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Comparing measurements methods of carbon dioxide fluxes in a soil sequence under land use and cover change in North Eastern Spain

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ABSTRACT

Carbon dioxide measurements from soil surface may indicate the potential for soil respiration and carbon consumption according to microbial biomass and root activity. These processes may be influenced by land use and cover change, and abandonment especially in the upper soil organic layer. Seven environments from cultivation to late abandonment, with the same soil type classified as Lithic Xerorthent, were tested to ascertain the respiration capacity according to the current use and cover, and to establish the ability to preserve and eventually increase the organic matter pools after abandonment. Given the importance of carbon dioxide measurements at soil surface, a comparison between the classic soda lime method (SL) and a rapid method based on infrared sensor analyzer (IR) was performed from autumn 2008 to autumn 2009 in the field. The field measurements of CO_2 proved significant correlations between the values from the two techniques under the same natural conditions and along the period of observation. However, the values of CO_2 measured by the soda lime method were always higher than those obtained by the infrared analyzer. This pattern was attributed to the difference in time of measurement, larger in the former method, and type of measurement technique. Despite that the trend of measured CO_2 values was rather similar along the year. On average, the highest values of CO_2 emission in the field were recorded in the warmest periods of the year and with soil surface moisture not lower than 3% independently on the method used. High soil surface temperature with soil moisture below 3% decreased drastically the CO_2 production from the dry soil. The cultivated environments and soil under forests have resulted higher CO_2 producers than abandoned soils depending on the age of abandonment, climatic conditions, and within abandonment perturbations. Those abandoned soils preserved by perturbations like wildfire showed a higher potential for accumulating organic carbon, as indicated by the lowest emission of CO_2 with respect to SOC content, during the period of observation. Results demonstrated the reliability of the methods used to evaluate the soil carbon dioxide production capacity and allowed to classify through environments with increasing potential for carbon sequestration. The classification was rather similar by using both methods indicating a higher susceptibility to carbon loss in the following order: soil under Vines (V) > under Olives (O) > under Pine trees (PI) > under Cork Trees (S) > under Pasture (PR) > under Cistus scrub (MC) > under Erica scrub (MB) by using the SL method and V > O > PI > S > MC > MB > PR by using the IR method. Indications about the need of management of abandoned areas were also considered in order to recover the landscape heterogeneity.

1. Introduction

The consumption of organic matter in soil by heterotrophic microorganisms and the respiration of plant roots are known to produce carbon dioxide, which is finally released to the atmosphere (Jensen et al., 1996). Thus mineralization and humification processes may affect atmospheric CO_2 concentration as much as mineralization exceeds humification. The humification process is based on the relative susceptibility of the type of fresh organic field wastes and decaying debris to biological decomposition, mainly depending on C/N ratio, soil moisture and temperature. This process implies also consequent biochemical transformations of labile (carbohydrates) and more stable (lignin and phenolic compounds) organic fractions into humus. In case of carbohydrates as starting point the carbon percentage that makes it to humus is less than 20%, so that mineralizable labile organic compounds are most easily lost as CO_2 through mineralization (Zech et al., 1997). If the starting point is lignin, tannins, or other phenolic groupings (mostly found in wood and leaves) the percentage may reach 75%. In this case, low-mineralizable humic substances may increase in the soil and be preserved in the form of stable organic compounds for decades or even centuries if the soil is accurately managed. Soil structure and porosity as well as bulk density are tightly related with mineralization and humification processes. When intensive soil tillage or mismanagement cause low structure and poorly distributed porosity, metabolization of labile or even stable organic

Abbreviations: SL, Soda lime method; IR, Infrared gas analyzer method.

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fractions into CO_2 is likely to increase, favoring mineralization, decline in structural stability and compaction. Conversely, a stable structure with positively sorted porosity tends to maintain better soil conditions through stable organic compounds persistence and structural stability improvement. To minimize the carbon dioxide emission by mineralization of organic matter fractions, any agricultural practice or spontaneous plant succession preserving humic compounds in the soil system, may favor this system to act as an organic carbon sink, playing an important role in balancing carbon dioxide concentrations in the global carbon cycle. There are contrasting opinions on how land use and cover change and land abandonment may influence soil organic carbon (SOC) content and consumption or either its redistribution in time and space, involving biological activity, porosity, soil temperature and water regimes (Bajracharya et al., 2000; Levy et al., 2004). According to the previous land use and management before abandonment, land use and cover change, and abandonment may increase erosion processes affecting SOC content (Chengere and Lal, 1995). However, eroded soils contain relevant amount of SOC which may be redistributed and preserved in depositional areas (Bajracharya et al., 1998). Also, farmland abandonment may lead to natural vegetation succession increasing plant species colonization and the recovery of suitable organic horizons. Because of the varied effects on SOC and microbial activity, land use and cover change and land abandonment have important influence upon soil respiration, C flux to the atmosphere and C sequestration in soil. Measurements of carbon dioxide fluxed from the soil are therefore important in order to add information on the carbon dynamics. Nevertheless, there is a considerable uncertainty in the CO_2 measurements from soil surface, mainly due to differences in the methods used (Raich and Schlesinger, 1992). Nannipieri et al. (1990) relate soil respiration to the general metabolic activity of soil microorganisms to obtain energy for their growth through the decomposition of decaying organic debris. The effectiveness of soil microbial activity on the organic substrate depends primarily on soil properties such as moisture, temperature, infiltration capacity, clay content, and soil aeration (Buchmann, 2000; Zibilske, 1994). Moreover, soil respiration rates may be very sensitive to seasonal changes in soil temperature and water content because of their influence in microbial activity and root growth (Davidson et al., 1998; Kelting et al., 1998; Lee et al., 2003). Several methods have been reported for the estimation of carbon dioxide emitted from soils. The soda lime absorption and the infrared gas analyzer methods for CO_2 estimation are the most common used in the field and laboratory (Keith and Wong, 2006; Rochette et al., 1992). Other methods like Eddy Covariance are being also used though need of more specific and sophisticated procedures (Baldocchi, 2003).

In the North East Iberian Peninsula, terraced soils have been maintained for many decades at low agricultural management and then progressively abandoned. The change in the previous mosaic of land exploitation, the increased erosion processes and the periodical occurrence of wildfire has changed the soil properties as well (Dunjó et al., 2004; Pardini et al., 2003, 2004) arising questions on how these changes may have affected the organic reserve and the C flux from the soil. Thus, the objectives of this study were the following: i) to measure and compare the CO_2 emission capacity by means of the soda lime static method and the infra-red gas analyzer method in these peculiar soils; and ii) to evaluate on statistical basis the efficiency of the methods to detect the ratio between the organic carbon pool and the C–CO_2 loss, along one year of measurements (2008–2009).

2. Materials and methods

2.1. Description of the study area

The area belongs to the Natural Park of Cap de Creus, NE Iberian Peninsula. It is located in the Alt Empordà region and occupies approximately 30 km², ranging from 30 to 300 m asl. All the area has a significant ecological value expressed by the impressive geological and biological features, with unique examples of maritime and littoral terrestrial environments (Franquesa, 1995).

In this area, the climate is severely affected by the effects of the *Tramuntana* wind, from the Northern Pyrenees Mountains. It is a cold wind with normally a high speed (up to 90 km h^{-1}) having a strong drying effect on the soil water content. Rainfall is seasonally irregular and the mean annual precipitation is around 450 mm with peaks increasing in spring and autumn. A Mediterranean xeroteric climate is common, with hot summers around 35 °C and cold winters rarely below 0 °C. The mean annual average temperature is set between the isotherms of 15–16 °C (Dunjó et al., 2003). Soils are shallow (0–40 cm depth) and poorly developed in agreement with parent material composition, mainly granodiorite and schists. The horizon sequence is generally Ap, C/R and according to Soil Taxonomy System (Keys to Soil Taxonomy, 1992) these soils are classified as Xerorthent lithic. The majority of soil environments are completely covered by old terraces created several centuries ago and exploited mainly for wine, olive oil and cork production. Agricultural release started progressively in the last century when diverse plant diseases decimated vineyards, olive groves and cork plantations, and then with the advent of mechanized agriculture the terraces were almost completely abandoned around the fifties. Spontaneous plant colonization of the old terraces conform a much disorganized territory, very sensitive to wildfire. Currently 90% is covered with two main stages of scrubs according to fire occurrence: The much longer preserved by fire scrub area covering ≈55% of surface and the more affected by fire scrub area covering ≈35% of surface and often showing regressive vegetation dynamics caused by wildfire during the natural plants succession. Cork and pine trees and pastures are also occupying small patches covering ≈1.7% each respectively, as small patches are still cultivated with vines and olive trees covering ≈2.5% each, respectively. Some new interest in recovering agricultural land has arisen recently by landowners as a measure to reduce fire risks. Land use change in this area has lead to changes in the hydrological cycle, increasing erosion and nutrients depletion (Pardini et al., 2003). In the environments under study representative sites were selected in each soil environment in order to have similar slope (~20%) and orientation (NE).

Finally, seven soil environments were selected in this study: Terraced soils under cultivated vines (V) (*Vitis vinifera*) and under olive groves (O) (*Olea europaea*) maintained at minimal agricultural management. The monitored area of soils under vines had an extension of 0.5 ha, 16% slope and 6% of canopy cover, and the soil profile was 40 cm deep. The area delimited for soils under olive groves was also 0.5 ha, 18% slope, 15% of canopy cover and the total soil depth was 38 cm. Terraced soils under stands of cork trees (S) (*Quercus suber*) representing residuals of ancient cultivation for cork production, and under stands of pine trees (PI) (*Pinus pinea*) reforested in 1955. Both types of trees are approximately 4–5 m tall with understory composed mainly by *Brachipodium retusum*. The area of soils under cork trees had 0.5 ha extension, 15% slope, 70% of plant canopy and a total soil depth of 37 cm. Similarly, the area of soils under pine trees had 0.5 ha of surface, 17% slope and 70% of canopy cover, with a soil profile 40 cm deep. Terraced soils under pastures (PR) with meadows of *B. retusum*, *Trifolium stellatum*, *Dactylis glomerata*, *Lavandula stoechas* are currently in transition to scrubland for gradual abandonment of grazing activity. The monitored area was 0.5 ha, 18%, 35% of canopy cover and a total soil depth of 25 cm. Terraced soils under scrub (MC) are mainly covered by *Cistus monspeliensis*, *Cistus albidus*, *Cistus salvifolius*, *Calicotome spinosa* and some patches of *B. retusum*. This site was devastated by fire repeatedly in 1984, 1988, 1990, and 1994 and it is satisfactorily regenerated by natural plants. The representative monitored area of this soil environment was 0.5 ha, with 20% slope, 50% of plant canopy and a soil profile 33 cm deep. Terraced soils under scrub (MB) are mainly covered by *Erica arborea*, *B. retusum*, *L. stoechas* and some sprouts of *Quercus coccifera*. This environment was not affected by fire since 1984 and shows more ancient vegetation. The representative area selected in this soil environment was 0.5 ha, with 18% slope, 35% of plant canopy and a total soil depth of 35 cm. Rainfall gauges allowed recording rainfall events in each environment.

2.2. Experimental layout

Relevant soil parameters were determined in the laboratory. Soil samples were collected at 0–15 cm depth, at the same time and place of CO_2 field measurements. Thirteen soil sampling were performed along autumn 2008 to autumn 2009, three replicates per each soil environment, and a total of 273 samples were processed. Each time soil samples were air dried, gently crushed and sieved at 0–2 mm for subsequent analyses. Textural class and water holding capacity (WHC) were determined according to Porta et al. (1994). Soil pH, soil organic carbon (SOC) and total nitrogen content (TN) were determined according to Forster (1995). Bulk density (BD) and soil moisture (SM) was determined by the core method at sampling. The continuous measurement of soil respiration over short time intervals is a necessity in accessing the biological activity of a soil and its capacity to process different organic matter fractions into CO_2 (Brooks and Paul, 1987).

One of the most common methods to measure carbon dioxide flux from soil is the soda lime method. Soda lime grains are composed of a mixture of Sodium and Calcium hydroxides with a minor percentage of water (about 20%). Water is molecularly required to form Sodium and Calcium carbonates, its formation recorded by an increase of soda lime weight. Soda lime is therefore used as a desiccant because of its ability to absorb carbon dioxide and water resulting from soil respiration. As only CO_2 is chemically bounded with soda lime its quantitative determination is possible after oven drying, thus allowing to know the concentration of carbon dioxide accumulated (i.e. produced), during exposure. This method has been successfully tested both in the laboratory and in the field (Edwards, 1982; Grogan, 1998; Keith and Wong, 2006). For our purpose, measurements of carbon dioxide fluxed from the soil were carried out periodically in the field. The soda lime method (hereafter called SL) was applied by using three PVC cylinders called Cover Box or Chambers having 11 cm diameter and 10 cm height, (covering a soil surface of 0.0095 m^2) in each soil environment. Cylinders were inserted 5 cm into the soil at a distance of approximately 15 m one another. The soda lime had been previously prepared in the laboratory as follows: 15 g of pure soda lime were placed in a glass cup, oven dried at 105 °C, cooled in the desiccator and weighed with the accuracy of 0.01 mg. Cups were soon hermetically closed and stored. At field site, the cups were opened and immediately posed on the soil surface (0.0095 m^2) inside each cover box, and the upper screw top lid of the cover box immediately closed. Generally this operation was done in the time interval between 13:00 and 15:00 pm. After 24 h the lid of each cover box was opened and the soda lime cups were collected and hermetically closed. Later on cups were transferred to the laboratory, opened, oven dried at 105 °C for 12 h, cooled in a desiccator and then weighed with the accuracy of 0.01 mg. The weight of carbon dioxide absorbed by soda lime was calculated by applying the correction factor proposed by Grogan (1998) accounting for water formed when soda lime reacts with CO_2 as following:

$$2NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O$$
$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$$

As observed each mole of CO_2 chemically bound to soda lime favors the formation of a mole of water, which is then lost by oven drying. Therefore dry mass increase before and after exposure underestimates CO_2 absorbed. The correction factor takes into account that 44 g of CO_2 react with a 74 g of $Ca(OH)_2$ to form 100 g $CaCO_3$ and 18 g H_2O. The measured increase in soda lime after oven-drying is 26 (i.e. 100–74). Thus the correction factor of 44/26 (i.e. 1.69) must be applied to the measured mass differences in order to obtain the true value of CO_2 absorbed. Carbon dioxide was calculated previously as mg CO_2 m^{-2} considering the soil surface inside the cylinder and then, according to measurement time, transformed in μmol CO_2 m^{-2} s^{-1}.

A recently commercialized gas analyzer apparatus was also used to detect CO_2 production by soil, namely the MultiRAE IR Monitor PGM54 from RAE Systems Inc., Sunnyvale, CA. This apparatus has been designed to provide continuous monitoring of gases including carbon dioxide. The equipment (Figure 1A) is composed of a newly developed miniaturized Non-Dispersive Infrared (NDIR) sensor measuring CO_2 concentration directly in ppm. The MultiRAE IR Monitor includes an integrated sampling pump, a diaphragm pump providing about 300 cm^3 min^{-1} flow rate. When the sampling pump is aspirating telluric air from the soil, infrared light is shone through the gas sample and the amount absorbed by the CO_2 is proportional to its concentration. The patented PID (Photoionization detector) uses an electrodeless discharge UV lamp as a high energy photon source to ionize the gas to be measured. The resultant electrical current is proportional to the gas concentration (Figure 1B). Previous calibration of the CO_2 sensor must be executed before any set of measurements. Generally it is zero calibrated with isobutylene (supplied by RAE) which is free of CO_2. Next, the CO_2 span gas is supplied, typically 5.000 ppm, to calibrate the upper level of CO_2 detectable. The multigas analyzer is easily portable in the field, reaching constant and reproducible CO_2 values from air and soil air, even at different depth. It has 12 h of continuous operation with a rechargeable Li-ion battery. The data storage capacity is up to 20.000 readings which can be downloaded to PC through RS-232 link. Moreover it can work in very extreme conditions as its temperature and relative humidity working ranges are −20 °C to 45 °C and 0% to 90% respectively. The MultiRAE IR monitor gives real time measurements and activates alarm signals whenever the exposure exceeds preset limits of calibration. The apparatus (hereafter called IR) was connected with a filter for retaining water and dust during measurements, connected with a hose (0.5 cm internal diameter) both to the filter and to the lid device (i.e. the chamber) having 10 cm diameter and 5 cm height. This was inserted a few mm into the soil surface in order to ensure a complete flow rate and measure a constant volume of air gas emitted from the soil surface (Figure 1A). Measurements were performed in the field in three different places (parallel to SL measurements sites) for each environment (at approximately 15 m distance one another) between 13:00 and 15:00 pm. The measurement of CO_2 in the aspirated soil air lasted until the volume of CO_2 concentration (expressed as ppm) was constant in the digital screen of the apparatus. Generally, this happened after 5 min. Values from IR measurements were reported as μmol CO_2 m^{-2} s^{-1} according to the following expression:

$$CO_2\text{flux} = (\Delta_c)\frac{Q}{V_{air}A}$$

where Δ_c is the difference between the air and soil chamber CO_2 concentration (μmol mol^{-1}) respectively, Q is the volume flow rate through the chamber (m^3 s^{-1}), A is the soil surface area covered by the chamber in contact with the soil (m²) and V_{air} the molar volume of air (m^3 mol^{-1}).

The temperature of soil surface was measured by non-contact laser thermometer in the field at any CO_2 measurements.

Statistical analysis has been carried out by using the program Statistics, Stat. Soft Inc. (2007).

3. Results

3.1. Relevant soil properties in soil environments

For each selected soil characteristic a mean value of the three soil replicates collected at each sampling date and environment was

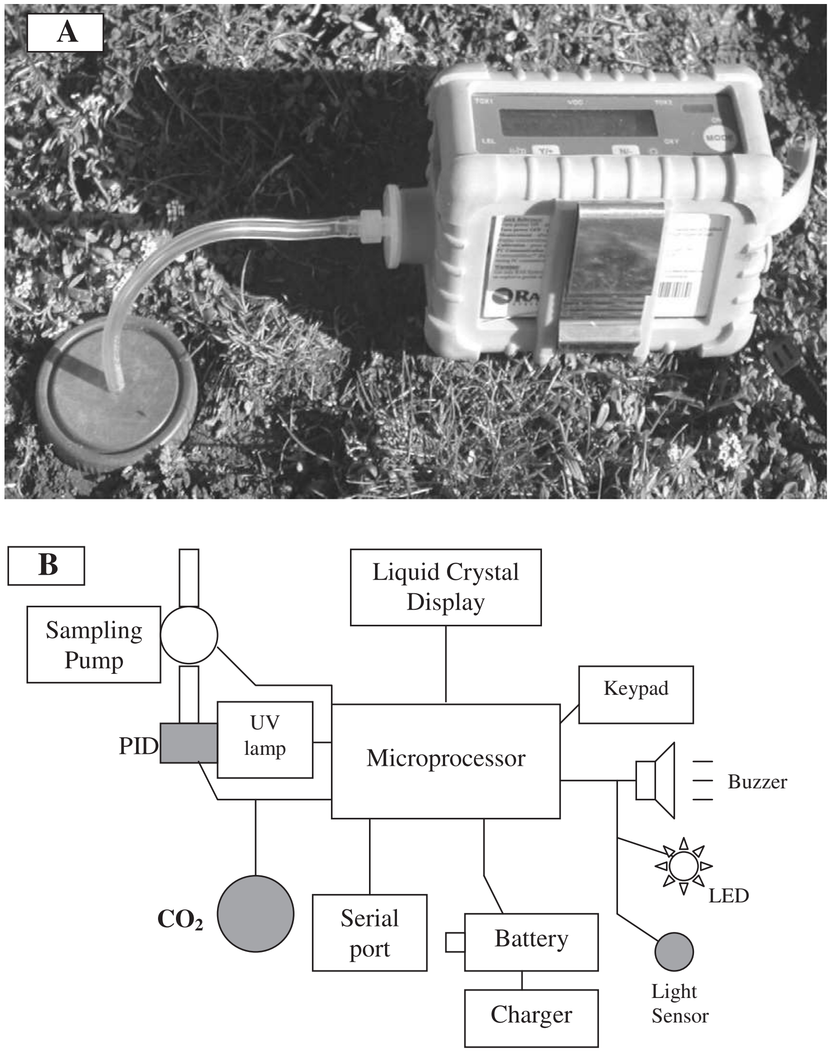

Fig. 1. A) The MultiRAE IRGA Monitor PGM54 apparatus in a measurement trial of CO_2 fluxed from soil surface in the field; B) Schematic block diagram of MultiRAE IR operating devices.

obtained. Then the mean annual value was calculated (± standard errors) from the thirteen temporal measurements, from autumn 2008 to autumn 2009 and reported in Table 1a. The variability in soil texture and bulk density indicates the variations that soil properties undertake probably due to the age of abandonment and the spontaneous plant colonization. Erosion processes may be therefore less effective, as less effective is fine particles removal by runoff. Nevertheless, abandoned soil environments may be often affected by fire occurrence, which increases erosion processes, temporarily altering some soil parameters like clay or SOC content. Soil environments PI and MC are an example for the reduction in organic carbon content. The fire effect on the former environment (PI) is indicated by a more variable content of SOC which can explain the 35% of variation with respect to cork trees (S environment) which is less affected by fire (Pardini et al., 2004) and shows more constant values of SOC (Table 1b). In the latter environment (MC) the fire effect is also clear by observing the Table 1b. However, the 17% less SOC in the MC with respect to MB soil environment may be probably more attributed to a faster recovering time after fire, also indicated by similar coefficient of variability between the two sites. Thus, according to the state of the soil, rainfall events may be more or less effective in terms of erosion. Along the time of observations the mean total amount of

Table 1a
Mean annual values of the selected soil characteristics (± standard errors) in the 0–2 mm soil fraction of the studied soil environments.

Env	Clay (%)	Silt (%)	Sand (%)	BD (g cm^{-3})	SM (%)	WHC (%)	ST (°C)	pH
V	3.75 ± 1.44	10.83 ± 1.44	85.00 ± 0.00	1.64 ± 0.19	3.68 ± 1.37	27.89 ± 5.58	19.55 ± 9.41	6.44 ± 0.18
O	16.25 ± 5.20	20.00 ± 6.61	66.67 ± 1.44	1.36 ± 0.04	7.78 ± 4.07	43.06 ± 4.28	27.18 ± 7.30	6.48 ± 0.16
S	11.25 ± 1.44	27.50 ± 2.50	61.67 ± 2.89	1.14 ± 0.08	11.28 ± 4.58	65.47 ± 7.65	18.85 ± 9.75	6.56 ± 0.32
PI	13.75 ± 3.82	20.83 ± 5.20	67.50 ± 2.50	1.16 ± 0.13	10.57 ± 4.12	56.24 ± 8.18	18.88 ± 9.94	6.22 ± 0.38
PR	18.75 ± 4.33	20.00 ± 4.33	62.50 ± 4.33	1.10 ± 0.10	17.87 ± 5.12	72.02 ± 7.37	15.00 ± 9.32	5.71 ± 0.16
MC	20.00 ± 3.82	26.67 ± 2.89	55.00 ± 2.50	1.13 ± 0.08	13.98 ± 3.06	64.92 ± 2.15	14.68 ± 8.91	6.33 ± 0.19
MB	13.75 ± 2.50	23.33 ± 2.89	64.17 ± 1.44	0.96 ± 0.09	15.45 ± 5.55	79.48 ± 9.14	14.25 ± 8.42	6.01 ± 0.19

Env: Environment; BD: bulk density; SM: soil moisture; WHC: water holding capacity; ST: soil surface temperature; V: soil under vines; O: soil under olives; S: soil under cork trees; PI: soil under pine trees; PR: soil under pasture; MC: soil under Cistus scrub; MB: soil under Erica scrub.

Table 1b
Descriptive statistical analysis of soil organic carbon (SOC) and total nitrogen (TN) for the selected soil environments along the carbon dioxide measurements from autumn 2008 to autumn 2009.

Env	Parameter	Mean ($mg\ g^{-1}$)	Max ($mg\ g^{-1}$)	Min ($mg\ g^{-1}$)	SD	CV (%)	C/N
V	SOC	2.64	2.95	2.16	0.58	12	4.88
	TN	0.53	0.84	0.08	0.13	59	
O	SOC	15.14	19.03	11.53	3.46	19	8.62
	TN	1.76	3.01	0.21	0.56	67	
S	SOC	28.96	33.70	24.28	4.49	13	12.12
	TN	2.16	3.45	0.33	0.68	60	
PI	SOC	18.62	25.29	12.93	3.11	32	11.67
	TN	1.46	2.03	0.19	0.52	54	
PR	SOC	36.97	39.07	32.91	1.53	7	10.28
	TN	3.34	4.82	0.47	0.71	54	
MC	SOC	30.58	37.75	24.59	3.86	17	11.67
	TN	2.09	3.10	0.35	0.71	53	
MB	SOC	37.06	43.53	27.71	3.45	16	13.54
	TN	2.35	3.63	0.41	0.78	54	

Env: Environment; SD: standard deviation; CV: coefficient of variation; C/N: carbon nitrogen ratio; V: soil under vines; O: soil under olives; S: soil under cork trees; PI: soil under pine trees; PR: soil under pasture; MC: soil under Cistus scrub; MB: soil under Erica scrub.

rainfall (measured with rain gauges at 1 m height) varied between the soil environments, reaching values of 61, 60, 34, 57, 41, 53, 43 $l\ m^{-2}$ for V, O, S, PI, PR, MC, MB respectively. This meant that on average cultivated soils (V, O), soils under pines (PI) and Cistus scrub (MC) received 1.5 fold more rainfall than other soil environments, which may be also explained by a cleared plant canopy. Accordingly, higher water storage capacity should be recorded within rainfall events in these soils. By contrast, frequent fire occurrence (MC environment), hardly mineralizable organic debris (PI environment) and insufficient agricultural management (V and O environment) lowered their organic matter content by 52% on average (Table 1b) with respect to S, PR, and MB soil environments, mainly affecting water holding capacity. Moreover, these soils showed higher erosion rates and nutrient decline (Pardini et al., 2003). It must be pointed out that the moisture regime is fundamental in these very shallow soils (0–40 cm depth) with a reduced A horizon (less than 15 cm) over a stony C/R horizon, in order to favor humus formation, nutrients dynamics and soil structure improvement. If the capacity to retain water decreases, it would drastically reduce soil moisture content within rainfall events during wetting–drying cycles, reducing soil evolution and increasing water loss by runoff and evaporation. Conversely, a higher potential for water storage was shown in soils with higher organic carbon content (MB, PR, and S environments), even receiving lower amount of rainfall, indicating a better water holding capacity. In fact, a proper soil evolution after abandonment is to be expected when natural plant colonization and succession is not interrupted and may be beneficial to soil itself by contributing to the development of a more organic profile.

According to soil reaction, Table 1a shows pH values lower than neutrality in all soil environments in agreement with the parent material composition. No carbonate content was found in all the studied soils and the lowest pH values were recorded in the soil under pasture (PR environment). Soil organic carbon (SOC) and total nitrogen content (TN) increased in the studied soils in the order: cultivated soils (O, V), forest soils (PI, S), soils under scrubs (MC, MB) and under pasture (PR). SOC in olive groves plantation was 5.7 fold higher than soil under vines. In forest SOC was 1.6 fold higher in soil under stands of cork trees than in soil under pines, whereas the soil under Erica scrub showed a SOC content 1.2 fold higher than the soil under Cistus scrub. The same trend was observed with total nitrogen. Within the cultivated soils, soil under vineyards indicated an extremely low organic carbon being total nitrogen (added often as chemical fertilizer) proportionally higher than organic carbon. This resulted in a very low value of C/N ratio along the year of observation with a yearly average of 4.98 for soils under vineyards and 8.60 for soils under olive groves. Periodical grazing occurring in soils under pines (PI) may partly explain the lower C/N ratio (11.67), which further decreases to 10.28 in soils under pasture (PR) where grazing is a common practice and animal manure is left onto the soil; this may have produced a higher mineralization enhancing microbial activity, despite the relatively acid pH. A more conservative carbon dynamics is likely to occur in S, MC and MB soil environments with C/N ratios of 12.12, 13.54 and 14.45 respectively.

3.2. Measurements of soil respiration

Soil respiration is widely accepted to be the most representative manifestation of the biological activity in the soil, and favors a good understanding of the variation occurring in the organic carbon pool due to land use and cover change (Sanhueza and Santana, 1993). Measurements of CO_2 concentration (three replications each measurement) were performed in the field in each soil environment from autumn 2008 to autumn 2009 by using both the SL and IR methods. Then two different data sets were obtained to evaluate the CO_2 respired from soil surface in different days of the year (DOY). Though the two methods may be conceptually different the degree of similarity in the emitted CO_2 values was noticeable. As may be observed in Fig. 2, the fluxed CO_2 expressed as $\mu mol\ m^{-2}\ s^{-1}$ recorded by both methods in each DOY (from DOY 312 corresponding to Autumn 2008 to DOY 310 corresponding to autumn 2009) show a rather similar trend along the temporal sequence of measurements. The highest values of fluxed CO_2 were recorded by the SL method probably because after 24 h a higher absorption of CO_2 by soda lime occurred in the closed chamber, with respect to the transient IR measurements made in a shorter time. IR method seems to measure the immediate CO_2 concentration in five minute constraints. Irrespective to this, the values from both methods were considered at a steady state, because: i) the soda lime collected after 24 h did ever show indications of CO_2 saturation (light pink color), ii) the CO_2 values from IR apparatus was always constant after 5 min. A descriptive statistics was carried out in order to check the standard deviation (SD) and the coefficient of variability (CV) either for all the analyzed soil environments/per each DOY (day of the year), or for each soil environment/per all DOY. In Table 2 the annual mean values show that the variation of CO_2 data is higher along the days of measurements, with respect to each day of measurement for all the soil environments. This indication was already clear reading Fig. 2. Linear relationships were then tried between all sets of data though the best linear fits were found from winter period (January, DOY 13) to the beginning of summer (June, DOY 167), as may be observed in Table 3. By using these data a total of 53 positive significant correlations were obtained. By relating the CO_2 values of both SL and IR method for each environment separately, 5 positive significant correlations were found. Linear regression equations showed $r=0.873$, $p<0.01$, $r=0.849$, $p<0.01$, $r=0.736$, $p<0.05$, $r=0.581$, $p<0.05$, $r=0.697$, $p<0.05$ for MC, MB, V, O, S, soils respectively. An example of the linear fitting on yearly basis may be observed in Fig. 3A, where CO_2 mean values from IR measurements are plotted against CO_2 mean values from SL measurements in MC and MB soil environments respectively. However, the slope of the equations depicts the proportionality of CO_2 data between the two methods with respect to 1:1 line (Keith and Wong, 2006). Soils under pines (PI) or pasture (PR) did not show any relationships when CO_2 flux data of IR vs SL method were tested for these two environments separately. Probably, fresh deposition of manure following grazing activity in PR soil environment during some periods of the year may produce stronger variability in CO_2 flux, altering the comparison with one another technique. Similarly, the particular composition of hardly decomposable pine tree leaves in PI soil environment may have hindered the temporal and

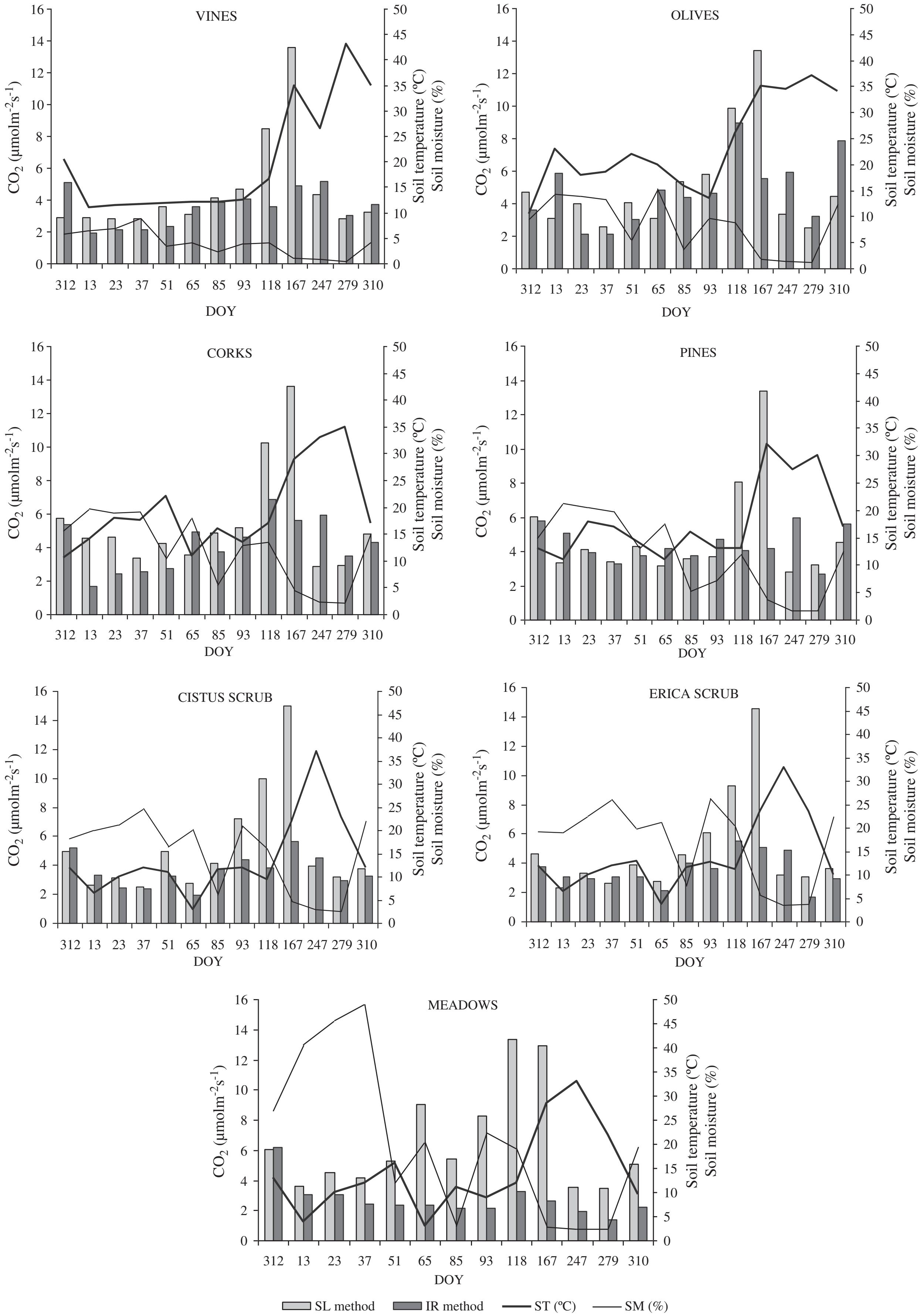

Fig. 2. Temporal mean values of CO_2 flux from soil (μmol m^{-2} s^{-1}) obtained by SL method (clear gray bar) and IR method (dark gray bar) along the period of study indicated by the day of the year (DOY) in the 7 selected soil environments. Trending of soil moisture (SM) (fine line) and soil temperature (ST) (coarse line) is also represented.

Table 2
Descriptive statistical analysis for the annual mean values (μmol CO_2 m^{-2} s^{-1}) of the two methods for all the analyzed environments/each DOY and for each environment/all DOY.

	SL method		IR method	
	Environments/ each DOY	Environment/ all DOY	Environments/ each DOY	Environment/ all DOY
X	5.36	5.36	3.81	3.81
SD	0.96	3.36	1.10	1.31
CV	20.24	63.17	28.33	34.42

X: Mean; SD: Standard deviation; CV: Coefficient of variation (%); DOY: day of the year.

spatial comparison of the two methods. Moreover, 21 positive significant correlations were obtained by relating values of fluxed CO_2 when using SL method between one another soil environments, i.e. V vs O, O vs S, S vs PI… (Table 3). Accordingly, as each environment was correlated with all the others, we may postulate that the SL method is detecting approximately the same amount of CO_2 respired from each soil environment at any measurement date independently of SOC content. By using the IR method with the same purpose, 6 positive significant correlations were obtained (Table 3) through the same soil environments, accounting for some diversity in CO_2 detection by this method, which may be probably due to the immediate response of soil to CO_2 measurements by IR apparatus. Linear fitting on yearly basis is shown in Fig. 3B with an example of CO_2 flux measured from two different soil environments (MC vs MB) by using data of SL and IR method. A rather similar sensitivity to CO_2 flux may be observed, also corroborated by both the slope of the SL graph (1.045) and the IR graph (0.824) with respect to 1:1 line. Data from SL method showed however a higher significance ($r=0.992$, $p<0.001$) with respect to IR method ($r=0.788$, $p<0.05$).

Another question to take into account is the soil temperature and soil moisture relationships and their interaction with CO_2 flux. Soil moisture showed a significant negative correlation with soil temperature in each soil environment and the period of study as showed by the following mean correlations (V, $r=-0.618$, $p=0.024$; O, $r=-0.626$, $p=0.022$; S, $r=-0.801$, $p=0.001$; PI, $r=-0.700$, $p=0.008$, PR, $r=-0.618$, $p=0.024$; MC, $r=-0.748$, $p=0.003$; MB, $r=-0.766$, $p=0.002$). Moreover, soil temperature was found to be more related to carbon dioxide emission than moisture which did not show any correlation neither with SL nor with IR method. Positive significant correlation coefficients were found between the SL method and soil temperature, but the latter was not correlated with the IR method. Values of CO_2 recorded with the SL method in V, O, PI, S, PI, PR, MC, MB soil environments gave the following statistical data respectively: V $r=0.954$, $p=0.000$; O $r=0.750$, $p=0.002$; S $r=0.694$, $p=0.03$; PI $r=0.819$, $p=0.007$; PR $r=0.437$, $p=0.204$; MC $r=0.766$, $p=0.01$; MB $r=0.826$, $p=0.006$. As indicated, only the soil under meadows (PR) did not show a significant correlation, which may be related to its particular dynamics: is the shallowest soil (25 cm depth) though is the more organic but subjected to rapid wetting–drying cycles because very exposed to wind and is periodically amended by grazing activity; the process of natural decaying vegetal debris comes mostly from the roots of herbaceous plants, though manure is another important source of organics. The values of CO_2 flux increased almost two or threefold either with SL or IR measurements at the beginning of summer season (a mean of 33 °C was recorded), compared with other periods of the year, which is in agreement with Rochette et al. (1991) stating that higher temperature and sufficient moisture content may enhance microbial activity in the soil system.

As reported elsewhere, SL and IR methods were used in the field to check the sensitivity of the measurements under natural conditions. For the SL method, the annual mean values of the thirteen averaged measurements of fluxed CO_2 in V, O, S, PI, PR, MC, MB soils, from autumn 2008 to autumn 2009 were the following: 4.57(±3.12), 5.10(±3.16), 5.44(±3.09), 4.90(±2.92), 6.53(±3.40), 5.24(±3.61), 4.92(±3.44) μmol CO_2 m^{-2} s^{-1}. A quantitative extrapolation of fluxed CO_2 values was obtained by using the molecular weight of carbon dioxide, resulting in the following annual means: 43.30(±30.24), 49.42(±30.64), 52.68(±29.93), 47.45(±28.26), 63.21(±32.93), 50.74(±34.95), 47.69(±33.28) Mg ha^{-1} $year^{-1}$ corresponding to the soil under Vines (V), Olive groves (O), Stands of cork trees (S), Pine trees (PI), Pasture (PR), *C. monspeliensis* scrubs (MC), and *E. arborea* scrubs (MB) respectively. The same procedure was applied when using the IR method. First, the annual mean values of CO_2 fluxed in V, O, S, PI, PR, MC, MB soils, from autumn 2008 to autumn 2009 were: 3.51(±1.14), 4.79(±2.07), 4.18(±1.57), 4.38(±2.99), 2.71(±1.16), 3.61(±1.10), 3.52(±1.12) μmol CO_2 m^{-2} s^{-1}, and the same quantitative extrapolation gave these results: 33.27(±10.78), 45.36(±19.57), 39.57(±14.83), 41.48(±9.43), 25.66(±10.99), 34.22(±10.41), 33.37 (±10.66) Mg ha^{-1} $year^{-1}$, corresponding to the soil under Vines (V), Olive groves (O), Stands of cork trees (S), Pine trees (PI), Pasture (PR), *C. monspeliensis* scrubs (MC), and *E. arborea* scrubs (MB) respectively. Subsequently, amounts of C–CO_2 (Mg ha^{-1} $year^{-1}$) were calculated for both SL and IR methods by multiplying the CO_2 values by the 12/

Table 3
Correlation matrix between CO_2 values from the SL and IR method for the selected soil environments, from DOY 13 (January 2009) to DOY 167 (June 2009).

		V		O		S		PI		PR		MC		MB	
		IR	SL	IR	SL	IR	SL	IR	SL	IR	SL	IR	SL	IR	SL
V	IR														
	SL	0.736**													
O	IR	0.446 NS	0.535 NS												
	SL	0.752*	0.983**	0.581*											
S	IR	0.806**	0.709*	0.711*	0.751*										
	SL	0.658 NS	0.983**	0.605 NS	0.985**	0.697*									
PI	IR	0.120 NS	0.058 NS	0.455 NS	0.068 NS	0.020 NS	0.102 NS								
	SL	0.616 NS	0.982**	0.451 NS	0.953**	0.615 NS	0.976**	0.001 NS							
PR	IR	−0.318NS	0.200 NS	0.462 NS	0.233 NS	0.084 NS	0.361 NS	0.227 NS	0.292 NS						
	SL	0.786*	0.837**	0.720*	0.853**	0.963**	0.829**	0.104 NS	0.774*	0.198 NS					
MC	IR	0.698*	0.825**	0.448 NS	0.846**	0.470 NS	0.802**	0.295 NS	0.758*	−0.026 NS	0.569 NS				
	SL	0.751*	0.977**	0.531 NS	0.981**	0.730*	0.960**	0.103 NS	0.944**	0.152 NS	0.846**	0.873**			
MB	IR	0.587 NS	0.827**	0.674*	0.890**	0.688*	0.869**	−0.011 NS	0.774*	0.321 NS	0.720*	0.788*	0.841**		
	SL	0.767*	0.991**	0.516 NS	0.992**	0.731*	0.974**	0.057 NS	0.961**	0.164 NS	0.843**	0.856**	0.992**	0.849**	

IR; infrared gas analyzer method; SL; soda lime method; V: soil under vines; O: soil under olives; S: soil under cork trees; PI: soil under pine trees; PR: soil under pasture; MC: soil under Cistus scrub; MB: soil under Erica scrub.
NS: Not significant.
** Probability level <1%.
* Probability level <5%.

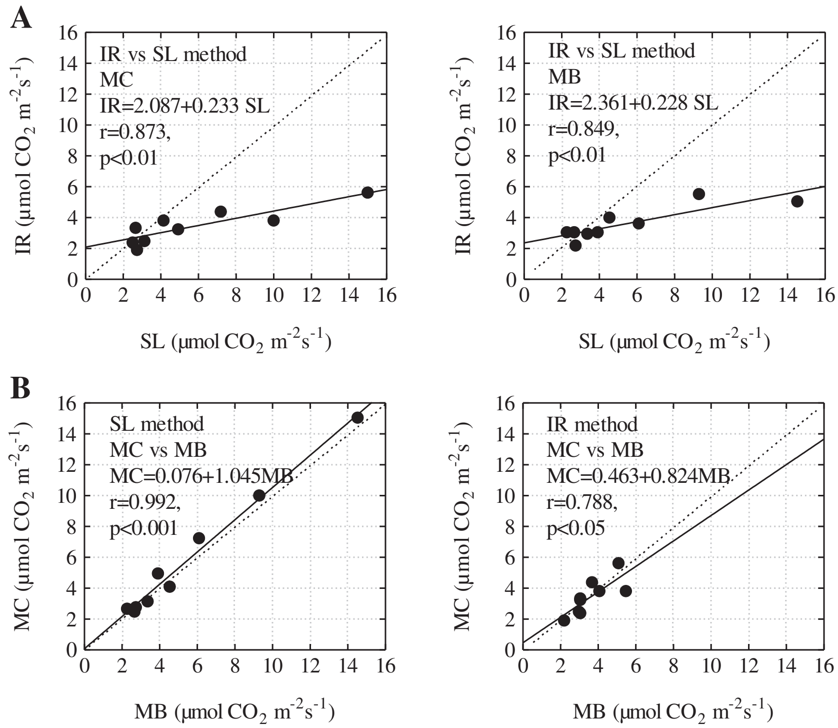

Fig. 3. Examples of fitting linear regression equations by using CO_2 values to compare IR vs SL method in MC and MB environment (A), and to check each method (SL and IR) with the two environments (B), from DOY 13 to DOY 167.

44 ratio being 12 the molecular weight of Carbon and 44 the molecular weight of CO_2 (Table 4). Finally, an indication of carbon loss potential (%) was obtained by relating the C–CO_2 amounts (Mg ha^{-1} year^{-1}) for each soil environment to the SOC stock (Mg ha^{-1}) of the same environment, calculated by using the average means of soil bulk density (Mg m^{-3}) and organic carbon (%) measured along the thirteen CO_2 experiments (Table 4). The dramatic differences in C loss (%) shown in Table 4 are produced by the differences in bulk density and organic carbon in the soil environments and may be informative, despite the high SD values, on the carbon dynamics occurring in the sequence of these peculiar studied soils. Effectively, an indication of the high variability in CO_2 field measurements by both methods was first observed by plotting the mineralization index (MI), calculated in each soil environment by dividing the mean value of C–CO_2 (Mg ha^{-1}) to the mean of SOC value (Mg ha^{-1}) from the initial three measurements at each DOY (Figure 4). In order to have a better reading of MI values, a logarithmic y-axis has been used and the x-axis crossed y scale at 1 MI. Despite the scatter of points of MI value for each soil environment, which defines the complexity of such type of measurements, stressed also by the SD values reported in Fig. 4, the findings may allow to compare the soil response to different land use and abandonment and the potential for carbon loss or storage (Romkens et al., 1999). Though MI values for soils under vines may appear meaningful, they may be justified by the fact that these soils are also often irrigated being the major CO_2 producers probably because of an extraordinary microbial activity with respect to the real SOC content. According to the presented results the sequence of susceptibility to carbon loss from the studied environments should then be the following: V > O > PI > S > PR > MC > MB by using the SL method and: V > O > PI > S > MC > MB > PR by using the IR method. Referring to the other soil environments they can be divided into soils showing a clear ability to preserve SOC, such as soils under meadows (PR), Erica scrub (MB) and cork trees, and soils with intermediate CO_2 emission because of historical fire perturbations (MC and PI environments) which may have affected their carbon storage capacity. Generally it is reported that environments with lower production of carbon dioxide are potentially carbon sequestering systems, though our data show that not always it may correspond with the highly variable field dynamics.

4. Conclusion

The reliability of the two techniques may be considered satisfactory, taking into account that these are the first data coming out from a sequence of both land use and cover change and abandonment in this

Table 4
Indicative percentage of carbon loss calculated on the basis of CO_2 fluxes from the studied environments along Autumn 2008–Autumn 2009.

Soil environment	SOC (Mgha^{-1})	SL method		IR method	
		C–CO_2 (Mg ha^{-1} year^{-1})	C loss (%)	C–CO_2 (Mg ha^{-1} year^{-1})	C loss (%)
V	6.49 ± 1.59	12.07 ± 8.07	185 ± 148	9.27 ± 2.94	143 ± 41
O	30.89 ± 5.56	13.47 ± 8.17	43 ± 34	12.65 ± 5.34	41 ± 22
S	49.49 ± 5.82	14.37 ± 7.98	29 ± 25	11.04 ± 4.05	23 ± 11
PI	32.39 ± 8.30	12.94 ± 7.54	39 ± 53	11.57 ± 2.67	36 ± 16
PR	61.00 ± 2.63	17.25 ± 8.78	28 ± 14	7.15 ± 3.00	12 ± 5
MC	51.83 ± 8.89	13.84 ± 9.32	26 ± 21	9.54 ± 2.84	18 ± 7
MB	53.37 ± 10.4	12.99 ± 8.88	24 ± 21	7.70 ± 2.91	14 ± 8

SL method: soda lime method; IR method: infrared gas analyzer method; SOC: soil organic carbon; C–CO_2: carbon–carbon dioxide; C loss; carbon loss; V: soil under vines; O: soil under olives; S: soil under cork trees; PI: soil under pine trees; PR: soil under pasture; MC: soil under Cistus scrub; MB: soil under Erica scrub.

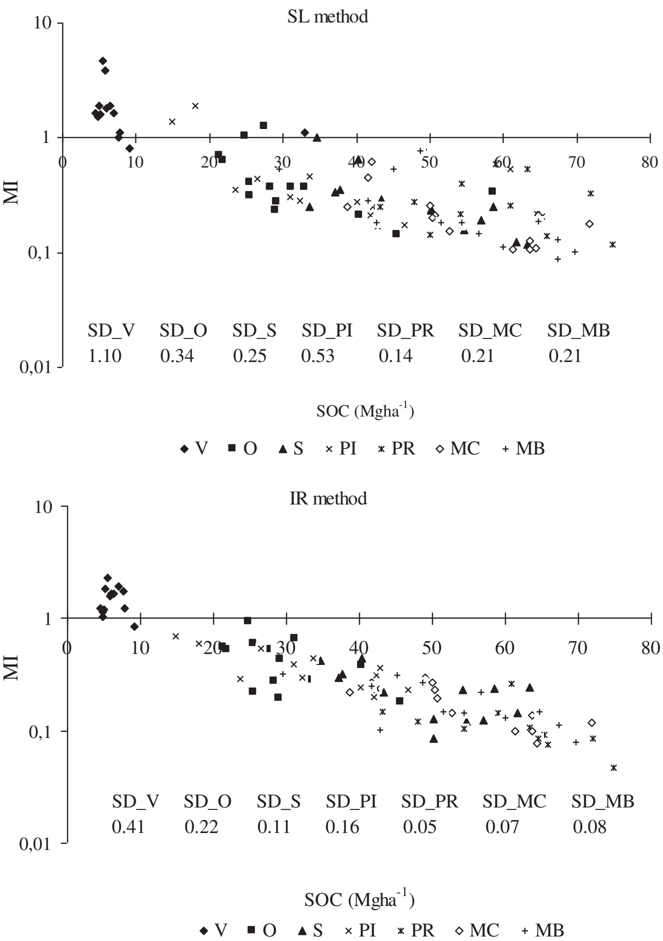

Fig. 4. Mineralization index (MI) vs soil organic carbon (SOC) stocks to emphasize the potential of soil carbon storage or loss by SL and IR method application in the studied soil environments.

part of NE Spain where very peculiar and vulnerable soils exist. Despite the great variability encountered during the measurements, data collected in this very fragile area under changing conditions should be carefully taken into consideration.

Field measurements of CO_2 showed a remarkable relationship between the IR and SL methods under the same field conditions, though the two methods differ on concept and time-dependence. The correlation between the two sets of CO_2 data were especially significant from DOY 13 to DOY 167 (from January to June 2009), and provided the best fits between SL and IR method. In this period, the two methods were compared successfully in five soil environments, except in soils under pines and meadows, though a factor of proportionality should be introduced. The correlation matrix reported high significant correlations when the CO_2 values (μmol m^{-2} s^{-1}) from SL method were tested among all the soil environments. Statistical significance at 0.01 probability level was found in all compared environments but PI vs PR which decreased to 0.05 probability level. The variations in the carbon dioxide production within the same environment and among different soil environments may explain the complexity of CO_2 production under different land use-cover change and age of abandonment. Despite that, findings showed that soils with a better soil structure and more stable organic compounds were assumed to best preserve organic carbon against CO_2 emission, and this was proved by applying the two methods. Labile forms of organic carbon were likely to be most easily mineralized by the microbial activity, and released as CO_2 from the soil surface to the atmosphere. This process seemed to be less effective in soils under S, MC, MB and PR environments with respect to soils under V, O and PI environments in terms of C–CO_2 loss. The attempt to estimate the percentage of C loss was performed by using the annual mean values of C–CO_2 calculated from CO_2 data of the SL and IR methods and the SOC pool for each environment. Data of C–CO_2 in Mg ha^{-1} $year^{-1}$ indicate a similar respiration capacity between the soil environments along the experimental period and may appear surprising due to the difference in soil characteristics. When these data are transformed as the proportion of the organic carbon pool, values allow differentiating the carbon storage potential of each environment along the land use-cover change and abandonment. According to current land use, four groups may be proposed: the first includes current cultivation (V and O environments) showing the highest loss of organic carbon. Moreover, the soils under vines should be amended with organic compounds addition as higher erosion rates have been generally recorded in this soil. The second group refers to forest soils and shows the difference in C preservation potential existing between *Q. suber* (S) and *P. pinea* (PI) probably due to a higher C/N ratio of the decaying debris of the latter one. In the third group the two types of soils under scrubs (MC and MB) are included. Though the difference in C loss percentage is not large between these two environments, it may be ascribed to the past effects of wildfire occurrence and the temporal regressive dynamics suffered by MC environment. Finally, the fourth group is formed by the soils under pasture (PR), representing the best situation as they may indicate the most favorable environment for carbon storage. At field conditions, the soil surface temperature plays an important role in CO_2 emission together with soil structure and organic matter. The increase of organic compounds in soils changes with soil use, land management, and abandonment, and this sequence may control the efflux of CO_2 from soil surface to the atmosphere. Soil use and management must be accurately planned and imbalance of CO_2 emission with respect to SOC content should be deeply studied in order to avoid land degradation processes. Land abandonment without management is not a suggestible practice because of the risk of fire occurrence in a disorganized scrubland. Nevertheless, it is proved that progressive colonization of spontaneous vegetation in abandoned agricultural land may have a primary role in recovering and preserving stable organic compounds and a good soil structure against degradation processes. This will suggest that also land abandonment is worth of special management in order to achieve a sort of mosaic of land use and prevent environmental damages.

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