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RADIOCARBON DATING REVEALS DIFFERENT PAST MANAGEMENTS OF ADJACENT FOREST SOILS IN THE CAMPINE REGION, BELGIUM.

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Abstract

The soils of adjacent first generation monospecific stands of Scots pine (*Pinus sylvestris* L.) and pedunculate oak (*Quercus robur* L.) in the Campine region, Belgium, apparently developed under the same forming factors, were studied for carbon dynamics to disentangle eventual different past land uses. In fact, visual observations suggested that the soil under pine experienced substantial addition of organic matter and ploughing, such to be considered a plaggen, opposite to the soil under oak, which is inexplicably much poorer in C. In order to prove this hypothesis, the soil organic carbon was quantified by horizons and, both bulk soil organic matter (SOM) and the least mobile SOM fractions – the humic acid and the unextractable fractions – were radiocarbon dated. Surprising was the marked difference between the mean SOM age from the two stands. In fact, while under oak this age is a few years or decades, under pine it amounts to more than a millennium, so confirming the hypothesis of a confined C supply occurred mainly in the Middle Age, or later using partly humified matter. The mean residence time (MRT) of SOM in the organic layers matches almost perfectly with that estimated via a mass balance approach and, as expected, was much lower in the oaks than in the pines. The humic acid fraction, generally the most stable fraction of SOM, in terms of both mobility and degradability, reflects the behaviour of the bulk SOM, showing higher radiocarbon ages under pine.

The findings of this work indicate that the large human-induced additions of organic material, in the area now occupied by the pine stand, probably occurred in the Middle Age and it continues to strongly affect the present soil C pools and their dynamics. Any study dealing with budgets and dynamics of C in soil should avail itself of a

careful reconstruction of the land uses and management history, in order to provide reliable conclusions about the real role of the current vegetation on soil carbon.

Keywords: forest soils, mean residence time (MRT), plaggic horizon, soil organic matter (SOM), soil management, radiocarbon dating.

Introduction

The global soil carbon (C) pool includes about 1550 Pg of organic C stored in terrestrial ecosystems (Lal, 2004). Spatially explicit studies are required for understanding the interactions between soil C and land use, as well as their contribution to the responses of terrestrial ecosystems to climate change (Meir et al., 2006). Past land uses, even when ceased centuries ago, can still exert strong influence on C cycling (Springob and Kirchmann, 2002; Fraterrigo et al., 2005). Plaggen soils (from Dutch: plag = sod) are a major reservoir of carbon, being characterised by a thick, black or brown, human-made C-rich diagnostic surface horizon – the plaggic (IUSS Working Group, 2006) or plaggen (Soil Survey Staff, 2006) – that evolved by long-continued manuring. In the past, sod and other materials were commonly used for livestock bedding. They were often mixed to faeces, and subsequently added, as fertilizer, to cultivated fields. Continuous addition eventually produced a dark soil mantle in places up to 1 m. The formation of such a thick layer implies an increase of the nutrient supply and the water retention of soil (Pape, 1970; Blume and Leinweber, 2004). Deep humiferous plaggen soils formed especially near villages, where the agriculture was more intense. In the north-western part of Belgium, soil management aimed to form plaggen was a common practice for about 3000 years, with the tendency to increase since the early Middle Age until the 17th century (Bastiaens and van Mourik, 1994). In an area of about 4000 km^2, plaggen soils cover about 550 km^2 (Conry, 1974; Fig. 1). Reconstructing the history of these built-up soils is often difficult, since documents and oral memories are scarce. Radiocarbon dating can be a useful tool to disentangle the timing of the plaggen formation and evaluating how soil organic matter (SOM) recycles once the large organic addition ceased. SOM is a heterogeneous mixture of organic compounds of plant, animal, and microbial origin at various stages of decomposition. As a consequence, caution is required for interpreting soil radiocarbon data. In fact, inputs of fresh organic material and selective leaching of humic substances may drastically alter the age of the SOM within a horizon (Mook and Streurman, 1983; Wang et al., 1996; Pessenda et al., 2001). Thus, the interpretation of ^{14}C data needs to be in function of the soil horizon under study and the events it experienced. Radiocarbon measurements performed on SOM fractions, having a narrower range of properties than the bulk SOM, can allow obtaining more precise information about the soil history (Orlova and Panychev, 1993; Kovda et al., 2001; Dalsgaard and Odgaard, 2001; Kristiansen et al., 2003; Tonneijck et al., 2006). Humic acid, soluble in alkali but insoluble in acid, and the fraction insoluble in alkali, generally are the least mobile fractions of SOM (Schulten and Schnitzer, 1997; Agnelli et al., 2002) and could be confidently studied in this purpose.

In this work we studied two adjacent forest soils of Belgium apparently developed under the same forming factors except the current vegetation. The aim was to relate the marked differences between the two soils to a different past soil management, rather than the standing forest cover. In this purpose we examined profiles and determined the depth-trend of some basic properties. The bulk SOM, the humic acid fraction and the residue of the alkaline extraction were radiocarbon dated.

Material and methods

Site description

The investigated forest, ”De Inslag”, is located close to Brasschaat, 20 km north-east of Antwerp, in the Campine region of Belgium (Fig. 1). De Inslag is 150 ha wide, and is dominated by Scots pine *(Pinus sylvestris* L.) and pedunculate oak *(Quercus robur* L.), which cover 50% and 35% of the total surface area, respectively. Two first generation and adjacent forest stands, one planted in 1929 and dominated by Scots pine, the other planted in 1936 and dominated by pedunculate oak, were studied in this work. Both stands are growing on an area that historically was a low-productive heatland, as witnessed by the de Ferraris 1771-78 map (Gemeentekrediet, 1965). Within the heathland small areas were cultivated and ploughed, and some others were not, but there should have never been forest in the past 1000 years in place where the stands are growing. Basic information on the sites are given in Table 1, while a more detailed description of the stands can be found in Janssens et al. (1999) and Curiel Yuste et al. (2005a).

Soil sampling and standard analyses

At either stand, three trenches were opened randomly. Two different samplings were carried out on opposite profiles of these trenches so as to have six replicates per sample. The first sampling was by genetic horizons, the second one, performed some months later, by depth intervals (0-4, 4-8, 12-22, and 35-45 cm). The sampling of depth intervals involved only the *solum*, hence excluding the C horizon, and was aimed to investigate some genetic horizons at two different depths. The description of a typic soil profile of either stand, made accordingly to Schoeneberger et al. (2002), is reported in Table 1. The soil profile described under pine is shown in Fig. 1. Samples of the organic horizon were taken by removing all the material present within a 19x19 cm frame, while the mineral soil was sampled using a cylinder of known volume (∅ = 8 cm, H = 10 cm), so as to determine the bulk density. All samples were air-dried and weighed. The mineral soil was sieved at 2 mm and the analyses were performed on the fine earth, the less than 2 mm fraction. The samples were analyzed for pH (potentiometrically in deionized water with a soil-water ratio of 1:2.5) and those from the mineral soil also for particle size distribution (pipette method). Finely ground (ball-mill) and oven dried (60 °C overnight) aliquots were analysed for total C by a Perkin-Elmer CHN Analyzer 2400 Series 2. Given the low pH, the presence of carbonates was excluded and total carbon in soils was assumed to be entirely in organic forms. Using data of bulk density, the concentration of organic C in the mineral soil was expressed on a volume basis. The fractionation of SOM was carried out on composite samples obtained by combining equal aliquots of each depth interval from the six examined profiles. Fractionation into fulvic acid fraction, humic acid fraction and unextractable fraction was done according to the procedure of Stevenson (1994). The fulvic acid fraction, which is the most dynamic of the three, was not taken into account for this work because we were mainly interested to the least dynamic SOM pools.

Radiocarbon dating

The bulk SOM, the humic acid fraction and the unextractable SOM were analysed for ^{14}C concentration at the “Centre for Isotope Research” of the University of Groningen, The Netherlands, by using the conventional radiometric method (homemade proportional gas counters; van der Plicht et al., 1992). The proportional gas counter determines the amount of ^{14}C present in a sample by measuring its radioactivity. The measurement uncertainty, which largely depends on the counting statistics and thus on the measurement time, is normally lower than 5‰. Samples preparation requires a process of combustion, during which organic C is converted to CO_2. To this purpose, each sample was placed in a quartz tube and flushed with N to remove any CO_2. Thereafter, the sample was heated to 1000 °C in a stream of O_2 gas, allowing complete oxidation of organic C to CO_2, which was dried and purified by passing through a series of water traps to remove impurities and water, and finally collected in a cryogenic trap (Goh, 1991). The samples were stored in sealed cylinders for one month in order to allow the decay of potentially trapped radon. Soil samples that did not provide the required minimum amount of CO_2 for performing the measurement by the proportional gas counter were analysed with an accelerator mass spectrometer (AMS, van der Plicht et al., 2000). In this case, prior to the analysis, the CO_2 obtained from the combustion was converted to graphite as described by Aerts-Bijma et al. (1997, 2001). The AMS system measures directly the isotopic ratios $^{14}C/^{12}C$ and $^{13}C/^{12}C$ of the graphite target, with typical measurements uncertainties around 4‰ (Meijer et al., 2006). The ^{14}C activity of the samples was expressed in $\Delta^{14}C$, that is the per mil deviation of the $^{14}C/^{12}C$ ratio in the sample from the same ratio of an oxalic acid standard prepared in 1950, corrected with respect to the $^{13}C/^{12}C$ ratio to account for isotopic fractionation effects (Stuiver and Polach, 1977). Because of the nuclear weapons tests, the concentration of ^{14}C in the atmosphere increased enormously in the 1950s and 1960s, the so called "bomb peak", to decrease later at a rate of about 8‰ per year (Levin and Kromer, 1997). A positive value of $\Delta^{14}C$ reveals the presence of ^{14}C produced by nuclear weapons testing, meaning that the sample was synthesized, at least partly, since 1950. Samples with positive $\Delta^{14}C$ were thus labelled as “modern”. They can not be dated due to the fast increase of the atmospheric ^{14}C concentration until 1963 (Meijer et al., 1994). Negative values of $\Delta^{14}C$ indicate that the organic material has resided in the soil long enough for significant radioactive decay of ^{14}C. In this case, conventional radiocarbon ages were calculated according to archaeological protocols using the Libby half lifetime (5568 years; mean lifetime 8033 years) and expressed in years before present (BP), such that 0 BP = 1950 AD. For determining the mean residence time (MRT) of the bulk SOM we used a time-dependent steady-state model as presented in detail by Gaudinski et al. (2000), referring to the ^{14}C concentration of the sample, the ^{14}C time record of the northern hemisphere air published by Levin and Hesshaimer (2000) for the period 1900-96, and direct atmospheric measurements (Smilde station, The Netherlands; unpublished continuation of the record in Meijer et al., 1994) for the period 1997-2003. In accordance with the model, the samples, being collected in 2003, showed two possible values of MRT when $\Delta^{14}C$ was > 69‰ (Fig. 2). The value of the two that is consistent with a theoretical CO_2 flux (as ratio between the amount of C in a given layer and the MRT of this carbon, according to Harrison et al. 2000) close to the one measured directly by Curiel Yuste et al. (2005b) was chosen.

Results and Discussion

Soil features and C storage

In the soil under pine, a large human-made addition of organic material is suggested by the sequence of horizons that consists of thick A1 and A2 horizons lying directly on a Cg horizon (Table 1). The latter horizon is occasionally affected by water stagnancy due to the presence of a clay layer at about 3 m depth, which allows the water table to raise during abundant rains. On the contrary, the soil under oak shows an A horizon passing gradually to the underlying B horizon and no evidence of water stagnancy within 1 m (Table 1). At a careful observation, the soil profiles under pine revealed weak evidence of past cultivation, apparently spade marks, which induce to call plaggic the carbon-rich horizon, according to the WRB (IUSS Working Group, 2006). However, the exact time the cultivation occurred and organic matter was copiously added to soil is unknown and no historical records, written or oral, can help in this regard. The soils of both stands are virtually stone and gravel free and have a sandy texture (Table 2). Their bulk densities cluster between 1.1-1.2 Mg m^{-3} except in the Cg horizon under pine where it is significantly higher. Soil pH is everywhere in the extremely acid range (Table 2). Such low pH guarantees about the absence of carbonates and, thus, the total C we determined is confidently all in organic form. The two soils differ substantially for the organic C content (Table 2). The organic horizon under pine contains much more C than that under oak, but it is the presence of the plaggic horizon that makes the pine soil twice richer in C compared to the oak soil. However, given the relatively young age of the forests and their low productivity (Curiel Yuste et al, 2005a; Xiao et al., 2003), it is unlikely that such a difference in soil organic C could originate from the standing vegetation only. A more plausible reason seems the addition of organic matter. The relatively low organic C concentration and the lack of any evidence of cultivation led to hypothesise a lower human impact on the soil under oak. Considering that both net primary production and soil CO_2 efflux are twice as high under oak than under pine (Curiel Yuste et al., 2005a,b), a shorter residence time of SOM in the oak stand is expected. Nevertheless, it cannot be the only reason for such a difference in soil C between the stands, which can probably be explained only if related to the historical practice of improving soil fertility by adding organic material. Radiocarbon analysis provided answers to most of these questions.

Radiocarbon age and mean residence time of bulk SOM

As expected, in both stands the radiocarbon ages increase with soil depth (Scharpenseel, 1993; Rumpel et al., 2002). In fact, decomposition rates decrease with depth as a consequence of reduced energy availability to sustain heterotrophic microbial biomass and activity (Certini et al., 2003; Fontaine et al., 2007), and increasing association of SOM with minerals, which reduces substrate availability to microbes (Paul et al., 1997; Kaiser et al., 2002). Moreover, in these very acid soils, earthworm activity is absent and bioturbation is marginal thus, there are only very few C inputs at depth except the obvious exudates and mortal remains of roots and dissolved organic C (DOC) coming from above.

In the soil under pine, the organic horizon comprises mainly “modern” C while the mineral soil contains prevalently “old” C, with mean ages in the A1 and A2 horizons of more than a millennium (Table 3). Under oak, both organic layer and mineral soil show ^{14}C concentrations largely influenced by modern C (Table 3). Even the BA horizon exhibits a clear influence of modern C, having a radiocarbon age of 16 years BP. Only in the B horizon the SOM appears to be marginally affected from the oak inputs, showing a mean age of 687 years BP. We speculated that the high SOM ages of the pine stand are the historical legacy of considerable additions of partially humified materials in more recent ages. Actually, it is plausible that in the past centuries, farmers have brought in organic material from drained peatlands, a practice that was common in these regions (Bastiaens and van Mourik, 1994). On the contrary, the soil under oak, which showed no signs of past cultivation, did probably experience no or minor addition of organic matter and that contained in the top mineral soil mostly derived from the present vegetation.

As expected, the radiocarbon-based MRT of the organic horizons differed between soils, with much higher values for the pine-covered soil (Table 3), in agreement with the recalcitrant nature of the pine litter, which decomposes more slowly than the oak litter (Berg and Ekbohm, 1991; Prescott et al., 2000). In the pine stand, the MRT of the organic horizon was 17 years, a value that matches perfectly the mass balance-based value found by Curiel Yuste et al. (2005a). In the oak stand, the radiocarbon-based MRT of the organic layer is 12 years and also in this case it matches well the mass balance-based value of 11 years (Curiel Yuste et al., 2005a). The good agreement in both sites between the MRT of the organic horizon calculated in two different ways is a confirmation of the fact that our ^{14}C approach produced realistic results. In the mineral soil under pine, the MRT of the bulk SOM was exceptionally high (1406 and 1712 years, respectively, in the A1 and A2 horizons), while in the mineral soil under oak the MRT was sensibly lower ranging from 115 years in the A horizon to 332 years in the BA and to 879 years in the B horizon, thus confirming for both sites the trend already showed by the radiocarbon ages.

SOM fractions' age

On a C basis, the extractable soil organic matter tends to be relatively more abundant under pine (44-54% of total SOC) than under oak (40-45% of total SOC). Under pine the humic acid fraction amounts to 20% of total SOC in the 0-4 cm mineral soil and its relative contribution increases with depth, representing 30% of SOC at 35-45 cm (Fig. 3). The same under oak, where the humic acid fraction, which account for 20-25% of total SOC, tends to slightly increase with depth. Consequently, the unextractable SOM represents the major fraction of SOC in both soils (Fig. 3). Radiocarbon measurements of the two SOM fractions of interest for this study revealed their heterogeneous nature throughout the profile (Table 4). Under pine, the apparent radiocarbon ages of the humic acid fraction and the unextractable SOM from the A1 horizon, suggest that the influence of the standing vegetation, despite the high C inputs from the pine trees, is mainly confined to the upper 4 cm of mineral soil. This information was not provided by the bulk SOM analysis because it was performed on the whole A1 horizon (0-8 cm), and evidently the high radiocarbon age of the SOM in the 4-8 cm layer would completely mask the young age of the SOM from the uppermost 4 cm (notice that the humic acid fraction and the unextractable SOM in the 4-8 cm layer are 708 and 1133 years BP, respectively). At 12-22 and 35-45 cm depth, both fractions show mean ages of more than a millennium (Table 4). In the A and BA horizons of the soil under oak, both analyzed SOM fractions are clearly influenced by C depositions that occurred after the “bomb peak”, as previously observed also for the bulk SOM (Table 3). The humic acid fraction and the unextractable SOM show modern values in the A horizon, while in the BA the humic acid is “modern” and the unextractable SOM has an apparent age of 603 years BP (Table 4). The age of the unextractable SOM increases progressively in the underlying two depths intervals being 1180 and 1690 years BP at 12-22 and 35-45 cm depth, respectively. On the contrary, the humic acid fraction age slightly decreases with depth, from 642 years BP at 12-22 cm to 523 years BP at 35-45 cm depth. Under pine the high radiocarbon ages of both SOM fractions support the hypothesis of old humified organic matter already in soil when the trees were planted. By the measurement of the net primary production and the soil respiration, Curiel Yuste et al. (2005a) showed that the soil under pine is an active C sink (much more C arriving on soil than decomposed), but evidently the ongoing C inputs from the pine trees to the A horizons are completely masked by the preponderant presence of ancient organic matter. On the opposite, under oak both SOM fractions from the A and BA horizons seems to be greatly affected from the standing vegetation, hence not suggesting any large human addition of organic matter to soil in the past.

Conclusions

Soil C measurements combined with the radiocarbon approach allowed identifying different past management types in the two studied forest soils, which we already hypothesized on the basis of the profiles observation. Under pine the SOM of the human-made plaggic horizon was assessed to have an extremely high mean age, which support the hypothesis of allocation of partly humified matter. Actually, the continuous bringing in of allochtonous humus was a very common historical practice in this area, albeit seldom documented. However, the adjacent soil under oak did not reveal such a human intervention. Here, in fact, no evidence of cultivation was observed in the soil profiles and, more importantly, the moderately abundant organic pool of this soil had a much lower age than that of the plaggic horizon.

The study of bulk SOM and its more stable components, humic acid fraction and unextractable SOM, indicated that the organic layer and the top 4 cm of mineral soil are the only compartments in which the recent SOM accounts for a percentage high enough to result in an overall low C age ("modern SOM"). On the contrary, in the deeper *solum* the amount of recent SOM is not enough to drive the bulk SOM to positive Δ^{14} values.

We conclude that the soil management that led to formation of a plaggic horizon, even when ceased for at least 74 years (but probably much longer), continues to affect the soil C pools and their dynamics in present day forests. This work demonstrates that radiocarbon dating is a powerful tool to reconstruct the history of anthropogenic soils, which needs to be evaluated to interpret soil C cycling in non-pristine ecosystems. This is relevant in view of the trendy research about soil C budget at a whole-country level, which often is based on current land use data only.

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