Characterisation of organic carbon in mire and heath soils at the Elgea-Urkilla Wind Farm, northern Spain

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SUMMARY

This paper describes a detailed comparative study of carbon storage in mire and heath soils within the Elgea-Urkilla Wind Farm (Basque Country, northern Spain). Different methods for estimating organic C stocks in the uppermost 15 cm of the soil profile were evaluated in an attempt to determine whether there was any spatial variability. The dominant vegetation of the study area was acidophilic and Atlantic heathland, with scattered areas of mire vegetation associated with spring lines. Soils were classified as Haplic Leptic Umbrisols (Oxyaquic, Molliglosic). Two sampling plots (900 m^2 and 600 m^2) were established adjacent to wind turbines. Mire vegetation was present in the larger plot (PLOT-A) and absent from the smaller one (PLOT-B). Fourier-transformed infra-red (FTIR) spectra indicated no noteworthy structural dissimilarities in the organic matter characteristics of the soils beneath the two types of vegetation. Soil samples were taken every week at systematically chosen points lying on fixed transects. Estimates of organic C stocks based on single sampling dates were 94–141 t C ha^{-1} for PLOT-A and 70–105 t C ha^{-1} for PLOT-B, and tended to increase as the weather became drier. When the estimates were derived from samples taken on several dates but from single transects, the range of the estimate for each plot was reduced to 111–116 t ha^{-1} for PLOT-A and 81–89 t ha^{-1} for PLOT-B. The results suggest that organic C stocks vary seasonally, and highlight difficulties that may be encountered in attempting to detect long-term changes in C storage.

KEY WORDS: Basque Country, Fourier-transformed infra-red spectroscopy, FTIR, organic carbon stocks, spatial variability, temporal variability.

INTRODUCTION

Mires are "active peat-forming wetland ecosystems". The formation and accumulation of peat occurs principally because the harsh environmental conditions in these habitats strongly limit microbial activity so that plant biomass production exceeds decomposition. In such environments, the soil is almost permanently water-saturated, O_2 availability and temperature are low, rainfall is high, and conditions are generally dystrophic (e.g. low nutrient content, low pH). The living mire vegetation is typically composed of plants that are highly efficient in terms of nutrient uptake and retention (Martínez Cortizas *et al*. 2008).

Peat soils represent an important carbon (C) reservoir because their organic components decompose slowly, and mires are amongst the most important ecosystems for sequestering C at the earth's surface. However, they may become a significant source of C emissions if mismanaged. Negative impacts on the environmental conditions of peatlands have long been induced by human activities such as drainage for agriculture, exploitation for forestry, and extraction of peat for use in horticulture or as fuel. Climate change may also affect the preservation of peatlands because it involves alteration of environmental variables that strongly influence these ecosystems. Most recently, the construction of wind farms has again placed these habitats at risk. Nonetheless, the risk assessment studies that are required prior to wind farm development provide a unique opportunity to learn more about peatlands and to prevent their degradation.

Given the importance of the topic in the current debate on global warming, there is a need for information about the changes in C storage in peatland ecosystems induced by all the factors mentioned above. Moreover, quantification of the changes is of particular interest because they may acquire economic value for carbon trading. However, those countries aiming to include soil C sequestration in their C accounting systems must have reliable methods for determining whether changes in C storage have actually occurred (Post *et al*. 1999). One major obstacle to detecting temporal C changes is the large quantity of C in most topsoils

- especially in mire areas - relative to annual C fluxes (Ellert *et al.* 2000), and spatial variability in organic C (Post *et al.* 1999).

In the study described here we evaluated the spatial variability in organic C concentration within a mire area located close to wind turbines in the Basque Country (northern Spain), related this to the hydrological conditions and vegetation types, and estimated organic C storage in the uppermost 15 cm of the soil profile using different methods. In-depth characterisation of the organic matter (OM) by Fourier-Transformed Infrared (FTIR) spectroscopy was also conducted at selected sampling sites. These investigations formed part of an environmental impact study associated with the construction of a nearby wind farm.

METHODS

Study area

The study area (42º 57' 37" N, 2º 24' 23" W) is located at the Elguea-Urkilla Wind Farm (Basque Country, northern Spain) in the east-west mountain range of the same name (Figure 1), which includes one of the highest peaks in the Basque Country (1291 m a.s.l.). The geology consists mainly of sandstones and other sedimentary rocks. The soils are classified as Haplic Leptic Umbrisols (IUSS Working Group WRB 2006) and Humic Lithic Dystrudepts (Soil Survey Staff 2006), and are characterised by low pH (3.5–4.7), low Effective Cation Exchange Capacity (ECEC) and low base saturation. The area lies on the boundary between the Atlantic and Mediterranean climatic zones (Heras & Infante 2008). During the period 2001–2004, annual precipitation ranged from 1074 to 1309 mm and mean annual temperature was 16 ºC, the monthly mean minimum of 5 ºC occurring in January and the maximum of 23 ºC in August.

The predominant vegetation is acidophilic and Atlantic heath (Aseginolaza *et al.* 1991) composed mainly of dwarf shrubs such as *Calluna vulgaris* L. (Hull), *Erica cinerea* L., *E. vagans* L. and *Daboecia cantabrica* (Hudson) C. Koch, with abundant common fern (*Pteridium aquilinum* [L.] Kuhn) and

Figure 1. The study area in the Elguea-Urkilla Wind Farm (Basque Country, northern Spain).

occasional *Ulex gallii* Planchon. Isolated patches of *Erica arborea* L. subsp. *riojana* (Sennen & Elías) Romo occur on more elevated ground. The grass *Agrostis curtisii* Kerguélen is very frequent, especially in the most open areas. There are small wet areas scattered throughout the study area. These biotopes always develop along more or less steep slopes below springs and are covered by mire vegetation. They are termed para-peaty habitats and cannot be regarded as true peatlands because they lack substantial peat deposits, but are nonetheless a valuable part of the local natural heritage (Onaindia & Navarro 1986, Heras & Infante 1990, Brugués *et al.* 2007). At least five *Sphagnum* species have been recorded in the vicinity of the study area (*Sphagnum auriculatum* Schimp., *S. capillifolium* (Ehrh.) Hedw., *S. fimbriatum* Wilson & Hook. f., *S. papillosum* Lindb. (Figure 2) and *S. subnitens* Russow & Warnst.), and other bog species such as *Polytrichum commune* Hedw., *Erica tetralix* L. and *Narthecium ossifragum* (L.) Hudson, also thrive here.

Figure 2. *Sphagnum papillosum* growing at the study site.

Soil sampling design

Two study areas were established, both located at an altitude of approximately 1000 m a.s.l. in an area known locally as Keixtuigana-Gainlabur. Despite the precautions taken by the developer, this area was damaged during construction of the access road to the wind turbines (Heras & Infante 2008). Specifically, a para-peaty habitat was partially destroyed, and this in turn stimulated interest that led to the detailed study of the locality reported here.

A 900 m^2 (15 x 60 m) plot which included two sub-areas with mire vegetation (Figure 3) - identified as PLOT-A - was established adjacent to a wind turbine. The slope of this plot was 20–30% and its aspect was south-easterly. For comparative purposes, a second plot 600 m^2 in area (15 x 40 m) was established adjacent to another wind turbine where the predominant vegetation was heath without *Sphagnum*, and identified as PLOT-B (Figure 3). The slope of this plot was also 20–30% and is aspect was south-westerly.

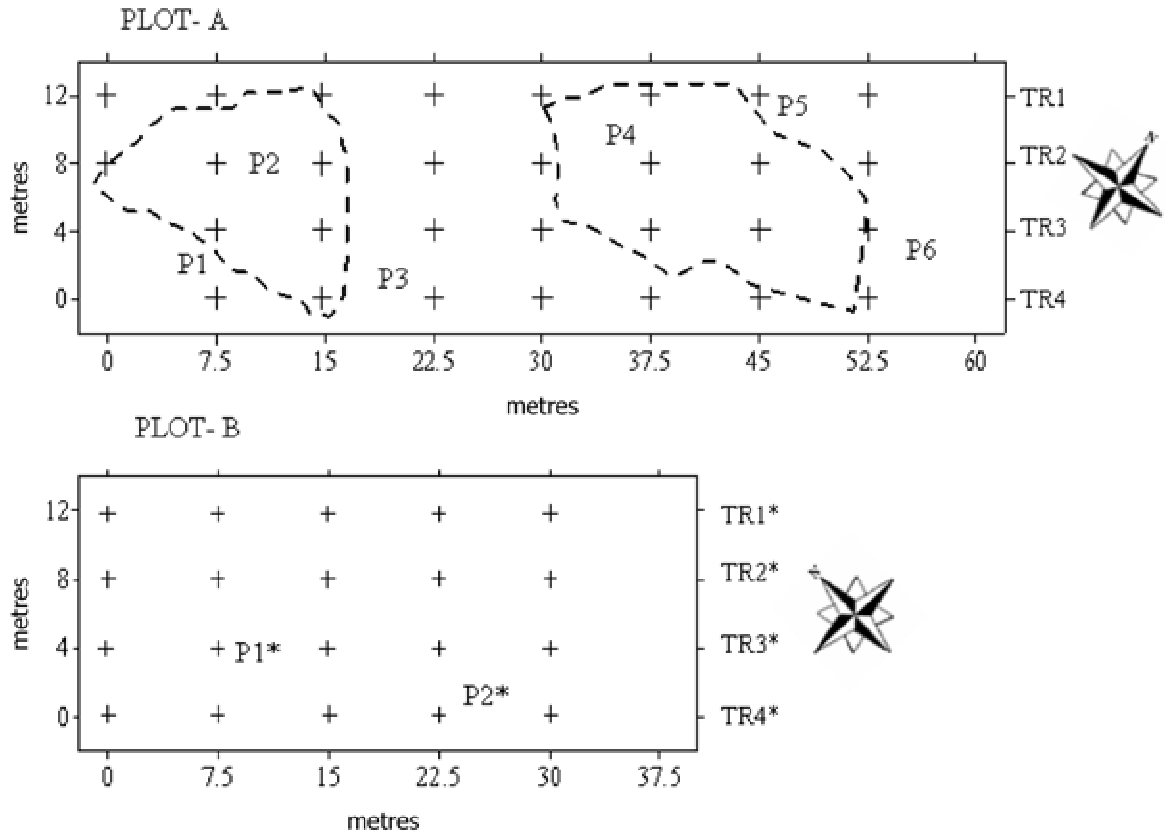

Figure 3. Layout of the two sampling plots PLOT-A and PLOT-B, showing transects (TR1–TR4 in PLOT-A and TR1*–TR4* in PLOT-B), sampling points on Day 1 (+), the locations of piezometers (P1–P6 in PLOT-A and P1*–P2* in PLOT-B), and the two para-peaty mire areas in PLOT-A; ⬭ indicates the two mire areas. Both plots were positioned so that the point defined by co-ordinates (0, 6 m) was at a distance of 15 metres from a wind turbine, and their dimensions were partly limited by local topography.

Six dipwells were installed in PLOT-A and two in PLOT-B. These were made from 100 mm internal diameter PVC tubing with perforated walls, and inserted to the base of the peat deposit (to 60 cm in the deepest profiles). The depth of the water table relative to the soil surface was measured at weekly intervals from 12 May to 18 August 2005.

The spatial variability in organic C content of the soils within the two plots was investigated using a modified version of the methodology of Ellert *et al*. (2002). These authors proposed a high-resolution method based on soil sampling across two transects within a rectangle, which involved collecting samples at intervals along the transects at time zero and repeating for the same transects - but displacing the sample locations by 1 m from the previous set - at the next sampling time. In the present study, the transects followed contour lines and there were four in each plot (Figure 3). The soil samples were taken weekly, at distance intervals of 7.5 m, displacing by 0.5 m each week. Finally, after 15 weeks, all transects were sampled every 0.5 m. In all cases the uppermost 15 cm of the soil profile, which is often considered to be the soil layer that is most susceptible to anthropic influence, was sampled.

Physicochemical and Fourier-Transformed Infra-Red spectroscopy determinations

Organic C was determined by dichromate oxidation in acid medium after heating (Nelson & Sommers 1996), and bulk density was determined at randomly selected sites following the methodology of Grossman & Reinsch (2002). The values of bulk density ρ (g cm^{-3}) fitted the following equation, which was obtained from data for 300 soil samples from the local region (R^2 = 0.996)

$$\rho = -0.2135 \, \text{Ln (OM)} + 1.3185 \qquad [1]$$

where OM is the organic matter content (g per 100 g of air-dried soil). This equation was used to estimate bulk density for all of the soil samples.

Fourier-Transformed Infra-Red (FTIR) spectra of selected soil samples from TR2 (PLOT-A) were also obtained. For this, pellets were prepared by mixing 0.5 mg of each sample with 49.5 mg of KBr until a homogeneous mixture was obtained. The pellets were then processed in a Brucker IFS-66V spectrometer (Brucker Daltonic GmbH, Karlsruhe, Germany), interfaced to a personal computer running Opus IR-2 software. All spectra were recorded from 4000 to 400 cm^{-1} with a resolution of 0.25 cm^{-1}. The frequency value for each band was obtained automatically by the software.

Estimation of organic C stocks

Organic C stocks were estimated using three different methods, all based on modifications of the method proposed by Ellert *et al.* (2002). As sampling took place during the spring and summer of a single year (2005), it was assumed that there would be no temporal changes in C stocks, and thus that the procedure could be used to investigate their spatial variability. Method 1 used all the data obtained from a single sampling visit; that is, from samples taken at 7.5 m intervals along all four transects within each plot. As there were 15 sampling visits, this yielded 15 estimates of the organic C stock for each plot. Because the area sampled moved by 7.5 m between the first and last sampling visits, data for locations within 7.5 m of either end of each plot were discarded for this analysis. Method 2 estimated the organic C stock using all the data obtained from a single transect; that is from samples taken at 0.5 m spacing along one transect during the whole experimental period. As there were four transects per plot, this method yielded four estimates of the organic C stock for each plot. Method 3 used all the data obtained from all four transects during the whole experimental period, and so provided a single estimate of the organic C stock for each plot.

Statistics

The data obtained at even and odd sampling visits were separated (thus minimising temporal differences beween the resulting subsidiary datasets) and compared using a Wald-Wolfwitz run test, in order to determine whether the two groups of observations were randomly sampled from the same population. A correlation test was performed to study the relationship between the C stock values obtained at each sampling visit and the number of days elapsed from the time of the first sampling visit. Statistical analyses were performed using Statview 5.0 (SAS 1998).

RESULTS

Vegetation in the plots

The para-peaty biotopes in PLOT-A were associated with a spring line which discharged into a small ephemeral brook running in a shallow channel with *Sphagnum auriculatum*, *Carex panicea* L. and *Juncus bulbosus* L. The damp soils at the sides of the channel supported a *Sphagnum* - rush community composed mainly of *S. auriculatum*, *Juncus effusus* L., *Carex echinata* Murray and *C. demissa* Hornem. The outer belt of drier soils was colonised by *Sphagnum capillifolium*, *S. papillosum* and *Calluna vulgaris* (dominant species), *Leucobryum glaucum* (Hedw.) Ångstr., *Hypnum cupressiforme* Hedw. var. *cupressiforme*, *Cladonia* sp., *Ulex gallii*, *Daboecia cantabrica*, *Erica cinerea*, *Juncus effusus*, *Potentilla erecta* (L.) Raeuschel, *Carex echinata*, *Agrostis curtisii* and *Juncus squarrosus* L. (very rare species). The site was already slightly disturbed by cattle (through grazing and trampling) before it was damaged during construction of the wind farm in 2003. The heath at PLOT-B was dominated by *Vaccinium myrtillus* L., and had no *Sphagnum* species.

FTIR spectroscopy

FTIR spectra for one non-mire and two mire locations are shown in Figure 4. In general, the spectra of the three samples were very similar and showed the same pattern of bands. The broad band centred at around 3410 cm^{-1} corresponds to the stretching vibration of bound and unbound hydroxyl groups of alcohols, phenols and organic acids, as well as N-H groups. Bands at 2921 cm^{-1} and 2850 cm^{-1} are attributed to symmetric and asymmetric stretching vibrations of C-H in CH_2 and CH_3 groups. The shoulder at 1710 cm^{-1} is attributed to the C=O stretching vibration of COOH, ketones, aldehydes and esters; this band indicates early metabolic products and commonly appears as a weak shoulder in fresh material (Gerzabek *et al.* 2006). The band centred at around 1625 cm^{-1} may be related to aromatic C=C stretching and C=O stretching of quinone and/or conjugated ketone and amide groups (amide I). The shoulder at 1426 cm^{-1} is generated by symmetric C-O stretching from COO^- or stretch and OH deformation (COOH) of carboxylate or carboxylic structures and/or C-H deformation of CH_2 or CH_3 groups (Chen *et al.* 2006). The band at 1384 cm^{-1} is attributed to O-H deformation, C-O stretching of phenolic OH, and to aliphatic structures. The broad band centred near 1085 cm^{-1} is related to C-O stretching of

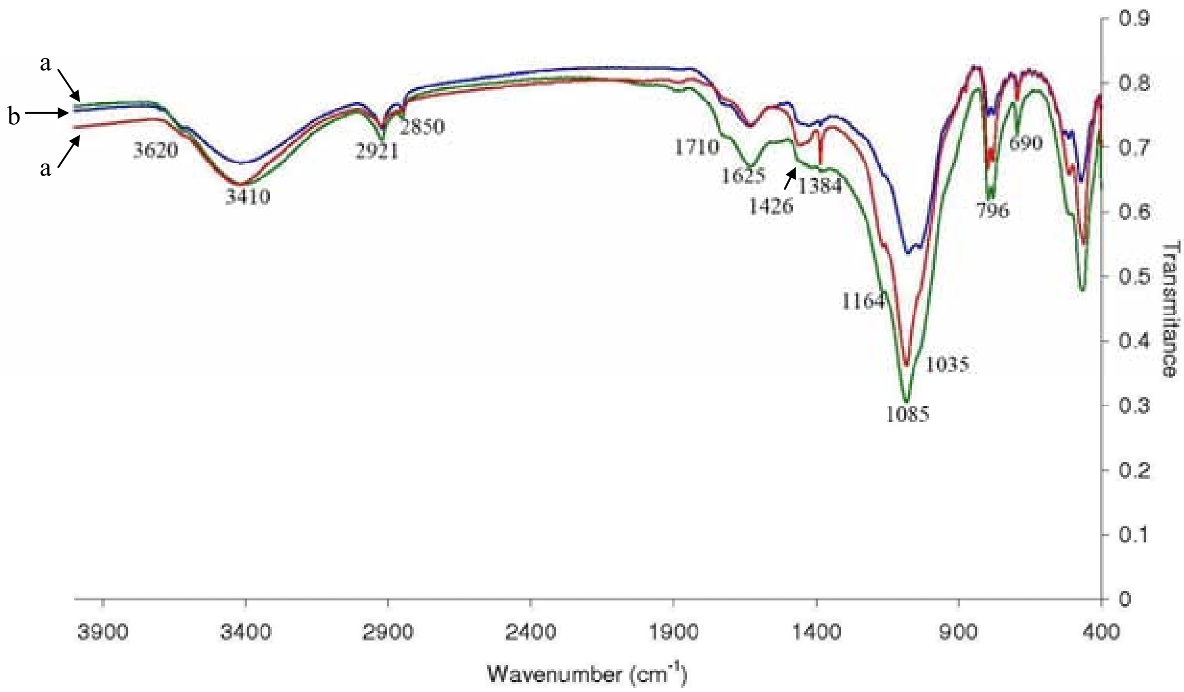

Figure 4. Fourier-Transformed Infra-Red (FTIR) spectra of three soil samples taken from one of the transects; (a) indicates soil samples from a para-peaty mire site and (b) the soil sample from a non-mire site.

polysaccharide or polysaccharide-like substances, as well as to silicate impurities. Bands at 1164 cm^{-1} and 1035 cm^{-1} are also characteristic of aromatic C-H in-plane deformation of syringyl and guaiacyl alcohols (Günzler & Böck 1990), two structural components of lignin. A sharp and intense band in the 1035 cm^{-1} region indicates the presence of polysaccharides and Si-O vibrations of clay minerals (Olk *et al.* 1999). Bands at 3620 cm^{-1} (Si-O-H vibrations), 796 cm^{-1}, 690 cm^{-1} and 470 cm^{-1} are indicative of inorganic materials (Smidt *et al.* 2002).

Spatial distribution of organic C stocks

The spatial distribution of organic C concentrations in the uppermost 15 cm of the soil profile within the two plots is shown in Figure 5. This diagram was constructed using all of the data acquired during the sampling period, i.e. from samples collected at 0.5 m distance intervals along all eight transects. Within PLOT-A, the organic C content of the soil ranged from 35 to 290 g C kg^{-1} soil and the mean organic C concentration for the whole area was 121 ± 41 g C kg^{-1}. When only the sub-areas with mire vegetation were considered, the values increased, especially in the north-western corner of the plot, where the maximum values of 290 g kg^{-1} were obtained (Figure 5). The lowest values for PLOT-A were obtained at its south-western corner, closest to the wind turbine but outside the boundary of the mire sub-area (Figure 5). In PLOT-B, the organic C content of the soil ranged from 25 to 146 g C kg^{-1} soil and the mean organic C content of the whole area was 80 ± 25 g C kg^{-1} (Figure 5). These values were, as expected, well below those obtained for PLOT-A. The results for both plots reflected the high spatial heterogeneity of organic C concentrations, with coefficients of variation of 34.2% for PLOT-A and 31.4% for PLOT-B.

Water table measurements

The sampling period included spring and summer seasons, and the latter was considerably drier than the former. Accordingly, the position of the water table in PLOT-A varied widely (Figure 6). The water level was initially high (in May), and at some locations it was at the soil surface with associated ponding. It then declined until the next precipitation events, which occurred in June, July and mid-August. As expected, the highest water levels were always recorded in the mire-like areas (Dipwells P2, P4 and P5). The dipwell with the lowest water level

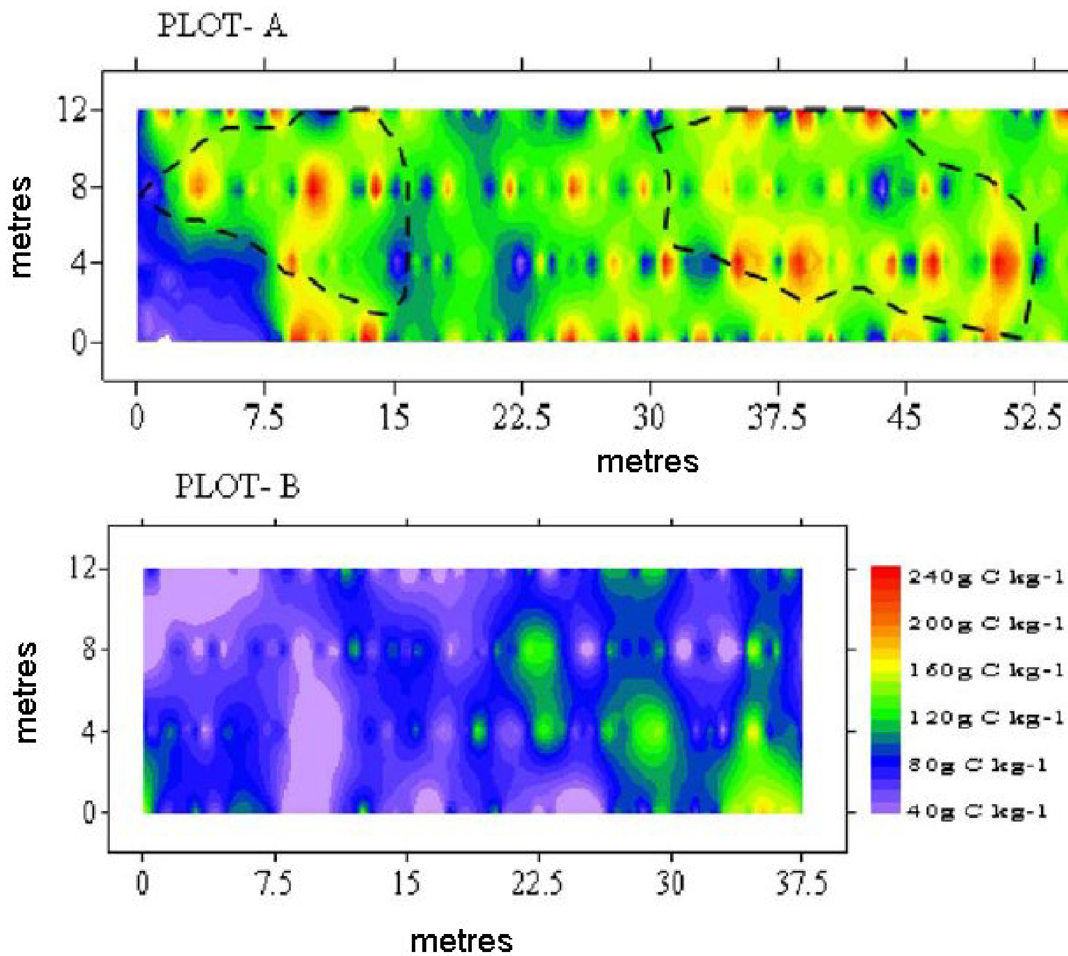

Figure 5. Spatial distribution of organic C concentrations within the study area, derived from the complete set of soil C data, including data from the two mire areas ⁚⁚⁚ .

(P6) was located in the most steeply sloping area, where internal soil drainage was presumably most intense. The soils in PLOT-B were well drained, as reflected by the type of vegetation, and a fluctuating water table close to the soil surface was absent except on one day after heavy rain at the beginning of July (Figure 6).

Estimation of organic carbon stocks

The fifteen estimates of the organic C stock in the uppermost 15 cm of the soil profile obtained using Method 1 ranged from 94 to 141 t ha^{-1} in PLOT-A, and from 70 to 105 t ha^{-1} in PLOT-B, with mean values of 118 ± 13 and 88 ± 11 t ha^{-1} respectively (Figure 7). This contrasts with the estimates of the organic C stocks obtained using Method 2 (based on data from single transects) (Table 1), which ranged from 111 to 116 t ha^{-1} in PLOT-A and from 81 to 89 t ha^{-1} in PLOT-B, with mean values of 113 ± 2.4 and 85 ± 3 t ha^{-1} respectively. Estimates of organic C stocks obtained by Method 3 (entire dataset) were 117 and 85 t ha^{-1} for PLOT-A and PLOT-B respectively. These results indicate that variability in the final estimate of the organic C stock for each plot was considerably smaller when all sampling sites on a single transect were considered (Method 2) than when selected sampling sites from all four transects were included (Method 1).

The Wald-Wolfwitz comparison of data from odd and even sampling visits tested whether spatial variability affected the results. According to this test there were no significant differences ($P < 0.05$) in the distributions of values for any of the sampling points. The results thus provide no evidence for the existence of spatial variability, although the spatial effect could not be fully tested with this procedure.

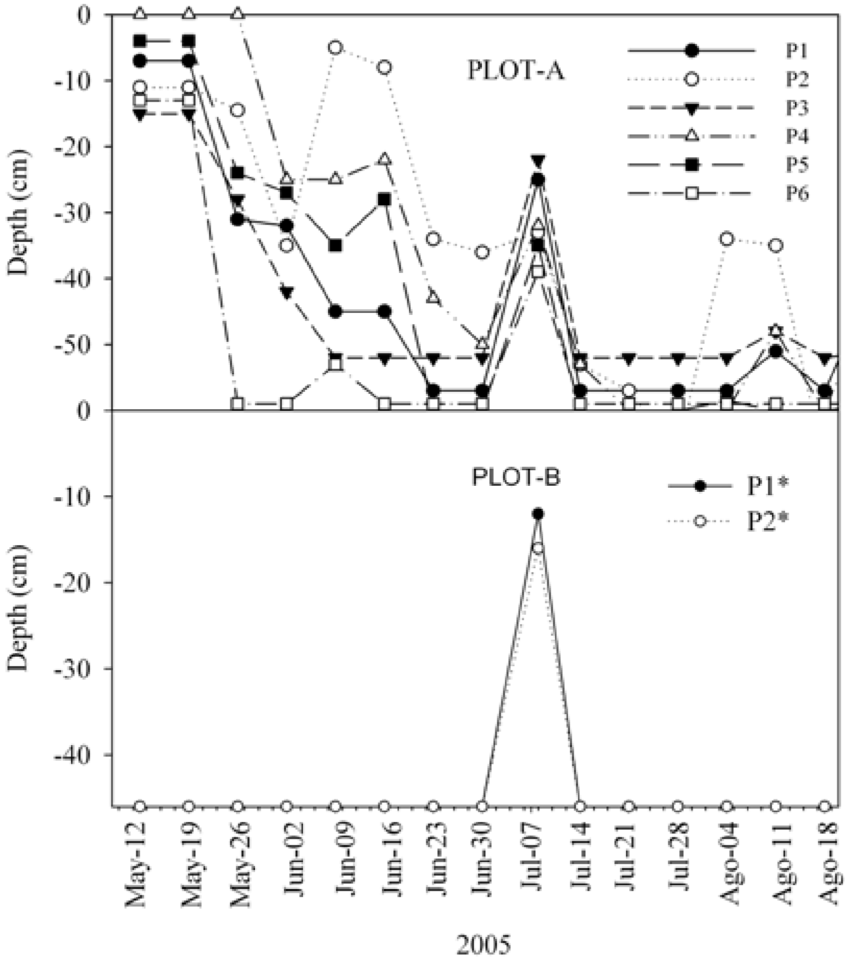

Figure 6. Water table data recorded during the study period.

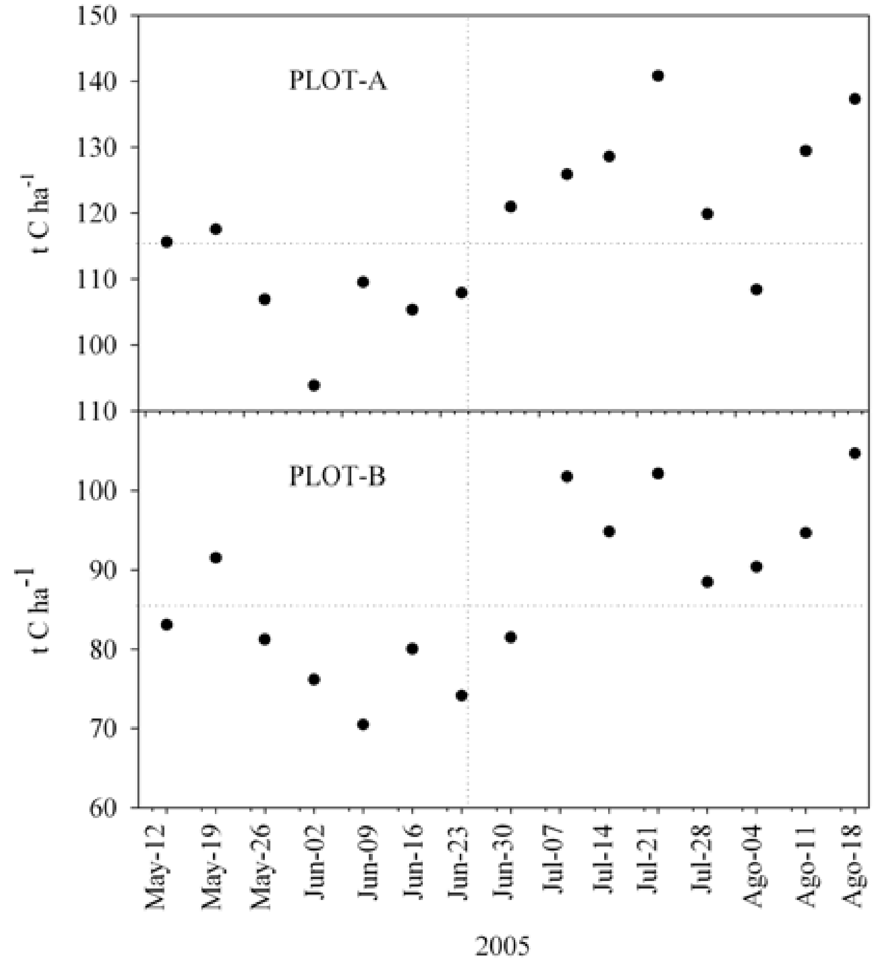

Figure 7. Organic C storage (t C ha^{-1}) for the two plots, calculated using all of the data obtained from each of the 15 weekly site visits (Method 1).

Table 1. Estimates of organic C stocks (t ha^{-1}) per plot derived from individual transect data (Method 2). STD: standard deviation; CV: coefficient of variation.

Transect	PLOT-A	PLOT-B
TR-1	114.3	81.4
TR-2	116.1	84.2
TR-3	111.8	88.9
TR-4	110.6	86.2
Mean	113.2	85.2
STD	2.4	3.2
CV	2.2	3.7

In this regard, it must be taken into account that soil samples were collected following a systematic sampling protocol. This means that if there was spatial variation in the concentration of organic C, areas with high or low values may have been systematically sampled along the different transects during individual sampling visits.

The correlation test for temporal effects revealed a significant positive correlation ($P < 0.10$) between the C stock values obtained at each sampling visit and the number of days elapsed from the time of the first sampling visit in 60% of cases for PLOT-A, and in 30% of cases for PLOT-B. Moreover, almost 50% of the sampling points in PLOT-A were positively and significantly correlated with each other, compared with 20% in PLOT-B. It may be concluded that there was temporal variation (accretion) in organic C stocks. This possible seasonal effect may be attributed to growth of vegetation in June–July, leading to more intense deposition of organic debris on the soil in the second part of the sampling period. This trend can be seen clearly in Figure 7 by comparing the values obtained before and after 01 July. In both plots, the values of most data points were higher during the second part of the sampling period than during the first part.

DISCUSSION

Para-peaty habitats are relic ecosystems and refugia for many mire plants, e.g. *Sphagnum* spp., that are common in north European countries but very scarce and localised in southern Europe. Thus they are highly valuable in terms of natural heritage.

FTIR spectra were obtained for soil samples taken from one of the transects that passed through both mire and heath habitat within PLOT-A, in order to determine whether the differences in vegetation were reflected in the organic matter chemistry of the underlying soils. Gerzabek *et al.* (2006) stated that three absorbance regions (around 2995–2887 cm^{-1}, 1614–1705 cm^{-1} and 1450 cm^{-1}) contribute substantially to molecular changes in soil organic C, but the spectra obtained did not show any noteworthy structural differences at the molecular level between mire soils and those beneath the typical heath vegetation nearby. Therefore this method, applied to bulk soil samples, seems to offer little utility for distinguishing differences in vegetation types. It may be necessary to use other techniques (e.g. pyrolysis-GC/MS) to determine whether there are any structural differences in soil organic matter due to differences in vegetation.

As indicated in the Introduction, quantification of soil organic C stocks is not an easy task, especially when the aim is to detect temporal changes in C, because of the high spatial variability and small size of annual C fluxes relative to background organic C levels. The lower variability in organic C estimates obtained here by Method 2 in comparison with Method 1 may have resulted from several factors:

(i) the existence of real spatial variability in the soils that were sampled;
(ii) the fact that more data were used in Method 2 (75–120 sampling sites) than in Method 1 (24 sampling sites);
(iii) the fact that in Method 2, samples taken at different times were combined, whereas in Method 1, the samples were taken at the same time (invoking a possible seasonal effect); and
(iv) the fact that Method 2 combined the results of chemical analyses carried out on different sample sets, whereas Method 1 combined the results of chemical analyses conducted on single sample sets only.

The last factor is probably the least important, however, as quality control procedures were applied.

CONCLUSIONS

1. Estimates of organic C stocks in the uppermost 15 cm of the soil profile derived for single sampling dates ranged from 94 to 141 t C ha^{-1} in PLOT-A (which contained mire vegetation) and from 70 to 105 t C ha^{-1} in PLOT-B (without mire vegetation), and tended to increase as the weather became drier.

2. When the estimates were derived instead from all samples taken at different sampling times along a single transect, the range of estimates within each plot was reduced (111–116 t ha^{-1} for PLOT-A and 81–89 t ha^{-1} for PLOT-B).
3. The results indicate that variability in estimates of organic C stocks may be due to (i) temporal variability in organic C concentrations caused by the new contribution of plant detritus during the experimental period, (ii) small scale spatial variability in organic C concentrations (not demonstrated), and (iii) a combination of both (i) and (ii).
4. Overall, the results obtained indicate that it is not possible to distinguish between spatial and temporal variability in C storage using the modified version of the method proposed by Ellert *et al*. (2002), which thus may not be suitable for detection of long-term changes in C storage in this type of ecosystem. We propose the use of single transects with samples taken in different seasons as an improved methodology for the comparison of temporal changes in C storage. This could be of especial interest in areas that have been disturbed by the construction of wind farms, as the impact of this activity on organic C stocks is still to be determined.

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