



Mineral controls on soil organic C stabilization in alpine and subalpine soils in the Central Pyrenees: Insights from wet oxidation methods, mineral dissolution treatment and radiocarbon dating

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ABSTRACT

Studies on the mineral stabilization of soil organic carbon (SOC) and the radiocarbon age of Pyrenean mountain soils have not been conducted to date. This study assessed the SOC concentrations, the interaction of SOC with the mineral phase (stabilization), the radiocarbon age and the relationships between SOC and dithionite-, oxalate-, and pyrophosphate-extractable Fe, Al and Si contents on four summits in Ordesa National Park (Central Pyrenees, Spain). The selected summits represented a gradient in plant community composition from subalpine (2242 m a.s.l.) to nival (3022 m) environments. Soil samples (0–10 cm) were physico-chemically fractionated and exposed to $\text{Na}_2\text{S}_2\text{O}_8$ and H_2O_2 oxidation and to mineral dissolution with HF. The average SOC concentration in the $<20\ \mu\text{m}$ fraction decreased with increasing elevation (from 80.1 to 15.0 g C kg dry soil $^{-1}$ at 2242 and 3022 m, respectively). The H_2O_2 - and $\text{Na}_2\text{S}_2\text{O}_8$ -resistant SOC fraction increased with elevation, and was mainly controlled by oxalate-extractable Si (Si_o), i.e., 69.3 and 66.7% of the variability was explained by the $\text{Na}_2\text{S}_2\text{O}_8$ -persulfate- and H_2O_2 -oxidation fractions, respectively. The oxalate-extractable Al (Al_o), dithionite-extractable Al (Al_d), pyrophosphate-extractable Al (Al_p), and indicators Al_p/Al_o and $\text{Al}_o + \text{Fe}_o$ were more important for SOC stabilization on the two lower summits (2242 m and 2519 m) than on the two higher summits (2800 m and 3022 m). In contrast, the HF-soluble SOC fraction was not correlated with the mineral indicators. The largest percentages of $\text{Na}_2\text{S}_2\text{O}_8$ - and H_2O_2 -resistant SOC fractions were found in one of the higher summits, where the greatest ^{14}C age was measured. The radiocarbon analyses indicated that the ^{14}C age of the mineral-associated SOC ranged from 825 and 830 yr BP on the two lower summits to 10,500 and ca. 16,000 yr BP on the two higher summits. Multivariate analysis, i.e., non-multidimensional scaling (NMDS) analysis, highlighted the relationship between plant community composition at the different summits and the facing slopes and the analysed soil variables. A clear detailed description of SOC and stabilization in mountain ecosystems is needed to enable further predictions of soil C gains and losses related to the expected global changes, i.e., T increase, precipitation regime and land use changes.

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1. Introduction

Mountain ecosystems are fragile environments due to their sensitivity to global climate change and the associated vulnerability of populations living in nearby areas (IPCC, 2014). In addition to climate change, other factors, such as nitrogen deposition, elevated carbon dioxide (CO_2) concentrations in the atmosphere, wind and water erosion, land use changes, and altered disturbance regimes are expected to affect plant community composition and cover, net primary productivity (NPP), soil decomposition rates, and soil carbon storage (Torn et al., 2009). The soil organic carbon (SOC) pool of mountain ecosystems represents on average 29% of the total 1502 Pg (Pg = Petagram = 10^{15} g) of

SOC stored globally to a depth of 1-m (Jobbágy and Jackson, 2000). The variability in abiotic and biotic factors, i.e., soil type, microclimate, stoniness, and slope, determine SOC storage and cycling in mountainous areas. A major part of the global SOC pool is assigned to stabilized fractions (Schmidt et al., 2011). Above and belowground plant production and decomposition determines C inputs to the soil, which can be low in alpine-nival environments (Hitz et al., 2001; van Groenigen et al., 2006).

Several factors are known to stabilize the SOC pool, i.e., selective preservation of biochemically resistant molecules, spatial inaccessibility of SOC for decomposers and enzymes (which reduces the probability – and therefore rate – of decomposition, thereby allowing the organic matter to persist, and interactions of SOC with surfaces and metal ions (von Lützow et al., 2007; Schmidt et al., 2011; Kleber et al., 2015). Different mechanisms for interactions of organic matter (OM) with mineral surfaces operate simultaneously at different scales via H-bonding and

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(Hassink et al., 1993) ligand exchange between carboxyl groups of OM and —OH groups, which are strong enough to form stable organo-mineral complexes (Kleber et al., 2015).

Physical fractionation methods do not satisfactorily recover the stable SOC fraction (Jagadamma et al., 2010). Consequently, chemical fractionation methods are normally used to isolate stabilized SOC fractions (von Lützow et al., 2007). Chemical recalcitrance was first viewed as the only SOC stabilization mechanism over long periods (Krull et al., 2003), but Marschner et al. (2008) also showed the opposite. Stabilization is now considered an ecosystem property rather than a question of recalcitrance (Schmidt et al., 2011). SOC adsorbed to soil minerals can be mobilized by hydrofluoric acid (HF) (Eusterhues et al., 2003), and the stable and functionally passive SOC can be isolated via oxidation with $\text{Na}_2\text{S}_2\text{O}_8$ and H_2O_2 (von Lützow et al., 2007; Bruun et al., 2008). However, most of the available fractionation methods do not yield homogeneous or functional OM pools. Since the currently available SOM fractionation methods generally isolate more than one functional SOM pool, they are only helpful in certain soil horizons where SOM is stabilized by a limited number of well-defined key mechanisms, like organo-mineral interactions and spatial inaccessibility of OM to the decomposer community, and where the fractionation method differentiates between these pools (von Lützow et al., 2006, 2007).

Sorption on mineral phases is also a very important control on the concentration and stabilization of organic C (Guggenberger and Kaiser, 2003). Oxalate-, dithionate-, citrate- and pyrophosphate-dissolution extractants are normally used to isolate Al, Fe, and Si minerals. Al_o , Fe_o , and Si_o (oxalate extractants) are derived from poorly ordered minerals, such as ferrihydrite, allophane, and imogolite (Hartwig and Loeppert, 1993). Al_p (pyrophosphate) is the Al solely from Al-humus complexes (Parfitt and Kimble, 1989). Dithionite-citrate-extractable Fe (Fe_d) represents both crystalline and poorly crystalline Fe oxides (Mehra and Jackson, 1960), and Al_d represents the Al substituted in Fe oxides (Kleber et al., 2005). A set of mineral indicators were obtained from these extraction techniques: Fe_o/Fe_d represents the poorly crystalline fraction of total Fe oxides (Kleber et al., 2005); Fe_d-Fe_o represents crystalline Fe oxides; Fe_o-Fe_p represents the content of ferrihydrite (López-Ulloa et al., 2005); Al_p/Al_o represents the ratio of organically-bound to total amorphous Al, or the relative amount of Al that is in the form of organic complexes; and $\text{Al}_o + 0.5\text{Fe}_o$ represents the amount of sesquioxides (Spielvogel et al., 2008).

The age of the stabilized SOC fraction can be on the order of thousands of years. Radiocarbon dating is one available tool to measure the mean age of the SOC pool of a certain fraction on decadal to millennial timescales. The longest time scales that can be addressed with ^{14}C are ca. 60,000 yr (Trumbore, 2009). At present, the factors influencing the stabilization of old SOC pool are not clear, especially for mountain soils. Much of the soil C is stabilized and decomposing only very slowly; highly stabilized OM, typically associated with mineral surfaces or very stable soil aggregates, persists in soils for thousands of years and is often referred to as the “passive” C pool (Torn et al., 2009). The “slow” C pool has turnover times in the range of decades to centuries, and it may consist of structural components of plants more resistant to decay, or organic compounds that have been stabilized by their association with soil minerals or aggregate structures (Torn et al., 2009). Slowly cycling C pools are highly correlated with clay particles and non-crystalline minerals, which stabilize and protect the OM (Paul, 1984; Torn et al., 1997).

The reduction of snow cover in alpine ecosystems due to the increasing temperatures associated with global warming will affect plant dynamics and vegetation cover via the progressive increase of warm mountain habitats and biota (Gottfried et al., 2012). Compared to other ecosystems, studies focusing on SOC storage and organo-mineral interactions in alpine-nival ecosystems are rare, and selective dissolution data are scarce for mountain soils, although some studies used alternative approaches to evaluate SOC stabilization and composition in alpine regions (Colombo et al., 2014; Leifeld et al., 2013; Saenger et al.,

2015; Catoni et al., 2016). The objective of this study was to quantify the SOC fractions stabilized by the association with soil minerals for several summits in the Central Pyrenees. Compared to other ecosystems, stabilization of C in alpine environments is less understood, and few data are available on the SOC concentrations in various particle-size fractions and the responsible mechanisms. We tested the hypotheses that relatively more C is stabilized in association with the mineral phase with increasing elevation, and that the ^{14}C age and proportion of stabilized and functionally passive SOC increases with elevation. A mechanistic understanding of SOC distribution and stabilization mechanisms in mountain soils is needed to quantify soil C gains and losses associated with a process of global change, including climate warming (IPCC, 2014) and land use changes in coming decades.

2. Material and methods

2.1. Study area

The study was conducted in the massif of Ordesa and Monte Perdido National Park (OMPNP). Four summits already included in the network of the Pan-European project GLORIA (“Global Observation Research Initiative in Alpine Environments”) were selected: Punta Acuta (ACU), 2242 m above sea level (a.s.l.); Custodia (CUS), 2519 m a.s.l.; Tobacor (TOB), 2779 m a.s.l.; and Punta de las Olas (OLA), 3022 m a.s.l. (Fig. 1). These summits are within the boundaries of the OMPNP, which is also a Biosphere Reserve and World Heritage Site (UNESCO), and they represent a gradient from subalpine environments (2242 m a.s.l.) to nival (3022 m a.s.l.) environments. The yearly average precipitation and temperature in the area (“Refugio Góriz”, 2200 m a.s.l., with data spanning the last 29 yr) are 1720 mm and 5 °C, respectively. No temperature and precipitation data are available for the four summits. The soil parent material is composed mainly of sandstone at ACU, CUS and TOB and limestone at OLA. The soils are defined (Soil Survey Staff, 2014) as humic Dystricrypts (Inceptisols) on the lower summits and lithic Cryorthents (Entisols) on the higher summits.

Extensive domestic grazing (cattle, goat and sheep) is permitted within the park and is still occurring on the two lower summits, whereas the two higher summits are moderately impacted by hiking activities. The study area has been heavily grazed for centuries. As a result, the upper tree line is ca. 300 m below its potential elevation as determined by climate (Camarero and Gutiérrez, 2004), similar to other mountainous systems (Fischling et al., 2007). Only scattered groups of Mountain pine (*Pinus uncinata* Ramond ex DC.) approach the summit ACU at 2180–2200 m; above this elevation no trees or shrubs are observed. The uppermost summit (OLA) reaches the subnival belt, and only pioneer plants such as *Saxifraga pubescens* Pourr. subsp. *iratiana* (F.W. Schulz) Engl. & Irmscher, *Androsace ciliata* DC., *Poa alpina* L. and *Leucanthemopsis alpina* (L.) Heywood colonize fissures of rocks with barely visible traces of herbivory.

The plant community composition (presence/absence) was documented for each aspect of each summit in the area where soil samples were taken. The inventory was performed 10 m below the summit point, and area size ranged from 16 to 100 m², depending on the available area in the summit (Appendix A).

2.2. Soil sampling protocol

For each summit a composite sample was obtained by taking 4 subsamples with a soil auger from the north-, south-, east- and west-facing slopes during various field trips in August–September 2008. All soil subsamples were taken in each aspect at least 15 m below the summit point, as vegetation assessments are periodically conducted within the GLORIA network. Soil samples were gently crumbled manually in the field, and taken to the lab for air drying at room temperature for several days. They were then passed through a 2-mm sieve for further analysis after removing visible root fragments and stones.

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