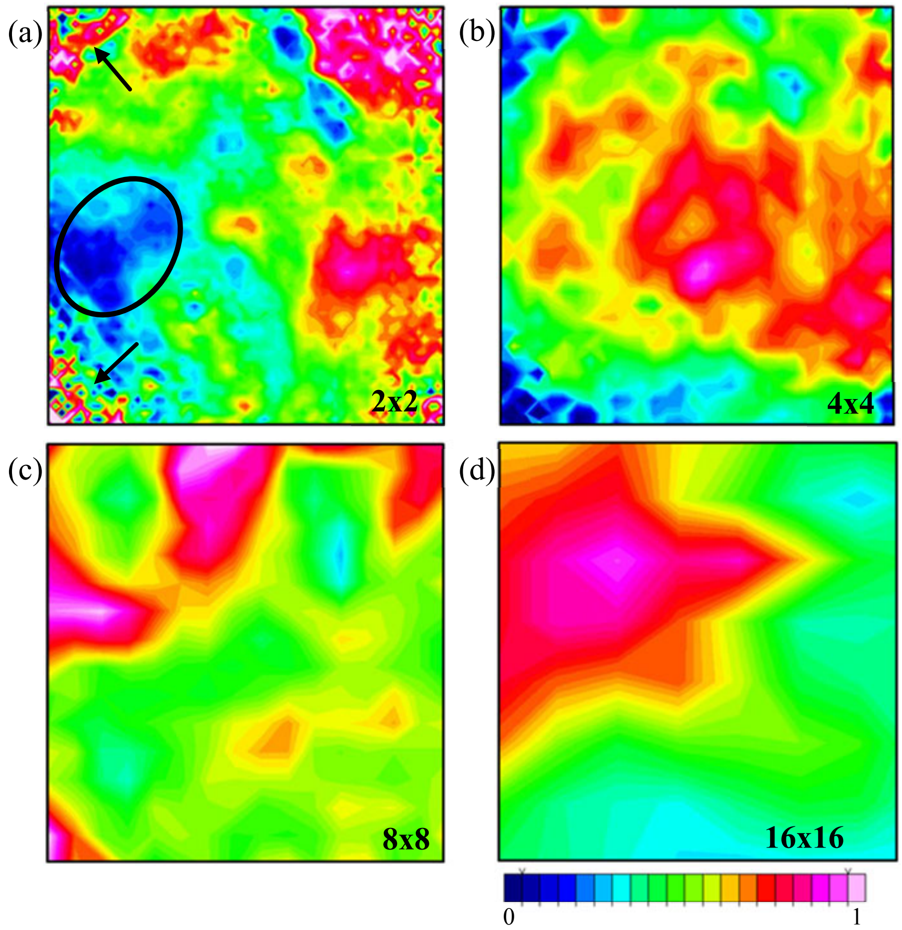

Figure 3. Chemical images for cotton fiber bundles as determined with a FTIR instrument equipped with a single reflection ATR accessory and a FPA Mid-IR detector. Images show data grouped into (a) 2x2, (b) 4x4, (c) 8x8, and (d) 16x16 pixels. Map tones reflect the integration intensity of the C-O bending region (1185-950cm^{-1}).

Cell Wall Development

The protocols established for cotton fiber bundle examination were used to examine cotton varieties at 4 different developmental time points, 18, 20, 22 days post anthesis (DPA) and mature (fully grown, 60+ DPA) fiber. These studies were undertaken to explore the potential use of this method for fiber maturity determinations. These time points were selected since they coincide with significant cell wall development. Two cotton varieties examined are shown in Figure 4. All varieties show a significant change in the relative intensity of one of the C-O bands, the 986 cm^{-1} peak. Previous studies have used changes in this peak intensity to monitor cell wall development and to quantify fiber physical properties (Liu Y et al., 2011).

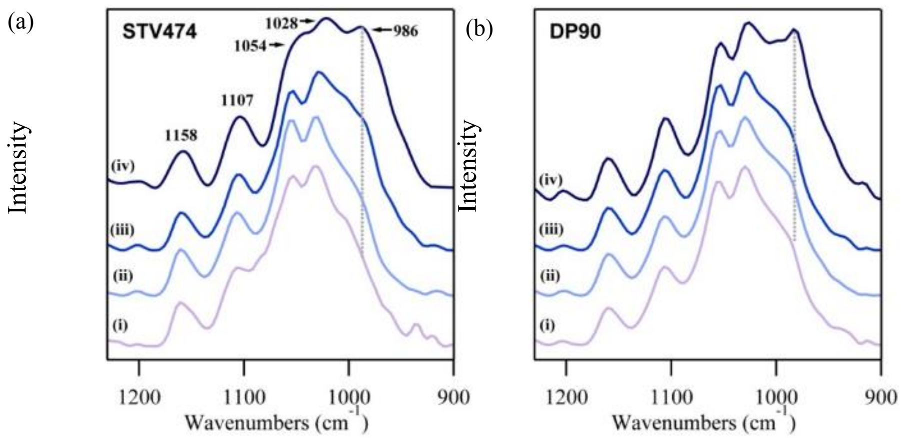

Figure 4. FTIR spectra of the C-O stretching region for cotton fibers harvested at different developmental time points. Spectra were collected utilizing a single reflection ATR attachment and a FPA detector. Two varieties were examined: (a) STV474 and (b) DP90. For each variety, 4 time points are shown: (i) 18 DPA, (ii) 20 DPA, (iii) 22 DPA, and (iv) mature fibers from open cotton bolls (+60 DPA). Indicated numbers are in wavenumbers.

Distribution of Cell Wall Development

The imaging protocol was used to image the chemical distribution in a cotton fiber bundle (Figure 5). Changes in the 986 cm^{-1} band were used as a marker for cell wall development. Spectral data were normalized to the dominant C-O peak at 1028 cm^{-1}, and the pixels integrated to the shoulder band centered at 986 cm^{-1}. Chemical images in Figure 5 serve to visualize the level of cell wall development in the examined fiber bundle. Figure 5a shows a chemical image of a 20 DPA cotton bundle (STV 474). The image shows mostly green tones that correspond to low intensity integrations of the C-O shoulder bands. However, the image is not homogenous in tone. Three additional tones are observed, an area with yellow (moderate high) integrations, and two small areas with red (high integrations) and blue (low integrations). Spectra taken from indicated points in the chemical image are shown in Figure 5b. The progression in intensity for the C-O shoulder band appears to mirror the cell wall development of portions of the bundle. Still, the vast majority of the chemical images correspond to minimally developed cotton fibers.

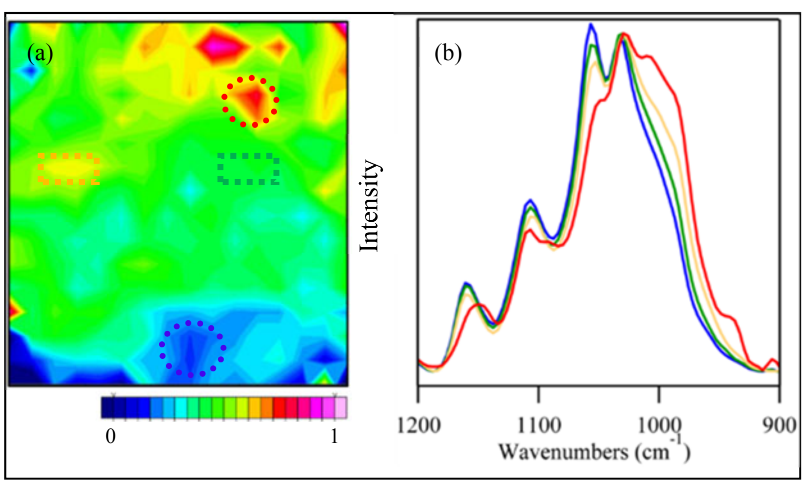

Figure 5. Chemical image for a cotton fiber bundle (20 DPA, STV 474), and FTIR spectra from the indicated sections of the map.

Summary

FTIR spectra and chemical images were acquired by the grouping of adjacent pixels in a focal plane detector array. The single reflectance FPA technique was employed to examine cotton fibers undergoing secondary cell wall development, and to visualize the distribution of this developmental process in a cotton fiber bundle. These examinations were quick (~10 minutes), required little in way of sample (~500 mg) and were not destructive to the cotton samples. While preliminary, our results point to the use of IR chemical imaging as a tool in the examination of cotton fiber bundles.

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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