

TEXTILE TECHNOLOGY

Preliminary Fourier-Transform Infrared Spectroscopy Analysis of Cotton Trash

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ABSTRACT

Ongoing research efforts at the United States Department of Agriculture (USDA) consist of developing methods for the identification of cotton trash. Botanical cotton trash (eg. leaf, stem, hull, bark) has been reported to be problematic in the processing efficiency and quality of cotton throughout ginning and textile processing. Fourier transform infrared (FTIR) spectroscopic methods are being utilized to develop a database for the classification of different trash found in cotton. This manuscript reports the effects of cotton trash as it is subjected to physical modifications (size reduction and thermal treatment) that may occur during ginning and textile processing on the FTIR spectroscopic properties. Results indicate that both thermal treatment and variations in trash particle size significantly affect the resultant FTIR spectra, which necessitates an expansion of the database to include these effects.

An important practice in ginning and textile processing of cotton is the separation of foreign matter from cotton fiber. Once detached from seeds, cotton fibers continue through processing and often contain additional foreign matter, particularly botanical trash, which becomes entangled within the fibers. Botanical trash (leaves, hulls, shales, stems, seed coat fragments, etc.) is known to influence the processing efficiency and quality of cotton from ginning to the finished fabric (Anthony, 1990; Brashears et al., 1992; Foulk et al., 2003; Foulk et al. 2004; Chung et al., 2004). Several methods have been used to evaluate the presence of trash in cotton. Researchers and textile mills use various systems, such as the Shirley Analyzer, Advanced Fiber Information System (AFIS), and High Volume Instrument (HVI), for identifying the presence of trash by gravimetric or geometric means. For

example, the HVI trashmeter measures the amount of trash by scanning the cotton surface, discriminating large trash particles from the lint, and then reporting the percentage of trash based on the total measured surface area (USDA-AMS, 2001). More innovative methods are needed to identify and classify specific types of trash for the development of new techniques to remove specific types of trash at earlier stages of processing. A new development of the Cotton Trash and Cotton Measurement (CTCM) system has exhibited an enhanced ability to categorize raw trash using more advanced imaging devices than those associated with HVI technology (Xu et al., 1997; Xu and Watson, 1999). Even though other methods have been explored, they are unable to provide the information, which is needed to clearly identify trash types at various cleaning stages and to determine their influence on ginning and textile processing.

In previous studies, a Fourier transform infrared (FTIR) spectrometer equipped with an attenuated total reflectance (ATR) attachment was used to characterize the chemical composition of cotton trash (Foulk et al., 2003; Foulk et al., 2004). Infrared (IR) spectroscopy is an effective method for the identification of molecular functional groups, such as methyl (C-H), hydroxyl (O-H), and carbonyl (C=O) groups. The evaluation of botanical cotton trash samples in the mid-infrared (mid-IR) region of 4,000 to 650 cm^{-1} has provided a means for the development of a spectral database (currently under patent consideration) that distinguishes the type of cotton trash based on chemical composition (Himmelsbach et al., 2006). Botanical trash is mainly comprised of cellulose but also includes other compounds, such as phenolics and organic acids. Infrared spectroscopy has the potential to determine distinct chemical components associated with various types of trash. Similar signature peaks are observed in the spectra of leaf, stem, hull, and seed coat fragments (Table 1) (Silverstien and Webster, 1998). Prior studies utilized the relatively small spectral differences among assorted foreign matter found in cotton to develop a spectral database (Himmelsbach et al., 2006). These studies, however, were conducted on trash samples that had not experienced any physical or thermal

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modifications known to occur during the ginning and textile processing. Therefore, it is of interest to explore the capabilities of mid-IR spectroscopy in the evaluation of trash samples obtained from lint that may have been physically and thermally modified by ginning and textile processes.

Table 1. List of common vibrational modes and mode assignments corresponding to components of botanical cotton trash

ATR-FTIR (cm ⁻¹) ^z	Mode assignment
~3300	O-H stretch
~2900	C-H (1) asymmetric stretch
~2850	C-H (2) symmetric stretch
~1700	C=O stretch of an carboxylic acid & ester
~1600	C=O stretch of an acid salt

^z These peaks are adapted from Chung et al. (2004) and Silverstein and Webster (1998).

To expand on continuing efforts to utilize FT-IR for trash evaluation, it is vital to subject the trash samples to simulations associated with the ginning and textile processing in order to determine any distinguishable spectral differences that are not represented in the existing spectral database. Throughout processing, trash is continuously altered into different physical states (Barker et al., 1995; Knowlton, 1997). For that reason, the consideration of size reductions from large trash particles (raw) to small particle sizes (pepper) and even smaller particle sizes (powder) is essential in understanding how these changes influence the IR absorption of chemical components present in trash. Furthermore, understanding the effect of gin drying and heating on processed cotton may lead to expanding the overall importance of the spectral database. Previous studies have determined that drying raw cotton to specific moisture levels enhances the ability of the ginning process to remove problematic trash (Griffin, 1977; Garner and Baker, 1977). To minimize potential heat damage to cotton lint, recommended drying temperatures should not exceed 177 °C (351 °F) (Griffin, 1977); however, the trash retains moisture differently than the lint (Fouk et al., 2004). There is a possibility that these changes can bring about peak reductions or new peak appearances within the mid-IR spectral region. The objective of this report is to determine if any distinguishable mid-IR spectral differences can be detected among different types of modified trash compared with the unmodified trash types.

MATERIALS AND METHODS

Cotton trash. Delta and Pine Land (DPL) Company in Scott, MS, provided cotton (DPL 33) that was commercially ginned (without heat) by the Southwestern Cotton Ginning Research Laboratory in Las Cruces, NM. Collected trash samples were separated by hand into specific categories (stem, leaf, hull, and seed). In addition, seeds were delinted by soaking in 70% (w/w) sulfuric acid until fibers were removed from the seeds. Then the seeds were neutralized with 10% sodium bicarbonate, rinsed with water, and allowed to air dry. Following seed delinting, the seed meat was separated from the seed coat. After the samples were separated, a Wiley mill (Scientific Apparatus; Philadelphia, PA) was used to grind each trash sample into smaller particle sizes. Pepper and powder sizes were prepared using 20 (0.841-mm std. diam.) and 80 (0.177-mm std. diam.) meshes, respectively. For all the treatments, trash samples were ground ensuring three replicates of raw, pepper, and powder sizes. Heating was accomplished by using a high performance Precision Scientific 625S oven (Precision Scientific, Inc.; Winchester, VA) fitted with a digital temperature controller. Following size reductions, trash samples were transferred to open weighing bottles and placed in the oven at desired temperatures for 20 min. Trash samples were exposed to temperatures of 22 °C (72 °F) (std. room condition) or 149 °C (300 °F).

Infrared (IR) spectroscopy. Spectra were collected on a bench FTS 3000MX spectrometer (Varian Instruments; Randolph, MA) equipped with a ceramic source, KBr beamsplitter, and deuterated triglycine sulfate (DTGS) detector. The spectrometer used a DuraScope (Smiths Detection; Danbury, CT) single-pass attenuated total reflection (ATR) diamond-coated internal reflection accessory. Sufficient amount of sample was used to cover the ATR element, and a stainless steel rod was used to give even contact pressure between samples and ATR element. To ensure that maximum internal reflectance was achieved, level 5 on the electronic load display scale was employed. Three replicate spectra were collected at a resolution of 4 cm⁻¹ co-adding 128 scans with a spectral range of 650 to 4000 cm⁻¹. The absorption spectra were the result of averaging the three replicate scans recorded at standard room conditions of 21 °C (70 °F) and a relative humidity of 65% (ASTM, 1997).

Data processing. Spectral integrations were performed in order to numerically emphasize the effects of size and heat to the results. For each heat and size reduction treatment, the ratio of the area under the total spectrum to the area under each of the IR bands of interest was determined. This integration was accomplished using GRAMS/AI 7.01 (Thermo Electron Corp.; Waltham, MA).

RESULTS AND DISCUSSION

Recent studies have employed mid-IR to detect differences in a range of raw, unmodified cotton trash samples based on their chemical composition (Allen et al., 2006; Foulk et al., 2003; Foulk et al., 2004; Himmelsbach et al., 2006). Figure 1 shows an empirical overlay of four unmodified trash types. As

shown in Table 1, the vibrational modes in the 3500 to 2800 cm^{-1} region are mainly due to stretching the O-H and C-H (Silverstein and Webster, 1998; Himmelsbach, 2003). The modes in the 1800 to 1500 cm^{-1} region are from the carbonyl functional group, as well as some overlapping involvements of C-H bending vibrations (Himmelsbach, 2003). The region below 1500 cm^{-1} , the fingerprint region, is made up of additional bands that correlate mainly to carbohydrates and other bio-constituents (Boeriu et al., 2004; Chung et al., 2004). During processing, raw samples of different trash types undergo several physical alterations, as well as thermal changes, making it harder to identify their source of origin. Quantitative changes in the FTIR spectra that occur as a result of size reduction and heat treatment are presented in Tables 2 and 3, respectively.

Table 2. Quantitative results of percentage area ratios between the areas under the individual peak to the area under the total spectrum (A_i/A_t) as the sample sizes are reduced

Trash type	Mode l size	O-H			C-H (1)			C-H (2)			COOH & COOR			COO ⁻		
		ATR band	A_i/A_t	\pm std error	ATR band	A_i/A_t	\pm std error	ATR band	A_i/A_t	\pm std error	ATR band	A_i/A_t	\pm std error	ATR Band	A_i/A_t	\pm std error
Stem	Raw	3288	0.0573	0.006	2918	0.0149	0.003	2850	0.0079	0.000	1733	0.0081	0.002	1593	0.0137	0.004
	Pepper	3287	0.1100	0.019	2920	0.0032	0.000	2853	0.0013	0.000	1734	0.0040	0.001	1595	0.0360	0.009
	Powder	3289	0.1121	0.008	2919	0.0011	0.000	2851	0.0006	0.000	1733	0.0031	0.001	1606	0.0275	0.002
Leaf	Raw	3276	0.0678	0.018	2921	0.0083	0.001	2852	0.0034	0.001	1730	0.0061	0.000	1601	0.0240	0.004
	Pepper	3271	0.1006	0.007	2918	0.0054	0.001	2851	0.0019	0.000	1731	0.0038	0.001	1607	0.0239	0.001
	Powder	3276	0.1006	0.006	2919	0.0023	0.000	2851	0.0005	0.000	1728	0.0014	0.000	1612	0.0225	0.000
Hull	Raw	3321	0.0443	0.001	2917	0.0200	0.001	2850	0.0091	0.001	1732	0.0153	0.001	1597	0.0158	0.001
	Pepper	3328	0.1105	0.004	2920	0.0016	0.001	2852	0.0007	0.000	1733	0.0034	0.001	1606	0.0207	0.001
	Powder	3328	0.1123	0.001	2920	0.0011	0.000	2851	0.0005	0.000	1734	0.0028	0.000	1602	0.0169	0.003
Seed coat	Raw	3281	0.0940	0.007	2919	0.0024	0.000	2851	0.0011	0.000	1715	0.0007	0.000	1603	0.0129	0.000
	Pepper	3289	0.1040	0.020	2922	0.0020	0.000	2853	0.0008	0.000	1711	0.0007	0.000	1614	0.0133	0.002
	Powder	3304	0.0932	0.011	2923	0.0020	0.000	2853	0.0008	0.000	1740	0.0006	0.000	1610	0.0105	0.000

Table 3. Quantitative results of percentage area ratio between the areas under the individual peak to the area under the total spectrum (A_i/A_t) associated with heat treatment

Trash type	Mode	O-H			C-H (1)			C-H (2)			COOH & COOR			COO ⁻		
		ATR band	A_i/A_t	\pm std error	ATR band	A_i/A_t	\pm std error	ATR band	A_i/A_t	\pm std error	ATR band	A_i/A_t	\pm std error	ATR band	A_i/A_t	\pm std error
Stem	No heat	3287	0.1100	0.019	2920	0.0032	0.000	2853	0.0013	0.000	1734	0.0040	0.001	1595	0.0360	0.009
	Heat	3332	0.0859	0.002	2919	0.0026	0.001	2851	0.0011	0.000	1733	0.0045	0.001	1612	0.0255	0.003
Leaf	No heat	3271	0.1006	0.007	2918	0.0054	0.001	2851	0.0019	0.000	1731	0.0038	0.001	1607	0.0239	0.001
	Heat	3329	0.0920	0.003	2922	0.0030	0.000	2853	0.0011	0.000	1732	0.0035	0.000	1603	0.0324	0.005
Hull	No heat	3328	0.1105	0.004	2920	0.0016	0.001	2852	0.0007	0.000	1733	0.0034	0.001	1606	0.0207	0.001
	Heat	3329	0.0951	0.004	2923	0.0013	0.001	2855	0.0005	0.000	1735	0.0066	0.000	1603	0.0198	0.002
Seed coat	No heat	3289	0.1040	0.020	2922	0.0020	0.000	2853	0.0008	0.000	1711	0.0007	0.000	1614	0.0133	0.002
	Heat	3312	0.1159	0.004	2922	0.0015	0.000	2854	0.0004	0.000	1741	0.0023	0.001	1616	0.0140	0.001

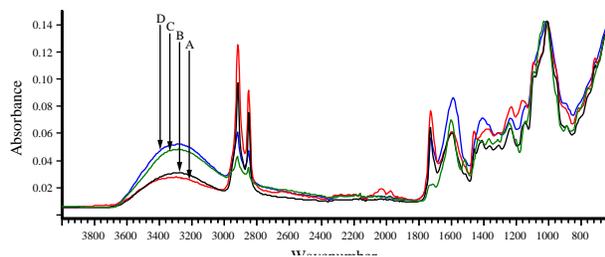


Figure 1. Experimental ATR-FTIR spectra of hull (red, A), stem (black, B), seed coat (green, C), and leaf (blue, D) raw samples displaying the similarities in absorption bands.

Size effect. The physical appearances of the raw samples were altered by grinding in order to generate smaller particle sizes (e.g., pepper and powder sizes). Spectral representations of the effect of pepper and powder sizes on individual trash samples are shown in Figures 2 to 5. A majority of the common characteristic modes are present in each sample, regardless of size (Table 1). This study concentrates on well characterized regions between 3500 to 2800 cm^{-1} and 1800 to 1550 cm^{-1} . Additional changes in absorption bands below 1500 cm^{-1} generate peaks that are more difficult to associate with specific functional groups.

3500 to 2800 cm^{-1} region. The vibrational modes around 3600 to 3200 cm^{-1} are attributed to O-H functional groups. Each trash sample shows a predominant broad band at $\sim 3300 \text{ cm}^{-1}$, which is indicative of a polymeric O-H or a hydroxyl group associated with hydrogen bonding (Coates, 2000). As the raw sample is reduced to pepper sizes, the spectra for the stem, leaf, and hull reveal an increased intensity (Fig. 2 – 4, respectively; Table 2). As the pepper samples are further reduced in size to powder, the stem, leaf, and hull exhibit intensities for the powder sizes that are similar to the pepper sizes, yet still higher than the raw sizes (Fig. 2 – 4, respectively; Table 2). The raw, pepper and powder seed coat spectra do not display any observable differences (Fig. 5, Table 2).

The two spectral bands around 3000 to 2840 cm^{-1} are indicative of the presence of waxes and oils (Himmelsbach, 2003). These strong to moderate spectral bands ($\sim 2900 \text{ cm}^{-1}$ and $\sim 2850 \text{ cm}^{-1}$) are exhibited in all four trash samples (Fig. 2 – 5). As the raw samples are reduced in size, the spectral intensities of stem, leaf, and hull all decline (Fig. 2 – 4, respectively). The leaf displays a relatively small change in intensity at these modes, whereas the stem and hull display a larger change from raw to smaller particle sizes. The changes are associated with the fact that waxes are located mainly on the outside portion, where it acts

as a barrier to microbial penetration and water loss. Consequently, when the size of either the stem or hull has been reduced, the exposure of wax is not as great, resulting in a reduction in the IR intensity. The seed coat demonstrates little change in absorbance as the sample size is reduced (Fig. 5).

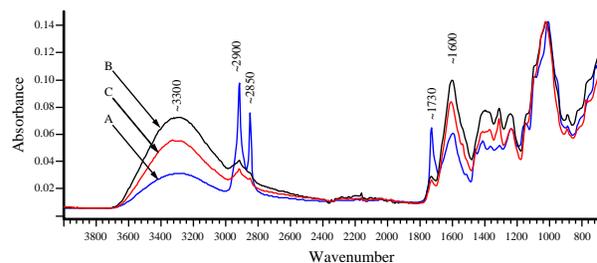


Figure 2. The effect of size reduction on the ATR-FTIR spectra of the stem sample, including ground raw size (blue, A), pepper size (black, B), and powder size (red, C).

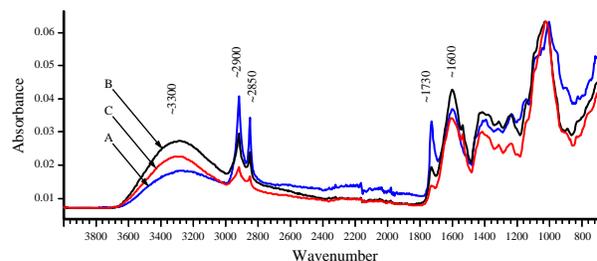


Figure 3. The effect of size reduction on the ATR-FTIR spectra of the leaf sample, including ground raw size (blue, A), pepper size (black, B), and powder size (red, C).

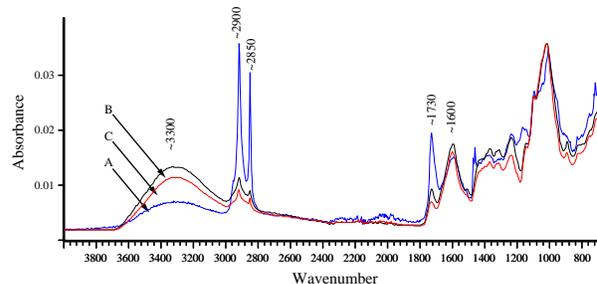


Figure 4. The effect of size reduction on the ATR-FTIR spectra of the hull sample, including ground raw size (blue, A), pepper size (black, B), and powder size (red, C).

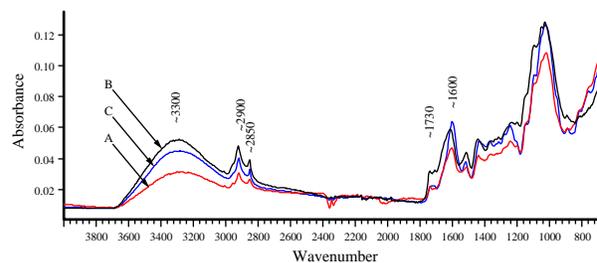


Figure 5. The effect of size reduction on the ATR-FTIR spectra of the seed coat sample, including ground raw size (blue, A), pepper size (black, B), and powder size (red, C).

1800 to 1550 cm^{-1} region. The presence of a carbonyl/carboxyl functional group is indicated by the presence of a strong band around 1650 to 1780 cm^{-1} (Himmelsbach, 2003). The stem, leaf, and hull exhibit the presence of this group at $\sim 1730 \text{ cm}^{-1}$. Similar decreasing absorption trends as the particle sizes are reduced can be observed for stem, leaf, and hull trash types (Fig. 2 – 4, respectively; Table 2). The matrix surrounding the functional group may be affected by grinding, which may in turn alter the intensity of the carboxylic band. In the case of the seed coat, the carbonyl/carboxyl functional group is present, but the splitting of the peak indicates the possible presence of more than one constituent (Fig. 5). The peak at 1630 to 1650 cm^{-1} is associated with the carboxylate anion (Himmelsbach, 2003). The frequency and shift of this peak is dependent on hydrogen bonding with water. Each trash sample spectrum shows a strong band at $\sim 1600 \text{ cm}^{-1}$, indicating a consistent frequency shift because of hydrogen bonding. For stem, hull, and leaf samples (Fig. 2 – 4, respectively; Table 2), as the particle size decreases from raw to pepper size, the carboxylate band shows a relative intensity increase. In the case of the seed coat, the splitting in the peak $\sim 1730 \text{ cm}^{-1}$ causes a difference between the spectra (Fig. 5) and the quantitative results (Table 2). Each trash sample exhibits a decrease in intensity as the particle decreases from pepper to powder sizes.

Heat effect. During ginning, heat is applied in order to dry cotton fibers to ease the task of separating the fibers from foreign matter (trash). As a consequence, trash components also experience heating, and it is desirable to understand how the IR spectra of each trash component are affected by the application of heat. This section represents heat treatments performed to pepper size trash samples. Upon heating, there was no observable discoloration in the trash samples. Following treatments, the samples were allowed to equilibrate back to standard conditions before testing, implying that observed changes are irreversible. The major IR modes mentioned previously will be used as the focus of this work.

3500 to 2800 cm^{-1} region. The leaf and seed coat spectra exhibit minute changes in the O-H band ($\sim 3300 \text{ cm}^{-1}$) with the application of heat (Fig. 7 and 9, respectively; Table 3). Conversely, the stem and hull spectra display a more drastic reduction in the same IR region (Fig. 6 and 8, respectively; Table 3). This effect is potentially because of the irreversible loss of water, seen most dramatically in the stem tissue. Each trash

spectrum shows a slight decrease in the C-H bands at 2900 and 2850 cm^{-1} . This decrease is likely because of the disappearance of volatile aliphatic compounds, which exhibit strong absorbance in this region.

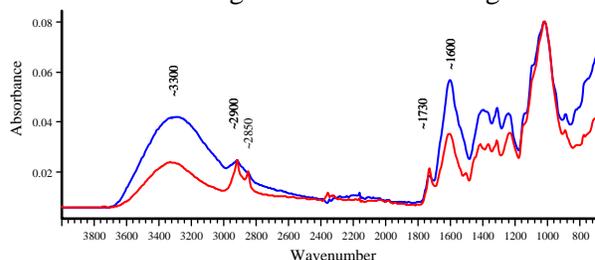


Figure 6. The effect of heat treatment on the ATR-FTIR spectra of the pepper-size stem samples. Samples at standard room condition (blue) were heated to 149 °C (300 °F) (red) for 20 min. Samples were reconditioned prior to analyzing.

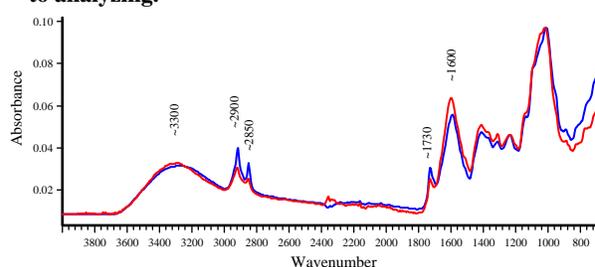


Figure 7. The effect of heat treatment on the ATR-FTIR spectra of the pepper-size leaf samples. Samples at standard room condition (blue) were heated to 149 °C (300 °F) (red) for 20 min. Samples were reconditioned prior to analyzing.

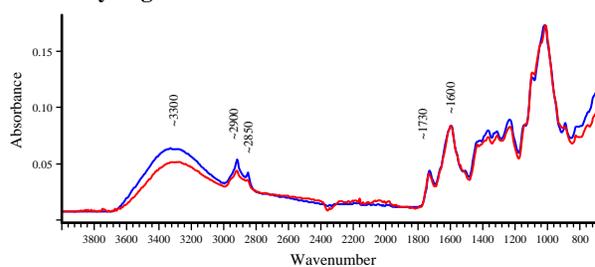


Figure 8. The effect of heat treatment on the ATR-FTIR spectra of pepper-size hull samples. Samples at standard room condition (blue) were heated to 149 °C (300 °F) (red) for 20 min. Samples were reconditioned prior to analyzing.

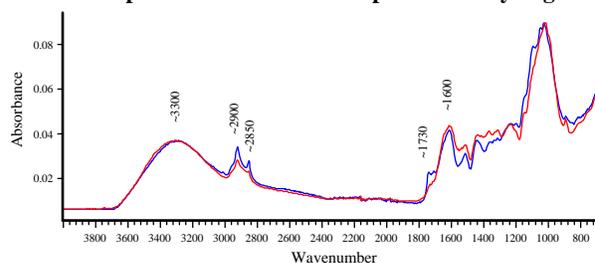


Figure 9. The effect of heat treatment on the ATR-FTIR spectra of pepper-size seed coat samples. Samples at standard room condition (blue) were heated to 149 °C (300 °F) (red) for 20 min. Samples were reconditioned prior to analyzing.

1800 to 1550 cm⁻¹ region. Each trash type shows various changes around the carbonyl/carboxyl region at $\sim 1730\text{ cm}^{-1}$. The stem, leaf, and hull samples exhibit no substantial change in peak intensity following the application of heat (Fig. 6 – 8; respectively, Table 3). In the case of the hull, however, the percentage area ratio for no heat treatment is 0.003 compared with a percentage area ratio of 0.007 for the heat treated sample (Table 3). The seed coat shows a more substantial change in this band compared with the other trash components (Fig. 9, Table 3). As mentioned previously, the seed coat exhibits structure in this region that suggests the presence of multiple carboxylic acid or ester constituents. After the application of heat, this region undergoes an irreversible decrease suggesting that the constituents responsible for the signal in this region may be volatile aliphatic esters.

Because of the presence of carboxylate groups, the band at $\sim 1600\text{ cm}^{-1}$ undergoes relatively minor changes in both the hull and seed coat samples as a result of heat treatment (Fig. 8 and 9, respectively; Table 3). In the stem samples, the intensity of the band is drastically decreased as a result of heating (Fig. 6; Table 3). It has been reported that stem trash has a moisture transfer rate lower than other botanical trash types (Barker et al., 1995). Barker et al. (1995) studied the effects of moisture on cotton trash samples by investigating the absorption and desorption rates. In this case, the moisture that is lost through heating will not be reabsorbed during reconditioning. In the case of leaf, an increase in intensity is observed as a result of heat treatment (Fig. 7; Table 3). Essentially, the leaf tissue appears to absorb additional moisture as the sample was allowed to re-equilibrate. The reason for this behavior is not known, but a tentative explanation is that the observed concomitant decrease in volatile aliphatic compounds as a result of heat treatment allows moisture to penetrate the leaf tissue more effectively.

CONCLUSION

In this preliminary study, two separate physical applications reflecting exposure to ginning and textile processing conditions were applied to selected cotton trash types. As the trash was reduced in size, previously masked interior tissues are exposed, revealing complex arrangements within each trash type resulting in observable changes in the FTIR spectra exhibited in the ratio differences of the

chemical functional groups analyzed. Application of heat treatments to the pepper-size trash likewise resulted in changes in the FTIR spectra because of the irreversible loss of volatile components associated with water and aliphatic moieties. Evaluation of these results indicates that the present FTIR spectral database must take into account particle size reductions and heat effects experienced by cotton trash during processing.

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DISCLAIMER

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