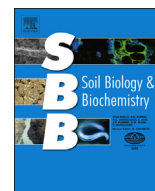




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Evaluating biodegradability of soil organic matter by its thermal stability and chemical composition

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ABSTRACT

The stability of soil organic matter (SOM) as it relates to resistance to microbial degradation has important implications for nutrient cycling, emission of greenhouse gases, and C sequestration. Hence, there is interest in developing new ways to quantify and characterise the labile and stable forms of SOM. Our objective in this study was to evaluate SOM under widely contrasting management regimes to determine whether the variation in chemical composition and resistance to pyrolysis observed for various constituent C fractions could be related to their resistance to decomposition. Samples from the same soil under permanent pasture, an arable cropping rotation, and chemical fallow were physically fractionated (sand: 2000–50 µm; silt: 50–5 µm, and clay: <5 µm). Biodegradability of the SOM in size fractions and whole soils was assessed in a laboratory mineralization study. Thermal stability was determined by analytical pyrolysis using a Rock-Eval pyrolyser, and chemical composition was characterized by X-ray absorption near-edge structure (XANES) spectroscopy at the C and N K-edges. Relative to the pasture soil, SOM in the arable and fallow soils declined by 30% and 40%, respectively. The mineralization bioassay showed that SOM in whole soil and soil fractions under fallow was less susceptible to biodegradation than that in other management practices. The SOM in the sand fraction was significantly more biodegradable than that in the silt or clay fractions. Analysis by XANES showed a proportional increase in carboxylates and a reduction in amides (protein) and aromatics in the fallow whole soil compared to the pasture and arable soils. Moreover, protein depletion was greatest in the sand fraction of the fallow soil. Sand fractions in fallow and arable soils were, however, relatively enriched in plant-derived phenols, aromatics, and carboxylates compared to the sand fraction of pasture soils. Analytical pyrolysis showed distinct differences in the thermal stability of SOM among the whole soil and their size fractions; it also showed that the loss of SOM generally involved preferential degradation of H-rich compounds. The temperature at which half of the C was pyrolyzed was strongly correlated with mineralizable C, providing good evidence for a link between the biological and thermal stability of SOM.

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1. Introduction

Soil organic matter is a key property related to ecosystem productivity and understanding its physical, chemical and biological properties is important for evaluating its role in C cycling in terrestrial ecosystems. The stability of SOM as it relates to resistance to microbial degradation has important implications in

ecosystem processes related to nutrient cycling, emission of greenhouse gases from soil, and C sequestration. Thus there is interest in developing new ways to accurately quantify and assess the labile and stable forms of soil organic C.

Carbon mineralization, determined as the total amount of CO₂-C released during laboratory incubation, provides information about the fraction of soil organic C that is readily available to heterotrophic organisms (Hopkins, 2008). Information about the degradability of the SOM is obtained when mineralized C is expressed per unit of soil organic C (Baldock and Broos, 2012). The resistance of SOM to biological decomposition is a function of its biochemical

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complexity (often referred to as recalcitrance (Marschner et al., 2008)), its location within the soil matrix and association with minerals, and the environmental conditions.

Thermal analysis methods such as thermal gravimetric, differential thermal analysis and differential scanning calorimetry (DSC) have been used to study the stability of SOM (Lopez-Capel et al., 2005; Plante et al., 2009). This approach integrates the energy input and yield from the breakdown of intra-molecular SOM bonds during combustion as well as that from the breakdown of bonds between SOM and mineral surfaces (Peltre et al., 2013).

Lopez-Capel et al. (2005) used thermogravimetry differential scanning calorimetry (TG-DSC) to evaluate the proportions of active and stable components in SOM fractions isolated by density. They concluded that soil physical fractionation was useful to identify the effect of land-use on SOM with greater precision than possible with whole (unfractionated) soil. Peltre et al. (2013) used TG-DSC coupled with measurements of CO₂ and compared them with biological stability of SOM as measured during long-term laboratory incubation. They hypothesized that the variability in published results was due to distinct stabilization mechanisms in the different materials analyzed, such as protection by organomineral interaction vs. chemical recalcitrance, resulting in different relationships between the thermal and biological stabilities.

Analytical pyrolysis provides information on the thermal stability and chemical composition of organic matter present in the sample (Leinweber and Schulten, 1999; Plante et al., 2009, 2011). Recent experiments by Albrecht et al. (2014) showed that major classes of organic constituents characterized by analytical pyrolysis using Rock-Eval were related to chemical structures identified by ¹³C PMAS NMR. Analytical pyrolysis is fundamentally different than other thermal degradation techniques in that it involves use of an inert atmosphere and rapid heating rates, and because it characterizes the reaction products from heating rather than properties of the bulk sample (Plante et al., 2009; Peltre et al., 2013). Under pyrolysis hydrocarbons are released by volatilization of labile organic matter or thermal cracking of more complex biomacromolecules (Gillespie et al., 2014b). Hence, in terms of its potential relationship to biological stability, we hypothesize that pyrolysis may simulate enzymatic depolymerisation and the monomers released at a particular temperature may be related to monomers available for microbial respiration.

Rock-Eval pyrolysis was originally developed for petroleum screening of geological (rocks, sediments) material (Lafargue et al., 1998) to characterize the nature of C molecules in samples. It has also been used in the analysis of organic matter in surface soils, sediments, and peats because it provides useful information on quantity and quality of C in a complex sample (Zaccone et al., 2011; Carrie et al., 2012; Saenger et al., 2012). Sebag et al. (2006) used Rock-Eval to characterize the SOM in 76 organic and organomineral soil samples from different ecosystems. They deconvoluted the pyrograms obtained into four components which were used to identify the contributions of immature 'bio-macromolecules'. Carrie et al. (2012) developed a library of Rock-Eval data on a variety of pure biochemical compounds (proteins, lipids, carbohydrates, and lignin) and biological standards (phytoplankton, copepods, tree bark and conifer needles) which would comprise different SOM fractions in recent sediments and soils. They compared the Rock-Eval data to petrographical and elemental analysis results and ascribed the obtained signals in Rock-Eval pyrograms to organic molecules. Saenger et al. (2013) used Rock-Eval pyrolysis to analyze 109 soils across France and observed a strong correlation between TOC measured by Rock-Eval and that measured by elemental analyzer; this correlation was strong even for calcareous soils (i.e., with relatively large amounts of inorganic C present).

Assessment of the chemical composition of SOM involving spectroscopic techniques like X-ray absorption near-edge structure (XANES) includes information on the chemical speciation and bonding environments of C and N compounds at the molecular and atomic scales (Solomon et al., 2005; Gillespie et al., 2011) and can be used to identify the fine structures of organic C in various molecular groups (Solomon et al., 2009). Recent XANES analyses identified useful biomarkers produced during the transformation and stabilization of organic matter by soil organisms (Gillespie et al., 2014a).

Our objectives in this study were to evaluate and describe relationships among the biodegradability, thermal stability and chemical composition of SOM under widely contrasting management regimes to determine whether the variation in chemical composition and resistance to pyrolysis observed for various constituent C fractions in SOM could be related to their resistance to decomposition.

2. Materials and methods

The soils used in this study were from a field experiment at Lincoln, Canterbury, New Zealand (43°40'S latitude, 172°28'E longitude). The experiment, which was on a Wakanui silt loam (Udic Dystocrypt, (Soil Survey Staff, 1999); soil pH ~6.0), was established in November 2000 to examine the effects of various management practices on SOM. Soils from three treatments with widely contrasting management regimes were sampled in February 2011 for this study: permanent pasture (pasture), arable cropping (arable) and chemical fallow (fallow). These treatments were selected to obtain soil samples with different SOM levels but the same parent material.

The site had been under permanent pasture (i.e., perennial ryegrass/white clover sward, grazed by sheep) prior to the experiment and replicated plots representing this land use were maintained as a control within the experiment. To establish the arable cropping treatment, the pasture was killed off using herbicide, after which a barley crop was sown by direct drilling. The arable crops grown during the 2000–2011 period included cereals (barley in 6 years and wheat in 2 yr), peas (2 yr) and ryegrass seed crops. The fallow plots were maintained plant free using herbicides and were not cultivated or physically disturbed during the experiment. Details of all treatments included in the experiment, along with management information, are provided by Fraser et al. (2013).

Soil samples (0–7.5 cm) were collected from three field replicate plots of each treatment using a stainless steel corer 5 cm in diameter (three cores were collected in each plot and composited). The samples were sieved (<4 mm) in field moist condition and any coarse residue fragments that passed through the sieve were removed by hand. Subsamples were air-dried and ground to <250 µm for determination of total C and N using a LECO Truspec analyser. The soils did not contain any inorganic C so total C is equivalent to organic C (TOC_L, g kg⁻¹). The soil texture was sand, 36%; silt, 44% and clay, 19%. Additional soil properties are provided in Table 1.

2.1. Particle size fractionation

Sub-samples (equivalent of ~30 g dry soil) were dispersed by overnight end-over-end shaking in de-ionized water containing 20 glass beads (3 mm diameter) to facilitate disaggregation. The sand fraction was separated by washing the soil suspension through a 50 µm sieve. The <50 µm fraction was further subdivided into 50–5 and <5 µm fractions by gravity sedimentation with settling time for particles of 5 µm equivalent spherical diameter calculated by Stokes' Law (Kroetsch and Wang, 2008). Complete separation of

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