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Tracing the source and fate of dissolved organic matter in soil after incorporation of a ^{13}C labelled residue: A batch incubation study

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ABSTRACT

While dissolved organic matter (DOM) in soil solution is a small but reactive fraction of soil organic matter, its source and dynamics are unclear. A laboratory incubation experiment was set up with an agricultural topsoil amended with ^{13}C labelled maize straw. The dissolved organic carbon (DOC) concentration in soil solution increased sharply from 25 to 186 mg C L^{-1} 4 h after maize amendment, but rapidly decreased to 42 mg C L^{-1} and reached control values at and beyond 2 months. About 65% of DOM was straw derived after 4 h, decreasing to 29% after one day and only 1.3% after 240 days. A significant priming effect of the straw on the release of autochthonous DOM was found. The DOM fractionation with DAX-8 resin revealed that 98% of the straw derived DOM was hydrophilic in the initial pulse while this hydrophilic fraction was 20–30% in control samples. This was in line with the specific UV absorbance of the DOM which was significantly lower in the samples amended with maize residues than in the control samples. The $\delta^{13}C$ of the respired CO_2 matched that of DOC in the first day after amendment but exceeded it in following days. The straw derived C fractions in respired CO_2 and in microbial biomass were similar between 57 and 240 days after amendment but were 3–10 fold above those in the DOM. This suggests that the solubilisation of C from the straw is in steady state with the DOM degradation or that part of the straw is directly mineralised without going into solution. This study shows that residue application releases a pulse of hydrophilic DOM that temporarily (<3 days) dominates the soil DOM pool and the degradable C. However, beyond that pulse the majority of DOM is derived from soil organic matter and its isotope signature differs from microbial biomass and respired C, casting doubt that the DOM pool in the soil solution is the major bioaccessible C pool in soil.

1. Introduction

The dissolved organic matter (DOM) in soils is a small but reactive fraction of the soil organic matter. The DOM concentration in soil solution is determined by the net result of its production, degradation, immobilisation and leaching. The contribution of these different processes to the DOM concentration is relatively unexplored (Kalbitz et al., 2000). Different methods have been used to identify the sources and sinks of DOM in soil. Seasonal and spatial variability in DOM concentration and composition indirectly suggests relative importance of different DOM sources such as litter decomposition and dissolution of humic substances. Alternatively, ^{13}C or ^{14}C isotope studies allow directly tracing the sources of DOM.

Data on DOM concentration and composition in soils of forest ecosystems suggest that throughfall, recent litter and/or root litter are major sources of DOM (Qualls et al., 1991; Yano et al., 2005). In contrast, other studies found a dominance of more humified compounds in DOM, suggesting that DOM originates from the large stock of native soil organic matter rather than from recent added litter (Fröberg et al., 2003; Hagedorn et al., 2002; McDowell and Likens, 1988). Litter or incorporated plant-residues release significant amounts of DOM, but this pulse is short-lived and hence, does not affect the composition and the size of DOM on the long term or in deeper soil layers (Hagedorn et al., 2004). Comparable results were found in agricultural soils. The amendment of corn and soybean residues initially increased water extractable organic C (WEOC) in surface soils, but this WEOC was decomposed in a few days and only very little was leached into subsoils (McCarty and Bremner, 1992). Similarly, surface application of oilseed rape residues at the surface of irrigated soil columns increased dissolved organic carbon (DOC) concentration in soil solutions sampled by rhizon samplers at 2 cm depth, while incorporating the residues in the top 10 cm of the column increased DOC concentrations at 10 cm. In none of the treatments, elevated DOC concentrations could be found at 18 cm depth (Coppens et al., 2006).

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Most of the pulse-labelling studies that allow determining source and fate of DOM have been set up with forest soils. A ^{14}C labelled residue experiment in forest soils showed that root litter as well as leaf litter may contribute to the leaching of DOC at 50 cm depth (Uselman et al., 2007). However, Fröberg et al. (2007) found that only 14% of the DOC leaching from a forest soil at 15 cm depth originated from recent litter. The DOM from fresh litter was largely retained or consumed in the surface layers while only a small fraction moved through the soil (Fröberg et al., 2009, 2007). A shift from a C3 to C4 crop in an agricultural field has been used to estimate the plant derived C in the DOC based on the $\delta^{13}C$ values (Steinbeiss et al., 2008). This fraction was only found in the soil solution in fall and early winter (22% at 10 cm depth when the litter was removed). Moreover, plant derived C was preferentially mineralised and adsorbed to soil particles and the main component of DOC transported down the soil profile was remobilised soil organic carbon (SOC) (Steinbeiss et al., 2008). John et al. (2003) estimated the contribution of younger (≤40 years) C4-derived and older (>40 years) C3-derived soil organic matter (SOM) to different C pools using agricultural soils with different history of maize cropping. The isotope signature suggested that the percentage of maize-derived carbon was highest in CO_2 (42–79%), followed by microbial biomass carbon (MBC) (23–46%), DOC (5–30%) and SOC (5–14%). A larger proportion of new C in MBC compared to DOC is consistently found in other studies investigating the natural abundance of ^{13}C after crop rotations (Accoe et al., 2002; Gregorich et al., 2000; Liang et al., 2002). This indicates that the DOM is partly derived from old SOM and not only from decaying biomass or recent residues. In addition, it also indicates that DOM is not uniformly metabolised, that the release of plant C in the soil solution is in steady state with its decomposition or that plant litter or young SOM can also be degraded by the biomass, without first going into solution.

No pulse-labelling studies have yet been set up that allow tracing newly released DOC after residue amendment and its relationship with CO_2–C and MBC in agricultural soils. In addition, as far as we are aware, no studies have combined isotope studies with DOM fractionation to relate differences in structural properties of DOM to their source. Such data facilitate the interpretation of the fate of the DOM pulse. The objective of this study was to investigate the source and dynamics of DOM in an agricultural soil amended with a ^{13}C labelled residue. The subsequent changes in respired CO_2, microbial biomass-C and DOC were measured along with changes in $\delta^{13}C$ values in these C pools. The composition of DOM was analysed by its specific UV absorbance. Finally, on selected samples, DOM was fractionated according to hydrophobicity with DAX-8 resin to monitor and identify the fractions of DOM in which the labelled plant derived C was present.

2. Materials and methods

2.1. Soil sampling and experiment set-up

Soil samples were taken from the topsoil of an agricultural luvisol in Leuven (Belgium) (Amery et al., 2008) after removing the top 2 cm and the plant cover. They were transported to the lab in plastic bags and sieved at 4 mm. After 4 days pre-incubation (20 °C, darkness, $\theta_g = 0.23$ g g^{-1}) the soil was divided over 53 glass jars (300 ml) containing 150 g soil each. To 29 jars, ^{13}C enriched maize straw was added at 2 g dry matter per kg dry soil. The other 24 soil samples were used as a control. The maize straw contained 1.7% N and 42.5% C with a ^{13}C atom excess of 2% and the fraction 2–4 mm was used. Nine percent of the maize carbon was soluble in a water extract (1/100, 2 h shaking, 0.45 μm filtration). The uniformly labelled straw was obtained by growing maize up to maturity in a gas-tight growth chamber (see details in Trinsoutrot et al. (2000)). The atmosphere in the chamber was enriched in ^{13}C by injecting CO_2 containing 2% atom excess ^{13}C–CO_2. The jars were closed with gas-tight rubber lids and all soil samples were incubated at 20 °C for 8 months under aerobic conditions in darkness. Approximately 4 h (0.2 days) after adding the plant residues and after 1, 3, 14, 57, 112 and 240 days of incubation, three replicates of each treatment were destructively sampled to analyse for moisture content, DOC and biomass-C. Respiration was measured more regularly (see below). At 0.2, 3 (amended soil only) and 57 days, two or three additional replicates were destructively sampled to obtain enough soil solution for hydrophobic fractionation. Respiration was also measured in these replicates, but no analyses of moisture content, DOC and biomass-C were performed on these additional replicates. At the end of the experiment, the soil residual ^{13}C in the last three replicates was determined by measuring 30–50 mg of ground soil on a C:N analyser–mass spectrometer (ANCA-GSL Preparation Module 20–20 Stable Isotope Analyser; Europa Scientific, Crewe, Cheshire, UK).

2.2. Dissolved organic carbon and microbial biomass carbon

At each sampling occasion, moisture content was determined after oven drying (105 °C; 16 h). Soil solution was isolated by centrifuging approximately 65 g of fresh soil (double chamber method, 1700*g*, 30 min; see isolation of pore water in Amery et al. (2007)). Soil solutions were filtered at 0.45 μm. The DOC concentration was assayed as the difference between the total dissolved carbon concentration and the dissolved inorganic carbon concentration (Multi N/C 2100S, Analytik Jena). The $^{13}C/^{12}C$ isotopic ratio of the DOC was measured with a TOC analyser coupled to an IRMS (De Troyer et al., 2010). The maize-derived DOC was calculated as described in Section 2.4.

The specific UV absorbance (SUVA, l g^{-1} cm^{-1}) of the DOM in the soil solutions at 254 nm was measured by UV–VIS spectrophotometry (Perkin–Elmer, Lambda 20, quartz cells) with a path length (b) of 1 cm. If the absorption at 254 nm (A^{254}) was higher than 2.0, soil solutions were diluted to assure linear relationships between A^{254} and DOC concentrations. The SUVA of the DOM was calculated as:

$$\mathrm{SUVA} = \frac{A^{254}}{b\ [\mathrm{DOC}]} \quad (1)$$

with A^{254} dimensionless, b in cm and [DOC] in g C l^{-1}. The specific UV absorbance is used as an estimate of the aromaticity of DOM (Weishaar et al., 2003).

The remains of the soil solutions sampled at day 0.2 and 57 (control) or day 0.2, 3 and 57 (amended) were pooled to be fractionated by DAX-8 into hydrophilic (HPI), hydrophobic acid (HPOA) and hydrophobic neutral (HPON) fractions (Amery et al., 2009). The total amount of DOC in the HPI and HPOA fraction was determined by measuring the DOC concentration in all subfractions and dividing the total amount of DOC in all subfractions by the original volume of the sample. The DOC present in the HPON fraction was calculated by difference between DOC in the original sample and the DOC quantities in fractions recovered (Amery et al., 2009). The $\delta^{13}C$ values of the three subfractions with the highest DOC concentration within in the HPI and HPOA fraction were determined using a TOC analyser coupled to an IRMS (De Troyer et al., 2010).

Soil microbial biomass was determined by the fumigation–centrifugation method based on van Ginkel et al. (1994). About 65 g of soil sample was fumigated for 24 h at 25 °C with ethanol-free $CHCl_3$. After fumigant removal, the soil solution was sampled as above, filtered at 0.45 μm and DOC concentration as well as the $^{13}C/^{12}C$ isotopic ratio of the DOC were analysed. The microbial biomass-C (mg kg^{-1}) was calculated as: