

Abstract

Conceptual models suggest that stability and age of organic carbon (OC) in soil depends on the source of plant litter, occlusion within aggregates, incorporation in organo-mineral complexes, and location within the soil profile. Various tools like density fractionation, mineralization experiments, and radiocarbon analyses have been used to study the importance of these mechanisms. We systematically apply them to a range of European soils to test whether general controls emerge even for soils that vary in vegetation, soil types, parent material, and land use. At each of the 12 study sites, 10 soil cores were sampled in 10 cm depth intervals to 60 cm depth and subjected to density separation. Bulk soil samples and density fractions (free light fractions – fLF, occluded light fractions – oLF, heavy fractions – HF) were analysed for OC, total nitrogen (TN), $\delta^{13}\text{C}$, and $\Delta^{14}\text{C}$. Bulk samples were also incubated to determine mineralizable OC.

Declining OC-normalized CO_2 release and increasing age with soil depth confirm greater stability of OC in subsoils across sites. Depth profiles of LF-OC matched those of roots, which in turn reflect plant functional types in soil profiles not subject to ploughing. Modern $\Delta^{14}\text{C}$ signatures and positive correlation between mineralizable C and fLF-OC indicate the fLF is an easily available energy and nutrient source for subsurface microbes. Fossil C derived from the geogenic parent material affected the age of OC especially in the LF at three study sites. The overall importance of OC stabilization by binding to minerals was demonstrated by declining OC-normalized CO_2 release rates with increasing contributions of HF-OC to bulk soil OC and the low $\Delta^{14}\text{C}$ values of HF-OC. The stability of HF-OC was greater in subsoils than in topsoils; nevertheless, a portion of HF-OC was active throughout the profile. The decrease in $\Delta^{14}\text{C}$ (increase in age) of HF-OC with soil depth was related to soil pH as well as to dissolved OC fluxes. This indicates that dissolved OC translocation contributes to the formation of subsoil HF-OC and shapes the $\Delta^{14}\text{C}$ profiles. While quantitatively less important than OC in the HF, consistent older ages of oLF-OC than fLF-OC indicate that occlusion of

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LF-OC in aggregates also contributes to OC stability in subsoils. Overall, our results showed that association with minerals is the most important factor in stabilization of OC in soils.

1 Introduction

The response of soil organic matter (OM) to environmental changes is determined by mechanisms regulating its turnover at different spatial and temporal scales. Early research evidenced that not all organic carbon (OC) in soils is similarly decomposable and that modelling its dynamics requires considering more than one OM pool (Gårdenäs et al., 2011). Many soil models are based on the idea of OC stability and turnover being determined by the complexity of organic molecular structures. However, because all OM in soil is potentially degradable and chemically labile organic compounds can have old ^{14}C ages, the concept of chemical recalcitrance has been challenged and more emphasis given to spatial separation of substrates and decomposers, stabilization of OC through association with minerals, and soil environmental constraints (Marschner et al., 2008; Schmidt et al., 2011; Fontaine et al., 2007; Dungait et al., 2012).

Density fractionation, which separates organic debris residing inside and outside aggregates (light fraction, LF) from OM bound to minerals (heavy fraction, HF), provides one method to study physical or physicochemical stabilization mechanisms (Golchin et al., 1994; Six et al., 1998; Sohi et al., 2001, 2005). Organic carbon in the LF basically consists of plant and animal residues at different stages of decomposition, while larger contributions of microbial residues are observed in HF-OC (Gregorich et al., 2006; Wagai et al., 2009; Golchin et al., 1994; Poirier et al., 2005; Kögel-Knabner et al., 2008). LF-OC released from soil upon disruption of aggregates (occluded light fraction, oLF) often has an intermediate age and decomposition stage between the physically unprotected free LF (fLF) and the stronger protected HF, but sometimes, oLF-OC can be even older than HF-OC (Wagai et al., 2009; Golchin et al., 1994; Poirier et al., 2005).

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Above- and belowground plant litter input is the main source of OC in the LF (Gregorich et al., 2006). But recent studies indicate that roots are more important for soil OC storage than aboveground litter (Schmidt et al., 2011). Organic carbon storage in the HF is controlled by the availability and nature of binding sites at mineral surfaces.

Iron (Fe) and Al hydrous oxides rather than other clay-sized particles were shown to be good predictors for HF-OC, especially in acid forest soils (Kögel-Knabner et al., 2008; Mikutta et al., 2006; Kaiser et al., 2002). Compounds contributing to HF-OC formation can be direct decomposition products of LF-OC (Sanullah et al., 2011), or dissolved OC (DOC) from the percolating soil solution (Kalbitz and Kaiser, 2008). Modeled estimates of the contribution of DOC to subsoil OC are highly variable and mostly limited to forest soils (Sanderman and Amundson, 2008; Kalbitz and Kaiser, 2008; Michalzik et al., 2003).

Most evidence for the different stability of OC in density fractions comes from stable isotope ($\delta^{13}\text{C}$) and radiocarbon analyses, with the HF usually having the oldest OC (Trumbore, 1993, 2009; Kögel-Knabner et al., 2008). However, studies following the incorporation of “bomb” produced ^{14}C , or determining the ^{14}C age of residues after oxidation or acid hydrolysis of the HF demonstrate that the bulk HF-OC is itself a mixture of younger and older components (Tan et al., 2007; Swanston et al., 2005; Kögel-Knabner et al., 2008; Trumbore, 1993).

Only a few studies relate OC in soil fractions to stability by combining density separation with mineralization experiments. Alvarez and Alvarez (2000), Janzen et al. (1992) and Hassink (1995) observed that microbial biomass and in vitro mineralization rates were positively correlated to LF-OC at grassland and cropland sites when pooling topsoil and subsoil layers. In contrast, Swanston et al. (2002) observed for forest topsoil layers that the decomposability of LF-OC was not different from that of HF-OC when incubating density fractions separately and normalizing mineralization rates to OC in the fractions. Crow et al. (2007) found similar specific decomposition rates for LF and HF-OC in a coniferous, but not a deciduous forest topsoil. They also pointed at methodological problems associated with the incubation of separated density fractions. None

of the studies relating density fractions with soil incubations analysed topsoil and subsoil layers separately.

Consideration of entire soil profiles is required to understand the processes controlling soil carbon storage and how vulnerable SOM is to environmental change. Subsoils contain a large fraction of total OC stocks and are sensitive, for example, to land use changes (Harrison et al., 2003; Don et al., 2009; Rumpel and Kögel-Knabner, 2011). Turnover of topsoil and subsoil OC is probably controlled by different mechanisms, as reflected in ongoing debate as to whether subsoil mineralization rates are limited by energy (Fontaine et al., 2007), nutrients (Fierer et al., 2003), or the physical separation between substrate and decomposers (Salomé et al., 2009). The few studies of density fractions over the entire soil profile show declining contributions of LF-OC to total OC with soil depth (Kögel-Knabner et al., 2008; John et al., 2005). Strongly increasing carbon ages, especially of HF-OC, with soil depth were assumed to be due to greater stability of HF-OC at depth (Kögel-Knabner et al., 2008) and can reflect differences in physicochemical protection between topsoil and subsoil layers. However, observed ^{14}C -depth profiles may also be explained by downward mixing and sorption of aged dissolved OC (DOC) to subsoil layers as suggested by Kaiser and Kalbitz (2012). As most studies on soil OC stability were restricted to topsoil layers, our understanding of subsoil OC is still limited (Rumpel and Kögel-Knabner, 2011).

In summary, density fractionation separates OC into functional meaningful fractions. As density separation is time-consuming, most studies were restricted to a small number of sites and to topsoil layers. In order to overcome those limitations, we conducted a large scale study of entire soil profiles to determine controls of soil OC stability and storage that hold across different soil types, soil depths, vegetation, and land use types. We aimed at verifying conceptual models that emphasize (1) the importance of root litter, DOC, and pedogenic Fe and Al oxides for OC amounts and depth profiles in the LF and the HF, (2) the rapid turnover of physically unprotected low density plant residues, and reduced decomposability of OC occluded in aggregates and in association with minerals, and (3) the relevance of soil depth for OC stability. Multiple replicates

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per site were analysed to quantify the importance of small-scale spatial variability for the OM fractions. Two independent measures were applied to determine OC stability: laboratory incubations, providing potential carbon (C) mineralization rates, and ^{14}C analyses as an indicator of OC turnover under field conditions. Our data set vastly expands available information on the spatial and depth-dependent variation of OC in density fractions, ^{14}C in density fractions, and mineralization-based measures of soil OC turnover.

2 Materials and methods

2.1 Soil sampling and sample treatment

The twelve study sites were selected from the 52 main sites of the CarboEurope IP project (<http://www.carboeurope.org/>) to give geographical spread, equal numbers of different land use types, and to assure reliable long-term eddy covariance measurements. The latter was required since these sites are part of a long-term soil monitoring program (Schrumpf et al., 2011). Details on location, soil type, bulk densities, stone content, climate, land use, and total C stocks of the study sites are given in Table S1 and Schrumpf et al. (2011).

Soil samples were collected in 2004. Deciduous forest sites (Hainich, Germany; Hesse, France; Sorø, Denmark), and coniferous forest sites (Norunda, Sweden; Le Bray, France; Wetzstein, Germany) were sampled from March to December, grassland sites (Laqueuille, France; Easter Bush, UK; Bugac, Hungary) from April to June; and arable soils (Carlow, Ireland; Grignon, France; Gebesee, Germany) in August and September, after harvest. Gebesee and Grignon sites were tilled before soil sampling but Carlow not. At all sites, except for Wetzstein and Norunda, 10 soil cores (8.7 cm diameter, 60 cm length) were randomly selected out of 100 extracted soil cores per site (Schrumpf et al., 2011). Soil cores were divided into seven depth increments: 0–5, 5–10, 10–20, 20–30, 30–40, 40–50, and 50–60 cm. At the stone-rich sites Wetzstein and

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Norunda, samples from 10 soil monoliths (25 cm edge length) were selected. At Wetzstein, three mineral soil layers were sampled: 0–10, 10–30, and 30–50 cm. Soils at Norunda were collected to a depth of 10 cm in two segments (0–5, 5–10 cm). A smaller auger (3 cm diameter) was used to extend the sampling depth to 40 cm with three replicates per site. These subsoil samples could not be used for bulk density determinations, so C stocks were only calculated for the upper soil layers at Norunda. Organic surface layers were collected at the forest sites by horizon (Oi, Oe, Oa), using a metal frame of 25 cm side length prior to mineral soil sampling.

Coarse roots (diameter > 1 mm) and gravel/stones (> 4 mm) were removed from field moist samples in the laboratory before separating a subsample for mineralization experiments. After drying the remaining soil material at 40 °C, another subsample was taken for density fractionation, then the rest of the samples was sieved to < 2 mm. Roots were dried at 70 °C and their weight determined.

2.2 Mineralization rates

Field moist subsamples of organic layers and mineral soils to a depth of 30 cm (corresponding to 16 and 100 g dry weight, respectively) were placed in plastic containers (50 cm² surface area, 466 cm³ volume) fitted with a lid with a 5 mm diameter aperture for gas exchange. These soil microcosms were incubated at 15 °C for 20 days. The soil moisture level was set to 60 % of the water holding capacity, either by addition of distilled water or by letting them dry up to the appropriate water content.

To determine C mineralization in the soil sample, the container lid was periodically replaced with an airtight lid with a rubber septum. Background gas samples were taken after 15 min from the headspace with a syringe and injected into a gas chromatograph (HP 5890, Hewlett Packard Company, Avondale, PA, USA). Measurements were repeated when an appropriate amount of CO₂ had accumulated, from 2 h (humus) to about 5 h (mineral soil), depending on the respiration rate. The mass of C evolved per container and hour was calculated according to Perrson et al. (1989) and Perrson and Wiren (1993), taking the pH-dependent solubility of CO₂ in the soil water into account.

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CO₂ measurements were performed once a week after the starting day, and the mean CO₂ evolution rate per day was based on cumulative estimates up to day 20. Mineralization rate was given as g CO₂-C (g dry weight)⁻¹ d⁻¹ or as a CO₂-C (g C)⁻¹ d⁻¹. Because roots and mycorrhizal mycelia were partly removed by sieving, and since there was a delay of 3 weeks between sampling and start of incubation, we considered the estimated C mineralization to be of heterotrophic and not autotrophic origin.

2.3 Density fractionation

Density fractionation was carried out for 720 samples, with two analytical replicates per sample. The fractionation was performed following ideas of Golchin et al. (1994) and Sohi et al. (2001), using sodium polytungstate (SPT, C and N poor, Tungsten Compounds, Grub am Forst, Germany) solution of 1.6 g cm⁻³ density.

Twenty five g of soil were placed into 750 ml centrifugation bottles and 125 ml SPT solution was added. Bottles were gently shaken by hand to release the free light fraction (fLF-OM). Suspensions were allowed to settle for one hour, and then centrifuged at 5500 g for 30 min. The floating fLF was decanted onto glass fibre filters (GF 6, Whatman GmbH, Dassel, Germany) and filtered under vacuum. The SPT solution was placed back into the centrifugation bottles after adjustment of density. The occluded LF (oLF) was obtained by treating the samples with ultrasound. Tests were performed for each site to determine the sonication energy required for complete aggregate disruption by observing the effects of stepwise increase of sonication energy (Schmidt et al., 1999; Wander, 2004; Cerli et al., 2012). Complete disruption of aggregates was assumed when no further occluded OC was released at the next sonication step and no aggregates were visible any more. OC concentrations of the oLF, visible plant debris still adhering to mineral particles in the HF, and scanning electron imaging were used as indicators for the full separation of oLF and HF-OC (results not shown). Calorimetric calibration of the sonicator (Bandelin GmbH, Berlin, Germany) according to Schmidt et al. (1999) was performed to provide an estimate for the energy applied. Energy input of 100 J ml⁻¹ was sufficient for sandy soils (Bugac, Bordeaux), and between 300 and

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450 J ml⁻¹ for most other soils. For the clay-rich Hainich soil, the energy input had to be raised to up to 900 J ml⁻¹. After sonication, samples were allowed to stand for one hour, and then centrifuged at 5500 g for 30 min. The floating oLF was collected on a glass fibre filter as described above. The fLF and oLF fractions were washed with deionized water to remove SPT until the conductivity of the rinsing solution was < 50 μS. The settled heavy fraction (HF) was washed with deionised water to remove SPT until electrical conductivity was < 200 μS for soils containing carbonates (steady dissolution of carbonates maintained the electrical conductivity at that level) and < 50 μS for all other soils. The SPT collected from the filtration of the light fractions (fLF, oLF) was cleaned and recycled as described by Six et al. (1999) to avoid transfer of C and N between samples. Light fractions were dried at 40 °C and subsequently ground in an agate mortar. The HF were freeze dried, sieved to < 2 mm and ground with a ball mill. Fractionation of samples from the andic soils at Laqueuille was performed on field-moist samples to avoid the occlusion of fLF material within aggregates forming during drying. The density of the applied SPT solution was adapted to the samples' water content to achieve a final solution density of 1.6 g cm⁻³.

2.4 Analyses of bulk soil samples and density fractions

Total C and N in < 2 mm soil separates and density fractions were determined by dry combustion (VarioMax CH analyser for bulk samples and heavy fractions; Vario EL for light fractions; both Elementar Analysensysteme GmbH, Hanau, Germany). At the sites Hainich, Gebesee and Carlow, the carbonate C of bulk samples was determined after dry combustion of the samples in a muffle furnace at 450 °C for 16 h. Carbonate C of bulk soil samples of the sites Bugac and Sorø, of heavy fractions of the sites Hainich, Gebesee, Carlow, Bugac and Sorø was analysed by determining the evolution of CO₂ upon treatment with phosphoric acid (C-MAT 550, Ströhlein GmbH, Viersen, Germany). Concentrations of OC were calculated as difference between total and inorganic C. Stocks of OC and TN were calculated based on the fine earth mass per area for each

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sample (Schrumpf et al., 2011). For $\delta^{13}\text{C}$ analyses, ground samples were weighted into tin capsules, and combusted in an oxygen stream using an elemental analyser (NA 1110, CE Instruments, Milan, Italy). Samples having carbonates were treated with sulphuric acid inside the capsules, and dried at 60 °C before combustion. Evolved CO_2 was analysed using an isotope ratio mass spectrometer (IRMS; Delta C or DELTA+XL, Thermo Finnigan MAT, Bremen, Germany). Isotope ratios are given as $\delta^{13}\text{C}$ in per mille (‰) relative to the international reference standard v-PDB using NBS19 (Werner and Brand, 2001).

Three to five replicates of three soil depths (0–5, 10–20, 30–40 cm) were analysed for ^{14}C of all sites except for Le Bray; these samples were lost during postal transfer. For sites where density fractions showed no contribution of fossil C, corresponding bulk samples were also analysed. Sample preparation and analyses were performed at the ^{14}C laboratory at Jena, Germany (Steinhof et al., 2004). Samples were weighed into tin capsules and combusted in an elemental analyser. Samples containing carbonate were decalcified prior to combustion (as for $\delta^{13}\text{C}$ analyses described above). The evolved CO_2 was transferred into a glass tube cooled by liquid nitrogen and containing an iron catalyst. Reduction of CO_2 to graphite was carried out at 600 °C under hydrogen gas atmosphere. The graphite was analysed by ^{14}C AMS (3MV Tandetron 4130 AMS ^{14}C system: High Voltage Engineering Europe, HVEE, The Netherlands). ^{14}C is given in per mille (‰) $\Delta^{14}\text{C}$, which is the relative difference in activity with respect to a standard (oxalic acid standard NBS SRM 4990C), after normalization to $\delta^{13}\text{C}$ (fractionation correction) and correction for decay between 1950 and analyses in 2009 and 2010.

Soil pH was measured in the supernatant after shaking a soil–distilled water suspension (1 : 1 by volume) for 2 h, followed by sedimentation for 22 h. Oxalate-extractable iron and aluminium (Fe_o, Al_o) were determined using 0.2 M oxalate solution (pH 3) (Schwertmann, 1964). Dithionite-extractable iron and aluminium (Fe_d, Al_d) were analysed by the cold dithionite–citrate method (Holmgren, 1967). Aluminium and iron in extracts were measured by Inductively-Coupled Plasma Atomic Emission Spectroscopy

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(ICP-AES, Optima 3300 DV, Perkin-Elmer, Norwalk, CT, USA). Particle size distribution was carried out following Schlichting and Blume (1964), with fractions $< 63 \mu\text{m}$ being analysed using a Sedigraph 5100 (Micromeritics Corp., Nocrass, USA).

2.5 Statistical analysis

5 Results are presented as means \pm standard error unless indicated otherwise. Analyses of variance (ANOVA) were used to test for significant differences between land use types or fractions ($p < 0.05$). Scheffé tests were used for post-hoc analysis. To determine relations between variables, Pearson correlation coefficients were calculated and tested for significance with a two tailed t-test ($p < 0.05$, or $p < 0.01$, as given in
10 figures). Statistical analyses were performed using the software-package SPSS 16.0 for Windows.

3 Results

3.1 Density fractionation

3.1.1 General trends for organic carbon, total nitrogen, and carbon isotopes in 15 density fractions of soil profiles

Mass losses and OC recoveries during density fractionation for each site are given in Table S2. General trends in depth distributions of OC contents, OC-to-TN-ratios, $\delta^{13}\text{C}$, and $\Delta^{14}\text{C}$ values for undisturbed soil profiles are presented in Fig. 1, while results of the cropland sites and the ploughed grassland site Easter Bush are shown in Fig. 2.

20 Results of all individual sites are given in Fig. S1 and Table S3.

The study sites cover a range of climatic and geologic regions, resulting in a variety of different soil types and characteristics (Table S1). Accordingly, distribution of OC over LF and HF varied between sites and total OC stocks in 0–60 cm ranged from 0.4 to 11.2kgm^{-2} for the LF and from 5.6 to 22.0kgm^{-2} for the HF (Figs. 1, 2, 3a).

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In accordance with total OC, stocks of LF-OC and HF-OC were largest in the uppermost layers and declined with depths at all sites. The decline with depth was more pronounced for LF-OC than for HF-OC. Accordingly, significantly larger proportions of total LF-OC ($43 \pm 5\%$) than of HF-OC ($29 \pm 2\%$) stocks were in the upper 10 cm of the mineral soil. On average across sites, the contribution of LF to total OC decreased from $30 \pm 16\%$ (S.D.) in the 0–5 cm layer to $14 \pm 12\%$ in the 50–60 cm layer.

The OC-to-TN ratios differed significantly ($p < 0.05$) between fractions in the 0–5 cm layer and decreased in the order fLF (28 ± 3) > oLF (24 ± 3) > HF (14 ± 2) across sites. The HF contributed on average $8 \pm 1\%$ more to TN stocks than to OC stocks in that layer. The OC-to-TN ratios of HF decreased with soil depth for most sites while OC-to-TN ratios of LF increased (Fig. 1).

The low $\Delta^{14}\text{C}$ values of the LF of the sites Easter Bush, Carlow, and Grignon compared to other sites suggest high contributions of fossil C (Fig. 2). This assumption is supported by wide OC-to-TN ratios (Fig. 2). Carlow and Easter Bush both developed on carboniferous sedimentary rocks and Grignon on loess over tertiary or cretaceous sedimentary rocks. Accordingly, fossil C from the parent material could contribute to OC at these sites.

Otherwise, most fractions from the 0–5 cm layer had positive $\Delta^{14}\text{C}$ values, reflecting influence of bomb-produced radiocarbon (except for Gebesee and Wetzstein), and $\Delta^{14}\text{C}$ decreased in the order fLF > oLF > HF. In the 0–5 cm layer, only the fLF and HF were significantly different ($p < 0.05$). Correlations between $\Delta^{14}\text{C}$ of fLF and of oLF as well as between $\Delta^{14}\text{C}$ of oLF and HF at 0–5 cm point at a link between turnover times of fractions in the uppermost but not in deeper soil layers (Fig. 4).

The $\Delta^{14}\text{C}$ of fLF showed no consistent trend with depth across sites and was usually above zero, while the $\Delta^{14}\text{C}$ of oLF decreased with depth (Fig. 1). Except for the croplands, where the soil profiles were disturbed by ploughing, also the $\Delta^{14}\text{C}$ of HF decreased linearly with soil depth (Fig. 1). Accordingly, differences between fractions became more pronounced at deeper soil layers.

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The comparatively heavy $\delta^{13}\text{C}$ values of fLF and oLF of the semiarid grassland site Bugac are due to the presence of C4 grasses, which were not observed at the other sites. Excluding that site and the sites affected by fossil C, the HF had significantly less negative $\delta^{13}\text{C}$ values than fLF and oLF at all soil depths, while no general trend for differences between $\delta^{13}\text{C}$ values of fLF and oLF was found (Fig. 1). The $\delta^{13}\text{C}$ of all fractions increased with depth.

3.1.2 Impact of land use and vegetation type

Homogenization of topsoil layers by ploughing was evident in the depth profiles of OC concentrations and stocks, OC-to-TN ratios, $\delta^{13}\text{C}$, and $\Delta^{14}\text{C}$ of density fractions at the cropland sites (Fig. 2). Former ploughing affected the OC depth distribution of the grassland site at Easter Bush and the conifer forest at Le Bray.

Coniferous forests and grasslands had the largest LF-OC stocks in the upper 0–10 cm of all studied land uses (average 1.38 ± 0.57 and $1.17 \pm 0.19 \text{ kg m}^{-2}$, respectively), while the croplands had the lowest stocks of LF-OC ($0.42 \pm 0.15 \text{ kg m}^{-2}$). The proportion of total OC stored as LF in the 0–10 cm soil depth was largest for the coniferous forest ($45 \pm 10\%$) and smallest for the cropland sites ($14 \pm 5\%$). Including the profiles down to 60 cm soil depths, croplands maintained the smallest LF-OC stocks of all land use types (Fig. 3). Greater contributions of LF-OC at Grignon were due to larger amounts of old (fossil) LF-OC, which does not reflect the current land use (Fig. 2).

The depth distribution of LF-OC also varied with land use. Contribution of LF-OC stored in the upper 0–10 cm to LF-OC within entire soil profiles was significantly larger at the grassland ($71 \pm 5\%$, excluding the ploughed site Easter Bush) than at the deciduous forest ($41 \pm 3\%$) and the cropland sites ($35 \pm 3\%$, excluding Grignon). Similarly, roots in the 0–10 cm layer of grasslands contributed more to total root masses within entire soil profiles than of deciduous forest soils ($78 \pm 3\%$ at grasslands; $43 \pm 4\%$ at deciduous forests) (Fig. 5). Overall, the relative contributions of the fLF-OC and oLF-OC in the 0–10 cm depth interval to the LF-OC and oLF-OC summed over the entire

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profile both correlated with the proportion of roots sorted out of the 0–10 cm layer compared to the whole profile (Fig. 6). The HF-OC declined less strongly with depth than LF-OC at the grassland sites, with $30 \pm 6\%$ of the HF-OC of the entire profiles located in the 0–10 cm layer (Fig. 5).

5 The link between the mass of sorted roots and OC storage in density fractions is further supported by the positive correlation between roots and fLF-OC as well as HF-OC in the 0–10 cm layer (Fig. 7). For fLF-OC of the coniferous forest sites and the sandy site Bugac, this relationship differed from those found for the remaining sites.

3.1.3 Soil properties influencing density fractions

10 The fraction of OC present as LF in the 0–10 cm layer depended on soil texture and pH (Fig. 8). The contribution of the LF to total OC declined with increasing clay content, while the proportion of HF-OC increased accordingly. The coniferous forests with low pH had larger proportions of OC in the LF than the other sites.

15 Absolute HF-OC concentrations across sites in the 0–10 cm layer did not depend on the clay content but were positively correlated to oxalate- and dithionite-extractable Fe as well as to oxalate-extractable Al (Fig. 9). Also, stocks of HF-OC to 30 cm depth related positively to stocks of oxalate-extractable Al ($r = 0.85$, $p < 0.01$) and Fe ($r = 0.82$, $p < 0.01$) and of dithionite-extractable Fe ($r = 0.85$, $p < 0.01$) at the same depth (data not shown). Figure 10 shows that more OC was with the LF at sites with larger OM loading on minerals in the uppermost soil layer. Relations between texture or pedogenic oxides and OC in density fractions were less clear in subsoils, and either not significant or dependant on one sampling site only (i.e. the Andosol at Laqueuille for pedogenic oxides).

3.2 Mineralization rates

25 Evolution of CO_2 per g soil showed large variation among sites (Fig. S2) and decreased with soil depth from a mean (\pm S.E. for all sites) of $13.7 \pm 8.4 \mu\text{gCO}_2$ -

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$C(gdwt)^{-1} day^{-1}$ in the 0–5 cm layer to $1.5 \pm 0.7 \mu g CO_2-C(gdwt)^{-1} day^{-1}$ in the 20–30 cm layer (Fig. 11). With the organic layers included, and summing over all depths, incubation-based total soil heterotrophic respiration rates were, on average, 3.26 ± 0.31 , 2.44 ± 0.19 , 1.43 ± 0.34 , and $0.91 \pm 0.11 g CO_2-C m^{-2} day^{-1}$ for the coniferous, deciduous, grassland, and cropland sites, respectively (Fig. 3b). The organic layers contributed on average $73 \pm 8\%$ and $37 \pm 8\%$ to the total CO_2 evolved in incubations of the coniferous and deciduous forest sites.

Mineralization rates for all sites and soil depths were positively correlated to OC concentrations of the incubated soil samples (Fig. 12). In the uppermost soil layer (0–5 cm), respiration rates were also positively related to OC of all three density fractions, although CO_2 -release per g OC in the fraction was greater for the LF than for the HF (Fig. 13). The sandy soils of the sites Bugac and Le Bray had mineralization rates lower than expected from the proportion of OC in the fLF and from total OC (Fig. 12 and 13). In addition, mineralization rates correlated positively with the amounts of roots extracted from the 0–10 cm layer across all sites ($r = 0.81$, $p < 0.01$, data not shown), indicating that mineralization rates relate to the input of fresh OC entering the soil as roots. No significant relation between absolute mineralization rates and the OC in density fractions was observed across sites in deeper soil layers.

As absolute mineralization rates depend not only on the decomposability of OC in the samples, but also on the total amount of OC in the samples, OC-normalized CO_2 -release (specific mineralization rates) should be used for correlations with OC quality indexes and with $\Delta^{14}C$. Similar to absolute mineralization rates, specific mineralization rates also declined consistently with soil depth (except for the grassland site Bugac), indicating reduced OC turnover at depth (Fig. 11, Fig. S2).

In order to determine the relation between density fractions and specific mineralization rates, we excluded the sites Grignon (large contribution of old OC not linked to current vegetation to LF) and Le Bray (high in subsoil LF due to deep ploughing before forest planting) from statistical analyses but still present them in Fig. 14. No significant relation between OC with density fractions and specific mineralization rates was found

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for the uppermost soil layer (Fig. 14). In deeper soil layers however, specific mineralization rates were positively correlated with the contribution of fLF to total OC, and negatively with the contribution of HF. Here, OC-normalized CO₂-release rates were also significantly positively related to the absolute amount of fLF-OC and the OC-to-TN ratio of the fLF. Specific mineralization rates also correlated significantly with the contribution of fLF and HF to total OC when values were integrated over the upper 30 cm of the mineral soil ($r = 0.76$, $p < 0.01$ for fLF and $r = -0.61$, $p < 0.05$ for HF, correlations not shown).

Release of CO₂ per unit OC was negatively related to bulk soil $\Delta^{14}\text{C}$ at the 10–20 cm depth, indicating faster turnover of more recent OC (Fig. 15). Variable contributions of bomb C and smaller differences in ^{14}C between fractions probably obscured correlation across sites for the 0–5 cm layer.

4 Discussion

4.1 Sources and controlling factors of organic carbon in density fractions

The results of our large-scale study support previous work showing that density fractionation separates total soil OC into fractions of different OC-to-TN ratios, $\delta^{13}\text{C}$ values, and $\Delta^{14}\text{C}$ values (Alvarez and Alvarez, 2000; Janzen et al., 1992; Swanston et al., 2005; Golchin et al., 1994). Isotopic signatures and the OC-to-TN ratios of density fractions were more similar in the uppermost than in deeper soil layers, indicating a stronger decoupling of fractions at depth. General characteristics of organic matter in density fractions of total soil profiles are summarized in Fig. 16.

Above- and belowground plant litter and animal residues are considered as main sources for LF-OC (Gregorich et al., 2006). However, also charred or fossil C can contribute significantly to LF-OC (Marschner et al., 2008; Wagai et al., 2009; Gregorich et al., 2006). Rumpel and Kögel-Knabner (2011) pointed out that substrate-derived geogenic C can influence bulk ^{14}C values of subsoils. Fossil C contributed to LF-OC at

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three out of our 12 sites. Greater contributions of old OC to deeper soil layers suggest that this fossil C (like coal) rather derived from the sedimentary parent material than from contamination by deposits of industrial old C as observed by Rethemeyer et al. (2004). Hence, possible contributions of old OC from the parent material need to be considered when interpreting ^{14}C data.

Our results show that roots rather than above ground litter are the main sources for LF-OC. The fLF-OC in upper soil layers is directly related to the root mass removed from the soil before analysis. Jobbagy and Jackson (2000) showed that vegetation type influences depth distributions of total soil OC through differences in C allocation. Our results suggest general correspondence in depth distributions between roots and fLF as well as oLF fractions for unploughed grassland and deciduous forest sites, with less of a correspondence for HF fractions. These results are overall in line with recent studies showing that roots are more stable and more important for soil OC formation than aboveground litter (Mendez-Millan et al., 2010; Bird and Torn, 2006; Kong and Six, 2010; Kramer et al., 2010).

Differences in the rooting systems of plant functional types therefore affect the depth distribution of LF-OC. Grassland soils had larger concentrations of roots and LF-OC in the uppermost layers than deciduous forest soils, which had relatively more roots and LF-OC in deeper soil layers. However, LF-OC contents of the coniferous forests were greater than expected from the amount of sorted roots from non-coniferous sites included in our study. Large LF-OC contents in coniferous forests have been observed before (e.g. by Crow et al., 2007; Laganière et al., 2011; Kaiser et al., 2002), and may reflect the additional influence of environmental constraints, such as soil acidity, or reduced litter quality (wide OC-to-TN ratio in conifers). The destruction of soil structure by tillage, subjecting formerly occluded OC to microbial degradation, has been assumed to be the reason for the reduced contributions of LF to total OC stocks in croplands compared to forests or grasslands (Balesdent et al., 2000; Alvarez et al., 1998; Roscoe and Buurman, 2003; John et al., 2005). Grubber-tillage before sampling caused mixing of fresh harvest residues into the upper soil layer at the sites Gebesee and Grignon.

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Accordingly, soil management, but also sampling time, influence the contribution of LF to total OC of croplands.

Increasing LF-OC with decreasing clay contents, and increasing LF-OC at higher OC loadings of clay particles or pedogenic oxides in the uppermost soil layers indicate a greater importance of LF-OC for OC storage at sites with limited sorption capacity of the HF. Gulde et al. (2008) showed in an agricultural experiment that increasing C input at some point only leads to increases in particulate OM, while other fractions stagnated. There seems to be a similar situation at our topsoils, but this does not imply that OC accumulation simply continues in the LF once HF is OC saturated. Due to the fast turnover of LF-OC in topsoils, increases in litter input will only lead to small stock changes in the LF. There were no clear indications of greater LF-OC stability with increasing amounts of HF-OC per g clay-sized minerals or pedogenic oxides across sites from ^{14}C data. Instead, low pH and litter quality reduce decomposition rates at the sandy coniferous forest sites, and environmental constraints like drought could have promoted accumulation of LF-OC at the sandy Hungarian site Bugac.

Our study confirmed previous observations that secondary hydrous Fe and Al phases are generally more important to OC accumulation in the HF than clay-sized particles (Kögel-Knabner et al., 2008; Torn et al., 1997; Kaiser and Guggenberger, 2000). The OC-loading of minerals typically decreased with soil depth as for example the ratio of HF-OC to $\text{Al}_\text{o} + \text{Fe}_\text{d}$ declined by roughly 60 % from the 0–5 cm to the 10–20 cm soil layer at the deciduous forest sites. Little to no relation between mineral indicators and OC can be expected when available binding sites at mineral surfaces are not occupied. This could be one explanation for weaker or no correlation between HF-OC and pedogenic oxides across sites for deeper soil layers.

Decomposition products of LF-OC are a possible source for HF-OC upon sorption to mineral surfaces. Positive relations between $\Delta^{14}\text{C}$ of fLF-OC and oLF-OC, and between oLF-OC and HF-OC indicate that these fractions are not entirely independent of each other in the topsoil. The relevance of roots as source of HF-OC is indicated by the relation between root mass and HF-OC in the uppermost soil layer and supported by

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several studies highlighting the role of roots for soil OC storage (Rasse et al., 2005; Sanaullah et al., 2011; Bird and Torn, 2006). As root-derived C is quickly incorporated into microbial biomass (Liang et al., 2002), and microorganisms in soils are directly attached to particle surfaces or in biofilms around them (Mills, 2003), microbial residues and metabolites contribute to HF-OC formation (Poirier et al., 2005).

Direct transfer of OC from the LF to the HF would result in roughly similar depth distributions of both fractions; however, subsoils had much more HF- than LF-OC, especially at the grassland sites. Several studies suggested that transport of DOC contributes significantly to OC accumulation in subsoils (Baisden and Parfitt, 2007; Sanderman and Amundson, 2008; Kalbitz and Kaiser, 2008; Kaiser and Kalbitz, 2012). Different sorption rates of younger DOC from overlying horizons to minerals in subsoils should be reflected in different depth profiles of the $\Delta^{14}\text{C}$ of the HF. The $\Delta^{14}\text{C}$ of the HF at our study sites declined almost linearly with soil depth down to the 30–40 cm layer at undisturbed sites but slopes of the decline varied among sites (see example in Fig. 17 and Fig. S1). These slopes were positively correlated with the DOC fluxes measured by Kindler et al. (2011) at some of the study sites, and with the amounts of dissolved OC sorbed in the subsoil (Fig. 17). A small increase in soil age with depth at sites with large fluxes and large sorption of DOC indicates an overall faster transport of younger OC into deeper soil layers. This is supported by the negative relation between soil pH and the slopes of the $\Delta^{14}\text{C}$ depth decline, which points at deeper penetration of younger OC with larger fluxes of dissolved OC in acidic soils (Fig. 17). Accordingly, the age-gradient of HF-OC in the upper parts of soil profiles can be an indicator for transport rates of DOC. As suggested by Masiello et al. (2004), our study shows, based on independent measures, that DOC is relevant to the formation of HF-OC in subsoils, irrespective of texture, acidity, or land use type. The $\Delta^{14}\text{C}$ values of the HF of subsoils, therefore, reflect not only OC turnover in the given layer but the entire residence time of OC in the soil profile.

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4.2 Link between organic carbon stability and density fractions in topsoils and subsoils

The combination of OC-normalized CO₂-release rates from incubations and $\Delta^{14}\text{C}$ values as indicators for OC stability was useful to overcome limitations of each approach. Incubations give potential mineralization rates of labile OC fractions and exclude interactions between plants and soil. Still, Kutsch et al. (2010) showed for the Hainich site that measured heterotrophic soil respiration rates in the field compare well to those obtained in our laboratory incubations, after correcting for soil temperature and moisture. The $\Delta^{14}\text{C}$ of soils is one of the most important tracer for cycling of soil C under field conditions on timescales of years to decades (Trumbore, 2009; Torn et al., 2009). However, at sites having charred or fossil OC in the LF, laboratory incubations gave better estimates of the fast cycling OC than radiocarbon measurements. Direct correlations between OC-normalized CO₂-release rates and $\Delta^{14}\text{C}$ values can only be expected for OC fractions with homogenous turnover times (Torn et al., 2009). This could be one reason why we did not find good correlations between $\Delta^{14}\text{C}$ data and incubation results in the uppermost soil layer.

Modern $\Delta^{14}\text{C}$ values of all fractions in the uppermost soil layer of sites not containing fossil C, and correlations between CO₂-release rates and OC in all three density fractions suggest that OC in all three fractions turns over fast enough to contribute significantly to mineralization rates. Nevertheless, stronger responses of respiration rates to contents of fLF-OC than of HF-OC hint at fLF-OC being more bioavailable than HF-OC. Also lower $\Delta^{14}\text{C}$ of the HF than of the fLF indicate that part of HF-OC in topsoils has longer turnover times, and thus, is stabilized against degradation. Alvarez and Alvarez (2000) showed that fLF-OC was closely linked to microbial biomass. Accordingly, it seems that the input of fresh, potentially energy-rich OM with plant litter promotes overall greater biological activity.

Declining specific mineralization rates and increasing C age with soil depth suggest a general increase in the stability of OC with soil depth across sites. Similar results have

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been obtained by Fang and Moncrieff (2005), Lomander et al. (1998) and by studies summarized in Kögel-Knabner et al. (2008). Especially HF-OC strongly increased in age with soil depth, while fLF-OC mostly remained modern (see e.g. studies in Kögel-Knabner et al., 2008). Reduced bioavailability of HF-OC at depth is supported by the negative correlation between OC-normalized CO₂ release and the proportion of OC with the HF, which is significant across sites, except for the uppermost soil layer. One explanation for stronger protection of mineral-associated OC in deeper soil layers is stronger sorption of OC to mineral surfaces with smaller OM loading, causing longer residence times of OC at mineral surfaces and reduced desorption, which is prerequisite for degradation of mineral-bound OC (Guggenberger and Kaiser, 2003; Kaiser et al., 2007; Kögel-Knabner et al., 2008). The correlation between OC-normalized CO₂-release rates and bulk sample $\Delta^{14}\text{C}$, and the negative correlation between bulk sample $\Delta^{14}\text{C}$ and the contribution of HF to total OC at 10–20 cm depth, confirms independently slower turnover of OC in deeper soil layers with more OC in the stabilized HF.

In case of HF-OC being less bioavailable at depth, LF-OC would become even more relevant as an energy and nutrient source in deeper soil layers. This is in agreement with the higher $\Delta^{14}\text{C}$ values and explains the positive relation between OC-normalized CO₂ release, fLF-OC, and the proportion of OC with fLF for deeper soil layers. Greater OC-normalized CO₂ release at sites with large OC-to-TN ratios in the fLF suggests that microbial growth in subsoils is N-limited, so that supplying N demands requires complete processing of the fLF. This is supported by increasing OC-to-TN ratios of fLF-OC with depth at most sites, probably resulting from lower fine root N-contents in deeper soil layers (Ugawa et al., 2010; Genenger et al., 2003; Göransson et al., 2007). The increase in TN relative to OC in HF with depth hints at unequal nutrient bioavailability in the two fractions, possibly because of better protection of OM in the HF, or large contributions of biologically unavailable mineral N in subsoils. Accordingly, the amount of OC with and the OC-to-TN ratio of the fLF were good indicators of OC turnover in subsoils. As absolute mineralization rates in subsoils were linked to total OC, which was dominated by HF-OC, a portion of that fraction seems to be active

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throughout the profile. This is in line with previous findings by Swanston et al. (2005), Tan et al. (2007) and Trumbore (1993).

Differences in OC-to-TN ratios and $\delta^{13}\text{C}$ values between fLF and oLF were not consistent enough to support the idea of the oLF being generally more degraded than the fLF. Overall, oLF rather resembled fLF than HF in terms of $\delta^{13}\text{C}$ and OC-to-TN ratio while having an intermediate ^{14}C age, ranging between that of the fLF and the HF. This hints at some stabilization of LF-OC by occlusion within aggregates, especially in deeper layers, as suggested by Rasmussen et al. (2005) and Salomé et al. (2009). The rather small portion of OC with the oLF as compared to HF-OC might explain why no correlation between specific respiration rates and oLF-OC was found. Also, it was not possible to identify single factors or processes explaining oLF-OC contents across sites possibly due to variable contributions of selectively preserved organic material with large OC-to-TN ratios (like charred material), as suggested by Wagai et al. (2009). In addition, methodological problems must be considered when attempting to distinguish between “aggregate-protected” and “unprotected” organic matter. For example, larger organic particles, such as degrading roots, may be partially within aggregates but also partially without. In addition, sonication treatments, while optimized for aggregate disruption, have also been shown to cause redistribution of OC across fractions by differential dispersion of HF material (Kaiser and Guggenberger, 2007). For example, the large amount of oLF-OC in the sandy soil at Le Bray (Fig. S1) likely was not from within aggregates (the soils are weakly aggregated) but originates from disruption of weak organic-sand associations upon sonication. Characterizing oLF-OC is therefore more difficult than the fully unprotected fLF-OC and the mineral-associated OC.

5 Summary

The distribution of OC over density fractions determined the overall stability of subsoil OC for all types of vegetation, land use, and soil. The combined application of density fractionation, laboratory incubations, and isotope analyses across a range of sites

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mostly confirmed conceptual models of OC stability in soils. The following conclusions and implications arise from our study:

5.1 Sources of LF-OC and HF-OC

Root distribution is a good predictor for LF-OC along the soil profile. Geogenic C and charcoal can also contribute to LF-OC at certain sites, strongly impacting $\Delta^{14}\text{C}$ values. Direct transfer of fLF or oLF-OC to HF-OC in the course of LF decomposition seems relevant to the formation of HF-OC, especially in topsoils. Fast turnover of young fLF-OC indicates that it probably contributes less to the old HF-OC found in subsoils. Instead slow downward migration of DOC is a more important source of HF-OC in subsoils, contributing to the greater difference of isotopic signatures and OC-to-TN ratios between fractions in deeper soil layers.

5.2 Association with minerals provides protection against mineralization

Our study confirmed association with minerals as the most important OC stabilization mechanism in the soil. The separation into a rather active fLF-OC and a largely inactive HF-OC was more pronounced in deeper soil layers than in the topsoil, probably as a result of greater stability of HF-OC at depth. Nevertheless, a more rapidly cycling component of the HF-OC contributed to CO_2 release in incubations, as already observed in some previous studies. In topsoils, HF-associated organic matter might be of more transient nature, prone to steady replacement by freshly produced reactive organic compounds.

5.3 Occlusion within aggregates increases the longevity of LF-OC throughout the soil profile

Occluded LF-OC often has an intermediate ^{14}C signature, between that of fLF-OC and HF-OC. The heterogeneous composition of oLF-OC does not allow for ascribing the reason for greater stability either to spatial separation from decomposers or the

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presence of refractory or charred compounds. Since far less of the total OC is found with the oLF than with the HF, binding to minerals is the more relevant stabilization mechanism, especially in subsoils.

5.4 fLF-OC fuels biological activity

The OC in the fLF and its OC-to-TN ratio were universal predictors of the biodegradability of soil OC in top- as well as in subsoils and represents fresh, unprotected OC. Enhanced root litter input will promote biological activity and turnover of a larger carbon stock as suggested by Bird et al. (2011). Whether, and how, root litter input facilitates the breakdown of HF-OC in subsoils is still to be resolved. The close correlation between fLF-OC and the CO₂-release per g soil OC indicates that decomposition was mostly independent of site-specific differences in root litter quality or mineral composition in deeper soil layers.

5.5 Limited OC storage capacity in soils

One consequence of binding to minerals being the most important stabilizing process in soils is that long-term soil OC storage is finite. This is supported by greater shares of LF-OC in soils with small sorption capacity for OC of minerals. Unless other environmental constraints hamper decomposition, the OC storage capacity of soils will depend on the availability of binding sites and, accordingly, on the soil's mineral composition and depth.

5.6 Interpretation of ¹⁴C data and modelling of OC turnover

Conversion of $\Delta^{14}\text{C}$ values of density fractions in a given soil layer into turnover rates needs to consider: (1) possible contributions of fossil C, either of industrial sources or inherited by the parent material; (2) transfer of OC between fractions, (3) OC transport along the soil profile, and (4) lags between the time of fixation and the time OC enters the soil (Gaudinski et al., 2001; Sah et al., 2011; Trumbore, 2009). As this requires

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a more complex modelling approach and further constraints, such as litter input, this is beyond the scope of this study and remains a future task.

Supplementary material related to this article is available online at:

<http://www.biogeosciences-discuss.net/9/13085/2012/>

[bgd-9-13085-2012-supplement.pdf](#).

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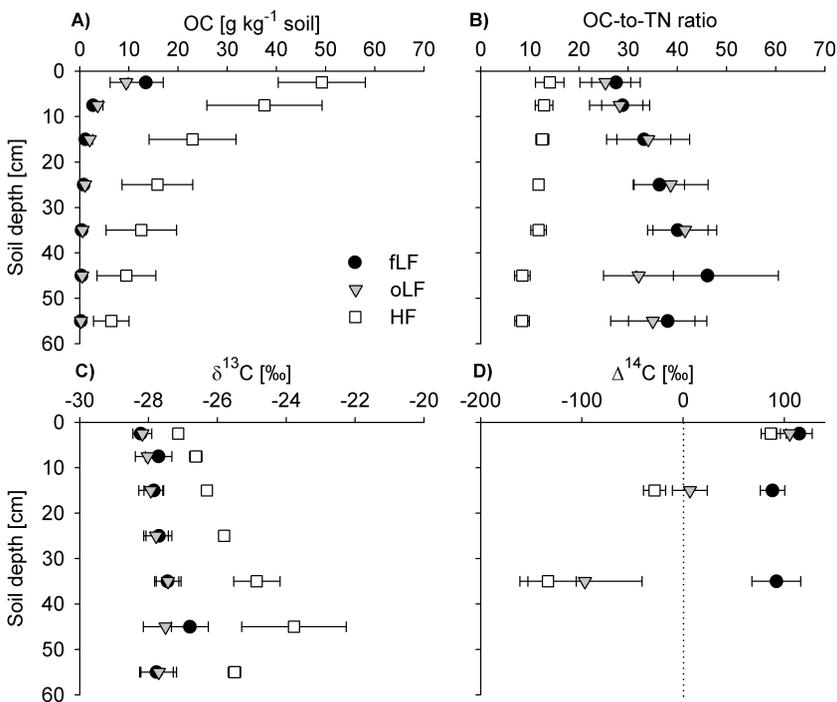


Fig. 1. Depth profiles of average **(A)** masses of OC in the fLF (free light fraction), oLF (occluded light fraction) and HF (heavy fraction) per kg soil, **(B)** OC-to-TN-ratios of fractions, **(C)** $\delta^{13}\text{C}$ of OC in fractions, and **(D)** $\Delta^{14}\text{C}$ values of OC in fractions of unplowed sites (Hainich, Hesse, Soroe, Bugac, Laqueuille, Norunda). As C₄ grasses occurred at the grassland site Bugac it was excluded from the average of $\delta^{13}\text{C}$ values. Error bars are standard errors ($n = 6$ except for $\delta^{13}\text{C}$ $n = 5$). Wetzstein was not included because of different depth increments analysed (see Fig. S1). TN contents close to the detection limit in some subsoils probably contributed to greater variability of OC-to-TN ratios at depth.

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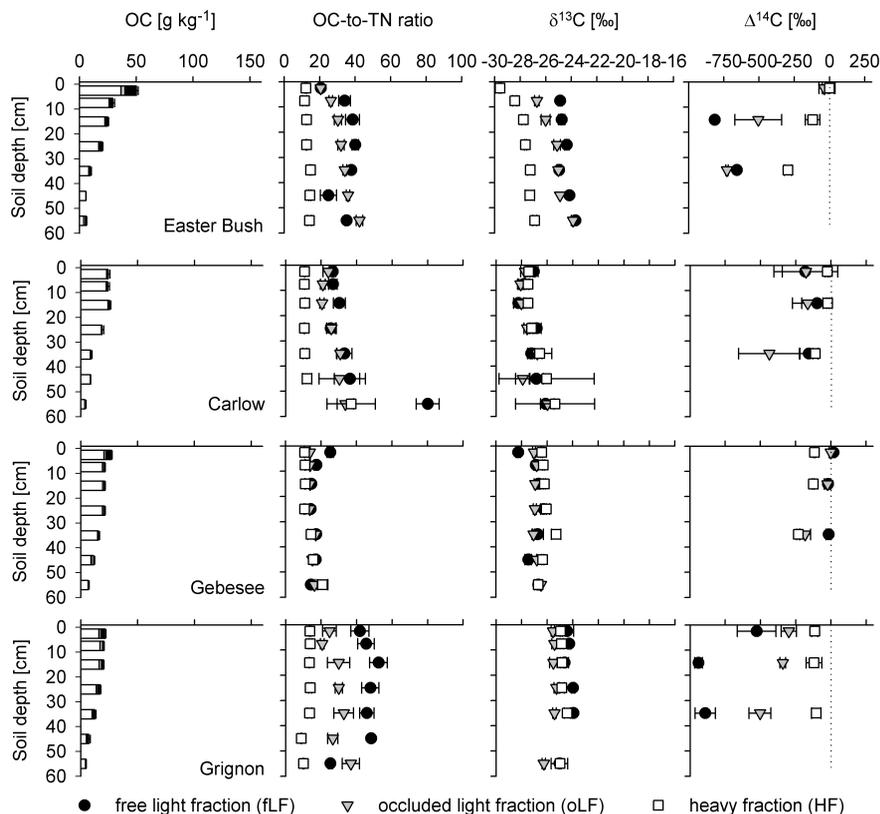


Fig. 2. Depth profiles of average masses of OC in the fLF, oLF and HF per kg soil, the OC-to-TN-ratios of fractions, the $\delta^{13}\text{C}$ of OC in fractions, and the $\Delta^{14}\text{C}$ values of OC in density fractions for the cropland sites Carlow, Gebesee, and Grignon, and the plowed grassland site Easter Bush (fLF: free light fraction, oLF: occluded light fraction; HF: heavy fraction).

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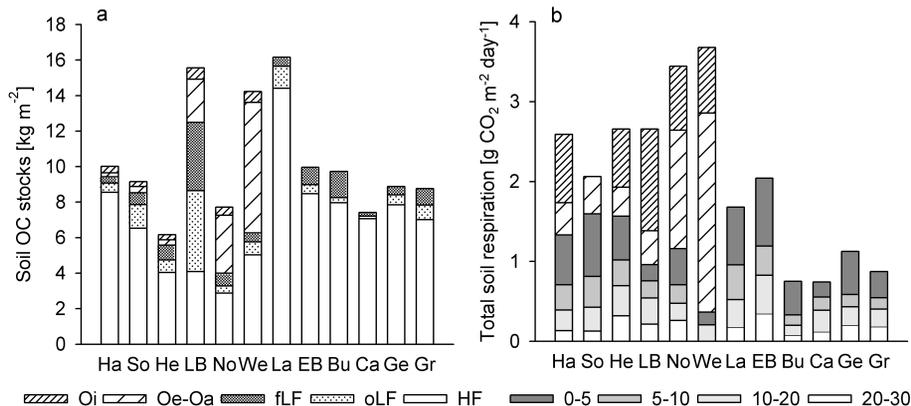


Fig. 3. The left graph **(a)** presents soil OC stocks of the litter layer (Oi, Oe-Oa) and in density fractions (fLF: free light fraction, oLF: occluded light fraction, HF: heavy fraction) of the mineral soil (0–30 cm); the right graph **(b)** C mineralization rates per unit area for each studied soil layer (Wetzstein: first bar below Oe-Oa is 0–10 cm, second bar 10–30 cm).

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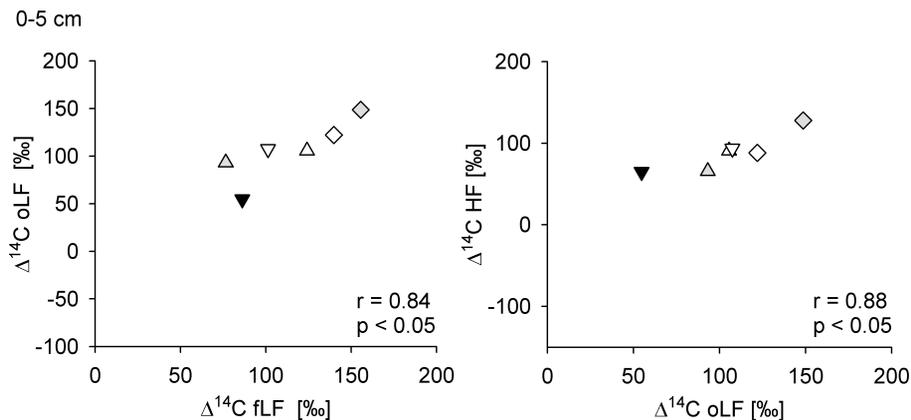


Fig. 4. Relation between the $\Delta^{14}\text{C}$ of fLF (free light fraction) and oLF (occluded light fraction) (left), and between the OC of the oLF and HF (heavy fraction) at 0–5 cm soil depth. Only undisturbed sites having no fossil C are shown (Hainich, Hesse, Soroe, Bugac, Laqueuille, Norunda).

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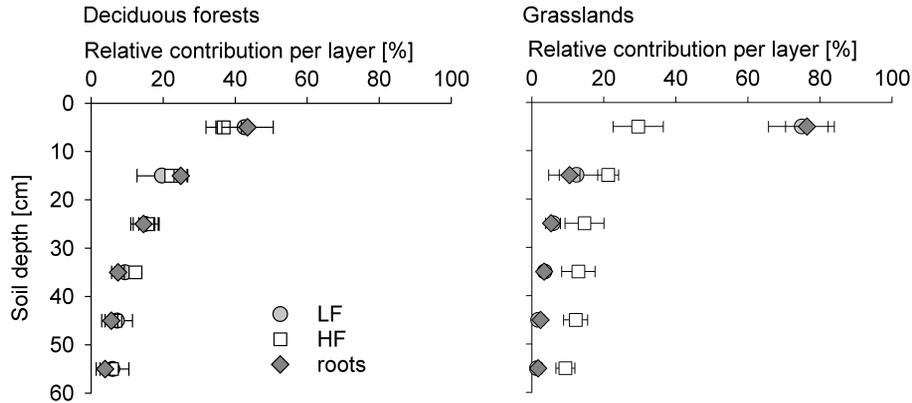


Fig. 5. Relative contributions of 10 cm depth increments to total stocks in 0–60 cm (100%) of OC with LF (light fraction) and HF (heavy fraction) and roots sorted out during sample preparation. Averages for deciduous forest and undisturbed grassland sites are shown.

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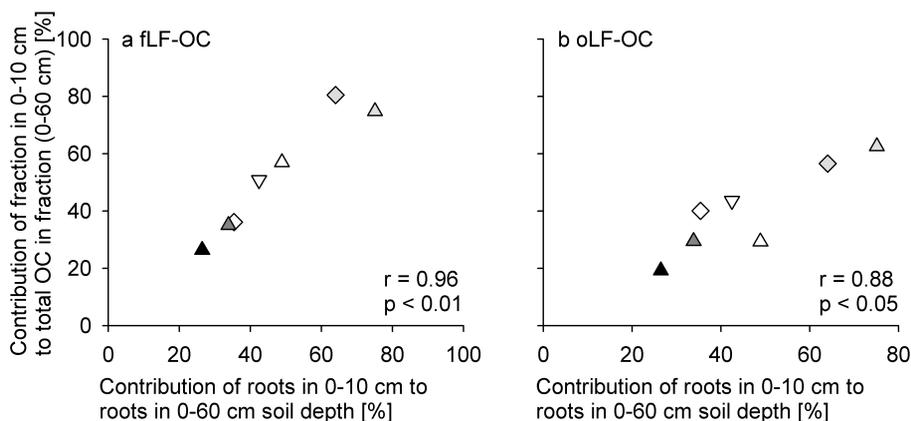


Fig. 6. The left graph **(a)** shows the correlation between the contribution of fLF (free light fraction) at 0–10 cm soil depth of totals soil OC and the proportion of sorted roots concentrated in the 0–10 cm layer. The right graph **(b)** gives the same relation for the oLF (occluded light fraction). Undisturbed sites sampled down to 60 cm soil depth are shown (black: coniferous forest, dark grey: croplands, light gray: grasslands, white: deciduous forests).

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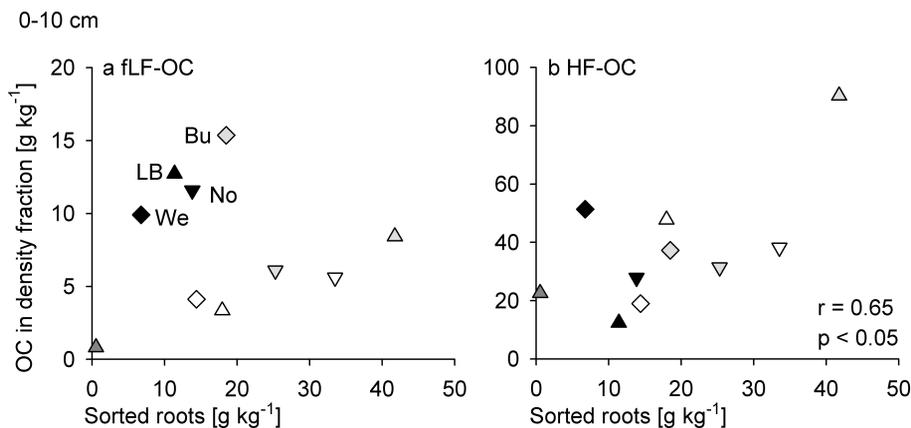


Fig. 7. Correlation between the amounts of sorted out roots in 0–10 cm with OC in the fLF (free light fraction, left graph) and OC in the HF (heavy fraction, right graph) for the study sites. Cropland sites Gebesee and Grignon were ploughed before sampling and therefore excluded (black: coniferous forest, dark grey: croplands, light grey: grasslands, white: deciduous forests).

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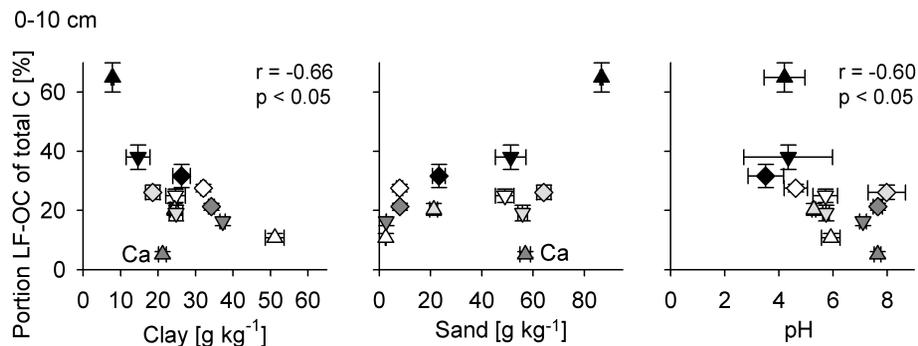


Fig. 8. Relation of the contribution of the LF (light fraction) at 0–10 cm soil depth to total OC to clay and sand content, and pH at 0–10 cm soil depth (black: coniferous forest, dark grey: croplands, light grey: grasslands, white: deciduous forests).

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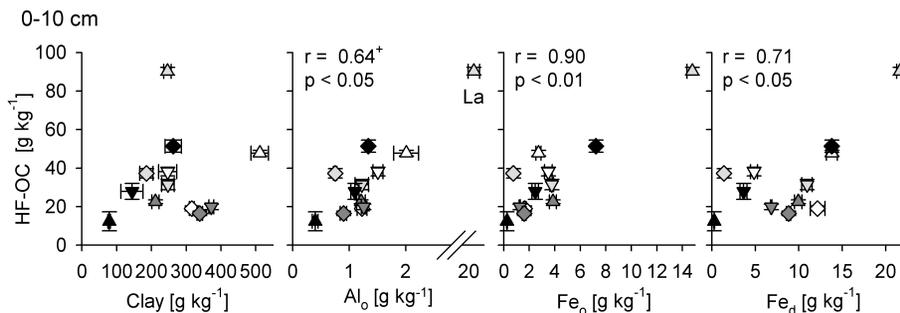


Fig. 9. Relation of the HF (heavy fraction)-OC per gram soil at 0–10 cm soil depth to concentrations of clay, oxalate-extractable Al (Al_o) and Fe (Fe_o), and dithionite-extractable Fe (Fe_d). Note, the x-axis break before the data point of the site Laqueuille (Andosol, La) for Al_o, which was excluded from the correlation analyses (black: coniferous forest, dark grey: croplands, light gray: grasslands, white: deciduous forests).

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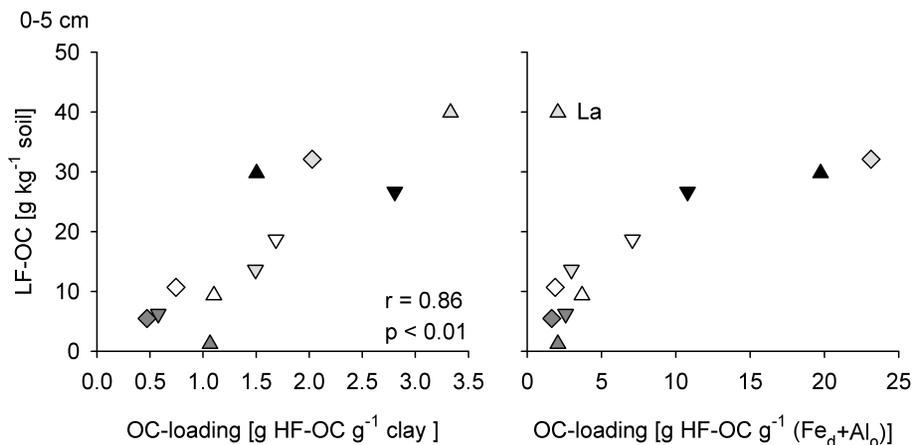


Fig. 10. Correlation between the amount of the LF (light fraction)-OC at 0–5 cm soil depth and HF (heavy fraction)-OC normalized to clay (left figure) and HF (heavy fraction)-OC normalized to dithionite-extractable Fe (Fe_d) plus oxalate extractable Al (Al_o) (black: coniferous forest, dark grey: croplands, light gray: grasslands, white: deciduous forests).

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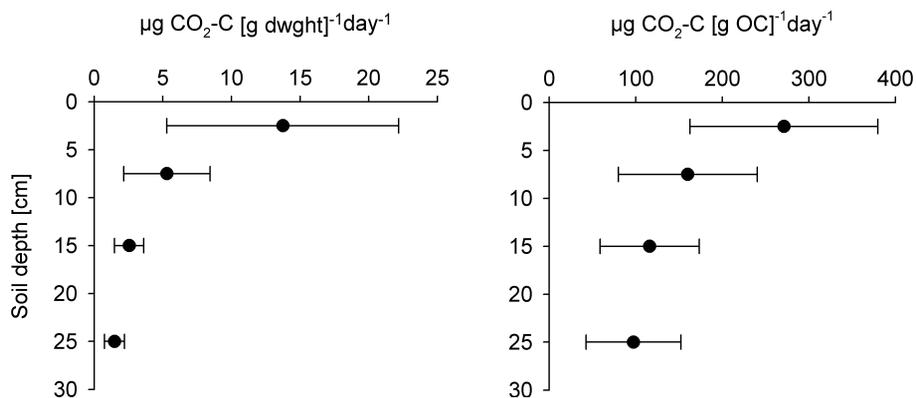


Fig. 11. Average C mineralization in the mineral soil over 20 days across all sites, given per g soil dry weight and per g OC (\pm standard deviation).

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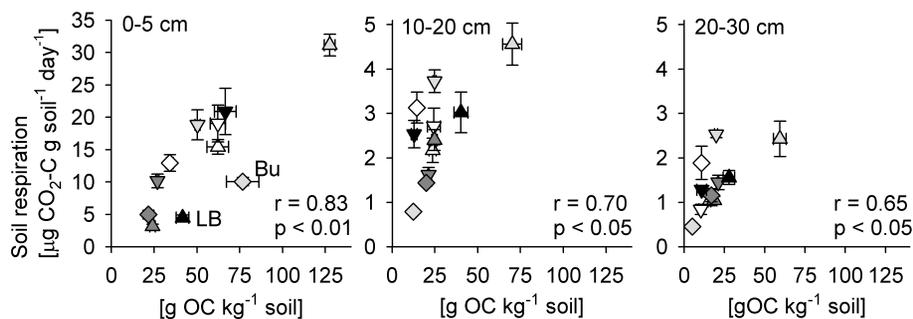


Fig. 12. Soil respiration per gram dry soil as related to OC concentrations for three soil depths (black: coniferous forest, dark grey: croplands, light gray: grasslands, white: deciduous forests).

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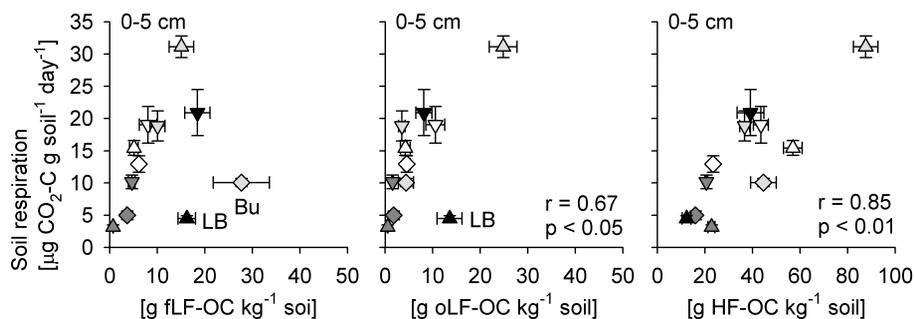


Fig. 13. Soil respiration per gram soil in relation to fLF-OC, oLF-OC and HF-OC per dry weight soil at 0–5 cm depth (fLF free light fraction, oLF occluded light fraction, HF heavy fraction, black: coniferous forest, dark grey: croplands, light grey: grasslands, white: deciduous forests).

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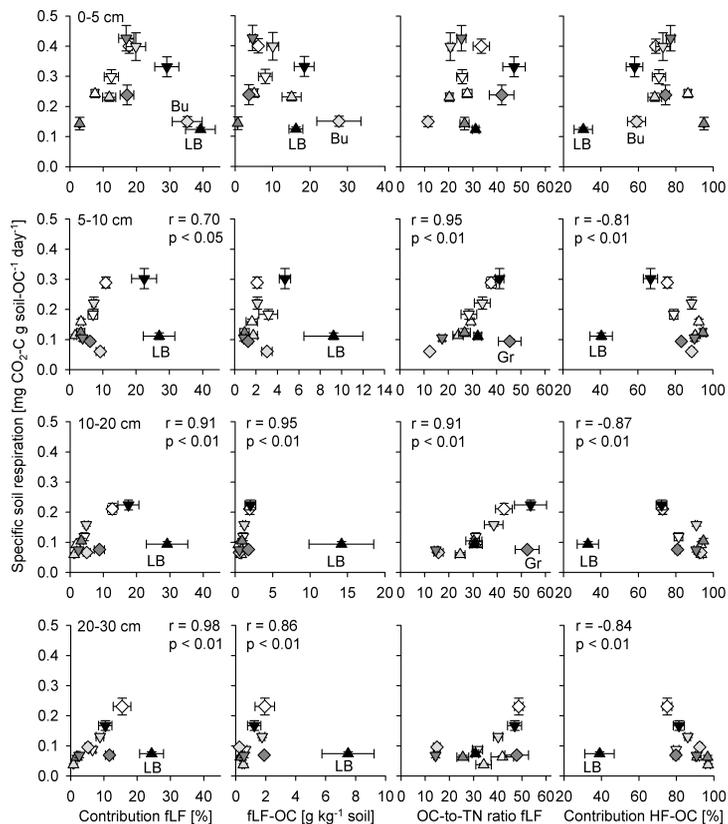


Fig. 14. Specific soil respiration per gram OC in relation to the proportion of free light fraction OC (fLF-OC), heavy fraction OC (HF-OC), the amount of fLF-OC per gram soil and the OC-to-TN ratio of the fLF for soil depths 0–5 cm, 5–10 cm, 10–20 cm, 20–30 cm. The sites Le Bray (black filled triangle up, LB) and Grignon (dark grey diamond, Gr) were excluded from correlation analyses (see text for details) (black: coniferous forest, dark grey: croplands, light gray: grasslands, white: deciduous forests).

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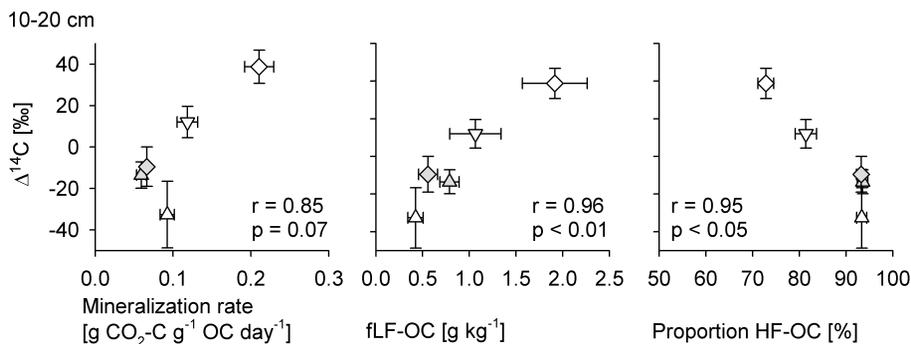


Fig. 15. Relation between average $\Delta^{14}\text{C}$ and specific mineralization rates per g OC, amount of OC with the fLF, and the proportion of HF (heavy fraction)-OC at 10–20 cm soil depth (only undisturbed sites Hainich, Hesse, Soroe, Bugac, Laqueuille containing no fossil C).

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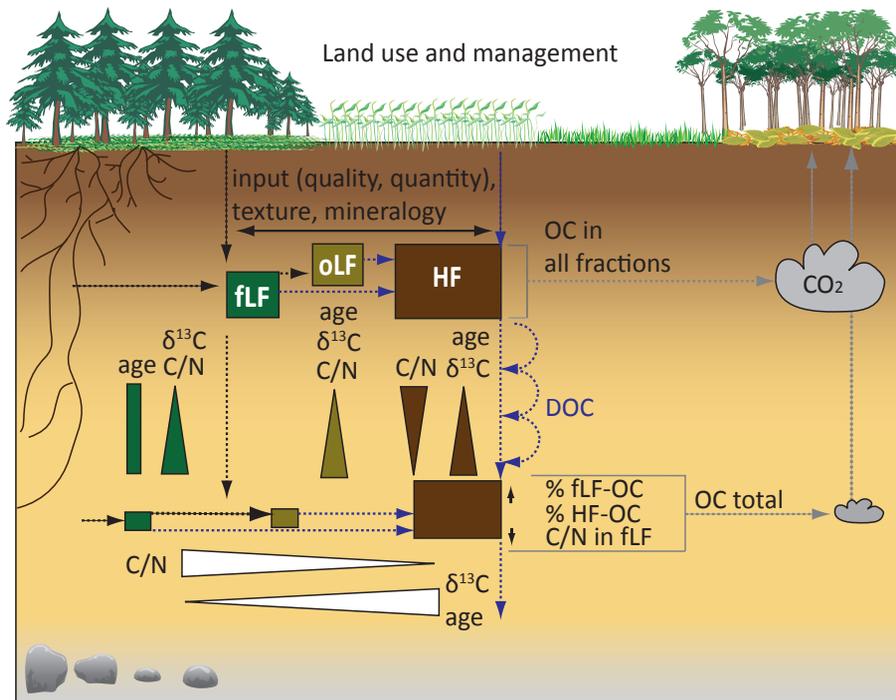


Fig. 16. Summary of characteristics of density fractions in topsoil and subsoil layers and their relation to soil respiration (fLF: free light fraction, oLF: occluded light fraction, HF: heavy fraction, C/N: OC-to-TN ratio, DOC: dissolved organic carbon).

Storage and stability of organic carbon in soil profiles

M. Schrumpf et al.

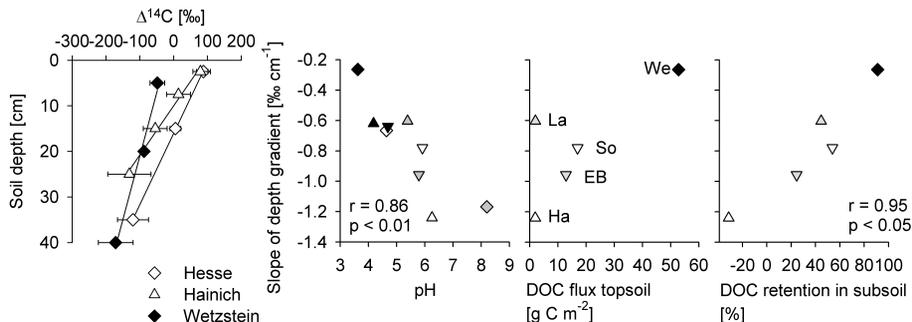


Fig. 17. The left graph shows examples for depth declines of $\Delta^{14}\text{C}$ in the heavy fraction for three of the study sites. The three graphs to the right show the relation between the slope of the decline of HF $\Delta^{14}\text{C}$ with soil depth and (1) pH values of the upper 30 cm of the soil, (2) dissolved OC fluxes in the topsoil (values taken from Kindler et al., 2011), and (3) OC retention in the subsoil (values taken from Kindler et al., 2011) (black: coniferous forest, dark grey: croplands, light gray: grasslands, white: deciduous forests).

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