



Pyrogenic carbon distribution in mineral topsoils of the northeastern United States



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ARTICLE INFO

Article history:

Received 6 September 2016

Accepted 20 February 2017

Available online 2 March 2017

Keywords:

Pyrogenic carbon

Climate change

Fire

Soil development

Soil organic matter

Bayesian regression kriging

ABSTRACT

Due to its slow turnover rates in soil, pyrogenic carbon (PyC) is considered an important C pool and relevant to climate change processes. Therefore, the amounts of soil PyC were compared to environmental covariates over an area of 327,757 km² in the northeastern United States in order to understand the controls on PyC distribution over large areas. Topsoil (defined as the soil A horizon, after removal of any organic horizons) samples were collected at 165 field sites in a generalised random tessellation stratified design that corresponded to approximately 1 site per 1600 km² and PyC was estimated from diffuse reflectance mid-infrared spectroscopy measurements using a partial least-squares regression analysis in conjunction with a large database of PyC measurements based on a solid-state ¹³C nuclear magnetic resonance spectroscopy technique. Three spatial models were applied to the data in order to relate critical environmental covariates to the changes in spatial density of PyC over the landscape. Regional mean density estimates of PyC were 11.0 g kg⁻¹ (0.84 Gg km⁻²) for Ordinary Kriging, 25.8 g kg⁻¹ (12.2 Gg km⁻²) for Multivariate Linear Regression, and 26.1 g kg⁻¹ (12.4 Gg km⁻²) for Bayesian Regression Kriging. Akaike Information Criterion (AIC) indicated that the Multivariate Linear Regression model performed best (AIC = 842.6; n = 165) compared to Ordinary Kriging (AIC = 982.4) and Bayesian Regression Kriging (AIC = 979.2). Soil PyC concentrations correlated well with total soil sulphur (P < 0.001; n = 165), plant tissue lignin (P = 0.003), and drainage class (P = 0.008). This suggests the opportunity of including related environmental parameters in the spatial assessment of PyC in soils. Better estimates of the contribution of PyC to the global carbon cycle will thus also require more accurate assessments of these covariates.

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1. Introduction

Climate change has triggered an increasing interest in biogeochemical carbon (C) cycling and the question of how global change affects biotic processes. Recent studies suggest that the biosphere currently acts as a C sink (Schimel et al., 2001; Pan et al., 2011). However, the sink strength may decrease over time, turning the biosphere into a C source (IPCC, 2014). The largest uncertainty in predicting C turnover in the terrestrial biosphere is the soil (Tian et al., 2015), which stores at least three times as much C as either the atmosphere or terrestrial vegetation (Friedlingstein et al., 2006; Schmidt et al., 2011). Hence, soil organic C (SOC) is the main component of the terrestrial C cycle and accounts for annual carbon dioxide emissions that are an order of magnitude higher than all anthropogenic carbon dioxide emissions taken together

(IPCC, 2014). Decomposition of SOC by microorganisms is likely to intensify through global warming, augmenting the release of carbon dioxide into the atmosphere (Davidson and Janssens, 2006). If, however, a larger fraction of SOC were to demonstrate slower decomposition rates than currently assumed, the soil respiration-warming feedback may have been over estimated and current models of global climate change would need to be revised (Lehmann et al., 2008).

Slow-cycling SOC is either protected by minerals (organo-mineral interactions, adsorbed OC, contained in aggregates) or chemically altered with a highly aromatic structure and few oxygenated functional groups (pyrogenic carbon), which makes it a less preferred energy source for microbial decay (Preston and Schmidt, 2006; Schmidt et al., 2011).

While the formation of stabilized plant residues may take a multitude of pathways (Kleber et al., 2007), pyrogenic carbon (PyC) is produced by partial combustion of plant material and is a major component of a continuum from charcoal to soot to graphite (Kuhlbusch, 1998; Schmidt and Noack, 2000; Preston and Schmidt,

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2006). Although PyC can be degraded both chemically and biologically, it decomposes at a slow rate, with mean residence time in soils estimated from decades to millennia (Lehmann et al., 2015; Wang et al., 2015). Therefore, it mineralizes significantly slower than other litter input (Ansley et al., 2006), providing a greater potential for PyC to act as a significant C sink from the more rapid bio-atmospheric C cycle to the slower (long term) geological C cycle (Forbes et al., 2006; Ohlson and Tryterud, 2000). Skjemstad et al. (2002) found that PyC can constitute a significant proportion of SOC with up to 35% in several long-term experiments in the United States. Despite these findings of the importance of PyC, most recent C-related studies focus merely on non-PyC components and, therefore, neglect to address the long-term environmental significance of PyC stock changes in the global C cycle. Additionally, most available PyC data are collected as point data without attempting to correlate these measurements to other environmental properties of the surrounding landscape (Murage et al., 2007). Transformational processes and products, initially driven by climate and geomorphology, define the landscape's natural potential which influences ecosystem characteristics, for instance the capacity to act as a C sink or source (Blümel, 2009). In order to quantify this potential and upscale point measurements to the landscape scale, accurate information about the spatial distribution of PyC in soils of different ecosystems and the relationship to other environmental parameters are important to support projections of future climate change (Lehmann et al., 2008). Consequently, so as to better understand the importance of PyC in the global C cycle, an understanding of the spatial distribution of PyC is required (Bird et al., 2015; Reisser et al., 2016; Santín et al., 2016). To date, studies assessing spatial patterns of PyC in soils over large areas have been scarce, may combine PyC estimates from many different analyses protocols (Reisser et al., 2016) and often only focus on soot-derived PyC (Shaoda et al., 2011; Paroissien et al., 2012).

Therefore, the aim of this study was to assess the amount of PyC in topsoils of the northeastern United States, to determine the importance of related environmental parameters in the overall distribution of PyC on a landscape scale and to evaluate the performance of different spatial models in predicting PyC distribution over large areas. Specifically, the suitability of Ordinary Kriging, Multivariate Linear Regression and Bayesian Regression Kriging was examined with the goal to obtain the best model to depict and quantify spatial patterns of PyC distribution in the landscape.

2. Materials and methods

2.1. Study region

The sample sites are located in the northeastern United States, a part of the humid temperate zone, which globally covers 9.7% of the global terrestrial landmass. Mean annual temperature ranges from 8 °C to 12 °C and mean annual precipitation from 600 mm to 1000 mm. Rainfall is broadly distributed throughout the year. Temperate broadleaf and mixed forests comprise the predominant natural vegetation type. The organic layer consists of slightly acidic to slightly alkaline mull, which is rich in nutrients and carbon compared to the underlying mineral horizons. Both climate and vegetation control soil formation in this ecoregion and primarily lead to the development of dystic to eutric Cambisols, Luvisols and Podzoluvisols (Goudie, 2001; Woodward, 2003).

2.2. Sample collection and analysis

A composite of the soil A horizon (ca. 1 kg of the uppermost mineral soil, up to 0.1 m depth for the study region, after removal of the organic horizon if present) was collected using either soil profiles or augers at 165 sample sites in the six New England States and New York State (Supplementary Fig. S1) as part of the U.S. Geological Survey's North American Soil Geochemical Landscapes Project (Smith et al., 2012,

2013, 2014). No additional field information was collected at the time of soil sampling. Additional soil and site information was obtained from other databases (Supplementary Tables S1 and S2). Field sites were selected using a generalised random tessellation stratified design that corresponded to approximately 1 site per 1600 km² (Stevens and Olsen, 2004). This is a low sampling intensity compared to the processes that are likely responsible for soil PyC distribution, but is a typical sampling density available for large-scale spatial analyses of soils (e.g., for Africa 1 site per 1122 km², Hengl et al., 2015; for Europe 1 site per 199 km², Ballabio et al., 2016). Our analyses therefore reflect assessments of PyC spatial distribution, the ability to understand its drivers and the opportunities to improve spatial interpolation using co-variables for a data density typical of soil inventories.

The samples were air-dried, disaggregated and sieved to <2 mm. This material was then finely ground prior to chemical analysis (Smith et al., 2013). Adapting the method of Briggs (2002), a USGS contract laboratory determined total sulphur (S) concentration by a near-total four-acid (hydrochloric, nitric, hydrofluoric, and perchloric) digestion at a temperature between 125 and 150 °C followed by inductively coupled plasma-atomic emission spectrometry (ICP-OES Optima 5300/7300, Perkin Elmer Inc., Waltham, MA, USA).

A chemometric prediction method was used to estimate PyC contents whereby mid-infrared (MIR) spectra were correlated with large dataset of lab-produced PyC quantification from soils in Australia (Baldock et al., 2013a, 2013b). Finely ground and homogenized samples (Retsch Ball Mill, MM400, Haan, Germany) were analyzed by diffuse reflectance MIR spectroscopy using the identical spectroscope for the Australian calibration set and the US soils presented in this study. Spectra between 8000 and 400 cm⁻¹ were recorded with a Nicolet 6700 FTIR spectrometer (Thermo Fisher Scientific Inc., Waltham, MA, USA) equipped with a KBr beam-splitter, a DTGS detector and an AutoDiff-Automated diffuse reflectance accessory (Pike Technologies, Madison, WI, USA). Subsequent partial least squares (PLS) regression analysis of PyC was carried out with the Unscrambler 10.2 software package (CAMO software AS, Oslo, Norway). In this, a standard set of 312 Australian soils, previously analyzed for PyC using HF treatment followed by solid-state ¹³C nuclear magnetic resonance (NMR) spectroscopy, served as PLS calibration data. Test set validation indicated a root mean square error of 0.324 mg C g⁻¹ with an R-square of 0.85 (Baldock et al., 2013b).

An outlier ratio based on Hotelling's T-squared distribution and an inlier ratio based on the Mahalanobis distance derived using the Unscrambler 10.2 software (CAMO Software AS, Oslo, Norway) were applied to determine how closely our field site PyC data in the US north-east aligned with the range of data used in the PLS calibration data set from Australia. Under the similarity assumptions associated with the PLS model fit to the calibration data, no more than about 5% of the calibration samples should be expected to lie beyond the threshold for each metric (i.e. having a ratio > 1). For the soils being predicted in this study, approximately 58% were found to be beyond the Hotelling's critical value for the outlier ratio and 8% beyond the Mahalanobis critical value for the inlier ratio (Fig. 1), thus providing a measure of distance between our data and the data used for the calibration. Consequently, 58% of our data lie beyond the current predictive range of this model and prudence should be exercised in interpreting the results. One possibility would be to down-weight the elements of the data that are statistically beyond the base calibration data set. This might be appropriate if the variables used for the prediction were scattered more or less randomly outside of its range. However, this is a spatial prediction, so sites with characteristics that lie beyond those of the calibration data are in fact aggregated in specific regions. Thus down-weighting or eliminating these data would leave gaps in the prediction. Even so the predicted means are not likely to change from those presented here, although their variance increases. Consequently, for our purposes of relating PyC contents to each other and to site variables, we assume that the established linear relationship is appropriate.

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