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Fourier-Transform Imaging of Cotton and Botanical and Field Trash Mixtures

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Abstract: Botanical and field cotton trash comingled with Upland cotton lint can greatly reduce the marketability and quality of cotton. Trash found comingled with cotton lint during harvesting, ginning, and processing is of interest to the textile community. In the current study attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopic imaging was employed as an analytical technique to analyze cotton trash. Some benefits of this technique were its non-destructive nature and lack of required sample preparation. The technique used in this study, specifically ATR-FTIR spectroscopic chemical imaging, allows for three-dimensional spectral and spatial data to be obtained. In the current study, cotton in mixtures with botanical and field trash types have been identified spectrally and spatially using ATR-FTIR imaging. Botanical trash types (trash derived from the cotton plant) were evaluated and identified independently from cotton, even though both contained cellulose. The field trash types were easily identified from cotton due to their differences in chemical composition. This study can complement current cotton qualitative studies by adding spectral and spatial information to sample analysis.

Keywords: cotton; Fourier-transform imaging; hyperspectral imaging; botanical trash; field trash

1. Introduction

Cotton is a global commodity with many uses (apparel, mattresses, personal care products, etc.). Contamination or cotton trash can occur in the ginned cotton due to both botanical trash (from plant sources) and non-botanical sources (such as plastics), making the study of its contamination very important [1]. Knowing the trash types present with cotton could lead to improved ginning and processing to decrease the deleterious effects cotton trash can cause on cotton lint [2]. Conventionally, the High Volume Instrument (HVI®), Shirley Analyzer, and human "classers" are used to classify cotton trash [3]. However, there are some limitations with these methods. The HVI® method must be carried out in a climate-controlled environment, has a high cost, lacks the specificity of identifying trash types, and gives the percentage of trash content by image analysis. The human "classers" can lead to a lack of reproducibility between measurements from person to person. On the other hand, the Shirley Analyzer, a gravimetric technique, is unable to provide specific information on trash types. Additionally, the Shirley Analyzer cannot identity individual trash types. Cotton contaminated with different types of trash affects the overall quality of the cotton. For instance, it was reported that when cotton yarn was contaminated with as little as 1% bark, yarn breakage increased nearly 66% [4]. In a different study, an imaging technique was previously used to assess cotton trash removal from cotton fiber revealed seed coat removal to be more challenging than removal of leaf, neps, and wood fragments [5]. Frey and Schneider reported on updated equipment, such as bale openers, coarse cleaners, and blenders, that were used to remove small particulate cotton trash [6]. The effect of using each of these machines was reported, however, the card was touted as the best machine to tackle seed coat fragments. Thus, a need to identify and eventually remove cotton trash is desirable.

Cotton trash types have been analyzed using many techniques. Previously, neural networks and clustering analysis were employed to study cotton trash [7]. Although the neural network approach was accurate, it was shown to require a long analysis time. Siddaiah and co-workers developed a geometric approach to identify bark, leaf, and pepper trash with a size of 0.841-mm standard-diameter [8,9]. This approach was 95% accurate by applying an imaging and intelligent pattern recognition method. A machine vision system comparing a camera- and scanner-based imaging technique was developed [10]. Their cotton trash identification system (CTIS) was compared to the High Volume Instrument (HVI®), the Shirley Analyzer, and the advanced fiber information system (AFIS), all of which are conventional techniques. The CTIS system, calibrated using "classer" calls (human visual identification), yielded accurate identifications 97% of the time for botanical trash, such as bark, grass, stick, leaf, and pepper trash.

It has been demonstrated that the presence of trash with lint can affect processing efficiency. Out of the three spinning methods, which include ring, open-end rotor, and vortex, open-end rotor spinning efficiency has been found to be affected the most [11]. In a recent study, hull, shale, and seed coat fragments were identified using Fourier transform mid-infrared spectroscopy [12]. The presence of cotton trash in the rotor was observed to yield yarn breakage, thick places in the yarn, and yarn entanglement. Complicating this issue was the reduction in size of cotton trash as processing proceeded, making it nearly impossible to visibly identify the individual cotton trash types present. The results of this study revealed that hull and shale trash types were found in rotor grooves rather than seed coat fragments.

Recent studies began to focus on identifying trash using different spectroscopy techniques, such as near-infrared, ultraviolet, and ATR-FTIR spectroscopy. Fortier et al. identified botanical trash using near-infrared spectroscopy [3]. By applying a subcategory between the hull and seed coat, a database composed of botanical trash including hull, leaf, seed coat, and stem was designed. This approach yielded greater than 98% accurate identification of the trash types. Ultraviolet spectroscopy was also used to identify botanical trash which yielded 67% identification accuracy [13]. Near-infrared spectroscopy was also used to classify and distinguish field trash and botanical trash previously using a spectral database [14]. Himmelsbach et al. garnered the identification of botanical and field trash using ATR-FTIR spectroscopy [15]. This report demonstrated the feasibility of using ATR-FTIR spectroscopy as a technique to identify pure trash components which could be used to start a database. Therefore, spectroscopy, in general, is an effective technique to identify trash present in minute amounts of cotton with little to no sample preparation.

A new study classified cotton trash using shortwave infrared hyperspectral reflectance imaging [16]. This technique applied a liquid crystal tunable filter, as well as linear discriminant analysis for identification of cotton from cotton trash. A different study demonstrated the practicality of using ATR-FTIR imaging to identify pure cotton trash types [17]. In addition, Santiago Cintrón and co-workers demonstrated the utility of applying ATR-FTIR imaging to study secondary cell wall development in cotton fiber bundles using a focal plane array detector [18]. This report utilized a binning technique which enhanced the signal to noise ratio of spectra and chemical images. The aim of the current study is to determine the identity of botanical and field cotton trash mixtures in the presence of cotton using ATR-FTIR imaging.

2. Materials and Methods

2.1. Cotton Fiber and Trash Samples

Upland cotton (*Gossypium hirsutum*) fiber samples that had been previously ginned were used in this study. The subsequent meticulous removal of trash from the cotton was critical to ensure straight-forward interpretation of the results without the effects of trash originating from the cotton sample as a contaminant. The field trash types (black mulch, twine, and blue tarp) were obtained from the USDA-ARS-Southwestern Cotton Ginning Research Laboratory in New Mexico and were cut to fit over the ATR crystal. The botanical trash (leaf, hull, and stem) were obtained from the USDA-ARS in Clemson, SC.

2.2. ATR-FTIR Imaging and Binning

All ATR-FTIR images were collected on a FTIR macro-imaging system equipped with a focal plane array (FPA) detector (Bruker Optics, Billerica, MA, USA). Spectra were collected with an 8 cm^{-1} resolution, with 32 air background scans, and 32 sample scans. The diameter of the ATR crystal (Bruker Optics, Billerica, MA, USA). was 15 mm which limited the sample size of this study's components. The spectral range scanned was 900 cm^{-1} to 4000 cm^{-1}.The ATR crystal consisted of zinc selenide (ZnSe). The ATR accessory was centrally located in the imaging macro chamber (IMAC). To run the sample on the ATR crystal in the IMAC, the thin samples (a few millimeters) of cotton, and botanical and field trash types were mounted on the crystal, with the sample weighed down using a metal plate and tightened with a screw to ensure good contact with the sample and the screw. Sometimes the samples were arranged horizontally or vertically on the ATR crystal to optimize sample signal collection; no glue or glass slides were used. The images consisted of cotton and cotton trash in the same viewing area for all mixtures. The extracted spectra, or spectra derived from the three-dimensional chemical imaging cube, were obtained in areas of high intensity of reflection based on the color legend with pink being the highest intensity and blue being the lowest intensity of reflection. The analysis time for each sample was nearly 5 min. Higher scan times were investigated, but no increase in resolution was observed. In addition, the macro-imaging in this ATR-FTIR imaging study was chosen to analyze larger samples compared to microscopic imaging, which is more suited for a single, or few fibers.

Previously, this ATR-FTIR spectra and chemical image study yielded spectra and chemical images with poor sensitivity and large amounts of noise (data not shown). To enhance the signal to noise ratio, an averaging technique was applied to the data known as binning, as previously reported in the literature by Santiago Cintrón and co-workers [18]. Herein, the spectra were placed into pixel groups. At first, an 8 × 8 binning size was investigated (data not shown). Yet, there was still quite a small amount of noise observed. Thus, 16 × 16 pixel groups were analyzed. This experimental setup proved to be successful in this study. It is important to note that both the background and sample runs were performed using the binning technique. Other pre-processing techniques used in this study were baseline correction and subtraction of the CO_2 band on the extracted spectra.

3. Results

3.1. Cotton and Botanical and Field Trash Types

Figure 1 depicted the three types of botanical trash commonly found with cotton (A), leaf (B), hull (C), and stem (D). Similarly, three types of field trash were investigated with cotton, including black plastic mulch (E), blue tarp (F), and twine (G). Based on their spectral peak shape and physical characteristics, the botanical trash was more challenging to investigate, whereas the field trash types were more amenable to this investigation.

Figure 1. Depiction of different types of botanical and field trash materials present with (**A**) cotton. Contaminated cotton included botanical trash, such as (**B**) leaf, (**C**) hull, and (**D**) stem. Field trash can also be found with cotton, such as (**E**) mulch, (**F**) blue tarp, and (**G**) twine.

3.2. ATR-FTIR Spectra

ATR-FTIR imaging was chosen as the analytical tool of choice since multiple spectra could be acquired simultaneously, as well as the ability to yield spectral and spatial information compared to a single spectrum using ATR-FTIR spectroscopy. In addition, the macro-imaging in this ATR-FTIR imaging study was chosen to analyze larger samples compared to microscopic imaging, which is more suited for a single or few fibers. As mentioned before, a binning technique was applied to the spectral and chemical data to enhance the signal to noise ratio. Figure 2 shows the effect of applying the binning technique to an Upland cotton sample. When the FT-IR spectra were analyzed, it was clear that the decreasing effect of noise was apparent. In addition, Table 1 shows the signal to noise ratio calculated using the Bruker OPUS software program. As the binning application increased from 1 $\times$ 1 up to 16 $\times$ 16, the spectral data depicted more distinct peaks and reduced noise.

Figure 3 showed the extracted spectra of cotton (A) and the studied botanical trash types. At first glance, the ATR-FTIR botanical trash spectra were very similar to the cotton spectra, making their identity challenging. Cotton and botanical trash had a broad peak at around 3300 cm^{-1} due to the O–H stretch. At 2900–2800 cm^{-1} the C–H stretch was present in both botanical trash spectral bands, as well as the cotton spectrum [15]. In the fingerprint region, including the CO and CH stretching region, between 1100–900 cm^{-1}, all spectra had the C–O bands and at 900 cm^{-1} a beta-linkage appears between two glucose units due to cellulose synthesis [19]. Cotton did not have a strong band at 1800–1500 cm^{-1} signifying the C=O stretch, whereas the trash spectra all have this fine structure [15]. Among each other, the botanical trash types were distinguished by the fingerprint region having different band structures. Leaf, Figure 3B, had a distinctive CO band at 1750 cm^{-1} and a CH doublet between 1500–1250 cm^{-1} [19]. In contrast, the hull, (Figure 3C), and stem, (Figure 3D), had a doublet in the same fingerprint CH region (1500–1300 cm^{-1}) with differences in intensities for these trash types, but hull had a C=O doublet at 1700 cm^{-1} [19], whereas the stem, (Figure 3D) has a singlet peak in that region. The presence of carbonyl compounds in a larger amount with the leaf compared to hull and stem was suggested.

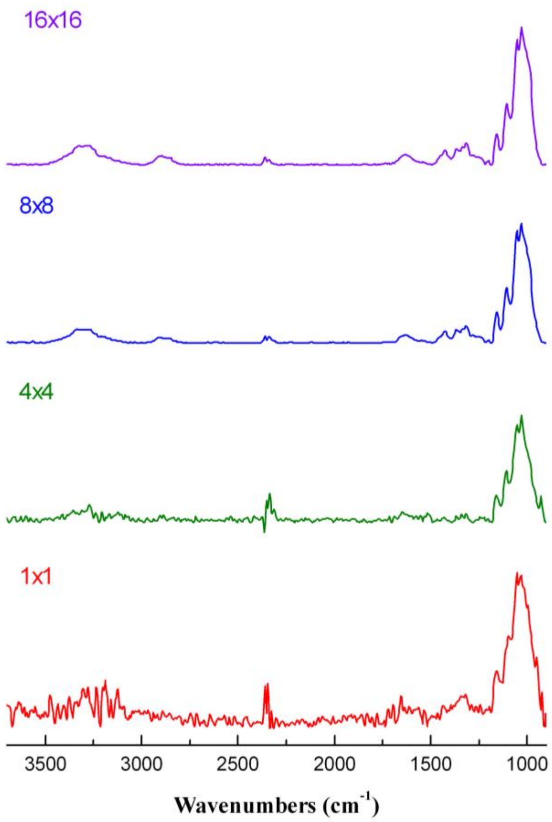

Figure 2. Effects of pixel binning on FTIR signal to noise ratio for an Upland cotton control sample at four levels of binning: 1 × 1, 4 × 4, 8 × 8, and 16 × 16.

Table 1. Effect of pixel binning on signal to noise ratio (S/N) of cotton fiber on FTIR spectra.

Binning (pixels)	Signal to Noise Ratio (S/N)
1 × 1	0.97
4 × 4	1.041
8 × 8	1.069
16 × 16	1.074

Figure 4 showed the extracted spectra of cotton and the studied field trash types. Clearly, the field trash can be distinguished from cotton based on their chemical makeup. The plastic polymer trash types had bands that did not overlap with cotton, as observed with the botanical trash types. In Figure 4A, the cotton spectrum, as described previously, had a broad band around 3300 cm^{-1} stemming from the O–H stretch. The band at 2900–2800 cm^{-1} represented the C–H stretch. The bands from 1500–900 cm^{-1} were overlapping C–H vibrations. The mulch represented by Figure 4B had a distinctive CH doublet around 2900–2800 cm^{-1}, characteristic of polyethylene. In Figure 4C, a similar spectrum was shown by blue tarp suggesting its chemical makeup was largely polyethylene. The singlet band observed in mulch and blue tarp just under 1500 cm^{-1} suggested the presence of an antisymmetric CH_3 deformation or scissoring [19]. In Figure 4D, the twine spectrum had a distinctive

CH_2 quadruplet band around 2900–2700 cm^{-1}. The doublet just below 1500 cm^{-1} also suggested CH_3 deformation. Based on this spectrum, the presence of polypropylene in twine was inferred.

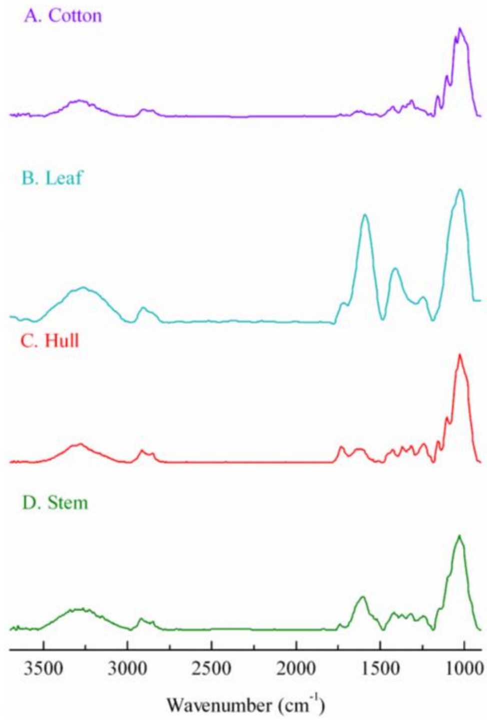

Figure 3. FTIR extracted spectra of different types of botanical trash materials present with cotton. The images displayed are (**A**) cotton, (**B**) leaf, (**C**) hull, and (**D**) stem, respectively.

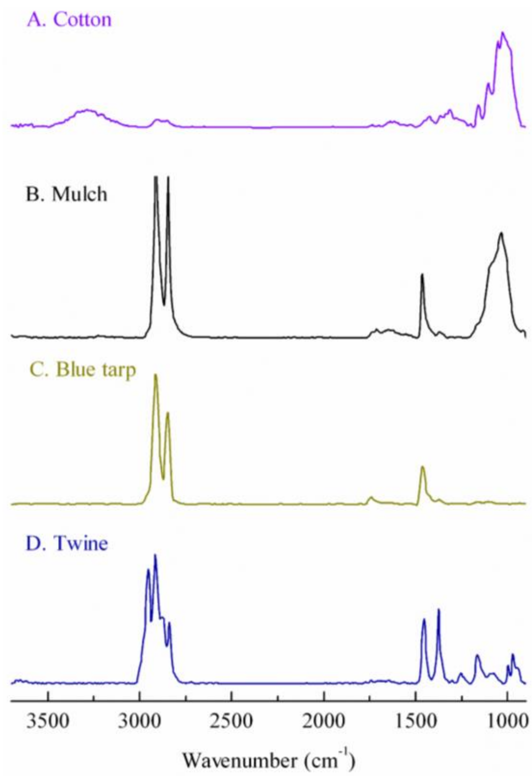

Figure 4. FTIR extracted spectra of different types of field trash materials commonly present with cotton. The images display (**A**) cotton, (**B**) mulch, (**C**) blue tarp, and (**D**) twine.

3.3. ATR-FTIR Chemical Images

The FTIR chemical images were initially collected for each binary mixture while integrating over a specific range. From each chemical image, an ATR-FTIR spectrum was extracted at a specific spatial position which represented the variation in the intensity of reflected light. Figure 5 displayed the FTIR imaging of different types of botanical trash materials present with cotton over integrated regions. Figure 5A,B shows the chemical images of cotton and leaf botanical trash mixtures. In Figure 5A, the chemical image of the cotton was integrated over the 1765–1480 cm^{-1} fingerprint region, which includes the COC spectral bands [15]. The color intensity for the cotton representation in the image is low. The relationship between the pseudo-colors of the chemical images and the samples being analyzed was that the pink color signified high spectral integration intensity and the blue color signified low spectral integration intensity. In Figure 5A, the cotton shown by bright colors suggested a strong reflectivity of cotton, which was not expected compared to leaf. In Figure 5B, integrating over 2968–2808 cm^{-1} represented the CH region. The leaf had colors which were less intense.

It was possible that a small amount of trash that was missed by visual inspection was picked up by the detector, since cotton was expected to have a lower reflectivity than trash. Or, it could be that the integration bands chosen for cotton were present in a higher intensity compared to the integration range chosen for the leaf trash. There were some overlapping features of the images for Figure 5A,B, but clearly the intensity properties were distinctive for the two samples studied. Figure 5C represented the cotton and hull mixture highlighting the cotton region. The integrated region for cotton was 3519–3098 cm^{-1} which was characteristic of the OH spectral band. The hull mixture, Figure 5D was integrated over the spectral region of 1847–1541 cm^{-1}, representing the CO functional group. Again, cotton was largely represented by areas of high and low intensity colors, whereas the hull in Figure 5D was also represented by pink and blue colors. Yet, for the hull, in Figure 5D, the color distribution were more spread throughout the image; signified by the light blue color as opposed to the dark blue colors in Figure 5C. Figure 5E shows the chemical image for the cotton and stem mixture highlighting cotton.

The integration region was 1187–935 cm^{-1}, representing overlapping CH vibrations. This region was a signature spectral range for cotton, as stated above in similar regions. When comparing the cotton image in Figure 5E to that of the stem image, Figure 5F cotton had less intense colors and the stem had strong intensity, signified by the light pink color in Figure 5F. The chemical image for stem was highly reflective based on the color pattern shown. The integrated region for stem consisted of 1472–1179 cm^{-1}, representing CH bands. The spectral bands covered in this study include the fingerprint region, the C–H region at 2800–2900 cm^{-1}, the overlapping CH regions from 900–1500 cm^{-1}, and the O–H region at 3300 cm^{-1}. Overall, the similar spectral regions were picked to highlight the cotton and botanical trash differences. This was done since the botanical trash had spectral similarities to cotton based on their cellulosic content. Clearly the intensity properties were distinctive for the two samples studied, with the stem being much more reflective than cotton.

Figure 6 depicts the FTIR imaging of different types of field trash materials present with cotton over the integrated regions. In Figure 6A,B, cotton and black plastic mulch make up the mixture. At first glance, the two images look very similar. Yet, the lighter pink in Figure 5B was more pronounced than the dark pink distribution for cotton in Figure 6A, as expected. Again, these two chemical images represent the same sample mixture, but they highlight different chemical components. In Figure 6A, the cotton in the mixture is highlighted in the mixture and was integrated over 3546–3002 cm^{-1}. This spectral region suggests the presence of the OH band. In Figure 6B, mulch is highlighted and the image was integrated over 2985–2739 cm^{-1}. When analyzing the image representing mulch, the doublet at 2900–2800 cm^{-1} is characteristic of polyethylene and the C–H band being present. In Figure 6C,D, cotton and blue tarp make up the mixture. Figure 6C represents the cotton in the mixture integrating over 1172–919 cm^{-1} in the fingerprint region and over 2991–2793 cm^{-1}, highlighting the blue tarp towards the left and right in the image. The blue tarp had the characteristic polyethylene peaks specifically at 2900–2800 cm^{-1} representing the C–H band, respectively [18]. In Figure 6E,F, twine and cotton were the mixture. In Figure 6E, cotton is strongly represented over the integration range

3521–1646 cm^{-1}. Figure 6F depicted the twine integrated over the region 3027–2796 cm^{-1}, representing the OH and CH bands. There appeared to be some similarity in Figure 6E,F, with the highly-intense portions directed toward the outside of the image. It should be noted that the twine physical properties made it challenging to analyze; thus, the lack of high intensity in more aspects of the Figure 6F may have been muted.

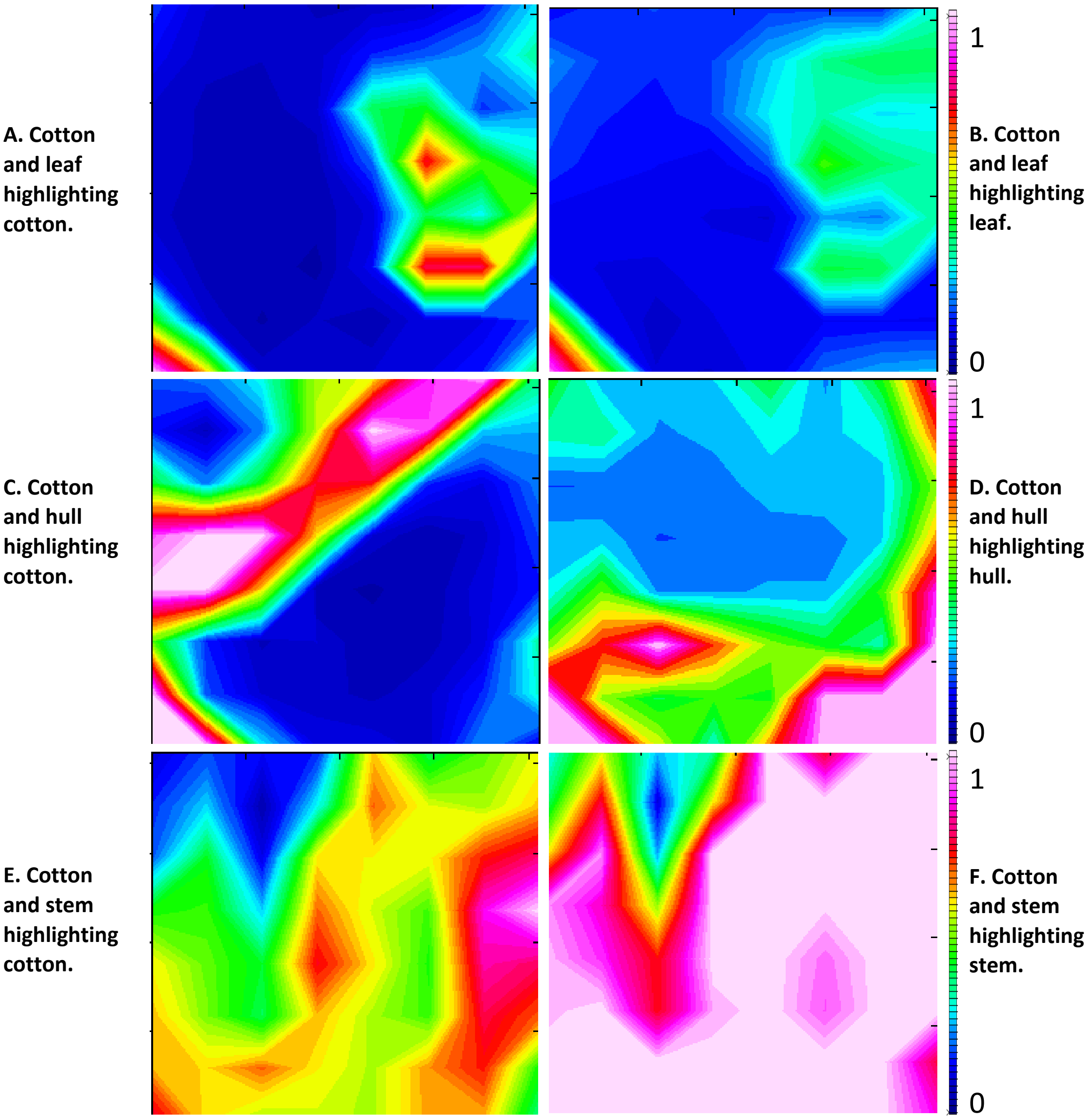

Figure 5. FTIR imaging of different types of botanical trash materials present with cotton over integrated regions. (**A**) The image shows cotton and leaf, highlighting cotton (1765–1480 cm^{-1}). (**B**) The image shows a blend of cotton and leaf, highlighting leaf (2968–2808 cm^{-1}). (**C**) The image depicts cotton (3519–3098 cm^{-1}) and hull, highlighting cotton, and (**D**) the image shows cotton and hull, highlighting hull (1847–1541 cm^{-1}), respectively. The images illustrate a blend of cotton and stem representing (**E**) cotton (1187–935 cm^{-1}) and (**F**) the image depicts cotton and stem, highlighting stem (1472–1179 cm^{-1}), respectively. Areas of high reflection in the images are represented with pink. Areas of low reflection in the images are represented with blue.

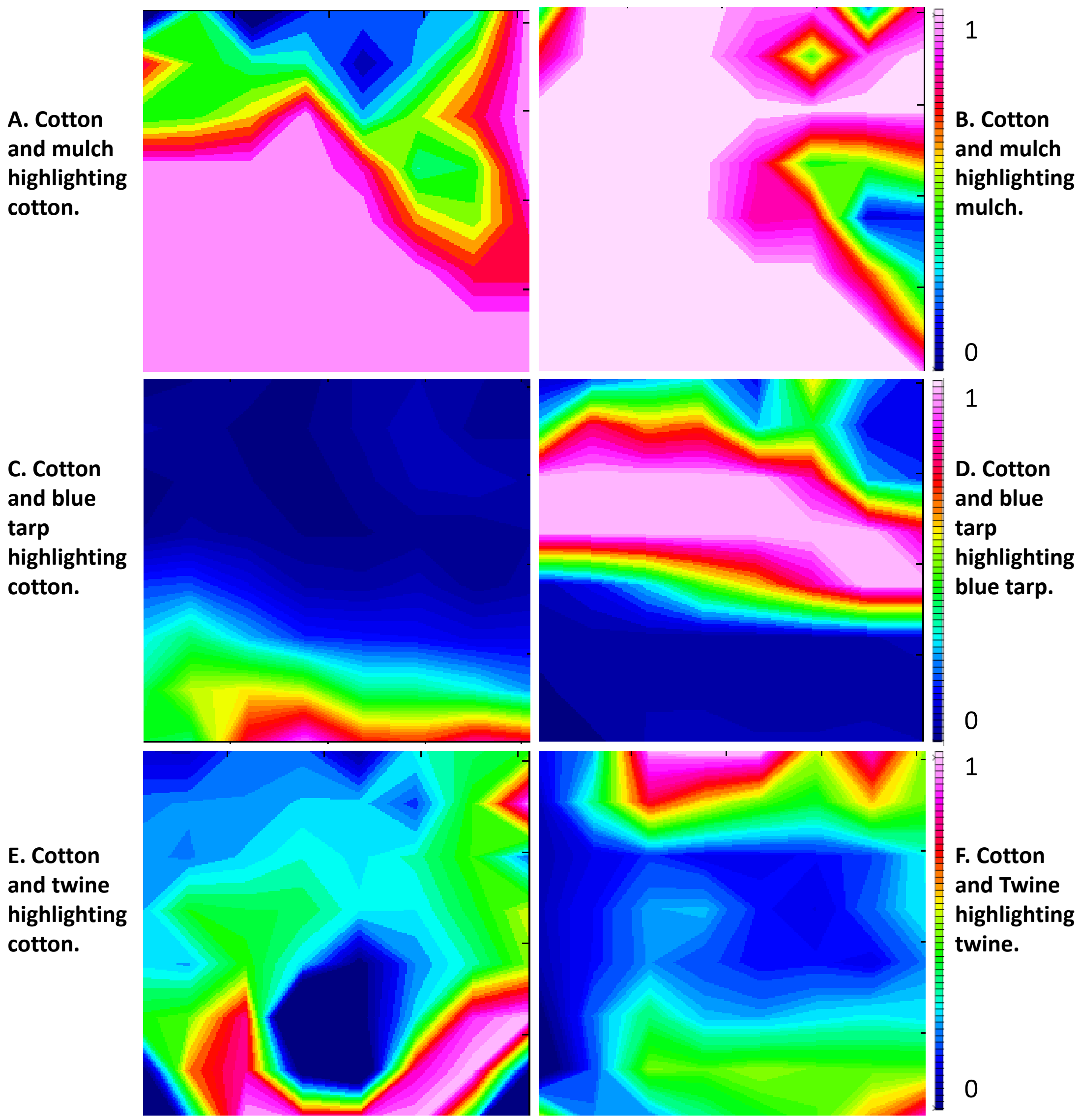

Figure 6. FTIR imaging of different types of field trash materials present with cotton over integrated regions. The images displayed a blend of cotton and mulch depicting (**A**) cotton (3546–3002 cm^{-1}) and (**B**) mulch (2985–2739 cm^{-1}), respectively. The images show a blend of cotton and blue tarp, highlighting (**C**) cotton (1172–919 cm^{-1}) and (**D**) blue tarp (2991–2793 cm^{-1}), respectively. The images illustrate a blend of cotton and twine representing (**E**) cotton (1646–3521 cm^{-1}) and (**F**) twine (3027–2796 cm^{-1}), respectively. Areas of high reflection in the images are represented with pink. Areas of low reflection in the images are represented with blue.

Along with the imaging software and computer, the focal plane array afforded the collection of numerous spectra simultaneously [20]. The choice of employing a focal plane array in this study gave an improvement over using an MCT detector, which measures point by point, and saved significant analysis time. The ATR-FTIR imaging technique was used to combine the averaging binning method to maximize the signal and minimize the spectral noise. Therefore, by integrating over a smaller range compared to the entire spectrum, sharp extracted spectra and images were acquired. The reflectivity of the samples was influenced by their sample makeup and surface properties. According to the chemical images, cotton did have some areas of high intensity, as signified by the legend color of pink, as well as areas of low intensity, signified by the dark blue color. This was unexpected based on earlier results using cotton balls which yielded quite low reflectivity (data not shown). The ATR crystal also greatly influenced the data acquired by limiting the sample area, allowing a shallow depth of IR penetration through the sample and requiring intimate contact of the sample and crystal by applying pressure with a screw. Some samples, such as the hull, stem, and twine, proved challenging to analyze based on their physical shape.

4. Conclusions

The aim of this study was to determine the feasibility of using ATR-FTIR imaging to identify cotton and cotton botanical and field trash in mixtures. The program demonstrated that this technique can, in fact, be used to reach this goal. The imaging of cotton and botanical trash came with challenges, since they were both largely made up of cellulose. However, cotton was easily deciphered from field trash due to the profound chemical makeup differences between them. The imaging of the cotton and trash can be complimentary to fiber analysis instruments (HVI, AFIS, and Shirley Analyzer) in the investigation of cotton fiber. This ATR-FTIR imaging technique offers a nondestructive, fast, and accurate method to analyze cotton and cotton trash. The current program provided the foundation needed by cotton textile manufactures, ginners, and farmers to identify and eventually sort cotton or other fibers from cotton trash. Specifically, this imaging technique afforded the user with the next level of classifying cotton trash. Future studies will include varying the composition of the sample by investigating the effect of different ratios of cotton to botanical and field trash types.

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Author Contributions: Michael Santiago Cintrón helped collect some of the data, was involved in the overall conception of the project, and wrote some aspects of the paper. James Rodgers was involved in conceiving the project and wrote some of the paper. Krystal Fontenot was involved in data analysis and presentation, and wrote some aspects of the paper. Donna Peralta was also involved in data analysis and wrote some aspects of the paper. The corresponding author, Chanel Fortier, collected and analyzed data and took the lead on writing the paper.

Conflicts of Interest: The authors declare no conflict of interest.

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