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ORGANIC MATTER CHANGES IMMEDIATELY AFTER A WILDFIRE IN AN ATLANTIC FOREST SOIL AND COMPARISON WITH LABORATORY SOIL HEATING

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Summary—The quantity, chemical composition and mineralization kinetics of the organic matter of an acid Humic Cambisol, developed over granite, under *Pinus sylvestris* L. were determined in 0–5 and 5–10 cm samples collected immediately after a high-intensity wildfire, and compared with those of an unaffected site nearby. Organic matter was characterized by different chemical fractionation methods, and the C mineralization was determined by aerobic incubation. A similar unburnt soil located in the same area was heated at the laboratory at 150, 220, 350 and 490°C to measure the losses of C content; the samples heated at 220 and 350°C were selected to determine chemical changes in organic matter composition. Surface and subsurface soil layers lost about 50% of their C content during wildfire. The C mineralized decreased in the surface layer; however, the percentage of total C mineralized increased in both layers. The cumulative CO_2-C mineralized fitted a double exponential first-order kinetic model, but the fire affected the kinetic parameters, increasing both the labile pool of the potentially mineralizable C and the mineralization rate constants of the recalcitrant and labile pools. Cellulose + hemicelluloses declined significantly after the burning, whereas lipids did not vary. The fire decreased the amount of unhumified organic matter and the alkali-soluble compounds, particularly humic acids, but there was a net increase of humin. The organic matter bound to Fe and especially to Al was much higher after the burning. In the soil heated under laboratory conditions the changes observed at 150°C were very low, whereas at 490°C almost all the organic matter disappeared. The changes exhibited by the samples heated at 220°C were the most similar to those observed in the samples from the wildfire. At 220 and 350°C the humification and metal complexation percentages of the organic matter increased, similar to the trend observed in the samples from the wildfire. © 1997 Elsevier Science Ltd

INTRODUCTION

The changes induced by fires on vegetation and soil characteristics have been studied for a long time. Vegetation fires are considered to be a natural phenomenon in many parts of the world, where repeated fires have resulted in maintaining particular ecosystems (Ahlgren and Ahlgren, 1960). In such areas controlled fires are widely used to reduce accumulated fuel and thus limit the risk of high-intensity wildfires. Reports are available on controlled fires (Chandler *et al.*, 1983; Khanna and Raison, 1986; Marion *et al.*, 1991) and soil heating under laboratory conditions (Kang and Sajjapongse, 1980), and their influence on soil properties such as pH, loss and availability of nutrients (Feller, 1988). Studies on high-intensity wildfires are scarce and, moreover, most were undertaken after some time had lapsed since the fire.

The northwest of Spain, which is located in the temperate-humid climate zone, contains soils which are rich in organic matter, and are acidic and sandy. This region had been considered in the past to be a relatively low-risk wildfire area, because of its climate and the nature of floristic composition of its forests. Deciduous species such as oak, chestnut, birch and alder in the natural forests of this area possess an intrinsic high resistance to fires. During the last two centuries the landscape of this region was greatly modified by human activities which included growing evergreen trees, particularly resinous, types which are more vulnerable to fire. Consequently, in recent decades the number of wildfires has dramatically increased. These fires adversely affect vegetation cover and soils. In some cases soil erosion produced irreversible damage, with consequent economic losses and landscape deterioration. Díaz-Fierros *et al.* (1982, 1987) found that the erosion risk increased considerably during the first year after a fire. These authors reported an average soil erosion of $56 \, t \, ha^{-1}$ for this period, the risk declining exponentially to negligible amounts after 12 months.

In sandy soils the main factor of cohesion and protection against erosion when the fires destroy the vegetation cover is a high content of organic

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matter which, in this area, is maintained even after high-intensity wildfires (Bará, 1981). The organic matter also produces the adequate conditions for the establishment of new vegetation contributing to soil regeneration. Studies on the effects of fires on organic matter content are quite common (e.g. Kang and Sajjapongse, 1980; Giovannini et al., 1987; Kutiel and Kutiel, 1989); however, those on changes in organic matter composition (Shindo and Urabe, 1993), structure (Almendros et al., 1992) and biological characteristics (Vázquez et al., 1993; Fritze et al., 1994; Acea and Carballas, 1996) are less so. There are studies on organic matter composition and mineralization in the soils of the northwest of Spain (Carballas et al., 1979, 1980, 1983; Beloso et al., 1993), but until now the effect of fires on these characteristics had not been studied.

Organic matter is responsible for forest ecosystems productivity because of its nutrient content and its influence on physico-chemical and biological characteristics. Because of this the aim of the study was to evaluate the effects of fire on the composition, stability and C mineralization of the soil organic matter immediately after a high-intensity wildfire.

MATERIALS AND METHODS

The soils selected for study were two Humic Cambisols under a pine forest of Pinus sylvestris, developed over granite and located in Cabeza de Manzaneda (Orense, northwest Spain) at 1740 m above sea level. The undergrowth consisted of Chamaespartium tridentatum (L.) P. Gibbs, Vaccinium myrtilus L., Erica spp and species of Gramineae. The burnt soil was sampled in September, 1 d after a high-intensity wildfire; simultaneously an unburnt soil from the same edaphic and pine stand located close to the burnt area was sampled and used as a control. Soil samples were collected at 0–5 and 5–10 cm depth all along a continuous profile 10 m in length. They were sieved (4 mm), and the fraction less than 4 mm was homogenized. Fresh samples were used for the study of C mineralization. Air-dried samples were employed for the determination of organic matter and the other soil characteristics.

For the experiments on soil heating under laboratory conditions a different unburnt soil from the same area, but far from the burnt zone, was used. The sample was collected in October, in the same way as the others from 0–5 cm, using a subsample as control. The soil was heated in an oven with a timer and a heating rate programme. Temperature in the soil was controlled by a thermocouple located into the bulk of soil. Conditions of heating were as follows: an increase of $3°C\,min^{-1}$ to prevent the sudden combustion produced when reaching the soil ignition temperature; 30 min of heating after

reaching the selected heating temperature; and a sample size of less than 1 cm depth to avoid a temperature gradient in the profile. The soil was treated at the ignition temperature (220°C; Salgado et al., 1995) and at temperatures lower (150°C) and higher (350 and 490°C) than the ignition temperature. Losses of organic matter were determined in all the samples. According to these losses the samples heated at 220 and 350°C were selected for subsequent organic matter fractionation. The same fractionation procedure was followed in the control sample.

The methods described by Guitián Ojea and Carballas (1976) were used to determine the following soil properties: field capacity (at 10 KPa in a Richard's membrane-plate extractor); pH in H_2O (1:2.5); pH in 1 N KCl (1:2.5); extractable Fe and Al oxides (by extraction with a mixture of hydrosulphite and Tamm's reagent). C content was determined by combustion and measurement of the CO_2 in a Carmhograph 12 with the primary oven at 900°C and the secondary oven at 400°C. The factor used to convert C into organic matter was 1.724 (Guitián Ojea and Carballas, 1976). Total N content was determined by Kjeldahl digestion using the method developed by Bremner (1965). The results are the average of three replicate determinations.

Analysis of C-containing compounds was performed by the method of Kononova (1961), except for water-soluble compounds that were extracted as recommended by Stevenson (1965). The water-soluble fraction was extracted by refluxing with boiling water for 2 h. Lipids were extracted from the residue with ethanol-benzene for 24 h in a Soxhlet. The residue was treated with H_2SO_4 to solubilize cellulose and hemicelluloses leaving lignin and mineral material as a residue. After ignition of this residue in an oven at 500°C for 6 h the lignin content was calculated by difference from the final residue weight (González-Prieto et al., 1993). The results were expressed as percentage of either soil weight or total organic matter.

To study the humified organic matter of the 0–5 cm soil layer a modified method of Duchaufour and Jacquin (1966) was used. After a densiometric separation of the unhumified and the humified organic matter using bromoform-ethanol with a density of 1.8, the humified organic compounds were extracted successively by 1% $Na_4P_2O_7$ (pH 9.8) and 0.1 N NaOH (pH 13). The extraction was performed by agitating soil with the corresponding reagent for 1 h, centrifugation and repetition of the cycle an additional time. The final residue, which was composed of the humin fraction (H), was dried at 40°C, ground and homogenized. In each extract humic acids (HA) were separated from fulvic acids (FA) by precipitation at pH 1 with H_2SO_4 and centrifugation after more than 4 h of standing (González-Prieto et al., 1993). C was determined in each

extract by Sauerlandt's method (Guitián Ojea and Carballas, 1976) which used $Cr_2O_7K_2 + H_2SO_4$ for digestion and further titration with $(NH_4)_2SO_4 \cdot FeSO_4 \cdot 6H_2O$. The residue C content was assessed using the Carmhograph method. Fe and Al bound to the organic matter was assessed by determining these elements in the alkali-extracts by atomic spectrophotometry. The per cent extraction $(C_{FA + HA}$ total $C^{-1})$, per cent humification $(C_{FA + HA + H}$ total $C^{-1})$ and indices of polymerization, fulvic acids to humic acids ratio (FA-to-HA), and metal complexation, (Fe + Al)-to-C ratio, were calculated.

Fresh samples or samples stored at 4°C for less than 7 d were used for the determination of the C mineralization kinetics by aerobic incubation of the unburnt and the burnt soil samples from the wildfire. The incubation method was that of Guckert et al. (1968) modified by Choné et al. (1974), who used an intermittent air flow for aeration. Five replicates of 50 g soil from each sample were placed into 500 ml Erlenmeyer flasks and maintained at 28°C and moisture at 75% of field capacity for 11 wk. The flask atmospheres were periodically renewed with CO_2-free air, and the CO_2 evolved was trapped by bubbling for 2 h in 40 ml of 2 N NaOH. From 5 ml NaOH solution, the Na_2CO_3 formed was precipitated by 20% $BaCl_2$ solution and then the remaining NaOH was measured by titration against 0.2 N HCl using a Metrohm 682 titrator. The CO_2 was then quantified by subtraction, using two empty flasks incubated under the same conditions as the control (Carballas et al., 1979; González-Prieto et al., 1991; Beloso et al., 1993). After 77 d of incubation, pH in water and KCl were determined in the burnt and the unburnt samples. The C mineralization, expressed as g CO_2-C evolved kg^{-1} soil, and the C mineralization coefficient expressed as a percentage of the total C were calculated.

The cumulative curves of CO_2-C release over time followed the double exponential model proposed by Andrén and Pauskian (1987) and employed by Updegraff et al. (1995) to quantify the kinetics of C release from different soils under different conditions:

$$C_t = C_0(1 - e^{-kt}) + (TC - C_0)(1 - e^{-ht}) \quad (1)$$

where C_t is the cumulative C released after time t (days), C_0 is the potentially mineralizable C in a labile pool with a instantaneous mineralization rate k (d^{-1}), TC is the total amount of C present in the soil sample $(g\ kg^{-1})$ and $(TC - C_0)$ is the amount of C in a recalcitrant pool with a instantaneous mineralization rate h (d^{-1}). This model explains the overall C mineralization process by considering two pools of C that decay independently and in parallel, with different degradation rates. The non-linear parameter estimation procedures in SPSS.5.0.1. for Windows were used to fit the experimental data to the model. To avoid errors in the parameter estimation the convergence criteria indicated by Updegraff et al. (1995) were used.

Two-way ANOVA test was applied to weekly C mineralization data to follow the kinetic variations as well as to quantify the variation of C mineralization attributable to the burnt soil and to the location of the layer in the profile. The least significant difference (LSD) test (Tukey, 1953) at the 95 and 99% probability levels was applied to the results. The χ^2 test was used for comparison of the results obtained in the different organic matter fractionations.

RESULTS AND DISCUSSION

Table 1 shows the main characteristics of the soil samples. The unburnt soil varied little in depth, was highly acid, had a high content of organic C and N, high values of cation exchange capacity (CEC), with a low percentage of base saturation and a moderate content of extractable Fe and Al oxides.

Immediately after the fire the increase of pH was moderate (Table 1) due to the high value of the buffer capacity index (Guitián Ojea and Carballas, 1976) of this soil (5.99) and the short time that had elapsed after the fire. Therefore, it is probably that pH had not yet reached its maximum value. This

Table 1. Main characteristics of the unburnt (UB), the burnt (B), the unheated (UH) and the heated (H) soil samples (mean values ± SD)

Sample	Depth (cm)	pH H_2O	pH KCl	C (g kg^{-1})	C-to-N	N (g kg^{-1})	Fe_2O_3 (g kg^{-1})	Al_2O_3 (g kg^{-1})	CEC (meq 100 g^{-1})	Base saturation (%)
UB[a]	0–5	4.19 ± 0.02	3.29 ± 0.01	158.2 ± 1.8	18	8.73 ± 0.10	5.5 ± 0.1	6.3 ± 0.4	51.8 ± 0.1	5.7 ± 0.4
UB[ss]	5–10	4.25 ± 0.03	3.45 ± 0.01	111.0 ± 1.8	18	6.30 ± 0.14	6.5 ± 0.1	7.8 ± 0.5	49.7 ± 1.5	3.3 ± 0.4
B[a]	0–5	4.74 ± 0.03	4.00 ± 0.00	74.4 ± 1.0	15	4.87 ± 0.15	4.9 ± 0.3	6.1 ± 0.3	24.2 ± 1.5	13.2 ± 0.3
B[ss]	5–10	4.58 ± 0.03	3.78 ± 0.03	51.1 ± 2.1	15	3.31 ± 0.07	4.7 ± 0.0	6.1 ± 0.1	33.8 ± 1.1	3.6 ± 0.2
UH	0–5	4.35 ± 0.03	3.30 ± 0.01	102.9 ± 1.1	19	5.49 ± 0.06	5.5 ± 0.1	6.3 ± 0.1	43.9 ± 0.4	3.1 ± 0.0
H[a]$_{150}$	0–5	4.00 ± 0.02	3.30 ± 0.03	105.1 ± 0.1	19	5.50 ± 0.02	5.6 ± 0.1	6.8 ± 0.9	ND	ND
H[a]$_{220}$	0–5	4.05 ± 0.02	3.15 ± 0.02	64.8 ± 0.1	12	5.40 ± 0.01	5.0 ± 0.2	6.2 ± 0.0	ND	ND
H[a]$_{350}$	0–5	5.70 ± 0.05	4.75 ± 0.03	10.7 ± 0.0	8	1.40 ± 0.01	5.4 ± 0.1	7.1 ± 0.0	ND	ND
H[a]$_{490}$	0–5	6.35 ± 0.04	5.45 ± 0.05	0.3 ± 0.0	ND	0.10 ± 0.00	5.6 ± 0.1	8.6 ± 0.1	ND	ND

[a]Heating temperatures (°C).
ND = not determined.

Table 2. Carbon mineralization capacity (mean values ± SD), kinetic parameters for C mineralization model based on equation 1 (estimate values ± asymptotic SE) and pH of the surface (s) and subsurface layer (ss) of the unburnt (UB) and the burnt (B) soil samples after 11 weeks of incubation (mean values ± SD)

Sample	Mineralized C $(g\,kg^{-1})$	Percentage of C mineralized $(g\,C\,100\,g^{-1}C_t)$	Kinetic parameters				pH	
			C_0 $(g\,kg^{-1})$	k	$h \times 10^4$	R^2	H_2O	KCl
UB_s	3.41 ± 0.09^c	2.15 ± 0.05^b	$0.67 + 0.02$	$0.105 + 0.005$	2.29 ± 0.02	0.9998	4.00 ± 0.0	3.15 ± 0.02
UB_{ss}	1.97 ± 0.07^a	1.77 ± 0.06^a	0.34 ± 0.01	0.159 ± 0.011	1.94 ± 0.02	0.9995	3.95 ± 0.0	3.26 ± 0.02
B_s	2.58 ± 0.07^b	3.47 ± 0.10^c	1.06 ± 0.02	0.184 ± 0.008	2.80 ± 0.06	0.9986	5.12 ± 0.0	4.05 ± 0.01
B_{ss}	1.99 ± 0.06^a	3.89 ± 0.12^d	0.48 ± 0.01	0.173 ± 0.008	3.98 ± 0.04	0.9996	5.00 ± 0.0	3.45 ± 0.03

[a–d]Values within columns with dissimilar letters are significantly different ($P \leq 0.01$).
C_0, potentially mineralizable C of the labile pool.
k, instantaneous mineralization rate of the labile C pool.
h, instantaneous mineralization rate of the recalcitrant C pool.

agrees with the higher pH in water exhibited by the burnt soil after incubation which was not found in the unburnt soil (Table 2). Chandler et al. (1983) attributed the increase of pH after burning not only to fire intensity, but also to soil characteristics. Ulery et al. (1993) gave the loss of organic acids during the fire and the addition of hydroxides and carbonates by the ashes as some of the reasons for the increase in pH. The percentage of base saturation increased and the CEC decreased as a consequence of the disappearance of organic matter due to fire (Table 1).

In the soil heated at 150°C there were no significant changes in most of the soil characteristics except a low decrease of pH which was also observed at 220°C (Table 1). At 350°C the pH was more than one unit higher and at 490°C it was two units higher than that of the control soil. The low decrease of pH at the lower temperatures as well as the high increase at the higher temperatures agreed with the findings of Nishita and Haug (1972), Giovannini et al. (1990) and Díaz-Raviña et al. (1992). Extractable Al oxides also decreased at 220°C, which was similar to that of the wildfire, and showed an increase at 350 and 490°C. In the case of Fe, a decrease was only observed in the soil sample heated at 220°C.

Organic matter content

The C content had decreased considerably after the fire. The decrease was approximately 50% in the whole depth studied (Table 1); however, it is likely that C losses were even higher in the surface layer, which was much more morphologically altered, and that they had been partially compensated by deposition of plant material not completely incinerated. Chandler et al. (1983) reported the great variability of the effect of fire on organic matter content ranging from a total destruction of the organic matter due to wildfires to increases that can reach 30% in the surface layers as a consequence of organic material redistribution in the profile. The loss of C was accompanied by a lower N decrease and consequently the C-to-N ratio also decreased

from 18 (characteristic of a moder forest humus) to 15 after the fire. The soil heated at 150°C did not exhibit a significant loss of C, whereas at 490°C almost all the organic matter had disappeared (Table 1). In the soil heated at 220°C, 37% of C was lost and at 350°C the loss was 90% of the total C. The C-to-N ratio dramatically decreased in the soil heated at 220°C, declining to a value of 8 in the soil heated at 350°C.

C mineralization activity

Figure 1, which shows the cumulated percentage of C mineralized, indicates that the wildfire not only affected the percentage of C mineralized by the end of incubation, but also altered the C mineralization kinetics during the incubation.

The C mineralization coefficient was relatively low ($\approx 2\%$ of total C after 11 wk) in the unburnt soil. At the end of the incubation this coefficient as well as the total amount of C mineralized were statistically different ($P \leq 0.01$) in both soil layers (Table 2). Low values for endogenous C mineralization coefficient are usual in soils over granite of similar characteristics from the same area (Carballas et al., 1979; Beloso et al., 1993).

The CO_2-C evolved from the surface layer of the burnt soil was significantly lower ($P \leq 0.01$) than that of the same layer from the unburnt soil (Table 2). This result agrees with that found by Almendros et al. (1984a) for soils under a mixed forest, collected 2 y after the fire. The C mineralization coefficient in the surface layer of the burnt soil was significantly higher ($P \leq 0.05$) than that of the corresponding layer of the unburnt soil throughout the incubation period (Fig. 1) as a consequence of the high loss of C after the fire. In the subsurface soil, fire did not significantly affect the amount of organic matter mineralized (Table 2). Nevertheless, like the surface soil, the C mineralization coefficient increased significantly ($P \leq 0.01$) and after 11 wk incubation was twice that of the unburnt soil (see Fig. 1 and Table 2).

After burning, the surface soil exhibited a high initial loss of CO_2-C that declined sharply after
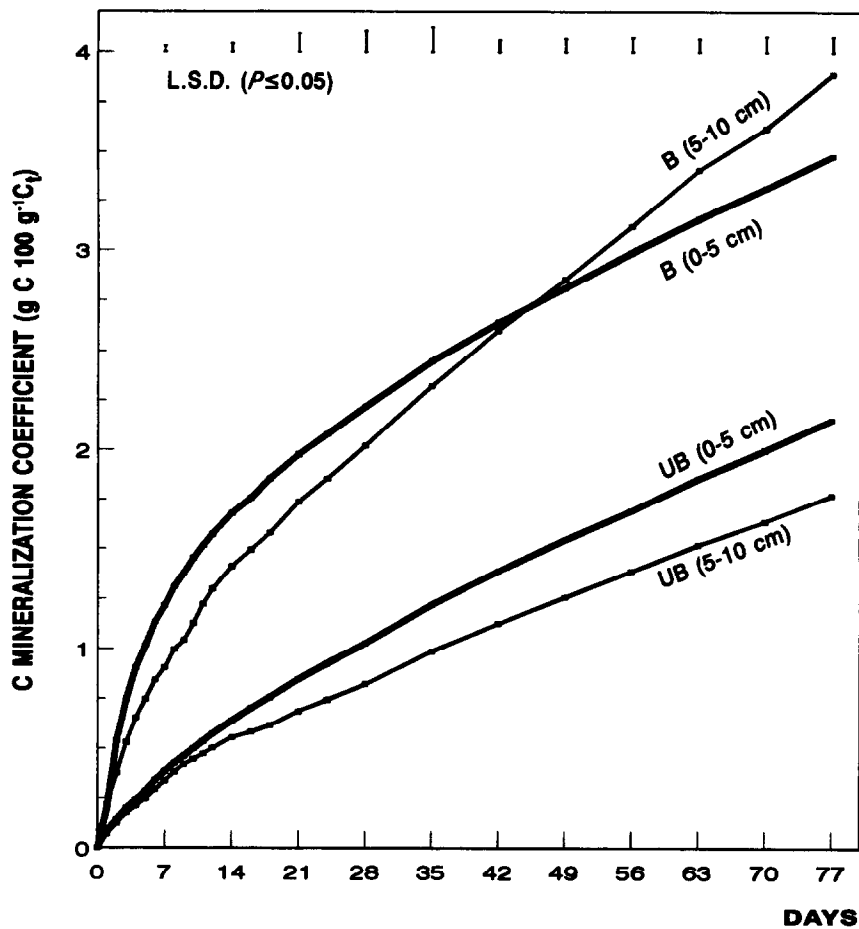
Fig. 1. Cumulative C mineralization coefficient during incubation of the 0–5 and 5–10 cm layers of the unburnt (UB) and the burnt (B) soils just after the wildfire. Least significant difference ($P \leq 0.05$) shown as bars at week intervals.

14 d of incubation; by this period the slope of the curve diminished and was practically parallel to that of the control soil (Fig. 1). The subsurface soil also exhibited an initial high value of CO_2 evolved, although lower than that observed in the 0–5 cm layer. After 42 d the CO_2 evolved by the subsurface soil was higher than that evolved by the surface soil, the difference between both values were significant from week 8 ($P \leq 0.05$) until the end of the incubation ($P \leq 0.01$). The two-way ANOVA test indicated a significant interaction between the burnt–unburnt factor and the depth of soil (0–5 cm/5–10 cm).

The cumulative curve of CO_2-C evolved by the different soil samples significantly fitted the first-order equation proposed by Stanford and Smith (1972), which was successfully used by Zak et al. (1993) to describe the mineralization kinetics in native soils and by Prieto-Fernández et al. (1993) in native and burned soils. However, as in the case of Díaz-Fierros et al. (1988) for N mineralization and Updegraff et al. (1995) for C and N mineralization kinetics, the CO_2-C data evolved by the soils stu-

died showed a superior fit to a double exponential model; the kinetic parameters obtained using the model proposed by Andrén and Pauskian (1987) are shown in Table 2. The values of these parameters showed the differences between the kinetics of the burnt and unburnt soils. The fire increased the potentially mineralizable C of the labile pool, which was 1.6- and 1.4-fold higher in the surface and the subsurface layer, respectively, of the burnt soil than that in the corresponding layers of the unburnt soil. In general the mineralization rate constants were also increased by the burning. For the labile pool the kinetic constant (k) was multiplied by 1.8 in the surface layer of the burned soil, whereas it did not vary in the subsurface layer. In contrast, for the recalcitrant pool the mineralization rate (h) duplicated after the fire in the subsurface layer and increased only 1.2-fold in the surface layer.

C-containing compounds

The organic matter content in the surface and the subsurface soil layers from unburnt site was 27.3
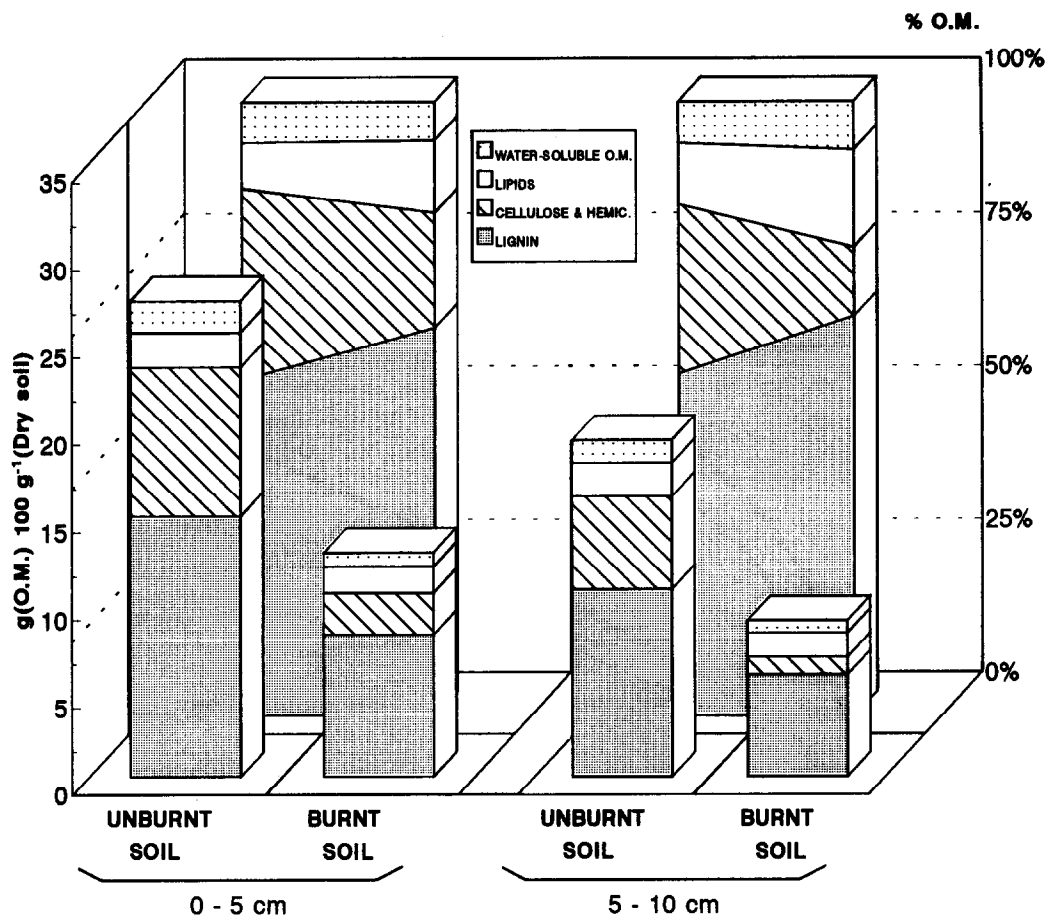
Fig. 2. Content (foreground of the figure) and changes in the percentage distribution (background) of the main groups of C-containing compounds of the organic matter from the 0–5 and 5–10 cm layers of the unburnt and the burnt soils.

and 19.1%, respectively, and the composition of the organic matter was similar in both soil layers as shown by the percentage distribution of the different fractions (Fig. 2). Lignin, which represented more than 50% of the total organic matter, was the predominant fraction followed by cellulose + hemicelluloses and a much small proportion of lipids and water-soluble compounds that did not reach 10% of the total organic matter. Therefore the most recalcitrant fractions to microbial attack predominated. This finding agreed with the C mineralization activity of this soil.

Burning affected all the organic matter fractions studied, although to a different extent (Fig. 2). In the surface soil, 72% of the cellulose + hemicelluloses disappeared, this fraction being the most affected by the fire followed by the water-soluble compounds (56% of losses), lignin (46%) and lipids (25%). The same trend was observed in the subsurface soil, but the disappearance of cellulose + hemicelluloses was higher (81%) and that of water-soluble compounds lower (46%), lignin and lipids presenting similar losses to those of the

surface layer (46 and 26%, respectively). Therefore the fire increased the more labile fractions and decreased the more resistant fractions (lignin + cellulose + hemicelluloses). The χ^2 test indicated no significant differences between the surface layers from the burnt and unburnt soils, but significant differences in subsurface layers ($P \leq 0.05$). Almendros *et al.* (1988) observed that fire increased the proportion of the shorter chain molecules of the lipid fraction, which may explain the increase of the C mineralization coefficient in both surface and subsurface soils immediately after the fire.

Although the number of samples is not enough to show possible correlations among the kinetic parameters (Table 2) and the different fractions of the organic matter, there was a high correlation ($r = 0.9420$) between the content of the water-soluble compounds and the C mineralization rate of the labile C pool (k), although this was at a low level of significance ($P = 0.058$).

Heating of the soil in the laboratory had similar effects on the distribution of the C compounds as
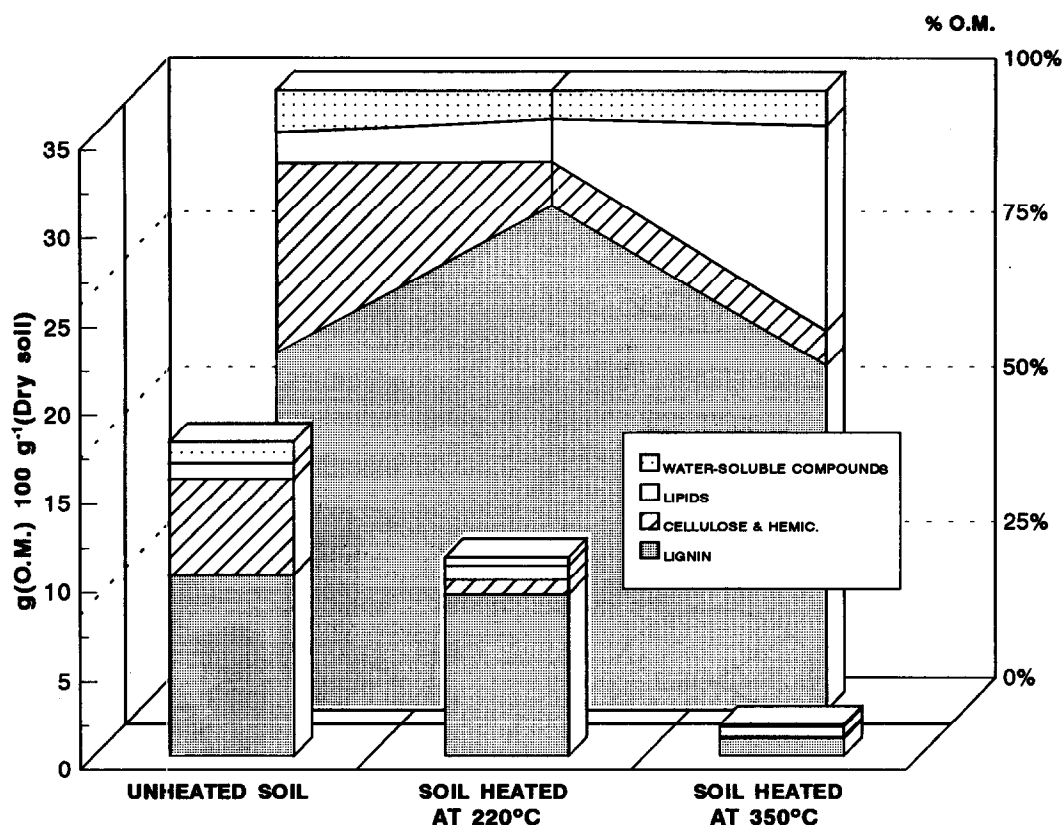
Fig. 3. Content (foreground of the figure) and changes in the percentage distribution (background) of the main groups of C-containing compounds of the organic matter from the surface layer of the unheated soil or soils heated at 220 and 350°C in the laboratory.

those of wildfire (Fig. 3). At 220°C, lignin, which was the most resistant fraction to the wildfire, only decreased by 11%. Lipids declined in the same proportion, whereas cellulose + hemicelluloses was the fraction most affected by the heating—85% of this fration disappearing during the heating process. At 350°C, when only 10% of the initial organic matter remained, lipids were the least affected fraction because only 33% of them disappeared, whereas 90% of water-soluble compounds, cellulose + hemicelluloses and lignin fractions were destroyed. The percentage distribution clearly showed the decrease of cellulose + hemicelluloses and the increase of lipids when the temperature increased. The χ^2 test showed that the organic matter composition of the soils heated at 220 or 350°C were significantly different ($P \leq 0.05$) from that of the unheated soil.

Humus

Figure 4 shows the percentage composition of the humified organic matter as well as the C content for the different fractions expressed as a percentage of dry soil. In the unburnt soil, although the content of unhumified organic matter was high, the humified fraction clearly predominated. Approximately 50% of the humified organic matter corresponded to humic acids, whereas the other

50% was quite uniformly distributed between fulvic acids and humin. The extraction and humification percentages as well as the FA-to-HA ratio (Table 3) were of the same order of magnitude as those found for other soils of the same area located at similar altitude and topography (Carballas *et al.*, 1983). The organic matter was quite well humified and moderately polymerized with a predominance of HA according to the FA-to-HA ratio. The extractable Fe and Al content of the different fractions was not very high, which implies a low degree of complexation of the organic matter. The extractable Al content in each fraction was always higher than that of extractable Fe, agreeing with the findings of Carballas *et al.* (1980) who studied the organo-metallic complexes in soils developed over granite from the same region.

The decrease of organic matter after fire was higher for the unhumified than for the humified fraction, and consequently the percentage of humification dramatically increased (see Fig. 4 and Table 3). There was a net increase of the humin fraction, as also observed by Almendros *et al.* (1990) in an oak forest affected by a fire. Humin was the predominant fraction in percentage distribution of the humic compounds, whereas HA and FA fractions decreased not only in percentage dis-
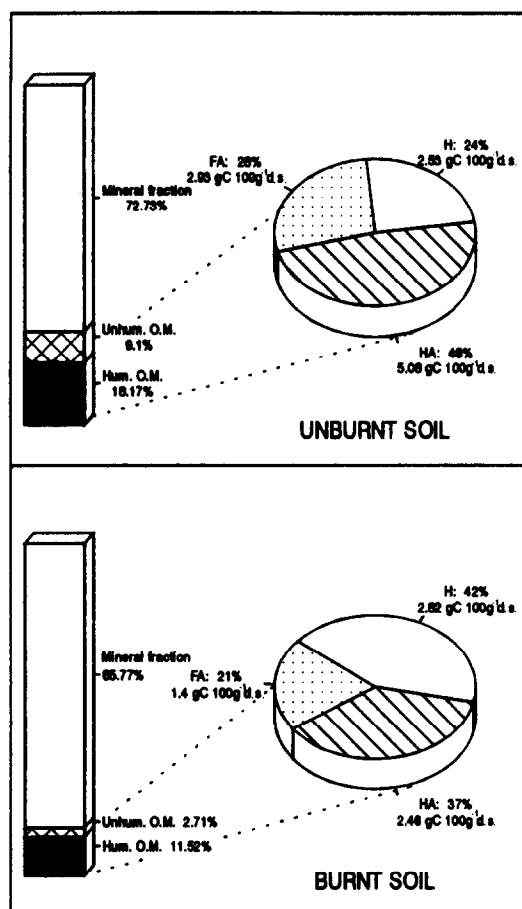
Fig. 4. Content and distribution of the different humic fractions from the 0–5 cm layer of the unburnt and the burnt soils.

Fig. 5. Fe and Al bound to humic (HA) and fulvic (FA) acids from the 0–5 cm layer of the unburnt and the burnt soils.

tribution, but also in absolute amounts—the decrease being higher for HA. The effect of the fire on the organic matter was similar to a climatic maturing (Almendros *et al.*, 1984b) because although the extraction percentage decreased slightly, the percentage of humification increased considerably as well as the degree of polymerization, all pointing to a more stabilized organic matter. The fire increased the Fe and Al bounded to organic matter (from 39 to 91 mg Fe g^{-1} C and
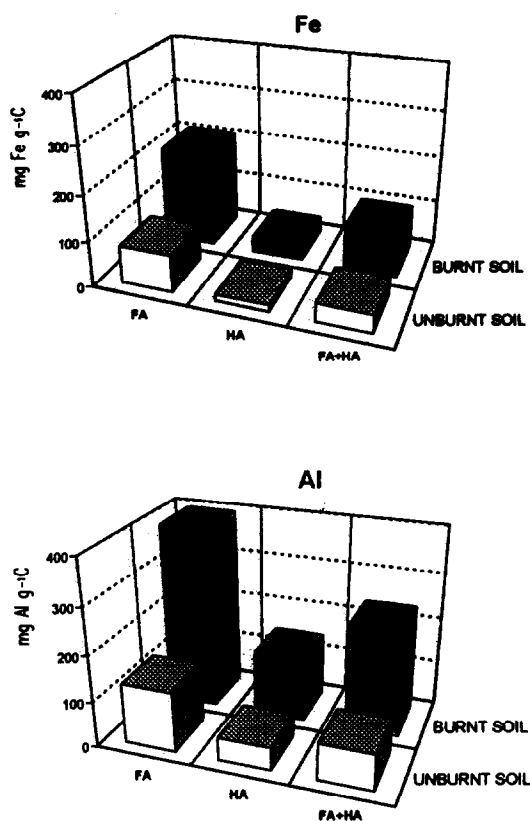
from 79 to 223 mg Al g^{-1} C considering the FA and HA fractions together) (Fig. 5). The complexation capacity of FA was higher than that of HA; moreover organo-Al complexes predominated over organo-Fe complexes. These findings also contributed to an increase in organic matter stabilization (Jacquin *et al.*, 1978; Cabaneiro *et al.*, 1987).

Heating the soil in the laboratory at 220°C resulted in a decrease of humified and unhumified organic matter, the losses being 22 and 55% of the total organic matter, respectively (Fig. 6). Although HAs were still the predominant fraction, they underwent the highest decrease, in contrast to

Table 3. Characteristics of the humified organic matter after a high intensity wildfire and after heating under laboratory conditions

Sample	Depth (cm)	Percentage extraction $(C_{(FA + HA)}C_t^{-1})$	Percentage humification $(C_{(FA + HA + H)}C_t^{-1})$	FA-to-HA	(Fe + Al)/C
UB[a]	0–5	51	67	0.58	0.12
B[a]	0–5	47	81	0.57	0.32
UH	0–5	44	54	0.46	0.16
H$_{220}^{a}$	0–5	48	67	0.52	0.29
H$_{350}^{a}$	0–5	8	70	0.60	12.89

UB, unburnt sample; B, burnt sample; UH, unheated sample; H, heated sample.
[a]Heating temperatures (°C).
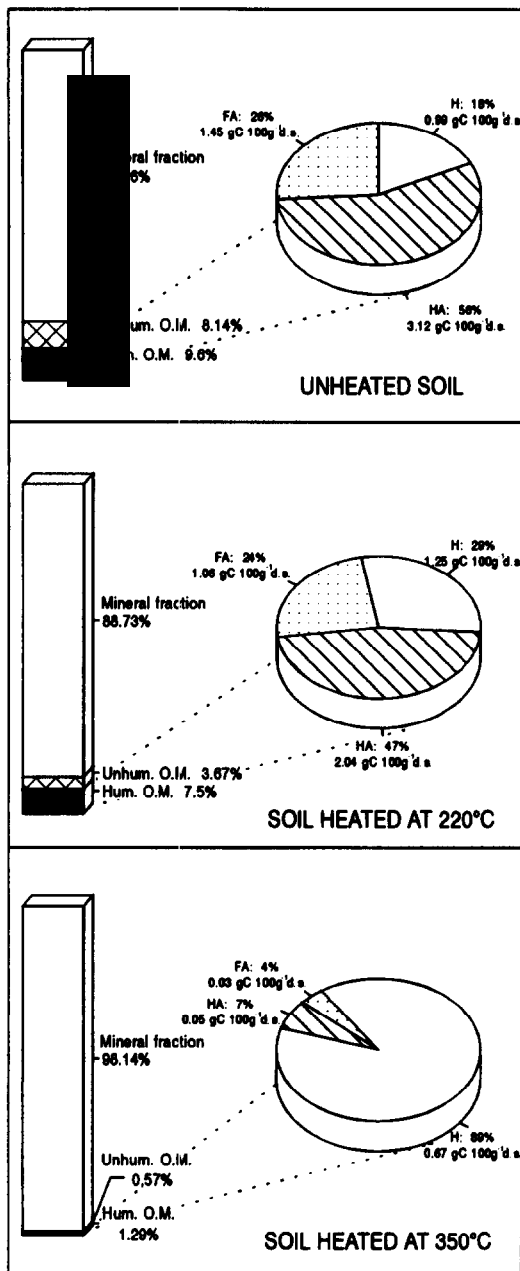FA, fulvic acids; HA, humic acids; H, humin.

Fig. 6. Content and percentage distribution of the different humic fractions from the surface layer of the unheated soil or soils heated at 220 and 350°C in the laboratory.

humin which increased; however, humin was not high enough to be the predominant fraction as it was after the wildfire. At 350°C the loss of organic matter was so high that the humin fraction was the only component of the organic matter that remained after the heating. Complexation of the organic matter with metals followed the same trend as in the case of the wildfire (Table 3). It increased after the heating, the association with Al being also favoured. At 220°C the increase of complexation

was similar to that found for the wildfire, whereas at 350°C it was considerably increased. Nevertheless the high increase of organic matter bound to metals, particularly to Al (Al-to-C = 12.29) observed at 350°C, could not be a real effect but rather a consequence of the alteration of Al inorganic forms that became soluble at high pH (NaOH at pH 13, in which fraction 93% of extractable Al was obtained). This did not occur for Fe complexation capacity since at 350°C the Fe-to-C ratio accounted for only 0.60.

The χ^2 test showed that the percentage variation of the different humus fractions from the wildfire soil was significantly different ($P \leq 0.05$) from that of the unburnt soil. Comparison of the unheated soil with the soil heated at 220°C at the laboratory showed that their composition were not significantly different, whereas the composition of the organic matter of the soil heated at 350°C was significantly different ($P \leq 0.01$) from that of the unheated soil.

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