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Short Communication

Refinement of the carbon to loss on ignition relationship in forest soils

Grady J. Boyle[a], **Evan S. Kane**[a,b], **Lucas E. Nave**[a], **Katherine A. Heckman**[b], **and Martin F. Jurgensen**[a]

[a]Michigan Technological University, College of Forest Resources and Environmental Science, Houghton, MI, USA; [b]USDA Forest Service, Northern Research Station, Houghton, MI, USA

Corresponding author: **Evan S. Kane** (email: eskane@mtu.edu)

Abstract

Loss on ignition (LOI) is a common method for determining organic matter in soils. Mineral soil organic carbon content has been shown to be approximately 50%–58% of the organic matter in many soils, but the carbon fraction of LOI can actually be much lower—particularly in Spodosols. We leveraged data available in the International Soil Carbon and National Ecological Observatory Networks to evaluate departures from the common 50% rule. We offer empirical equations that more accurately predict C:LOI with depth and genetic horizon in Spodosols. These equations enable more accurate soil C stock appraisals than using assumed C:LOI ratios.

Key words: organic matter, soil carbon, International Soil Carbon Network, Podzol, Spodosol, Inceptisol

Introduction

Managing forests for carbon (C) storage is gaining traction as a means for C offset programs (Domke et al. 2017). In temperate forest ecosystems, the majority of C is located in soil pools (Grigal and Ohmann 1992). A common means for estimating soil organic C (SOC) concentration is through the loss on ignition (LOI) method, as a proxy for soil organic matter concentration (Hoogsteen et al. 2015). LOI is an attractive method owing to being relatively inexpensive compared to elemental analysis. The estimation of SOC from LOI, based on a generalized ratio between the two metrics, has long been employed, with a value of 0.58 (referred to as the "van Bemmelen factor") being historically prevalent and values closer to 0.50 being deemed more accurate in more recent study (see Pribyl 2010, and references therein).

While the conversion factor of 0.58 has worked well in a large variety of soil types, evidence suggests that it may be overpredicting SOC in some acidic forest soils, such as Spodosols (or Podzols) (Huntington et al. 1989; Pribyl 2010). In their study of forest soils in New Hampshire, Huntington et al. (1989) concluded that conversion factors based on depth may be more accurate. However, it is not known whether these observed changes in SOC:SOM with depth in Spodosol profiles are localized or if they can be generalized throughout this important forest soil type.

Spodosols are of limited extent in North America (~3.5% of United States land area), but they are economically and ecologically important due to their extensive forest cover. The effects of forest harvesting on soil carbon are, in general, neutral to slightly negative depending on depth in the profile. Spodosols are highly leached and defined by illuvial (spodic) horizons with accumulations of organometallic complexes beneath eluvial horizons (Schaetzl and Thompson 2015). The accumulation of partially oxidized metals (sesquioxides) in spodic soil horizons would suggest a lower relative amount of C as a proportion of the total organic matter, as dissolved organic matter forms complexes with illuvial iron and aluminum (see, for example, chelate-complex model (Schaetzl and Thompson 2015)). As such, it is likely that studies employing a C:LOI conversion factor of 0.58 to quantify SOC stocks in Spodosols are overestimating soil C concentrations and pools, which could hinder attempts to determine effects of timber harvesting on SOC in this soil type (cf., Nave et al. 2021).

In this study, we investigated soil properties that may influence SOC:SOM ratios by using available data in the International Soil Carbon Network (ISCN) database (cf. Domke et al. 2017) and the National Ecological Observatory Network (NEON) (Nave et al. 2021) to compare results from forested Spodosols to those of Inceptisols (or Brunisols or Cambisols), which do not exhibit strong eluviation or illuviation. We focused on these two Orders as they are both well represented in forested sites in the ISCN, while also exhibiting very different mechanisms for pedogenesis. We also examined the relationship of SOC:SOM, based on LOI, to soil physical properties (texture and depth) by genetic horizon. A deviation from traditional conversion factors for LOI to SOC has been noted in prior research in Spodosols, and the goal of this study was to identify whether these trends noted in case studies transmit across the landscape. Should this trend exist in our dataset, we aimed to identify whether it was a consistent trend based on depth and or horizon that can be utilized to establish

Table 1. Coefficients and fit statistics for negative exponential relationship predicting the C:LOI ratio from depth to horizon (cm) for Spodosols and Inceptisols.

Model	Model *n*	Mean C:LOI	CI	Equation	Slope SE	Intercept SE	R^2	*P*-value
Spodosol								
All horizons	479	0.52*	0.013	$y = 0.5616e^{-0.017x}$	<0.001	0.015	0.531	**<0.001**
Mineral horizons	113	0.38*	0.010	$y = 0.4852e^{-0.015x}$	0.001	0.043	0.563	**<0.001**
O	367	0.57	0.010	$y = 0.5587e^{0.0013x}$	0.002	0.011	<0.001	0.591
A	33	0.55*	0.049	$y = 0.5882e^{-0.067x}$	0.019	0.055	0.287	**0.001**
E	13	0.44	0.077	$y = 0.4572e^{-0.006x}$	0.004	0.096	0.179	0.149
B	47	0.31*	0.023	$y = 0.3503e^{-0.006x}$	0.003	0.082	0.096	**0.033**
BC/C	19	0.21*	0.039	$y = 0.4267e^{-0.013x}$	0.005	0.316	0.316	**0.012**
Inceptisol								
All horizons	722	0.53*	0.003	$y = 0.5301e^{-0.01x}$	<0.001	0.015	0.133	**<0.001**
Mineral horizons	110	0.41*	0.010	$y = 0.4617e^{-0.013x}$	0.003	0.085	0.183	**<0.001**
O	612	0.55	0.009	$y = 0.531e^{0.0014x}$	0.001	0.01	0.003	0.176
A	55	0.44	0.070	$y = 0.4772e^{0.0062x}$	0.005	0.05	0.034	0.178
E	4	0.52	0.033	$y = 0.4353e^{-4E-04x}$	0.01	0.179	0.001	0.97
B	44	0.30	0.020	$y = 0.3149e^{-0.006x}$	0.005	0.217	0.032	0.242
BC/C	7	0.28	0.198	$y = 0.0473e^{0.0158x}$	0.025	1.88	0.074	0.55

Note: The models comprised of all mineral soil horizons are as depicted in Fig. 1. Statistically significant ($P < 0.05$) models for mean C:LOI are designated by an *. CI represents the confidence interval about the mean C:LOI value. Standard Errors (SE) are provided for slope and intercept coefficients.

a more effective conversion factor. Specifically, we assessed empirical models correcting for this departure based on soil physical properties, leveraging the full extent of available forested Spodosol and Inceptisol data in the ISCN and NEON databases.

Materials and methods

Dataset

For our analysis we extracted data for individual horizons of forested Spodosols and Inceptisols from the ISCN Version 3.0 database (Domke et al. 2017). In addition, five forested Spodosol and five forested Inceptisol profiles were included from NEON (Nave et al. 2021). We removed any datapoints that did not include total C (carbon from elemental analysis), LOI, or soil depth values. We then further trimmed the dataset by removing all datapoints that occurred outside of two standard deviations of the mean C:LOI ratio (<5% of the total data). Some of the genetic horizon types that were excluded were buried organic horizons and sediment (L horizon). The final dataset consisted of 1201 horizons from 738 different soil profiles across 32 different states (USA) and Puerto Rico.

Statistical analysis

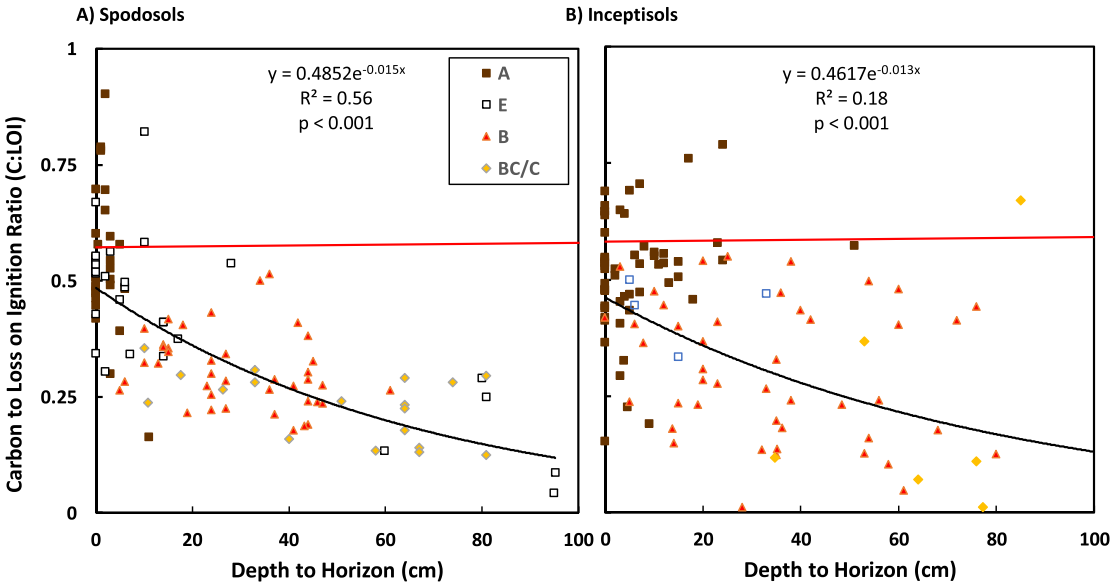
We used a general linear mixed model approach, which enables statistical models to be fit to data where the response is not necessarily normally distributed (PROC GLIMMIX; SAS version 9.4, SAS Institute, Cary, North Carolina, USA). We used the Univariate Procedure to perform a Kolmogorov–Smirnov test to analyze the distributions of the response variables. We then assigned the appropriate data distribution to satisfy normality in the mixed effect model (with distribution (dist) assigned as normal; link = Gaussian function). We considered Type-3 tests of fixed effects significant at $\alpha = 0.05$. We examined the effects of depth, Order, horizon, pH, and textural attributes on C:LOI in the mixed effect models. Depth to horizon and horizon type were the most available datasets, and given their importance in the general linear modelling approach we leaned on these variables for empirical models. We determined C:LOI versus soil depth trends using the Excel exponential trendlines and regression data analysis tool. We used these empirical models to evaluate C concentrations measured with elemental analysis (as reported in the ISCN and NEON).

Results

The main effect of pH was significant in predicting C:LOI when included in the general linear mixed model ($F = 4.82$, $P = 0.03$), but exhibited interactions with soil Order ($F = 5.51$, $P = 0.02$), suggesting possible variation explained by this factor was autocorrelated with Order. Texture (clay content) was not significant in the model ($F = 0.53$, $P = 0.47$). We found significant main effects on C:LOI by Order ($F = 9.40$, $P = 0.002$) and genetic horizon type ($F = 14.05$, $P < 0.001$). We found that depth and Order had a strong interaction in explaining C:LOI ($F = 12.95$, $P < 0.001$), and there was an interaction among depth, Order, and genetic horizon type ($F = 4.86$, $P < 0.001$). Based on these model results, we built empirical equations predicting C:LOI by depth and genetic horizon for Spodosols and Inceptisols (Table 1).

In Spodosols, the spodic horizon and the transition to parent material (BC horizon) exhibited the lowest mean C:LOI (Table 1). The relationship between depth in the mineral soil profile and C:LOI was best fit by an exponential equation ($R^2 = 0.56$; $P < 0.001$) (Table 1; Fig. 1). Comparing the soil horizon depth-based equation for Spodosols with the conven-

Fig. 1. Carbon (C) % to loss on ignition (LOI) ratios modeled by order and depth in the mineral soil. The red line represents the commonly assumed ratio of 0.58; values below this line represent cases of overestimation of C by using the assumed ratio, which is particularly true in illuvial horizons (B; BC).



tional conversion factor of 0.58 (Fig. 1*a*) showed this factor tended to overestimate C content in deeper illuvial soil horizons (B, BC) while underestimating SOC in some shallower horizons (O, A, and E). Inceptisols also exhibited a similar, but weaker, trend between C:LOI and depth (Fig. 1*b*). This trend was only evident across all mineral soil horizons, and was not evident for any specific genetic horizon in Inceptisols (Table 1).

Discussion

Our findings suggest that the conventional C:LOI conversion factors fail to accurately predict C content in Spodosols, with significant overestimation occurring in the illuvial horizons. Our results show a similar trend to those found in earlier case studies (e.g., Huntington et al. 1989), and suggest that these patterns are more broadly generalizable to forested Spodosols as identified across 12 different states (USA) in the ISCN and NEON databases. Additionally, our horizons follow a similar pattern of decreasing C:LOI ratio from the organic horizons to the parent material, only breaking for an increase in the E horizon. This creates an overall downward trend where horizons transition from underestimation to overestimation after the eluvial horizon. Based on these findings, we suggest that processes involved in the translocation of organometallic complexes associated with spodic horizons are a likely cause of the departure from conventional conversion factors.

Shortcomings with the LOI method are likely associated with the loss of masses other than organic matter with heating. The application of LOI to understand the thermal stability of minerals and mineral-associated organic matter has an extensive history of use in earth science (see review by Plante et al. 2009). In particular, mineralogists have studied the thermal stability of iron and aluminum oxyhydroxides (e.g., ferrihydrite, goethite, allophane, imogolite, and gibbsite), and have observed mass loss associated with the release of structural water with heating. In general, the poorly crystalline Fe- and Al-organometallic complexes associated with spodic horizons can have a high abundance of structural water and hydroxyl groups. Those waters and hydroxyl groups would be driven off at the temperatures used for LOI, converting the poorly crystalline forms of oxyhydroxides to more crystalline forms (see, for example, Karathanasis and Harris 1994). This mineral phase conversion is part of what causes the noticeable color change in samples combusted in LOI. While a detailed literature review characterizing the different chemical properties of Spodosol horizons was outside the scope of this Short Communication (see Schaetzl and Thompson 2015, for details), it is likely that the loss of structural water and hydroxyl groups is likely contributing to the observed low C to LOI in highly weathered, illuvial spodic horizons.

In contrast, Inceptisols have only slight profile development and exhibited a weak trend in declining C:LOI ratios with depth. The processes involved in creating spodic horizons through illuviation are not typically associated with Inceptisols. The implications of this analysis are (i) in more pedogenetically advanced soils, organic matter has a relatively lower C content in mineral horizons, relative to other elements (particularly Fe and Al), which means that (ii) depth and genetic horizon type should be taken into account when assuming the carbon content for a given amount of combustible organic matter.

We indicate that when examining highly weathered forest soils, using depth-corrected conversions will provide more accurate determination of C content (Table 1). For example, using the assumed "van Bemmelen factor" of 0.58 would likely overestimate the C:LOI ratio by 2.5 times at a depth of 50 cm

in Spodosols. We demonstrate overall trends by depth as well as trends by horizon, with the principal contribution of this work being refined equations for C:LOI in forested Spodosols. The significance of genetic horizons in our study re-emphasizes the importance of sampling by genetic horizon for soil C stock assessments.

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Author information

Author ORCIDs

Evan S. Kane https://orcid.org/0000-0003-1665-0596

Author contributions

Conceptualization: GJB, ESK, MFJ
Data curation: GJB, ESK, LEN
Formal analysis: GJB, ESK
Funding acquisition: ESK
Investigation: LEN, KAH, MFJ
Methodology: ESK, LEN
Validation: KAH
Writing – original draft: GJB, ESK
Writing – review & editing: GJB, ESK, LEN, KAH, MFJ

Competing interests

The authors declare there are no competing interests.

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