EXAMINATION OF COTTON FIBERS AND COMMON CONTAMINANTS USING AN INFRARED MICROSCOPE AND A FOCAL-PLANE ARRAY DETECTOR

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Abstract

The chemical imaging of cotton fibers and common contaminants in fibers is presented. Chemical imaging was performed with an infrared microscope equipped with a Focal-Plane Array (FPA) detector. Infrared spectroscopy can provide us with information on the structure and quality of cotton fibers. In addition, small contaminants can be routinely identified given the spatial resolution provided by the array detector system. Examples of our ongoing efforts to use FTIR imaging for cotton fibers and common contaminants will be presented.

Introduction

Infrared spectroscopy has been previously used for the characterization of cotton fibers, synthetic textiles and common agricultural and foreign contaminants (Foulk JD et al., 2003; Allen A et al., 2007). More recently, FTIR spectrometers that rely on reflection accessories (e.g., Attenuated Total Reflection or ATR) have been use for these characterizations (Abidi N et al., 2010; Liu Y et al., 2011). ATR accessories allow for the quick examination of cotton samples while also requiring little sample preparation. FTIR spectrometers with ATR attachments, however, offer little in the means of spatial resolution of the examined samples. Advances in microspectroscopy have resulted in the availability of FTIR Microscopes that afford spatial resolution. Unfortunately, FTIR microscopes studies with standard detection systems can be time consuming, since spectra for the sampled points are acquired separately. More recently, high-resolution FTIR microscope systems have been commercially introduced. These systems often contain a FPA detector that performs differently than the classic mercury-cadmium telluride (MCT) detectors found in standard microspectrometers or the deuterated triglycine sulfate (DTGS) detector common in bench-top FTIR spectrometers (Lewis et al., 1995). FPA detectors are composed of arrays of pixels that simultaneously examine thousands of points along a sample. When combined with the high spatial resolution already afforded by the microscope, FTIR microscopes equipped with an FPA detector allow for the creation of chemical maps of large sample areas in a reduced amount of time. For this initial study, an FTIR multi-point analysis of cotton fibers and a common contaminant were performed. In addition, a high resolution chemical distribution map is presented.

Material and Methods

The cotton fiber samples used were grown in 2009 in New Orleans, LA. The contamination sample was sourced from fabrics and bundles submitted to the USDA-ARS-Southern Regional Research Center (New Orleans, LA) for examination. The contaminant was visually identified by observation with an optical microscope, and manually isolated with laboratory tweezers. Cotton fibers and contaminants 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 and a DTGS detector. Samples were individually placed either on an aluminum plate with an opening or atop a NaCl IR sample card (International Crystal Laboratories, Garfield, NJ). FTIR microscope FPA data was collected in the transmission mode by acquiring 64 scans at a resolution of 8 cm^{-1}. Spectra for each sample were corrected for atmospheric gases and for their baseline using the OPUS spectroscopy software (Bruker; version 6.5). Three dimensional data analysis and chemical images were developed using the 3D software package in 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 Vertex 70 (left) and Bruker Hyperion 3000 miscrospectrometer (right).

Results and Discussion

Cotton Fiber Examination

Cotton fibers where examined with an FTIR microscope equipped with a FPA. Multiple points along two cotton fibers were examined simultaneously. Figure 2 shows the optical image of two cotton fibers (left) along with Mid-IR spectra extracted from various points along the fiber sample (right). The examination of the sample took less than 8 minutes.

While only a few points are presented in Figure 2, the examination explored a total of 16, 384 points (128 x 128 pixel detector). A similar examination with a standard FTIR microscope and detector would require a significantly longer acquisition time since the instrument 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 and ATR attachment (Abidi N et al., 2010; Liu Y et al., 2011) and those of cellulose, the major component of cotton. However, combination bands (1500 – 1000 cm^{-1}) appeared somewhat deformed and with reduced intensity. Improved resolution can be obtained by using a roller pen to flatten the samples.

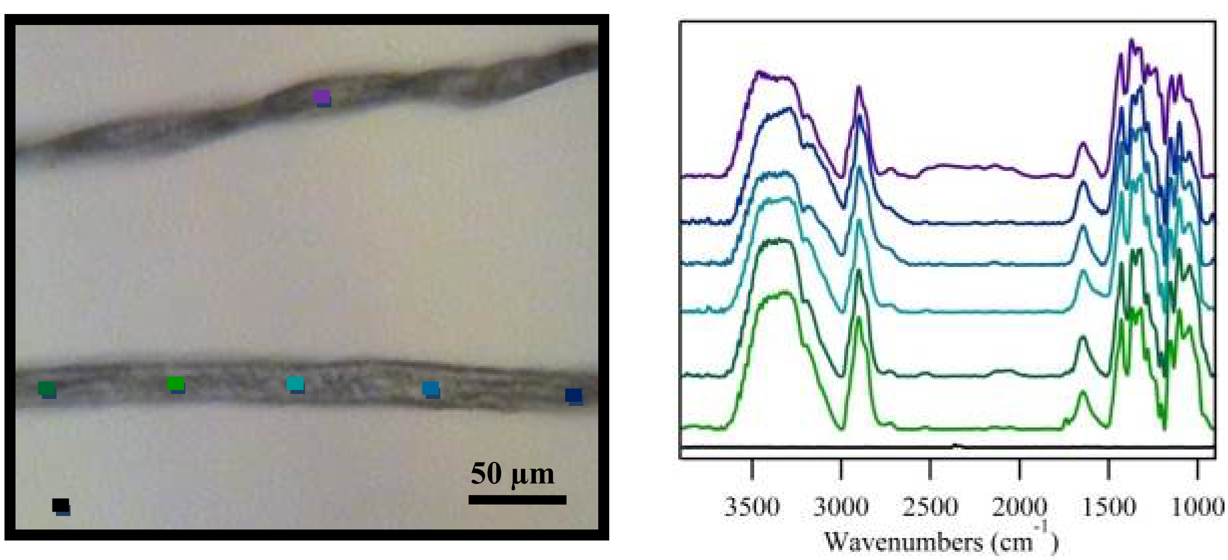

Figure 2. Multiple-point examination of mature cotton fibers with an FTIR microscope equipped with a FPA Mid-IR detector. Video image (20x objective) of two mature cotton fibers (left) and FTIR spectra of various sampling points (right).

Fiber Contaminant Identification

The FTIR microscope-FPA detector system was used for the identification of a common cotton contaminant. Since the detector provides highly sensitive and spatially resolved spectra, it follows that it could be used for the identification of small contaminants. Contamination sources in cotton products vary widely, but include agricultural sources from the field and industrial sources. In many cases, these contaminants are fairly small and difficult to examine with a standard FTIR spectrometer. For the proof-of concept study presented herein, a cotton fabric with a synthetic fiber contamination was examined with a conventional FTIR spectrometer and the FTIR microscope equipped with the FPA detector. The red contamination in the cotton fabric was isolated and examined. Spectra acquired with the conventional FTIR ATR system (Figure 3, top) showed a complex collection of bands, including OH stretching bands and other bands usually associated with synthetic fibers. Unfortunately, this result left unclear if the impurity was a synthetic copolymer with OH bands or if a small amount of the cotton fabric was sampled along with the impurity during the ATR experiment. The sample was then examined with the FTIR microscope. An average spectrum of the contamination is shown in Figure 3, bottom; the spectrum closely matches reference spectra of polyethylene terephthalate, a type of polyester. No OH Band is observed.

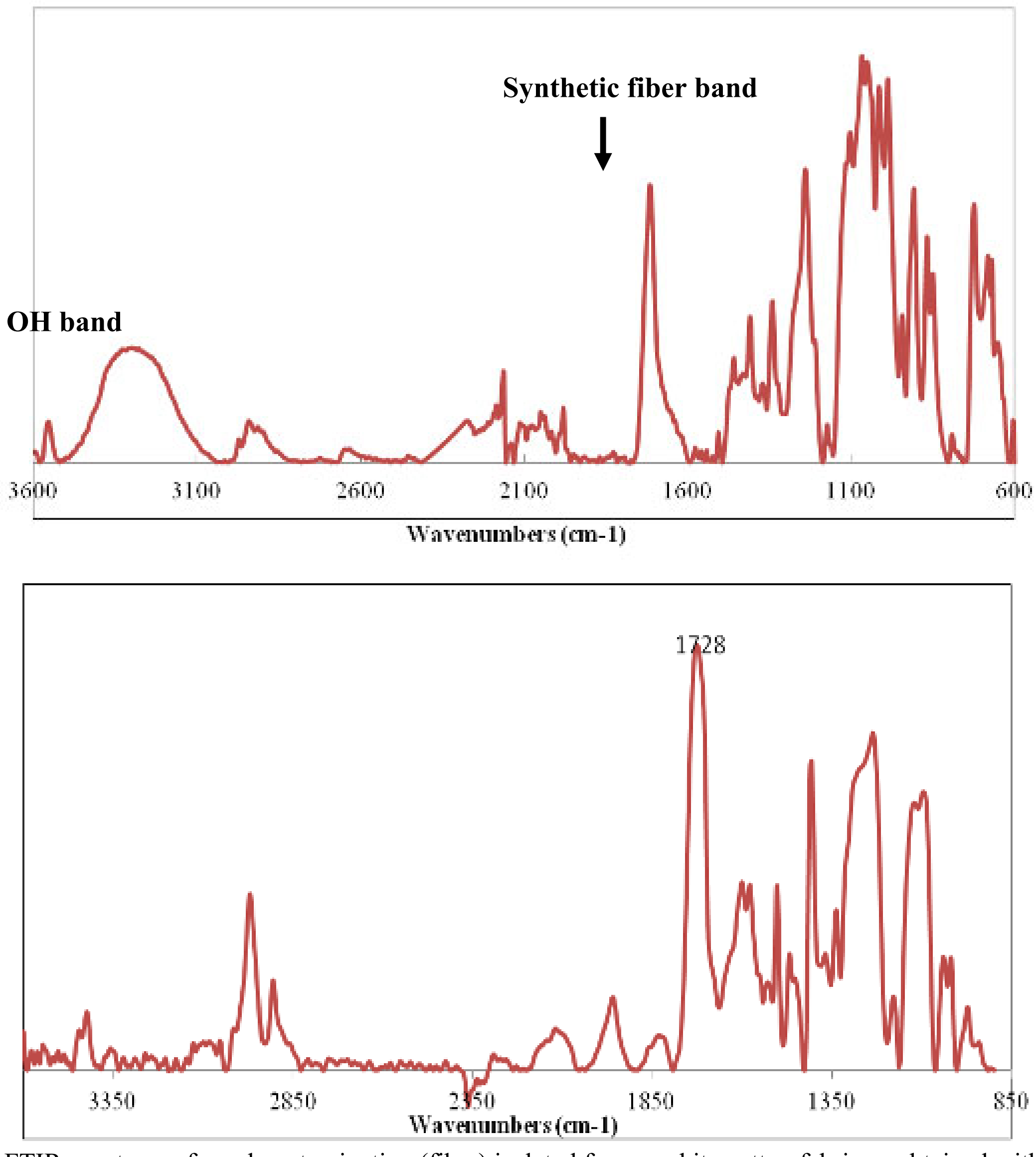

Figure 3. FTIR spectrum of a red contamination (fiber) isolated from a white cotton fabric as obtained with an ATR accessory (top), and FTIR spectrum of the same contamination as observed with FTIR microscope equipped with a FPA detector.

A chemical image of the isolated red fiber is shown in Figure 4. Chemical images serve to show the chemical composition of a sample. To obtain the image in Figure 4, all collected spectra were integrated to the 1728 cm^{-1} band associated with the polyester contaminant (Figure 3). Per the scale of the image, areas of moderate and high polyester presence will appear red or orange in the chemical image; areas with low or no presence of polyester will demonstrate a blue color. The red and yellow areas closely correspond to the areas where the red fiber was observed. This finding clarifies that the red contamination was not a copolymer.

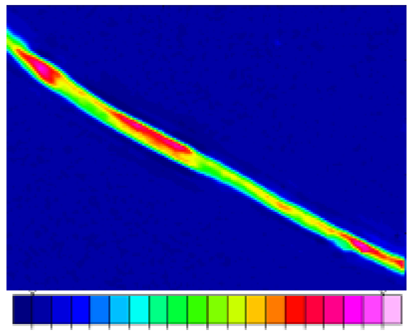

Figure 4. Chemical image of the isolated red contamination that has been integrated to the polyester peak at 1728 cm^{-1}. The red and yellow shadings represent areas with high integration or presence of the polyester, while blue shadings represents areas with low integration.

Summary

An FTIR microscope equipped with a FPA detector can be used to identify and image cotton fibers. Measurements are fast, usually taking less than 8 minutes, and require little sample preparation. Our results also suggest that FTIR microspectroscopy with a FPA detector could be used to examine cotton fiber and for the identification of common textile contaminants.

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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