Chemical Characterization of Cotton Plant Parts for Multiple Uses

Zhongqi He, Hailin Zhang,* Haile Tewolde, and Mark Shankle

Abstract: Cotton (*Gossypium hirsutum* L.) is an important crop in the southern and southeastern parts of the United States, but cotton plant biomass residues are underutilized because the high-value lint receives the most attention. In this study, whole cotton plants were collected at midseason and just before harvest and were chemically characterized to explore multiple uses. The plant samples were separated into six (midseason) or eight (pre-defoliation for harvest) biomass fractions. We determined the macro- and trace elements, protein, fiber, and lignin contents in the biomass materials. Growth stages affected the relative contents of some, but not all, of the measured parameters. Correlation coefficient analysis of the measured data revealed that some of the parameters were well related to each other, whereas some were quite independent. The information reported in this work will be helpful in exploring and optimizing management practices and processing strategies for best utilization of these types of cotton crop biomass materials as renewable natural resources.

Cotton (*Gossypium hirsutum* L.) is the United States’ number one high-value crop, with its primary production areas located in the southern and southeastern United States (He et al., 2013; Tewolde et al., 2015). Recently, studies have shown that cotton biomass materials other than the fiber are also useful as a soil amendment, animal feed, bioenergy sources, and industrial raw materials (He et al., 2014a, 2016; Holt et al., 2014; Ren et al., 2015; Wanjura et al., 2014). To identify the amounts of biomass available before and after seed cotton harvest, as well as the physical location of the material, Wanjura et al. (2014) measured the biomass yields from five collection groups: (i) pre-harvest standing stalk, (ii) pre-harvest material from the ground, (iii) post-harvest standing stalk, (iv) post-harvest material from the ground, and (v) material they referred to as “bur trail material” collected following harvesting using a stripper with an onboard field cleaner. Furthermore, they separated cotton crop biomass in each collection group into four components: seedcotton (lint and seed), burs, sticks/stems, and other vegetative matter. Analytical characterization of the individual biomass components indicated that stick and bur had properties more favorable for utilization as feedstock for biofuel or bio-based composite production, whereas other vegetative matter was better used as a soil amendment or animal feed ingredient.

Chemical composition is a critical parameter for assessing the product quality and exploring new uses of cotton plant biomass (He et al., 2013, 2014b). In this study, field-grown whole cotton plants collected at mid-season and just before defoliation were analyzed for chemical composition in roots, main stems, branches, petioles, leaf blades, and reproductive parts (burs, peduncles, and seeds). The objectives were (i) to document the chemical characteristics of the individual biomass components in terms of plant nutrients and animal feed quality and (ii) to improve the understanding of chemical ingredient accu-

**Core Ideas**

- Cotton plant parts can be used for multiple purposes depending on their compositions.
- Cotton stems with less ash are better suited for lignocellulosic feedstock of bioenergy and bio-products.
- Feed quality characteristics of cottonseed are comparable to those of forage crops.

**Abbreviations:** ADF, acid detergent fiber; ADL, acid detergent lignin; ATR-FTIR, attenuated total reflection Fourier transform infrared; NDF, neutral detergent fiber.
mulation over cotton plant growth and development. The information presented in this work will be helpful to the cotton industry in making decisions to maximize profitability through better use of cotton biomass resources.

Materials and Methods

Whole cotton plants were taken from cotton plots of a series of cropping management trials at the Mississippi Agricultural and Forest Experiment Station near Pontotoc, MS (34°48'30" N, 88°59'36" W) (Tewolde et al., 2015). As reported previously (Liu et al., 2016), a no-till cotton was grown in an Atwood silt loam soil (fine-silty, mixed, semiarboreal, Typic Paleudalfs) without irrigation. Four to eight whole plants were collected from each of four plots that received conventional inorganic fertilization according to regional recommendations. The samples were taken twice, once at midseason and repeated at pre-defoliation, by digging and loosen the soil to recover as much of the fine root material as possible, rinsed with tap water and separated into roots, leaf blades, petioles, branches, main stems, and reproductive parts. The reproductive parts included the boll (fruit), bracts, and 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 parts sampled at pre-defoliation were further separated into (i) cottonseed, (ii) burs, and (iii) peduncle/bracts. The cottonseed was further separated into seed and lint (fiber) by ginning using a small tabletop gin. The chemical composition of the cottonseed, (ii) burs, and (iii) peduncle/bracts. The cottonseed was further separated into seed and lint (fiber) by ginning using a small tabletop gin. The chemical composition of the reproductive parts did not include the data of lint. The reproductive parts included the boll (fruit), bracts, and 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 parts sampled at pre-defoliation were further separated into (i) cottonseed, (ii) burs, and (iii) peduncle/bracts. The cottonseed was further separated into seed and lint (fiber) by ginning using a small tabletop gin. The chemical composition of the reproductive parts did not include the data of lint. The reproductive parts taken at midseason were composed of squares (flower buds), flowers, and small immature fruits and were not separated into their component parts.

Concentrations of 10 elements were measured by a Spectro CirOs ICP spectrometer (He et al., 2013) after digesting 0.5-g ground biomass samples using the Environmental Express HotBlock digester in 10.0 mL of concentrated trace metal grade HNO₃. Total nitrogen (N) content of each sample was determined using a LECO Truspec dry combustion Carbon/Nitrogen Analyzer. The crude protein content in the samples was calculated by multiplying the total N by a factor of 6.25 (He et al., 2015). The acid detergent fiber (ADF), neutral detergent fiber (NDF), and acid detergent lignin (ADL) contents were determined using the filter bag methods with an Ankom Fiber Analyzer (Ankom Technology) (He et al., 2014b).

Results and Discussion

Within each of the six plant parts, the concentrations of calcium (Ca) and potassium (K) were the highest two of all measured macroelements, and sodium (Na) concentration was the lowest (Fig. 1). Generally, the contents of macroelements in different parts of biomass harvested in the midseason were in the order leaf blades > petioles > reproductive > branches > roots ≈ main stems. This order was also reflected in the ash contents of those parts. The order remained the same for plants collected at the end of the season (pre-defoliation), although the differences in the concentrations between the two growth phases were inconsistent among these six macroelements. The concentration of K, for example, decreased in five of six biomass parts as the plants grew older. On the other hand, the concentration of Ca in petioles and leaf blades increased over the growing stage. The distribution of the four trace elements basically followed the same trend of macroelements, in the order leaf blades > petioles > reproductive > branches > roots ≈ main stems. However, roots showed by far greater iron (Fe) levels than the other parts, which became even greater as plants matured. This is consistent with observations reported by Tewolde et al. (2005), who found that Fe accumulated in roots of potted cotton plants probably because of poor translocation to upper plant parts. Tewolde et al. (2005) found that supplying additional macroelements increased the translocation of Fe from roots to upper plant parts, but its concentration in roots remained very high and decreased rapidly with vertical movement to upper plant parts.

The pattern of protein content was similar to those of minerals. Protein content is highly related to the contents of sulfur (S), zinc (Zn), and manganese (Mn), with the correlation coefficients of 0.91, 0.90, and 0.65, respectively. This relation implies these elements could be part of the cotton protein structure in the form of protein S–S bond and metalloproteins.

The ADF, NDF, and ADL contents varied among the biomass parts in reverse order to those of protein and minerals (Table 1). For the midseason samples, for example, the ADF content was in the order main stems > branches > roots > petioles > reproductive > leave blades. As the crop matured, the contents of ADF, NDF, and ADL all increased in these biomass parts collected at pre-defoliation. The protein and fibers contents are typically used to evaluate the nutrition values of agricultural products and by-products (He et al., 2014b, 2015).

A previous study characterized the compositional features of these biomass parts by attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy (Liu et al., 2016). Principal component analysis of these ATR-FTIR data revealed that those biomass parts could be separated into two clusters: (i) stem cluster, including main stems, roots, branches, and petioles, and (ii) leaf cluster, including leaf blades and reproductive parts. The biomass parts in the first cluster served mainly the transportation functions, and the biomass parts in the second cluster seemed more involved in biological synthesis and storage functions. Thus, the mineral contents in the first cluster were generally low and constant, whereas their contents in the second cluster, where minerals are required for biological activities, were high. In contrast, ADF, NDF, and ADL could be regarded mainly as major structural components of these biomass parts so that their concentrations were high in the first four of the six plant parts. The cluster classification may provide some explanations on the distribution pattern and changes of these elements and compounds during growth. However, some exceptions were also observed, especially with petioles. This was probably because the petiole is the plant part between the first cluster and the second cluster.
Cotton stalk residues comprising stems, branches, burs, bracts, and peduncles are normally separated from the marketable commodity (i.e., cotton bolls) in the field. In the United States, the cotton plant that remains in the field after harvesting the cottonseed is frequently shredded and left in the field for soil conservation and health (Mitchell et al., 2012). In developing countries, the majority of cotton stalk is burnt in the field and the remainder used as cooking fuel (Windeatt et al., 2014). Even though a conservative estimate of 70% crop residue is required to remain in situ for soil health and nutrient recycling (Lindstrom, 1986), there are still considerable amounts of cotton crop residual biomass for value-added utilization, such as for biochar and biofuel production (Windeatt et al., 2014). While GIS-based biomass assessment has been used in the analysis of supply logistics for a sustainable biorefinery with cotton stalks in the southeastern United States (Sahoo et al., 2016), increased knowledge of chemical composition of cotton crop biomass is needed for optimal utilization of different biomass components. For example, low minerals and ash contents of biomass have been suggested to improve the operation conditions and the quality of biochar and bioenergy (Aquino et al., 2007; Rehrah et al., 2014). On the other hand, high lignocellulosic biomass was considered a preferred feedstock (Ali et al., 2015; Liu et al., 2014). Wanjura et al. (2014) thus proposed that stick and bur have properties more favorable as feedstock for biofuel or bio-based composite production, whereas other vegetative matter is better used as a soil amendment or animal feed ingredient.

This current study provides more information on the differential utilization of cotton plant biomass parts. Specifically, the leaf blade of cotton leaves contained high levels of protein (N) and ash minerals. This observation shows an extra benefit of defoliation practice prior to harvest: returning the N and mineral nutrients contained in leaf blades and petioles back to the soil. In addition to serving as feedstock for bioenergy, high-quality lignocellulosic biomass, stems and branches could also be suitable for fast pyrolysis to produce favorable biooil (He et al., 2016), or biomass liquefaction with phenolic reagents (Fidan et al., 2014). Many studies have suggested that defoliation increases the availability of nutrients for the remaining biomass (Lindstrom, 1986; Mitchell et al., 2012). This current study provides additional evidence that defoliation can increase the nutrient content of the remaining biomass, which could be beneficial for soil health and nutrient recycling.

Fig. 1. Concentrations of (A) macroelements, (B) trace elements, and (C) ash and feed quality characteristics of cotton plant biomass at two growth stages. The contribution of lint (fiber) is not included in the data of reproductive parts. Data are averages of four replicates, with vertical bars showing SD values ($n = 4$). ADF, acid detergent fiber; NDF, neutral detergent fiber; ADL, acid detergent lignin.
2010). Such bio-oil and liquefied biomass can be used in bio-based wood adhesives (Mao et al., 2017; Wan et al., 2017). Chemically, petioles fall in the transition zone between leaf blades and stems including branches. Morphologically, petioles resemble leaf blades more than stems or branches. When defoliants are applied to prepare the crop for harvest, the petiole along with the leaf blade separates from the stem and branches and falls off to the ground. Due to the cost of harvest and separation, petioles should be left as part of the leaf blade that return to the soil. If harvested together with the main stems and branches for biochar production, the produced biochar (with higher ash and mineral elements) could be more suitable for soil improvement than as absorbents for other environmental applications.

The reproductive parts collected in the later growth stage can be further separated into fiber, burs, peduncles/bracts, and seed. The elemental contents of bur, peduncles/bracts, and seed (Fig. 2) were not equally distributed in the three seed parts as the highest ash contents were with peduncles/bracts, equivalent to that of petioles. High fiber but low protein contents were found in bur and peduncles/bracts so that the two parts may be used for biogas, biochars, and other cellulosic feedstock (Demirbaş, 2001; Sutivisedsak et al., 2012). On the other hand, cottonseed was high in protein

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*, **, *** Significant at P = 0.05, 0.01, and 0.001, respectively.
† ADF, acid detergent fiber; NDF, neutral detergent fiber; ADL, acid detergent lignin.

Fig. 2. Concentrations of selected elements, ash, and nutritive values in cotton reproductive parts collected pre-defoliation. Data are averages of four replicates, with vertical bars showing SD values (n = 4).
content (25%) and moderate in fiber contents (ADF 30% and NDF 43%). Protein and fiber contents are normally used for evaluating the feed nutritive value of agricultural products and by-products (He et al., 2014b, 2015). Yari et al. (2012) reported that the protein, ADF, and NDF contents in alfalfa are at about 22, 33, and 43%, respectively. In comparison, Hymes-Fecht et al. (2013) reported the protein, ADF, and NDF contents with respective averages of 22, 27, and 35% in alfalfa (Medicago sativa L.) and 18, 28, and 43% in red clover (Trifolium pratense L.). Thus, cotton seed has feed quality characteristics comparable to those observed in legume forage crops. It is evident, then, that cotton plant parts can be used for multiple uses depending on the chemical compositions of each part.

References


