



Contents lists available at ScienceDirect

Science of the Total Environment

journal homepage: www.elsevier.com/locate/scitotenv

Pyrogenic carbon in Australian soils

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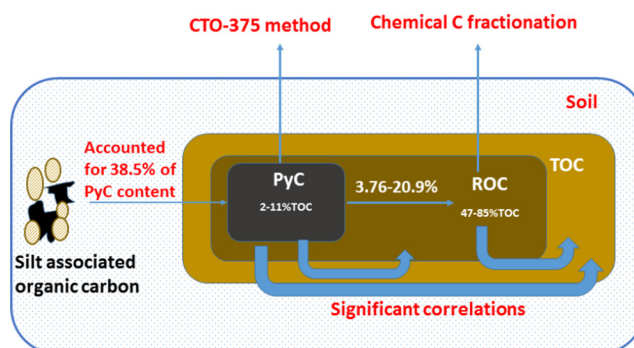
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HIGHLIGHTS

- The chemo-thermal oxidation method (CTO-375) was applied to quantify pyrogenic carbon (PyC) in Australian soils.
- Principle component analysis - multiple linear regression (PCA-MLR) gave a good prediction for soil PyC levels.
- PyC was a key fraction of TOC and chemically recalcitrant organic C accounts for a significant proportion of soil TOC.
- Chemically recalcitrant organic carbon was an order of magnitude greater than that of thermally stable organic carbon.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 20 November 2016

Received in revised form 6 February 2017

Accepted 7 February 2017

Available online xxxxx

Editor: Simon Pollard

Keywords:

Soil

Pyrogenic carbon (PyC)

Content

Distribution

Soil properties

ABSTRACT

Pyrogenic carbon (PyC), the combustion residues of fossil fuel and biomass, is a versatile soil fraction active in biogeochemical processes. In this study, the chemo-thermal oxidation method (CTO-375) was applied to investigate the content and distribution of PyC in 30 Australian agricultural, pastoral, bushland and parkland soil with various soil types. Soils were sampled incrementally to 50 cm in 6 locations and at another 7 locations at 0–10 cm. Results showed that PyC in Australian soils typically ranged from 0.27–5.62 mg/g, with three Dermosol soils ranging within 2.58–5.62 mg/g. Soil PyC contributed 2.0–11% (N = 29) to the total organic carbon (TOC), with one Ferrosol as high as 26%. PyC was concentrated either in the top (0–10 cm) or bottom (30–50 cm) soil layers, with the highest PyC:TOC ratio in the bottom (30–50 cm) soil horizon in all soils. Principal component analysis - multiple linear regression (PCA-MLR) suggested the silt-associated organic C factor accounted for 38.5% of the variation in PyC. Our findings suggest that PyC is an important fraction of the TOC (2.0–11%, N = 18) and chemically recalcitrant organic C (ROC) obtained by chemical C fractionation method accounts for a significant proportion of soil TOC (47.3–84.9%, N = 18). This is the first study comparing these two methods, and it indicates both CTO-375 and C speciation methods can determine a fraction of recalcitrant organic C. However, estimated

Abbreviations: CEC, cation exchange capacity; CTO-375, chemo-thermal oxidation method; IC, inorganic carbon; MA, microbial activity; MLR, multiple linear regression; NPOM, non-PyC organic matter; PCA, principal component analysis; PyC, pyrogenic carbon; ROC, recalcitrant organic carbon; SOM, soil organic matter; TOC, total organic carbon.

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<http://dx.doi.org/10.1016/j.scitotenv.2017.02.064>

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Please cite this article as: Qi, F., et al., Pyrogenic carbon in Australian soils, *Sci Total Environ* (2017), <http://dx.doi.org/10.1016/j.scitotenv.2017.02.064>

chemically recalcitrant organic carbon pool (ROC) was approximately an order of magnitude greater than that of thermally stable organic carbon (PyC).

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1. Introduction

Pyrogenic carbon (PyC), alternatively termed black carbon, char, charcoal or biochar (Bird and Ascough, 2012; Cusack et al., 2012; Zimmermann et al., 2012) depending on different aspects of focus, is the residue from incomplete combustion of fossil fuel and biomass (Bird et al., 2015; Goldberg, 1985). As a part of the inert fraction of soil organic matter (SOM), it can resist to a high degree microbial degradation and persist for centuries to millennia in the natural environment (Baker et al., 2011; Singh et al., 2012). PyC is widespread in soils and sediments (Kasozi et al., 2010; Schmidt and Noack, 2000) due to its recalcitrant nature and the common occurrence of combustion (Gustafsson and Gschwend, 1998; Reisser et al., 2016; Schellekens et al., 2017). It plays an important role in a wide variety of biogeochemical processes including acting as a fire history tracer in sediments and ice cores (Schmidt and Noack, 2000; Wang, 2010), a permanent carbon sink for climate change mitigation (Lehmann et al., 2005; McBeath and Smernik, 2009), a soil conditioner that increases soil nutrient-holding and cation exchange capacity (Liang et al., 2006; Qu et al., 2016) and a geo-sorbent for potentially hazardous contaminants in soils and sediments (Pignatello et al., 2006; Semple et al., 2013; Wang et al., 2010).

Accurate quantification of soil PyC content is essential in order to parameterize its role in biogeochemical processes (Falloon and Smith, 2000; Falloon et al., 2000). However, unified and standardized PyC analytical approaches are currently lacking. This is because PyC is not a unique C substance but a continuum including char, charcoal, soot and graphite (Schmidt et al., 2001; Thevenon et al., 2010), while varying methods often measure different fractions of the PyC continuum relying on operational definitions with clear-cut boundaries (Agarwal and Bucheli, 2011a; Hammes et al., 2007; Ponomarenko and Anderson, 2001). Chemo-thermal oxidation at 375 °C (CTO-375) initially reported by Gustafsson and Gschwend (1997) has been widely applied in PyC quantification and PyC relevant studies since its inception. This method has enabled findings of significant correlations between PyC and heavy metals including Cd, Cu, Pb, Zn, Cr, V, Mo, Sc (Cai et al., 2011; Chen et al., 2010; Wang, 2010) as well as organic contaminants such as polyaromatic hydrocarbons (PAHs), polychlorinated benzenes (PCBs) and polychlorinated dibenzo-dioxins and furans (PCDD/Fs) in soils and sediments (Gustafsson and Gschwend, 1997; Lohmann et al., 2004; Persson et al., 2002). Further, the CTO-375 method has successfully demonstrated the PyC - non-PyC organic matter (NPOM) dual sorption model improved quantitative prediction of solid-water distribution, mass transfer, bioavailability and bioaccessibility of various organic contaminants (e.g. PAHs, PCDD/Fs, PCBs) compared to the bulk TOC only sorption model used in numerous studies (Accardi-Dey and Gschwend, 2002; Apell and Gschwend, 2014; Bucheli and Gustafsson, 2001; Gustafsson et al., 1996; Koelmans and Jonker, 2011; Lohmann et al., 2004; Moermond et al., 2005; Oen et al., 2006; Pee et al., 2015; Werner et al., 2010). However, in Australia, where PyC levels can vary from 0 to 82% of TOC (Lehmann et al., 2008) due to repeated historical burning of grasslands, open woodlands and agricultural crop residues by indigenous people (Skjemstad et al., 2002), PyC quantification has been assessed by ultraviolet photo-oxidation + solid-state ¹³C NMR spectroscopy methods (Skjemstad et al., 2001; Skjernstad et al., 1999; Smernik et al., 2000). The CTO 375 method has only been applied in 8 Australian soils in a comparison with five other methods (Schmidt et al., 2001). Hence, it is essential to introduce the CTO-375 method into PyC quantification for more Australian soils. The CTO-375 estimates the stable pool of organic carbon in soil using combustion. Similarly, chemical fractionation methods may estimate the stable pool of organic

C (Bolan et al., 2012; Hedley et al., 2004). Hence, it is essential to introduce the CTO-375 method into PyC quantification of more Australian soils, and to compare chemical and thermal estimates of the stable pools of carbon. In addition, previous studies on PyC in Australian soils covered only a limited number of soils. Studies on the distribution of PyC in Australian soil profiles in varying land-uses, as well as the relationship of PyC with major soil properties, are lacking.

In this study, we aim to (a) evaluate PyC levels in Australian agricultural, pastoral, bushland and parkland soils as influenced by soil depths, soil types and land-uses, (b) characterize potential correlations between soil properties and PyC content by applying multivariate statistics, (c) identify common groups of soil properties that influence PyC levels via principal component analysis (PCA), and (d) investigate the relationship between stable organic carbon pool obtained by CTO-375 method and the chemical C fractionation method. This study represents the first detailed interrogation of soil stable organic carbon content by both CTO-375 and chemical C fractionation methods in such a wide variety of Australian soils.

2. Methodology

2.1. Soil sampling and analysis

In total 30 soil samples were used in this study. These soils were sampled from South Australia (SA), New South Wales (NSW), Victoria (VIC) and Queensland (QLD), Australia. Among them, 23 soil samples were from 5 locations with depths of 0–10, 10–20, 20–30 and 30–50 cm and another location with depths of 0–20, 20–30, 30–50 cm. Another seven soil samples at depths of 0–10 cm were also collected to cover a wide range of soil types and soil properties. The soil types included Calcarosols, Dermosols, Ferrosols, Sodosol, Tenosol and Vertosols (Isbell, 1996). These 30 soil samples were air dried and sieved through a 2 mm sieve and assessed for pH, cation exchange capacity, soil texture and microbial activity analysis. Their <425 μm counterparts were used for measurement of total carbon (TC), total organic carbon (TOC), PyC, active/amorphous Fe, Al and Mn oxides.

Soil pH and electric conductivity (EC) was determined using a 1:5 soil to water ratio. Soil texture was determined by the hydrometer method (Gee et al., 1986). Basic cation exchange capacity (CEC_b) of the soils was measured by the BaCl₂/NH₄Cl compulsive exchange method as described by Gillman and Sumpter (1986). Amorphous Al, Mn and Fe (Al_{oxa}, Mn_{oxa}, Fe_{oxa}) was extracted by 0.2 M ammonium oxalate/oxalic acid following Rayment and Higginson (1992). Microbial activity (MA) was obtained in the form of CO₂-C emission during a 20-day incubation period following Cheng et al. (2008). Soil TC was determined by combusting 0.2 ± 0.05 g soils at 1100 °C in a Leco TruMac CNS elemental analyzer. TOC was determined the same way as TC except a preceding 1 M hydrochloric acid (HCl) treatment process was applied to remove soil inorganic carbon (IC). Soil IC was estimated by the difference of TC and TOC. Dissolved organic carbon (DOC) was measured by shaking 2 g soil in 10 ml Milli-Q water for 16 h, following detection of soluble OC by a TOC analyzer (TOC-LCSH, Shimadzu). Measurement of PyC by the CTO-375 method was carried out in Switzerland by the same laboratory and procedure as Agarwal and Bucheli (2011b). This method involved combustion of soils (<425 μm) at 375 °C for 24 h under air atmosphere to remove soil NPOM, followed by removal of carbonates using acid fumigation for 4 h by 12 M HCl. The residual C content was lastly determined by a CHN elemental analyser. The carbon fractionation method adapted from Hedley et al. (2004) and Bolan et al. (2012) was applied to 18 selected soils to examine the recalcitrant fraction of organic

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