

Development and Use of an Attenuated Total Reflectance/ Fourier Transform Infrared (ATR/FT-IR) Spectral Database To Identify Foreign Matter in Cotton

DAVID S. HIMMELSBACH,^{*,†} JOHN W. HELLGETH,[‡] AND DAVID D. MCALISTER[§]

Quality Assessment Research Unit, Richard B. Russell Research Center, Agricultural Research Service, U.S. Department of Agriculture, P.O. Box 5677, Athens, Georgia 30605-5677; Hewlett-Packard Company, MS711A, 1000 N.E. Circle Boulevard, Corvallis, Oregon 97330; and Cotton Quality Research Laboratory, Agricultural Research Service, U.S. Department of Agriculture, Clemson, South Carolina 29631

The presence of foreign matter in cotton seriously affects the cotton grade and thus the price per bale paid by the spinner to the grower, the efficiency of the spinning and ginning operations, and the quality of the final woven product. Rapid identification of the nature of the extraneous matter in cotton at each stage of cleaning and processing is necessary to permit actions to eliminate or reduce its presence and improve efficiency and quality. Although several instruments are being successfully employed for the measurement of contamination in cotton fibers based on particle size/weight, no commercial instrument is capable of accurate qualitative identification of contaminants. To this end, ATR/FT-IR spectra of retrieved foreign matter were collected and subsequently rapidly matched to an authentic spectrum in a spectral database. The database includes contaminants typically classified as “trash”, cotton plant parts (hull, shale, seed-coat fragments, bract, caryx, leaf, bark, sticks, and stems) and grass plant parts (leaf and stem); “foreign objects and materials”, synthetic materials (plastic bags, film, rubber, bale wrapping and strapping); organic materials (other fibers, yarns, paper, feathers, and leather); plus entomological and physiological sugars and inorganic materials (sand and rust). The spectral matching resulted in consistently high-score identification of the foreign matter based on chemical composition, irrespective of its particle size. The method is envisioned to be employed with stand-alone rugged infrared instrumentation to provide specific identification of extraneous materials in cotton as opposed to only general classification of the type by particle size or shape.

KEYWORDS: Attenuated total reflectance; mid-infrared; FT-IR; spectral matching; upland cotton (*Gossypium hirsutum* L.); Pima cotton (*Gossypium barbadense* L.); cotton trash; foreign matter; ginning; spinning

INTRODUCTION

With the trend toward automation of cotton-processing equipment, the biggest problem has become cotton cleanliness. According to the International Textile Manufacturer Federation’s *Cotton Contamination Surveys (1)*, foreign matter, stickiness, and cotton seed-coat fragments in raw cotton continue to be the most serious problems affecting the cotton-spinning industry. This survey covers 16 sources of contamination plus cases of stickiness and cotton seed-coat fragment contamination. Overall, of cotton bales evaluated in 2005, 7% were found to be seriously contaminated by the 16 different sources of foreign matter, and

a further 15% were moderately contaminated, leaving 76% insignificantly contaminated. In addition, 17% displayed stickiness and 44% had cotton seed-coat fragment contamination. Sources of contamination in this report are categorized as (1) fabrics made of woven plastic, plastic film, jute/hessian, and cotton; (2) strings made of the same materials; (3) organic matter including leaves, feathers, paper, leather, etc.; (4) inorganic matter including sand/dust, rust, and metal wire; and (5) oily substances including grease/oil, rubber, stamp color, and tar. The extent of contamination by these components ranged from 5% for tar to 40% for organic matter. Thus, organic matter is the most prevalent source of contamination. This is rather a limited list of contaminants. In addition, the first category represents two groups of synthetic and natural materials that differ by only their physical organization and can be chemically redundant.

A more extensive list of 10 categories has been proposed by Etheridge (2). These categories are (1) foreign objects and

* Author to whom correspondence should be addressed [e-mail dshimmel@qaru.ars.usda.gov; telephone (706) 546-3233; fax (706) 546-3607].

† USDA-ARS, Athens, GA.

‡ Hewlett-Packard Co., Corvallis, OR.

§ USDA-ARS, Clemson, SC.

materials (fibers, yarns, fabrics, paper, oil, metal, rust, rubber, etc.), (2) trash (leaf, bark, bracts, noncotton vegetation, etc.), (3) motes, seeds, and seed-coat fragments, (4) dirt and dust, (5) entomological sugars, (6) physiological sugars, (7) neps, (8) short fibers, (9) dead/immature fibers, and (10) discoloration. The first six of these categories are primarily related to chemical properties of nonlint materials associated with cotton, and the last four are primarily related to physical properties of lint cotton.

To date, only systems that classify foreign matter on the basis of physical (gravimetric or geometric) analysis are currently generally employed. These include instruments such as the Shirley Analyzer (SDL International Ltd., Stockport, U.K.) and the Advanced Fiber Information System (AFIS) (Zellweger Uster, Uster, Switzerland) that are based on gravimetrics and “trashmeters” of the high-volume instruments (HVI) type based on geometric (image) analysis. The cotton trash and cotton color measurement system (CTCM) combined with clustering analysis has been demonstrated to be capable of categorizing foreign matter (trash) as leaf, bark, and seed-coat particles (3, 4). This appears to be the extent to which foreign matter has been identified by geometric-based instrumental methods. Limited attempts have been made to further identify contaminants based on chemistry. These methods include detection of entomological (melezitose and trehalulose) sugars by near-infrared reflectance (5), trehalulose and physiological sugars (fructose, glucose, sucrose, and turanose) by the potassium ferricyanide reducing sugar test (6), oil (motor oil and grease) by mid-infrared transmission and high-performance liquid chromatography (7), and bark, seed-coat fragments, and polypropylene by X-ray image analysis (8). No one system has been successfully applied to the identification of all types of foreign matter in cotton.

With the maturation of Fourier transform infrared (FT-IR) and the introduction of microattenuated total reflectance (ATR) devices in the past few years (9), essentially a universal sample-handling system has been developed for liquids and solids. These systems, with an appropriately chosen internal reflectance element (IRE), provide ease in sample handling along with ease of cleaning of the crystal surface and the ability to obtain a reproducible path length. The use of a thin diamond IRE supported on top of a larger optical element made of a material of a similar refractive index yields a hard inert surface, good optical performance, good mechanical support, and sample-focusing capability. This has permitted the use of the high discriminating power of mid-infrared spectroscopy to be applied to a wide diversity of samples. Attenuated total reflectance (ATR)/FT-IR is ideal for use in the identification of the various types of foreign matter associated with cotton that differ widely in physical state and chemical composition, except for those that give no absorbance in the mid-infrared range, such as metals. This work investigates the applicability of ATR/FT-IR for the chemical identification of a wide variety of foreign materials that can be present in cotton by matching their spectra in a searchable spectral database containing authentic samples of these materials. It is anticipated that this spectral database could be used by cotton gins, spinning facilities, and textile manufacturers to identify sources of contamination in cotton so that methods can be developed to reduce specific sources of contamination.

MATERIALS AND METHODS

Foreign Materials. Organic foreign materials associated with upland cotton (*Gossypium hirsutum* L.) and bale wrapping materials were provided by Stanley A. Anthony, USDA-ARS, Stoneville, MS. Organic foreign materials associated with Pima cotton (*Gossypium barbadense*

L.) and components of upland and Pima cotton from the 2005 New Mexico crop (used for testing the database) were obtained from Ed Hughs, USDA-ARS, Mesilla Park, NM. Physiological sugars, melezitose and trehalulose, were obtained from Sigma-Aldrich (St. Louis, MO). Trehalulose was obtained from Gary R. Gamble, USDA-ARS, Clemson, SC. Grasses and weeds were obtained from cotton fields near Watkinsville, GA. Asphalt was obtained from the National Center for Asphalt Technology, Auburn University, Auburn, AL. Soils were obtained from Dewayne Mays, National Soil Survey Center, Lincoln, NE. Road (patch) tar and plastic shopping bags were obtained locally (Athens, GA).

Infrared Spectroscopy. FT-IR spectra for the database library and test samples were collected using a Nicolet Magna 850 FT-IR bench (Thermo Nicolet, Madison, WI) employing a DuraScope (Smiths Detection, Danbury, CT) ATR sampling device equipped with video imaging. The IR spectrometer bench was equipped with a global source, a KBr beam splitter, and a deuterated triglycine sulfate (DTGS) detector. The ATR sampling device utilized a diamond internal reflection element (IRE) embedded into a ZnSe support/focusing element in a single-reflection configuration. Contact pressure was applied by way of a stainless steel rod employing the same pressure, level 5 on the electronic load display scale. Spectra collected for the database were obtained over the range of 4000–650 cm^{-1} at 8 cm^{-1} resolution, co-adding 128 interferograms. The spectra of six to eight subsamples of natural materials, three subsamples of synthetic materials, and one sample of homogeneous purified chemicals were acquired. Interferograms were processed with Happ–Genzel apodization and Mertz phase correction and ratioed against the open-beam single-beam spectrum collected through the ATR device with a clean crystal surface. No ATR correction, to make relative intensity adjustments, was applied to the spectra. The spectral database was initially collected using Omnic E.S.P. 5.2 (Thermo, Madison, WI) and put in that software’s library format. The spectral data were subsequently converted to GRAMS (Thermo Galactic, Salem, NH) and JCAMP formats to permit use with other IR spectrometer systems.

All searches of the database were conducted with the Omnic-developed version of the database using the correlation algorithm contained in that software’s library search routine.

RESULTS AND DISCUSSION

Figure 1 shows images of cotton plant components that largely contribute to organic foreign matter found in cotton lint. When cotton initially arrives at the cotton gin, these materials are large enough to be identifiable. The ginning process removes the bulk of these materials. Those materials that survive the gin and remain with the lint are smaller, making the identification of their source more difficult. During the spinning process, more of the contaminants are removed, but some of them still adhere to the yarn, whereas others become pulverized and collect as dust. Those that stick to the yarn can cause defects in the final woven fabric. Those that become dust clog the rotors in rotor-spinning equipment, which can cause “ends-down” in which the yarn breaks in the process of being spun. At this point in processing, it is virtually impossible to visually identify the source of the contamination. This problem suggested that the use of an infrared method of analysis, which identifies materials by their spectra that are indicative of their chemical composition, might address this problem. Considering the ease of use of ATR for sample preparation and the increased availability of portable infrared spectrometers using this technology, it seemed logical to develop a mid-infrared spectral database of the contaminants to make compositional identification possible. This was not without special challenges, because many of the contaminants were of natural origin and gave very similar spectra.

As can be seen in **Figure 2**, even though the organic cotton plant materials are initially anatomically very different, their

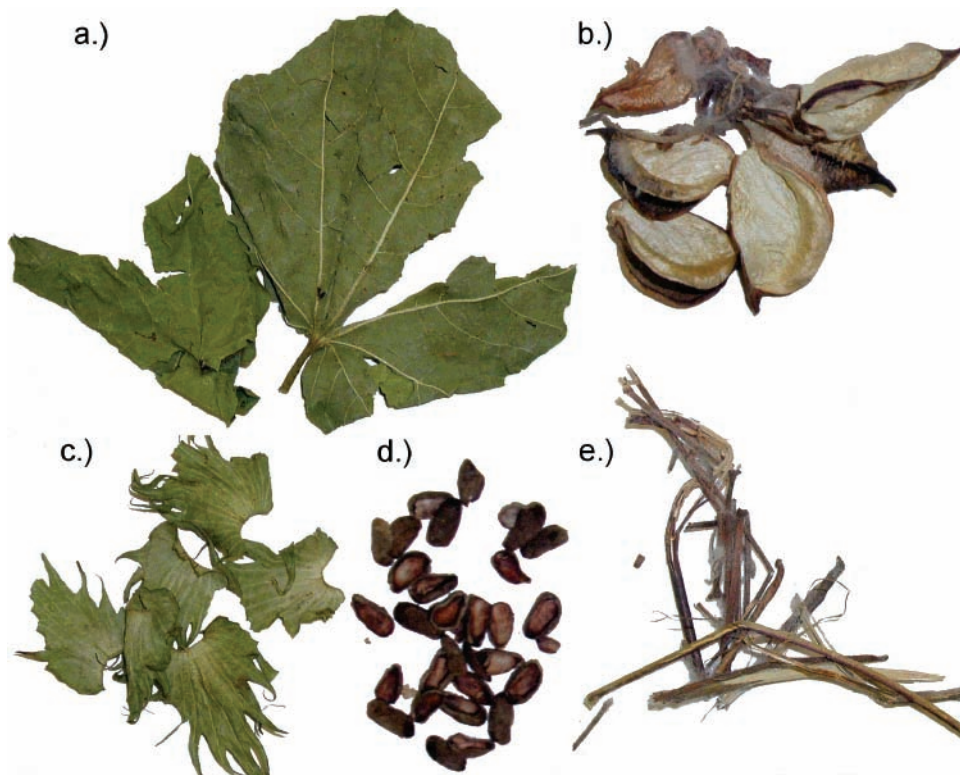


Figure 1. Examples of parts of the cotton plant that can become organic foreign matter: (a) leaves; (b) hulls; (c) bract; (d) seed coats; (e) stems.

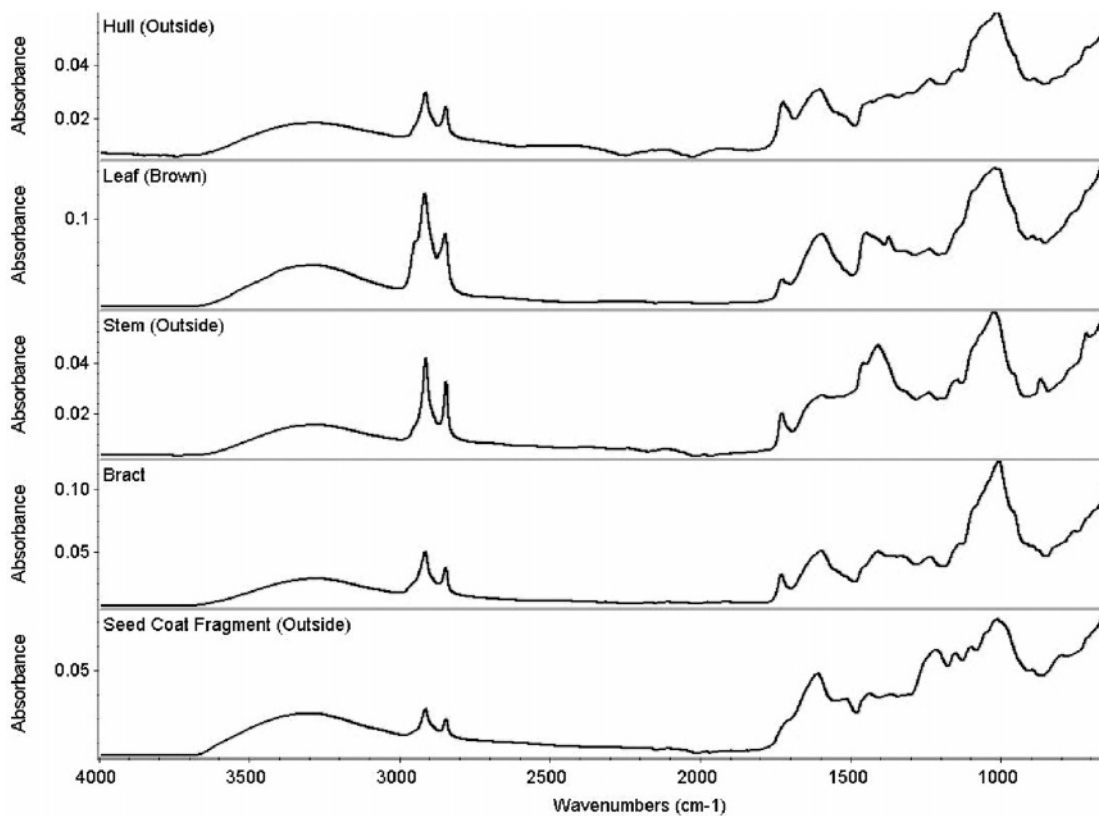


Figure 2. Examples of ATR/FT-IR spectra of parts of the cotton plant that can become organic foreign matter present in cotton lint.

infrared spectra are very similar. Because these are primarily cellulosic materials, except for those containing smaller amounts of waxes, phenolics, and some other minor constituents, their spectra are very much like those of the cotton fibers that they need to be distinguished from. Because these are natural materials, each type does vary slightly in composition from sample to sample and even among repetitive spectra of the same

sample. Samples that retain their pigments (e.g., green versus brown) also give slightly different spectra. It was also found that opposite sides of many of these materials give slightly different spectra. Much of this latter difference is due to the presence of more wax on the upper versus under surfaces of leaves and outer versus inner surfaces of hulls, seeds, and stems. Surfaces that are more exposed to the environment tend to build

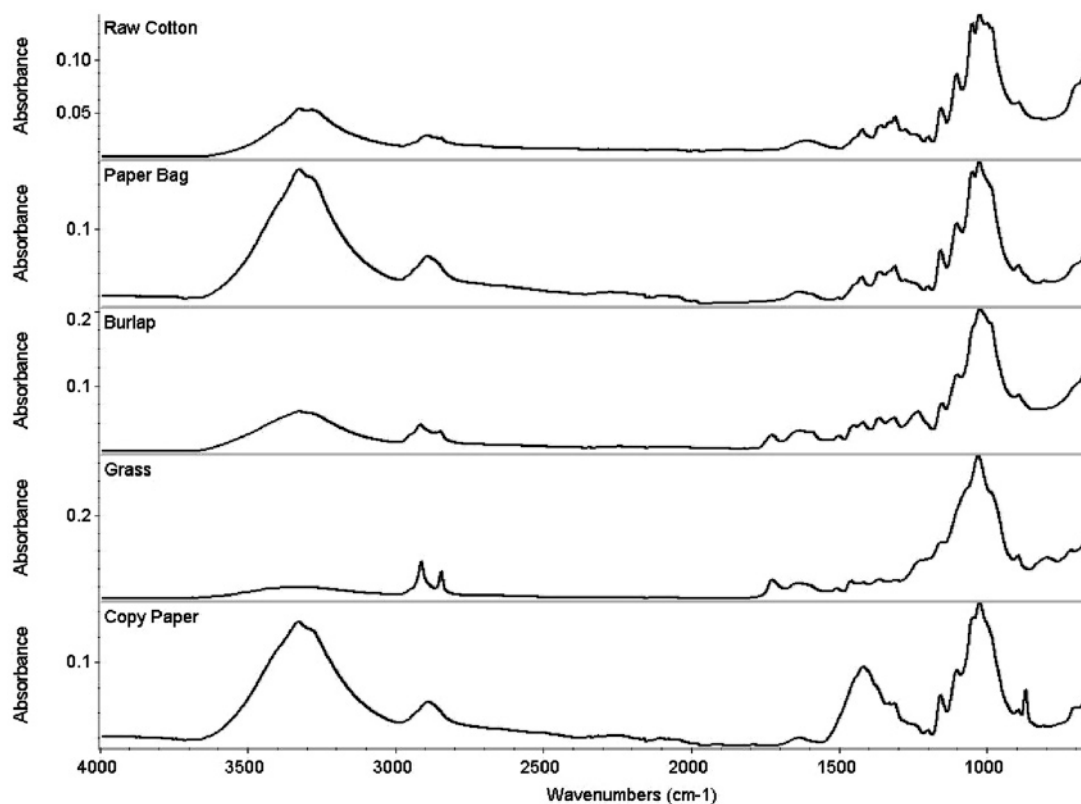


Figure 3. Examples of ATR/FT-IR spectra of raw cotton and other cellulosic materials that can become foreign matter in cotton.

up more wax as a protective barrier and for the prevention of water loss under heat stress. Because cottonseeds are not exposed to the environment as much as the latter materials, they do not have as much wax on their outer surfaces and their spectra show less intensity in C–H ($2900\text{--}2800\text{ cm}^{-1}$) and C=O (1735 cm^{-1}) stretch vibration regions (see **Figure 2**). Unlike synthetic or pure materials, several repetitions of spectra of each sample were obtained to account for their natural variation. Normally, this variation is reflected in intensity changes in bands and not in the appearance of bands at different frequencies. Therefore, it was found that the collection of six repetitions of each of the plant materials was necessary to be included in the database of spectra that formed the library.

In **Figure 3**, the spectrum of raw cleaned cotton is compared to those of other cellulosic materials that show very similar spectra. The spectrum of a paper bag, derived from wood tissue, displays the most similar bands to those of cotton. However, the spectrum of the paper bag does differ in the intensity ratio of the O–H (3330 cm^{-1}) to C–H (2900 cm^{-1}) stretch vibrations, the band shape of the C–O, C–O–H, and C–O–C bands around 1000 cm^{-1} , and the small band at 813 cm^{-1} , which may be due to other carbohydrate components not found in processed cotton. The spectra of the paper bag, burlap, and grass all show a weak band (displaying a maximum between 1512 and 1504 cm^{-1}) that most likely indicates the presence of lignin. Lignin is a polyphenol that is not known to be present in cotton lint. Thus, this band becomes a distinguishing feature of the spectra of other (primarily cellulosic) materials from that of cotton.

Cotton lint is essentially composed of pure cellulose ($93\text{--}95\%$), except for its containing $\approx 5\text{--}7\%$ of pectin and $\approx 0.7\text{--}1.1\%$ of wax on its surface (10). Lignin is also a component of all of the materials for which spectra are shown in **Figure 2**, but its presence is obscured by other components. Lignin has previously been spectroscopically shown to be present in hull material by subtraction of the spectrum of shale from the

spectrum of hull (11). The spectrum of the sample a copy paper, shown at the bottom of **Figure 3**, differs from all of the other components in that this particular paper appears to contain a blue dye coating. This is indicated by the bands centered at 1410 and 871 cm^{-1} . Without this additive, the spectrum of the copy paper would look much like that of the paper bag. This is just an example of another paper type. Papers have various coatings that contain pigments, adhesives or binders, and other additives. To include all paper types would be beyond the scope of this work. An extensive list of different coatings and papers and their infrared spectra, in absorbance mode, may be found on-line (12). Entomological and some physiological sugars, which are included in the database, are shown in **Figure 4**. Spectra of the entomological sugars (trehalulose and melezitose) that are deposited on cotton, in the field, by aphids (*Acyrtosiphon* spp.) and whiteflies (*Bemisia* spp.) are shown at the top of this figure. They are involved in what is called “sticky” cotton. The presence of sticky cotton is such a serious problem for the cotton industry that is often dealt with separately from other contamination (1). If these sugars are present in sufficient quantities in the cotton at the gin, they can be indicative of stickiness that can ruin saw blades by making the teeth become razor-sharp and cut the cotton lint. Sticky cotton can stop the gin and be the cause of gin fires. These entomological sugars can survive the spinning process and work their way into the final woven fabric. Even in small quantities, they can cause imperfections in the woven fabric and inhibit the speed and efficiency of processing machinery. They are often found with normal plant (physiological) sugars: glucose, fructose, and sucrose. Therefore, spectra of all five of these sugars are shown in **Figure 4** for comparison. The most distinguishable feature of the entomological sugars is that they contain bands at 825 ± 3 and $785 \pm 3\text{ cm}^{-1}$. Thus, entomological sugars can be distinguished among a mixture of mono- and oligomeric sugars. They can also be distinguished from the polymeric cotton lint

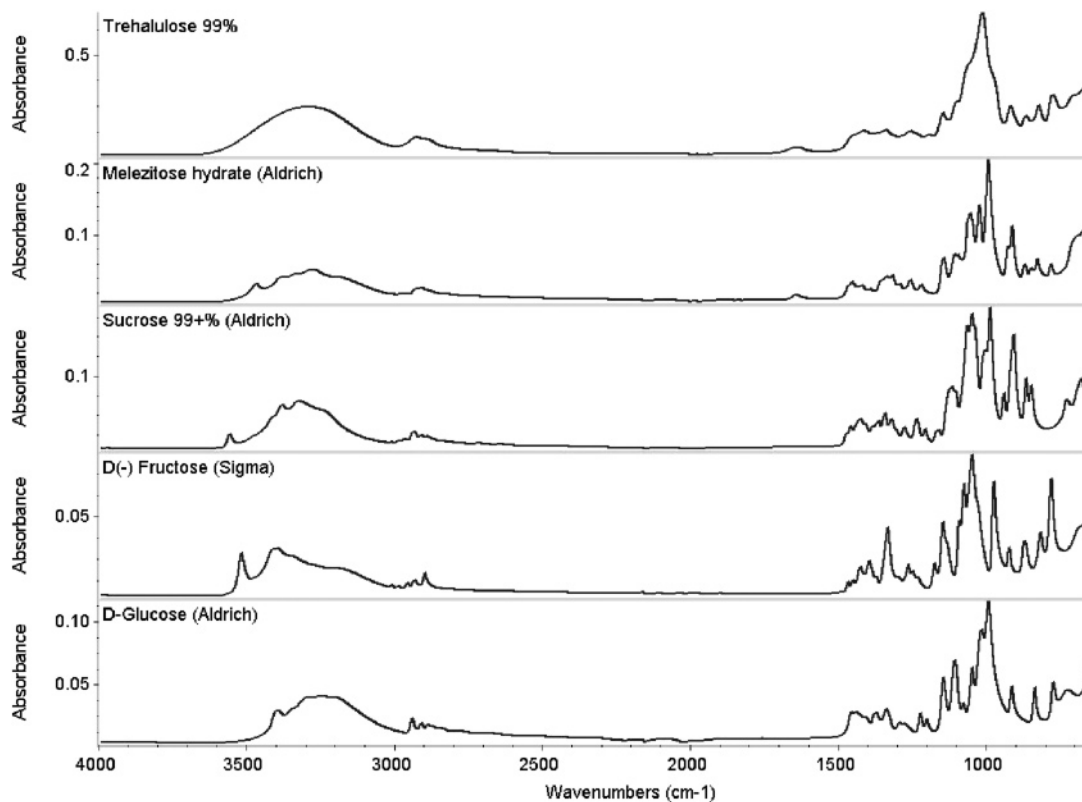


Figure 4. ATR/FT-IR spectra of entomological and physiological sugars that can naturally occur on cotton fibers.

cellulose, the spectrum of which displays only a shoulder at 895 cm^{-1} in the $900\text{--}750\text{ cm}^{-1}$ region of the infrared spectrum.

Oily substances also are frequent contaminants of cotton. Grease and motor oil can originate from the cotton-picking and transportation equipment plus any equipment used in spinning or weaving. Also, road tar tends to find its way into cotton fields from the tires of equipment or from direct road-spraying operations. Leather can come from belts on many types of equipment. Feathers from poultry operations are also common, because these operations are often in close proximity to cotton fields. Spectra of all of these materials are shown in **Figure 5**. Because the former materials are all derived from petroleum, their spectra are very similar, as they are all hydrocarbons. They display characteristic CH_3/CH_2 stretching bands in the $3000\text{--}2800\text{ cm}^{-1}$ and CH_2 deformation band around 1460 cm^{-1} . They do not display the carbonyl band that is present in the spectrum of cotton seed oil at 1744 cm^{-1} (not shown). However, cotton seed oil can also contaminate cotton fiber when seeds are broken open to expose the meat.

Animal-based materials, such as leather and feathers, are characterized by the presence of protein. These materials give characteristic bands for secondary proteins that appear around 3200 cm^{-1} for NH_2 stretch plus amide $\text{C}=\text{O}$ ($1670\text{--}1620\text{ cm}^{-1}$), *trans*-CHN bend-stretch ($1570\text{--}1510\text{ cm}^{-1}$), or *cis*-CHN bend-stretch ($1490\text{--}1400\text{ cm}^{-1}$), CHN stretch-open ($1330\text{--}1200\text{ cm}^{-1}$), and NH_2 rocking ($1150\text{--}1100\text{ cm}^{-1}$) (13).

Synthetic or manmade materials also constitute foreign matter. Examples of spectra of some these materials are shown in **Figure 6**. The yellow (woven) bale wrap, plastic shopping bag, and bale strapping are chemically identifiable, using a database such as Sprouse Polymers by ATR library, consisting of isotactic polypropylene, low-density polyethylene, and poly(ethylene terephthalate), respectively. Clear plastic sheeting (not shown) is also used as a bale wrap. Like the plastic shopping bag, it is made of low-density polyethylene. The only difference is that

shopping bags often contain dyes that are detectable by infrared spectroscopy, which permits their differentiation from clear plastic. Rubber, from a retread tire, is included here as a contaminant spectrally similar to the synthetic materials shown in **Figure 6**. This sample does not by any means cover all of the possible rubber compositions that may be encountered, but it is one of the mostly likely to be found as foreign matter in cotton. A more complete collection of infrared spectra of rubbers, plastics, and thermoplastic elastomers is available from Rapra (14). Because these materials are often somewhat variable, being contaminated with soil or possessing several different dyes, three repetitions of spectra of these materials (as actually encountered in the cotton) were included in the database so as to include these potential variabilities.

Because soils are often a co-contaminant, the spectrum of a clay soil is included in this figure as an example of nonplant natural material that can become foreign matter associated with cotton lint or as part of many of the other contaminants. Soil was handled as in the same manner as were the plant components, because it can also be extremely variable. The soils included in the database represented three basic types: sandy, sandy-loam, and clay, which were uncontaminated with insecticides or other materials. Sands are distinguished by their Si-O-Si and Si-OH stretching vibrations that occur between 1110 and 1000 and $910\text{--}830\text{ cm}^{-1}$, respectively (13), plus at 778 and $670\text{--}690\text{ cm}^{-1}$, being due to Si-O deformations. Clays show bands in about the same regions, $1160\text{--}1000$ and $960\text{--}730\text{ cm}^{-1}$, due to in-phase CO_3 stretch and CO_3 deformation, respectively (16). A more extensive spectral database of these materials is available commercially (Mineral and Clay Spectral Database, Bio-Rad/Sadtler, Philadelphia, PA), if a more precise identification of minerals and clays is desired. However, the three soil types included in the database developed are sufficient to generally identify soil as a contaminant in cotton.

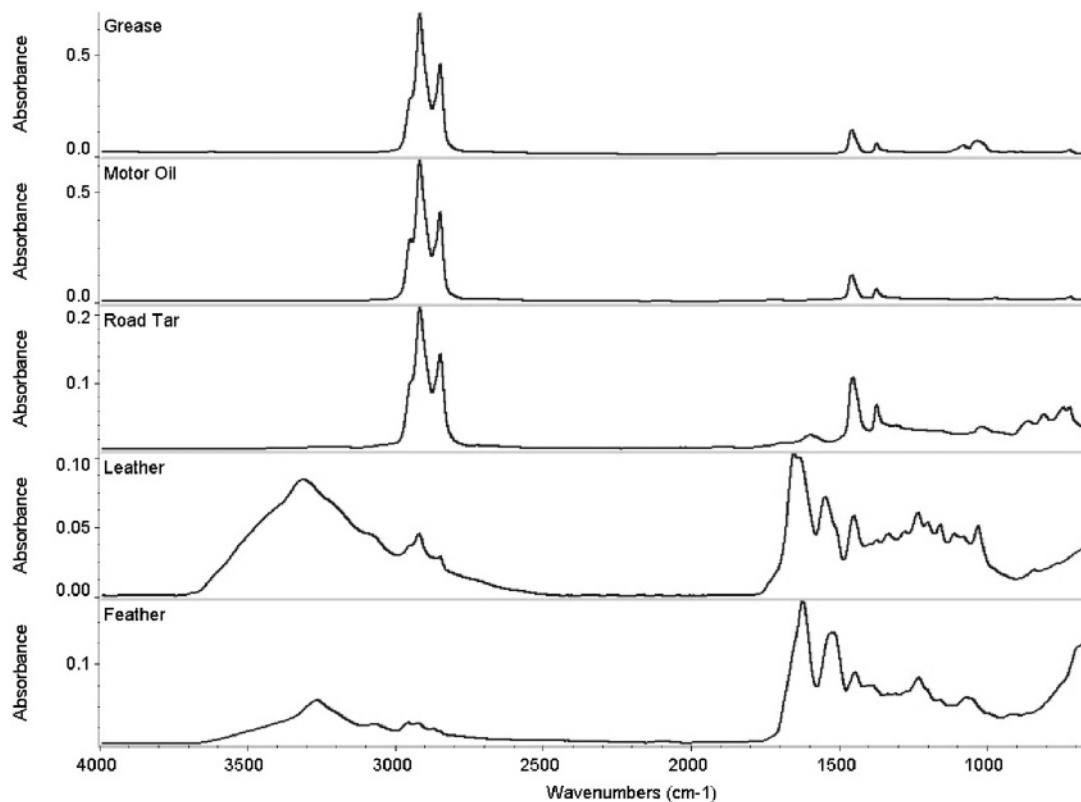


Figure 5. Examples of ATR/FT-IR oily substances and animal-based materials that are potential contaminants in cotton.

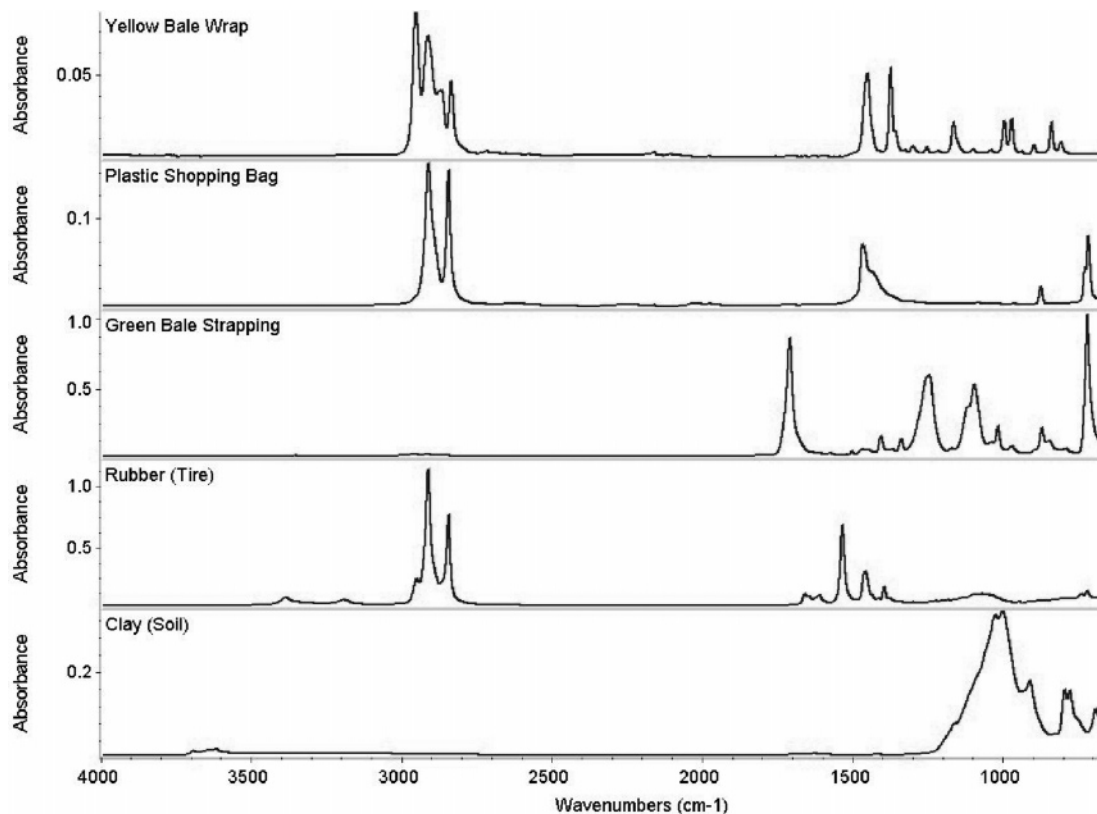


Figure 6. Examples of other ATR/FT-IR nonplant materials that are potential contaminants in cotton.

The use of this database resulted in the identification of unknown materials provided from a textile manufacturer; foreign matter in cotton received at the USDA-ARS, Southwestern Cotton Ginning Research Laboratory, Mesilla Park, NM; and foreign matter retrieved from the spinning equipment located at the USDA-ARS, Cotton Quality Research Unit, Clemson,

SC. In addition, authentic components of cotton plants the 2005 Pima and upland cotton crops grown near Mesilla Park, NM, were used as verification samples to test the database. The results of searching to identify these materials in the database are shown in **Table 1**. The table includes the general source of the sample along with a simple physical description of the sample, the IR

Table 1. Identification of Foreign Matter Found in Cotton by ATR/FT-IR Database

source	physical description	IR region ^a	match	identification
textile ^b	yellow fiber	FP	99.16	yellow bale wrap
textile ^b	blue fibers in sliver	FP	90.35	bale strapping
ginning lab ^c	green speck	FP	95.44	green leaf
ginning lab ^c	brown speck	FP	91.64	bract
spinning lab ^d	gray dust	FP	90.64	shale (vein)
spinning lab ^d	white dust	FP	95.58	hull (stem)
spinning lab ^d	yellow dust	FP	93.69	shale (inside)
spinning lab ^d	spot on bag	EX	93.60	motor oil
spinning lab ^d	ground dust	FP	95.22	hull
upland field ^e	brown hull	EX	94.03	hull (outside)
upland field ^e	yellow shale	FP	95.92	shale (inside)
upland field ^e	brown bract	FP	93.29	bract
upland field ^e	gray bract	EX	94.32	stem (outside)
		FP	93.29	bract
upland field ^e	yellow hull	FP	96.55	hull (inside)
upland field ^e	brown stem	FP	94.21	stem (outside)
upland field ^e	seed coat (outside)	FP	96.30	seed coat (outside)
upland field ^e	seed coat (inside)	FP	96.10	seed coat (inside)
upland field ^e	seed meat	FP	95.28	seed meat
Pima field ^e	stem	FP	94.21	stem (outside)
Pima field ^e	seed coat	FP	92.33	seed coat (outside)

^a FP, fingerprint region (1800–650 cm⁻¹); EX, extended region (3700–2700, 1800–650 cm⁻¹). ^b Textile manufacturing plant (name withheld). ^c USDA-ARS, Southwestern Cotton Ginning Research Laboratory, Mesilla Park, NM. ^d USDA-ARS, Cotton Quality Research Unit, Clemson, SC. ^e 2005 cotton crop, Mesilla Park, NM.

spectral region that was used for the search of the spectral database, the match quality to the database, and the resulting identification based on the best match. It is critical to use the appropriate computational method available to search the database, as it has a direct impact on the ability to discriminate these materials, since many samples are closely related. It was found that, of the generally available methods, the “correlation” method in the Omnic software gave the best results. Also, choosing to search the fingerprint region of the spectrum (1800–650 cm⁻¹) generally gave the higher matches. However, for the spectra of samples where the fingerprint region was weak or showed few features, the addition X–H stretch region of the spectrum (3700–2700 cm⁻¹) to the search increased the match quality to the database library. The region from 2700 to 1800 cm⁻¹, in both region searches, was avoided, because diamond absorbance bands occur in this region and no samples were found to display bands in this region. In all cases, both regions, designated FP and EX, were interrogated and the highest match result was reported. Only matches above 90 were considered to be viable.

All of the matches to samples included in **Table 1** were correct, except for one. This one was for the gray (aged) bract from upland cotton from the 2005 crop year. The highest match in this case was to stem material, and the second highest was to bract. Both of these matches are shown as they were separated by only one match unit. The apparent reason for this result is that no gray bract was included in the database. Thus, this type of sample needs to be added in the future. A database of this nature needs to be constantly updated until it includes samples from many years and environmental conditions. It can also be helpful to develop variety-specific versions to obtain better matches, if the variety is known.

For the dust samples, only the highest match is reported in **Table 1**. However, lower matches (but above 90) should still be considered for these samples. Dust is usually composed of more than one type of foreign matter. In our previous work on

dust samples obtained from spinning equipment that has experienced various ginning conditions, the five top matches were considered as potential components of the sample (11, 15). Both of these studies involved the use of a beta version of this database to analyze dust that was collected from rotors in rotor-spinning equipment. Part of the first study (15) involved 12 lots of Pima cotton with 3 replications of 4 different ginning conditions. Under two ginning conditions, the cotton was spiked with hulls (prior to ginning). Analysis, using the infrared database, indicated that the dust contained various hull components from Pima cotton in all but the lowest values of the top five matches. The second study (11) was more extensive, involving 23 ginning conditions. It proved that dust that collected in the rotors of rotor spinning was, on a qualitative basis, composed of hull and shale components. If it is desired to gain an understanding of the quantitative composition of cotton dust, each individual dust particle must be examined in a statistically representative sample of dust. Although this could theoretically be accomplished using the database, it would represent an extensive undertaking. A qualitative idea of dust composition should be sufficient for most requirements. Use of the database was generally successful in distinguishing between upland and Pima cotton derived organic matter as being the source of the foreign matter. However, distinguishing varietal features is rather tenuous and could be suspect in samples that contain a mixture of varieties or substantially different varieties. The database, as developed, was found not to be useful in identifying cotton-derived organic matter from experimental cotton varieties but could be extended to cover such varieties.

The current form of this ATR/IR database includes 601 spectra of foreign matter that can potentially be present in cotton. All materials, except for metals, that are listed in the *ITMF Cotton Contamination Survey (1)* are included in the database.

In conclusion, although this type of infrared database needs to undergo continual update, until all types of cotton plant component spectra from multiple crop years and multiple varieties are included, this work has proven that it is a viable conceptual approach for the identification of foreign material in cotton that, so far, is unavailable by any other method of analysis. The method appears to be applicable all the way from the cotton field to the final manufactured textile product. It should be useful in suggesting modifications of conditions at the gin, the spinning mill, and the manufacturing plant to reduce the negative influence of foreign matter at each stage of cotton production. With portable ATR/IR devices becoming more available, this type of analysis can be accomplished in all locations and under most conditions, with the exception of outside in wet conditions.

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