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Short communication

Compositional features of cotton plant biomass fractions characterized by attenuated total reflection Fourier transform infrared spectroscopy

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ABSTRACT

Cotton is one of the most important and widely grown crops in the world. In addition to natural textile fiber production as a primary purpose, it yields a high grade vegetable oil for human consumption and also carbohydrate fiber and protein byproducts for animal feed. In this work, attenuated total reflection (ATR)-Fourier transform infrared (FTIR) spectroscopy was applied to characterize the cotton plant biomass fractions. Furthermore, principal component analysis (PCA) of these ATR-FTIR spectra were performed to evaluate the similarity or dissimilarity of these biomass fractions from different parts of cotton plants, and the same plant parts collected between mid-season and pre-defoliation stages. These data revealed the more rapid accumulation of major carbohydrate components in main stems, roots, and branches than in petioles and leaf blades, increased our understanding of the cotton growth mechanism. PCA revealed that the ATR-FTIR spectra of cotton biomass fractions fell into two distinct clusters representing two types of cotton plant parts: Stem cluster (main stems, roots, branches, and petioles) and Leaf cluster (leaf blades, bracts, bur, and/or reproductive parts). Such clustering information could be helpful in selecting the FT-IR measurement parameters for assessing cotton plant growth, nutrition management, and biomass production.

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1. Introduction

Cotton is one of the most important and widely grown crops in the world. It is a well-traded agricultural commodity mostly for textile fiber purpose, but it also yields a high grade vegetable oil from cottonseed for human consumption as well as cottonseed meal and whole seeds as animal feed (Bellaloui et al., 2015; He et al., 2013). Recently, studies have shown the potential of cottonseed and other cotton byproducts as industrial raw materials and soil amendments (He et al., 2014a, b; Wanjura et al., 2014). For this purpose, Wanjura et al. (2014) separated cotton crop biomass into four components [i.e., seedcotton (lint and seed), burs, sticks/stems, and other vegetative matter (OVM)]. They quantified and chemically characterized them in order to produce information that helps cotton producers and processors make decisions to maximize profitability through the use of un-harvested biomass resources.

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http://dx.doi.org/10.1016/j.indcrop.2015.11.022 0926-6690/Published by Elsevier B.V. Fourier transform infrared (FTIR) spectroscopy is a nondestructive instrumental technique. It has been widely used in agricultural research (Himmelsbach et al., 2006; Waldrip et al., 2014). With the aid of such multivariate data tool as principal component analysis (PCA), FTIR spectra are able to enhance small differences between two plant samples that could not be distinguished visually (Liu et al., 2015; 2012). One feature of PCA is to reveal the correlations between spectra (or samples) by their scores on new principal components (PCs). In the score–score plot, similar samples appear as clusters, whereas dissimilar samples tend to be separated from each other.

For increasing the understanding on the distinct characteristics among the different cotton biomass byproducts, in this work, we separated the whole cotton plant into six (mid-season) or nine (predefoliation, i.e., pre-harvest treatment) components and analyzed them by ATR-FTIR technique. We hypothesized that the fast, nondestructive, and structure-rich nature of FTIR spectroscopy make it a very attractive option to use for evaluating the structural features of these cotton biomass components, and revealing the structural difference between them. Such information would be helpful in







Wavenumber (cm⁻¹) Fig. 1. Mean of normalized ATR-FTIR spectra from different cotton plant parts representing main stems, leaf blades, branches, petioles, roots, reproductive, bur, bracts, cottonseeds, and cotton fibers. For the purpose of general comparison, each spectrum was the average of those from different varieties in mid-season and predefoliation stages.

1200

1800

1030

3280, OH/ NH str.

2924, CH₂ asym. str.

2850, CH₂ sym. str. 1745, C=O str.

1620, HOH bending

1235, OH/NH def.

Cotton fibers, R = 1.49

1030, C-O str.

Branches, R = 1.02

Petioles, R = 1.07

Roots, R = 1.00

Bracts, R = 1.19

Bur, R = 1.11

600

Main stems, R = 1.02

Leaf blades, R = 1.16

Reproductive, R = 1.09

Cottonseeds, R = 0.48

+ amide I

developing better management strategies for on- and off-field utilization of the cotton biomass. Information on the comparison of the FTIR features of the in-season and pre-harvest samples could be also useful in developing an ATR-FTIR procedure as an effective diagnostic tool in monitoring plant biosynthesis for cotton plant physiology and management practices, ultimately to help producers grow a high-yielding and high-quality crop. Thus, this study collected and compared the ATR-FTIR spectra of different biomass fractions (parts) of cotton plants at the mid-season and pre-harvest phases of cotton growth. PCA approach was applied to reveal the relations of the ATR-FTIR spectral features of these cotton biomass parts. The knowledge derived from this study would benefit the future implementation of ATR-FTIR technique for discriminating/monitoring the biomass conditions of cotton crop in a greenhouse or field environment for the purpose of best cropping management practices and beyond.

2. Materials and methods

Whole cotton plants including roots were taken from four cotton plots of a cropping management trials at the Mississippi Agricultural and Forest Experiment Station near Pontotoc, MS (34°8'30" N, 88°59'36" W) (Tewolde et al., 2015). The soil was an Atwood silt loam soil (fine-silty, mixed, semiactive, thermic Typic Paleudalfs) with \approx 5% slope and has been under no-till cultivation for a minimum of 8 years. Cotton plants that received conventional inorganic fertilizers according to recommendations of the Mississippi State University Extension Service soil testing laboratory were collected twice on 8 Aug. 2014 (mid-season) and on 19 Sept. 2014 (pre-defoliation). Cotton crop samples (i.e., randomly selected four to eight whole plants) were collected from each experimental plot (four 1.02 m wide rows with 21.3 length each plot) of four replications by digging and loosening the soil to recover as much of the roots as possible. The plants were placed in large plastic bags and stored in a walk-in cooler (~42 °C) until processed. The plants were then processed by first cutting the root from the shoot of each plant



Fig. 2. (A) Plot of PC2 versus PC1 scores from normalized ATR-FTIR spectra of cotton plant parts assigned to four clusters, Fiber (+), Stem (\blacktriangle), Leaf (\blacklozenge), and Seed (\bigcirc). (B) PC2 versus PC1 plot of those assigned to Stem (encircled) and Leaf (non-circled) clusters only between mid-season (in red, underline, and linked) and pre-defoliation stages (in black).

at soil level using hand clippers, washing the roots in tap water, and placing in paper bags. The remaining shoots were also rinsed with tap water in large plastic tubs, loaded into new clean plastic bags, and returned to the cooler allowing excess water to drain. Then, the shoots were separated into leaf blades, petioles, branches, main stems, and reproductive parts. The reproductive parts included the fruit, bracts, and the peduncle. The separated samples were placed in paper bags and dried in a forced-air oven at 80 °C to constant weight. Following drying, the reproductive samples taken at near maturity (pre-defoliation) were further separated to seedcotton, burs, and peduncle and bracts. The seedcotton was further separated into seed and lint by ginning using a small tabletop gin. The reproductive parts taken at mid-season were composed of squares or flower buds, flowers, and immature fruits and were not separated into their component parts. All samples but cotton fibers were ground and conditioned at a constant relative humidity of $65 \pm 2\%$ and temperature of $21 \pm 2 \circ C$ for at least 24 h, prior to ATR-FTIR spectral acquisition.

All spectra were collected with an FTS 3000MX FTIR spectrometer (Varian Instruments, Randolph, MA) equipped with a ceramic source, KBr beam splitter, and deuterated triglycine sulfate

3600

3000

3280 2924 2850

Cotton fibers

Branches

Main stems

Petioles

Roots

Bracts

Leaf blades

Bur

Reproductive

Cottonseeds

2400

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