Carbon concentration predicts soil contamination of plant residues

Alan J. Franzluebbers

USDA, ARS, NCSU Campus Box 7620, Raleigh, NC 27695, USA

Correspondence
Alan J. Franzluebbers, USDA-ARS, NCSU Campus Box 7620, Raleigh, NC 27695, USA. Email: Alan.Franzluebbers@usda.gov

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Abstract
Dry matter estimations of field-harvested plant materials can be complicated by varying degrees of soil contamination. A quick and simple method of screening materials for potential soil contamination would avoid unnecessary analyses. Harvested forage biomass and soil-surface crop residues were collected from cool-season perennial pastures, a silvopasture, and no-tillage cropland to determine ash concentration. Samples were also analyzed for total carbon (C) and nitrogen (N) concentrations as part of routine plant analysis. When assessing ash and C concentrations along a gradient of soil contamination among 260 samples, a common regression was developed that had <1% coefficient of variation \[ \text{Ash (g kg}^{-1}\) = 999 - 1.89 \times C (g kg}^{-1}), r^2 = .99, p < .001\]. This association was consistent with available data in the literature, which also showed a negative relationship between ash and C concentrations of organically derived materials. Soil contamination of plant biomass and surface residues can be effectively estimated with this regression approach, allowing for more accurate organic matter estimates.

1 INTRODUCTION

Conservation agricultural systems retain more carbon (C) produced by plants at the soil surface (Ranaivoson et al., 2017). Estimating C inputs to soil requires both plant biomass production and transformations of plant-produced C into various decomposition states at the soil surface. Plant materials may undergo varying degrees of decomposition, and this process often leads to contamination of plant residues with soil that, if not accounted for, would overestimate plant biomass inputs. Accounting for soil contamination typically requires determination of ash concentration of the sample in question (Steiner, Schomberg, Unger, & Cresap, 1999). However, a simpler and more efficient method of determining soil contamination when routinely determining C concentration of plant materials might be possible given the relatively uniform and stable C concentration of plant materials, i.e., interquartile range of 447–495 g C kg}^{-1} dry matter (DM) among more than 2,000 analyses of different plant sources (Ma et al., 2018). Any reduction of C concentration from \(~450 \text{ g kg}^{-1}\) would, therefore, indicate potential soil contamination (Gale & Cam bardella, 2000). This suggests that biomass correction of soil-contaminated plant material is feasible when C concentration of uncontaminated material is constant and the relationship between ash and C concentrations is known. However, this association appears not to have been tested in the literature. Defining such a relationship could assist researchers by giving them a method to make adjustments to biomass and surface residue estimations with routine plant analysis of C and nitrogen (N) concentrations. The objective of this study was to test if a common association occurred between ash and carbon concentrations of plant materials.
C concentrations among different agricultural plant and soil-surface residue components.

2 MATERIALS AND METHODS

Plant samples were collected from (a) tall fescue [Schiedonorus arundinaceus (Schreb.) Dumort.] forage from an on-farm no-tillage cropping study, (b) surface residues from an on-farm no-tillage cropping study, and (c) surface residues from a silvopasture study. Plant material was suspected to have some soil contamination due to direct contact with surface soil in the field and/or collection method.

In the pasture study, tall fescue forage was collected in December 2018 to January 2019 with a rear-bag mower at >10-cm height (n = 17 samples) and 5-to-10-cm height (n = 21 samples) from locations in Georgia, North Carolina, South Carolina, and Virginia. Samples were a subset of those reported in Franzluebbers and Poore (2020).

In the no-tillage cropping study, surface residue was collected in May–June 2016 from three fields in North Carolina. Surface residue was collected from within a 30-cm-diam. ring; this was repeated at eight locations separated by ~2 m in a transect 3° diagonal to previous crop rows to form a composite sample. Crop and cover crop residues at the soil surface were cut at ground level if attached or simply picked up by hand if detached and placed into a labeled paper bag. Scraping the soil surface by hand to collect all residues meant that some mineral soil would have been attached. Twelve samples were collected from each of the three fields, except one field, which had 19 samples collected. A field in Stanly County, NC, was managed with long-term no-tillage with corn (Zea mays L.) as the previous crop and a history of single-species cover crop use. Six samples were collected from strips without cover crop, and six samples were collected from strips with recent multispecies cover crop (crimson clover [Triticum incarnatum L.], radish [Raphanus sativus L.], triticale [×Triticosecale Wittm. ex A. Camus], and ryegrass [Lolium multiflorum Lam.]). A field in Edgecombe County, NC, was recently managed with no tillage, had soybean [Glycine max (L.) Merr.] as a previous crop, and had rye (Secale cereale L.) cover crop in two strips and multispecies cover crop in two strips from which six samples each were collected. A field in Nash County, NC, was previously tilled with soybean as previous crop and had no history of cover cropping but had strips of no cover, rye cover crop, and multispecies cover crop (turnip [Brassica rapa L.], pea [Pisum sativum L.], hairy vetch [Vicia villosa Roth], radish, triticale, crimson clover, black oat [Avena strigosa Schreb.], and rye) from which six to seven samples were collected.

In the silvopasture study, surface residue was collected in December 2015 from a total of 180 individually placed 30-cm-diameter rings under both trees and pasture at the North Carolina Department of Agriculture Cherry Research Farm in Wayne County, NC. Twelve samples were taken, separated by 1.7 m, in each of 15 transects from under one line of trees across a grass alley to another line of trees. The intervening grass pasture was a mixture of big bluestem (Andropogon gerardii Vitman), switchgrass (Panicum virgatum L.), indiangrass [Sorghastrum nutans (L.) Nash], and eastern gamagrass (Tripsacum dactyloides L.). Trees were longleaf pine (Pinus palustris Mill.), loblolly pine (P. taeda L.), and cherrybark oak (Quercus pagoda Raf.) planted in 2007 (9yr prior to sampling) in five replications. Location and management details can be found elsewhere (Franzluebbers, Chappell, Shi, & Cubbage, 2017).

All samples were dried at 55 °C until constant mass (≥3 d) and ground to <1 mm prior to analyses. Surface residue samples were initially chopped coarsely into ~2-cm pieces with an electric chipper (Durostar ES1600, 14-amp), representatively subsampled, and then ground further to pass a 1-mm screen with a cyclone mill (Udy Corp.). At each subsampling step, samples were thoroughly mixed to obtain a representative sample. Prior to analyses, samples were again dried at 55 °C overnight. Samples were analyzed for C and N concentration from 0.5-to-1-g subsamples on a Leco TruMac CN combustion analyzer (Leco Corp.). A 5-g (± 0.0500 g) subsample was weighed in a 50-ml beaker and combusted in a Muffle furnace at 500 °C for 12 h (Model F-A1740, Thermolyne). After cooling in a desicator, mass of ash remaining was determined. Fraction of original dry mass as ash was calculated.

Data were subjected to regression analysis with SigmaPlot v. 14 (Systat Software Inc.). Regressions from individual sets of data (i.e., cropland, silvopasture, and pasture) were tested for significance of difference using SAS v. 9.4 (SAS Institute Inc.). Differences were declared at p < .05. Additional data of biomass C and ash concentrations were gleaned from the literature and plotted in a similar manner.

Core Ideas

- Soil contamination can be predicted from total C concentration of a plant sample.
- Ash and C concentrations were negatively associated across a diversity of sample types.
- Little practical difference existed among plant sources in the association between ash and C concentrations.
- Plant biomass can be corrected for soil contamination knowing C concentration.
3 | RESULTS AND DISCUSSION

Tall fescue forage had C concentration of 459 ± 29 g kg\(^{-1}\) DM. This was not a large range of variation but led to ash concentration of 109 ± 41 g kg\(^{-1}\), or coefficient of variation of 38\%. Adjusting nutritive value of forage on an organic matter basis (i.e., ash-free) is commonplace and allows for better estimations of true nutritive value (Arthington & Brown, 2005). If ash were 150 g kg\(^{-1}\) and estimate of forage mass were 1,000 kg ha\(^{-1}\), ash-free forage mass would be 850 kg ha\(^{-1}\).

Ash concentration of forage at ≥10-cm height (101 ± 32 g kg\(^{-1}\)) was not different from ash concentration at 5-to-10-cm cutting height (115 ± 46 g kg\(^{-1}\)), suggesting little evidence overall of soil contamination. Across 2,022 leaf samples of different plants in China, the interquartile range of ash concentration was 57–142 g kg\(^{-1}\) (Han et al., 2012). Main mineral constituents are N, Si, Cl, K, and Ca (Monti, Di Virgilio, & Venturi, 2008). Clean plant biomass samples can vary in ash concentration (i.e., mineral composition and concentration) with plant age, part, and cultural treatment (Humphries, 1956).

Surface residue C concentration with and without cover cropping in no-tillage cropland was 375 ± 70 g kg\(^{-1}\). Ash concentration was elevated at 302 ± 130 g kg\(^{-1}\), indicating significant soil contamination compared with an expected ash concentration of uncontaminated organic residues of ~100 g kg\(^{-1}\) (Gordon, 1989). Uncorrected surface residue mass estimates would have been 6.6 ± 3.3 Mg ha\(^{-1}\), but ash-free surface residue mass would have been 4.8 ± 2.8 Mg ha\(^{-1}\). A standardized 30% reduction with average ash fraction would have been misleading too, as individual samples had ash concentration ranging from 120 to 600 g kg\(^{-1}\). Ash concentration of surface residues varied within fields due to cover crop management (data not shown). Across all three fields, there was a significant negative association between ash concentration of surface residues and ash-free surface residue mass (\(r = -0.63\), \(p < .001\)). This suggests that the proportion of a sample contaminated with soil was greater when surface residue mass was low.

Surface residue C concentration in the silvopasture was 256 ± 87 g kg\(^{-1}\). Ash concentration was elevated to 516 ± 163 g kg\(^{-1}\), suggesting large magnitude and extent of variation of soil contamination. As with the cropland study, ash concentration of surface residues in the silvopasture was negatively associated with ash-free surface residue mass (\(r = -0.56\), \(p < .001\)). Ash concentration of surface residues was greater in the grass alleys of this experiment than under trees (559 vs. 474 g kg\(^{-1}\), respectively; \(p < .001\)). Ash-free surface residue mass was lower under \(Q.\) pagoda than under \(P.\) spp. (6.6 vs. 15.6 Mg ha\(^{-1}\), respectively; \(p < .001\)). Ash concentration of surface residues under perennial grass established with no-till was lower than when established with disk tillage prior to seeding (456 vs. 627 g kg\(^{-1}\), respectively, \(p < .001\)), despite no significant difference in ash-free surface residue mass between establishment methods (4.1 Mg ha\(^{-1}\)).

Association between C and ash concentrations of all plant samples (i.e., forage biomass and surface residue) was strongly negative (Figure 1a). A common regression among all types of samples was calculated, and the coefficient of standard error from the regression slope was <1\%. Despite this close association, and perhaps also due to it, regressions among the three studies were statistically different. Regression slope from forage biomass in tall fescue pastures was significantly lower than that of surface residues from cropland with cover crops and silvopasture (−1.91 vs. −1.84 g ash g\(^{-1}\) C, respectively, \(p < 0.001\)). Regression slopes between surface residues from cropland and silvopasture were not different. It seems likely that the difference in regression slope was a function of the different ranges of C concentration in each study; forage biomass had a narrow range of 387–505 g kg\(^{-1}\) compared with 99–462 g kg\(^{-1}\) for surface residues in the other two studies. The overall relationship among all three sources of plant materials does not suggest there should be a
difference between fresh plant residues (i.e., cut directly from living plants in tall fescue forage) and aged plant residues (i.e., surface residues that have undergone some microbial decomposition). Some of the surface residue samples in the cover crop treatments would have also been relatively fresh plant residues with little exposure to microbial decomposition prior to sampling.

Results from this study were compared with those available in the literature. A rich literature exists on elemental composition of organic materials due to the efforts to characterize heating value of raw, processed, and waste materials for energy sources. Therefore, data were compiled from several sources, including wood and food processing residues (Parikh, Channiwala, & Ghosal, 2005, 2007), grain harvest residuals, food and wood processing residuals, and straw (Shen, Zhu, Liu, Zhang, & Tan, 2010), sewage sludges in Thailand (Thipkhunthod et al., 2005), and paper products, food wastes, trees, and solid waste incinerator composites (Meraz, Dominguez, Kornhauser, & Rojas, 2003). Ash and C concentrations of these organic materials were also highly associated (Figure 1b). Results followed a very similar pattern as from the forage and surface residue samples in Figure 1a. These literature values had more variation in the overall regression, but this could be expected from the diversity of organic materials and analytical laboratories involved. Therefore, the common regression approach in Figure 1a is supported by existing literature.

From suspected soil-contaminated surface residues (or any plant biomass sampling, such as forage harvested near ground level with significant soil splashing), the regression equation in Figure 1a can be applied directly to remove soil mass from the biomass estimate in one of two ways. To calculate on an ash-free basis, a mildly contaminated sample with 400 g C kg⁻¹ DM and uncorrected mass of 5.0 Mg ha⁻¹ would be adjusted to an ash-free DM of 3.8 Mg ha⁻¹ (5.0 × 400 × 1.89/999). A highly contaminated sample with 200 g C kg⁻¹ DM and uncorrected mass of 1.5 Mg ha⁻¹ would be adjusted to an ash-free DM of 1.0 Mg ha⁻¹ [1.5 × (200 × 1.89 +100)/999].

Results from this study suggest that either determination of ash or C concentration is necessary to estimate soil contamination of plant biomass, but both are not needed. A relatively low-cost and simple method of obtaining ash is from the traditional use of heating to 500–600 °C in a muffle furnace for several hours, although standardization of the method is important to accurately estimate ash (Matthiessen, Larney, Selinger, & Olson, 2005). This study showed that ash concentration can be effectively quantified from plant biomass and surface residue samples even with significant soil contamination using routine determination of C and N concentrations with dry combustion (Figure 1a). Although this method may be more expensive and require greater technical expertise, having this capability essentially allows a single processing of C and N concentrations and prediction of ash concentration based on the inverse relationship with C concentration.

4 | CONCLUSIONS

Prediction of ash concentration from C concentration of plant samples, especially those considered dirty or contaminated with soil, is possible. Therefore, a correction factor for soil contamination can be applied, simply knowing the C concentration of the sample. Knowing this association will lead to more and better estimations of surface residue mass with conservation agricultural management. Carbon concentration of surface residues and plant biomass <450 g kg⁻¹ would be an indicator of potential soil contamination. A strong negative association between C and ash concentrations resulted in a highly predictive relationship that could adjust estimates of contaminated residues to true biomass. Surface residue mass estimations are important for quantifying the impacts of management on soil C sequestration, nutrient cycling, soil water retention, biological habitat, greenhouse gas emissions, and off-site water quality.

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CONFLICT OF INTEREST

The author declares no conflict of interest.

ORCID

Alan J. Franzluebbers https://orcid.org/0000-0003-0739-0913
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