Changes in a Humic Cambisol heated (100–500 °C) under laboratory conditions: The significance of heating time

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Abstract

The influence of the temperature and its duration, that are among the most important components to determine the fire effects on soil properties, were studied in the 0–5 cm layer of a Humic Cambisol developed over siliceous conglomerates and under a shrub of *Erica australis*. Soil was sandy loam with 8.5 g 100 g^{-1} of organic matter, pH of 4.7 and available P about 0.16 mg kg^{-1}. The water stability index (WSI) was 64.9, which was considered good. Differential thermal analysis (DTA) showed the presence of two peaks, one endothermic until 177 °C and other exothermic between 229 °C and 579 °C. Samples were heated at 100 °C, 200 °C and 500 °C during 5, 15, 30 and 60 min after reaching the selected temperature. To avoid a temperature gradient in the profile a sample layer of 2 cm depth was used. Soil heating to 100 °C at the four heating times and to 200 °C for 5 min did not produce significant changes in pH, organic matter, total N, C/N ratio, available P, extractable cations and WSI. Soils heated to 200 °C for 15, 30 and 60 min and 500 °C for 5 min, showed a significant increase in available P (from 0.9 to a maximum of 17.6 mg kg^{-1}) and divalent cations (Ca^{2+} from 4.4 to 46.3 and Mg^{2+} from 1.5 to 8.7 mg kg^{-1}). For soils heated to 500 °C for 15, 30 and 60 min, a clear increase in soluble Na$^+$ content (from 0.5 to 1.8 mg kg^{-1}) and pH (from 5.5 to 8.1), in addition to a significant decrease in organic matter (from 8.7% to 2.4%) was detected. As a consequence an important decrease in WSI was found (from 64.9% to 54.3%).
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1. Introduction

Forest fires are one of the main disturbances in Mediterranean ecosystems and their number, size and intensity have increased over the last few decades (Moreno et al., 1998; Cruz and Moreno, 2001). Wildfires can burn vegetation and litter, affecting the soil by different thermal gradients depending on heating duration, depth and fire intensity (Iglesias et al., 1997). The impact of fire is usually evident only in the upper 5 cm of soil, since temperature rarely exceeds 150 °C below this depth (Raison et al., 1986; Miranda et al., 1993). Although fire effects depend on many factors such as soil characteristics, soil–water conditions, vegetation type, etc, the changes caused in the chemical characteristics of the soil are directly related to fire intensity and fire residence time (Josa et al., 1994). It is very difficult to control fire intensity and fire residence time under field conditions due to irregular fuel distributions and climatic conditions. For this reason, several authors such as Sertsu and Sánchez (1978), Giovannini et al. (1990) and Soto (1993) developed laboratory heating programmes in order to record the influence of temperature on soil physical and chemical characteristics, or only on the physical ones (Ulery et al., 1993). Other studies analysed the effect of adding ashes on the soil nutrients (Ohno and Erich, 1990; Erich, 1991; Huang et al., 1992; Marcos et al., 1993–94) or even the effect of the heat–ash interaction (Sharrow and Wright, 1977; Raison and McGarity, 1980; Kutiel and Shaviv, 1989; Soto and Díaz-Fierros, 1993).

However, few of these heating programmes considered the time the heating remains, which can be considered the most damaging component of fire severity to soil (Robichaud et al., 2000). Heat duration not only has significant effects on the changes in soil characteristics (Wade and Lundsford, 1990), but also determines the germination rate of the seeds buried in the soil and therefore the regeneration capacity of the burned areas (Bradstock et al., 1992). The main objective of this study is thus

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to analyse the influence not only of the temperature but also its duration on the changes produced in the soil chemical characteristics and on aggregate stability.

2. Materials and methods

2.1. Soil sampling

The soil studied was a Humic Cambisol developed over siliceous conglomerates and located in Corcos (León, NW Spain). Soil was sandy loam with 8.5 g 100 g^{-1} of organic matter, pH 4.7 and available P about 0.16 mg kg^{-1}. The structural stability index (WSI) was 69.9, which was considered good (Marcos et al., 2000). The UTM coordinates are: 30TN284277. The area is situated at 1063 masl. The climate is subhumid Mediterranean with a mean annual temperature of 10 °C and a mean annual rainfall of 840 mm, with 732 mm annual evapotranspiration. The dominant species in the area were *Erica australis* L. subsp. *aragonensis* (Willk.) P. Cout., *Arctostaphylos uva-ursi* (L.) Sprengel and *Calluna vulgaris* (L.) Hull. Mean cover of herbaceous species was about 5%.

Mineral soil samples from a depth of 0–5 cm were collected in June, after removing the litter. The soil sample consisted of 40 subsamples taking at random, covering the study area (640 m^2) in order to avoid spatial variability.

2.2. Soil heating

Before heating, the sieved soil samples (<2 mm) were subjected to a differential thermal analysis (DTA) to learn the most characteristic reactions occurring in the soil when the temperature increases. The DTA is a technique based on measuring the thermal effects associated with the chemical or physical transformations which are checked in a sample during its heating. Simultaneous DTA and differential thermogravimetry (DTG) were performed on a Mettler Thermo Analyzer 1 using Pt–Pt Rh thermocouples: 1 g of soil was analysed to compare with calcined quartz sand as reference material. The apparatus was run at a constant heating rate of 10 °C min^{-1} under static air (Giovannini and Lucchesi, 1984). The final temperature was 1000 °C.

Once the reactions occurring in the soil were known, soil samples were heated in a muffle furnace. The soil (with a particle size <10 mm) was homogenised but not sieved before heating. The selected temperatures were 100 °C, 200 °C and 500 °C with heating times for each one of 5, 15, 30 and 60 min after reaching the selected heating temperature. To avoid a temperature gradient in the profile a sample layer of 2 cm depth was used. Each treatment consisted of three replications. No unheated samples were used as a control, because no significant differences were previously recorded between the unheated samples and those heated to 100 °C for 5 min (Marcos et al., 1993–94).

2.3. Soil analyses

After cooling, the samples were passed through a 2 mm sieve. The following properties were determined: pH in H_2O

(1:2.5); soil organic matter (SOM) using the Walkley and Black method (1934); total N by a semimicro-Kjeldahl procedure (Bremner and Mulvaney, 1982); and available P (Olsen et al., 1954); water-soluble Ca^{2+}, Mg^{2+}, K^+ and Na^+ were extracted from the soils with H_2O in a 1:5 soil: water ratio, allowed to stand for 24 h at room temperature with occasional shaking, and, after filtration, determined by atomic absorption spectrophotometry; and cation exchange capacity (Peech et al., 1947). Aggregate stability was measured by the modified method of Malqouri and Cecconi (Giovannini and Sequi, 1976). This method is based on a wet-sieving analyser which involved two immersions of the soil 2-1 mm fractions in water for 5 and 60 min and provides a water stability index (WSI).

2.4. Data analysis

To determine whether significant differences exist among treatments, the results were analysed using two-way (temperature and heating time) ANOVA and statistical significance determined at the $P < 0.05$ level. All treatments were compared by a principal component analysis (PCA), using STATISTICA 5.0.

3. Results

3.1. Differential thermal analysis

Thermal analysis of the soil studied (Fig. 1) shows the presence of two peaks. The first, which is endothermic, starts with heating the sample and ends at 177 °C. This peak is attributed to removal of hygroscopic water and loss of volatile substances. Between 229 °C and 579 °C there is a second, exothermic, peak which is due to decomposition and combustion reactions of organic matter. The mass loss of the sample corresponding to the temperature range, in which the main thermal reactions are produced, was calculated from the curve
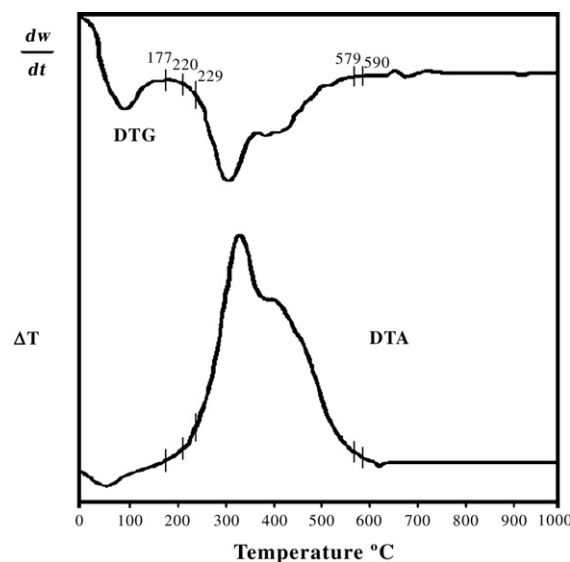

Fig. 1. Differential thermal analysis (DTA) and differential thermogravimetric (DTG) curves of soil sample before heating.
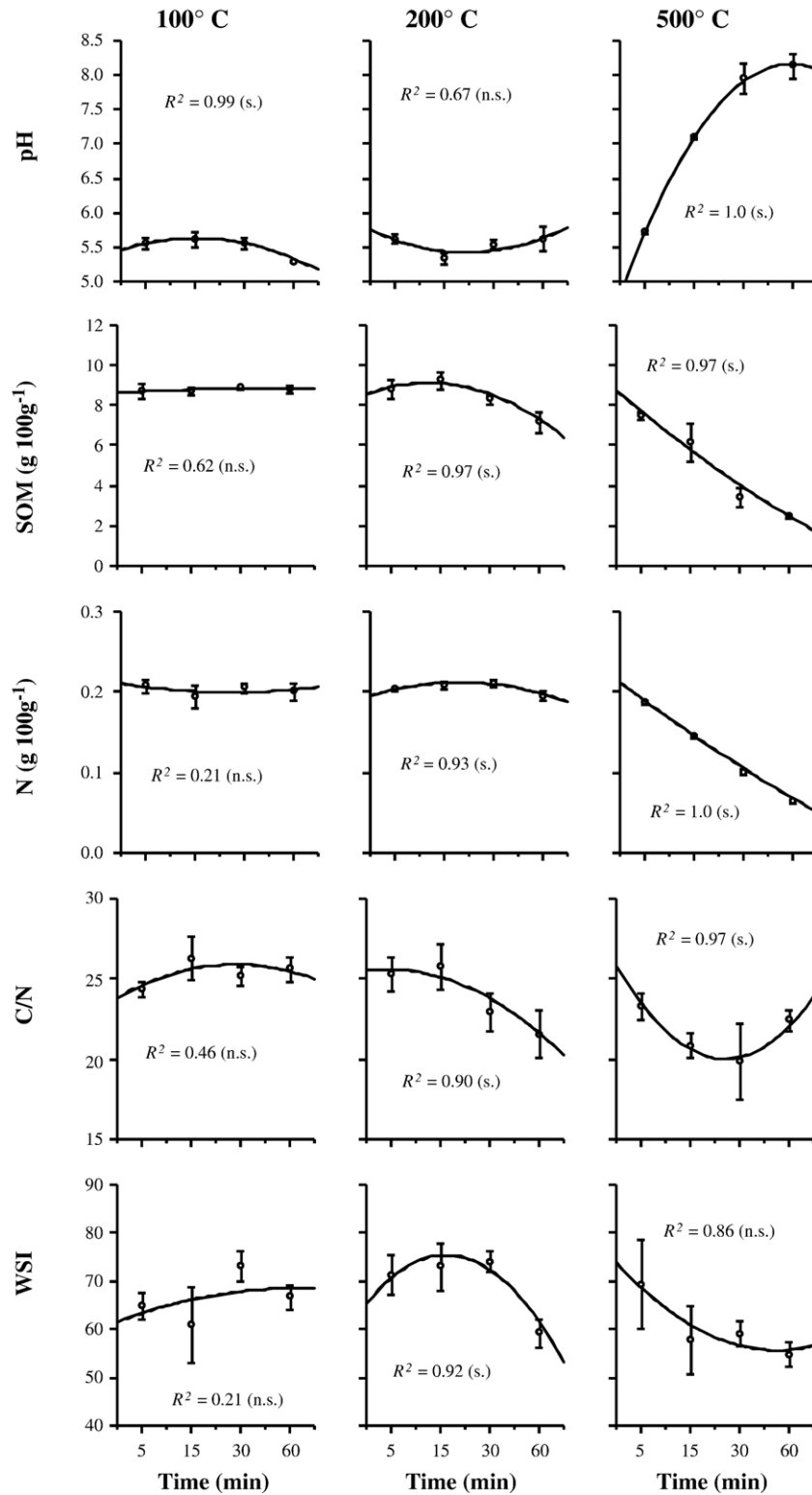
Fig. 2. Changes in pH, soil organic matter (SOM), total nitrogen, C/N ratio and water stability index (WSI) in relation with heating time. Mean values and standard error bars from the three replications of each treatment. Quadratic polynomial curve fitting is also included (s = significant fit, $P < 0.05$; n.s. = no significant fit, $P \geq 0.05$).

of the DTG analysis (Fig. 1). These occur between 279 °C and 578 °C with an 11.9% mass loss due to SOM combustion and between 578 °C and 950 °C with 0.1% loss. This mass loss was correlated with the exothermic region attributed to organic matter oxidation.

3.2. Soil heating

The pH of the soil shows a significant increase ($F = 64.3$; $P < 0.05$) starting with the 500 °C for 15 min treatment and reaching 2.6 units when the exposure time is 60 min, with a

significant polynomial curve fitting between pH and exposure time at 500 °C (Fig. 2). Soil heated to 200 °C for 60 min lost 17% of SOM ($F=4.4$; $P<0.05$), whilst the loss at 500 °C for 60 min is 72% of the total SOM ($F=19.4$; $P<0.05$). A significant negative correlation was detected between SOM content

and heating time at 500 °C. SOM loss was accompanied by a significant decrease ($F=351$; $P<0.05$) in total N, which occurs only at 500 °C. Therefore a decrease in the C to N ratio is to be expected; however, no clear tendency is observed when the treatments are applied, which may be due to the great sample
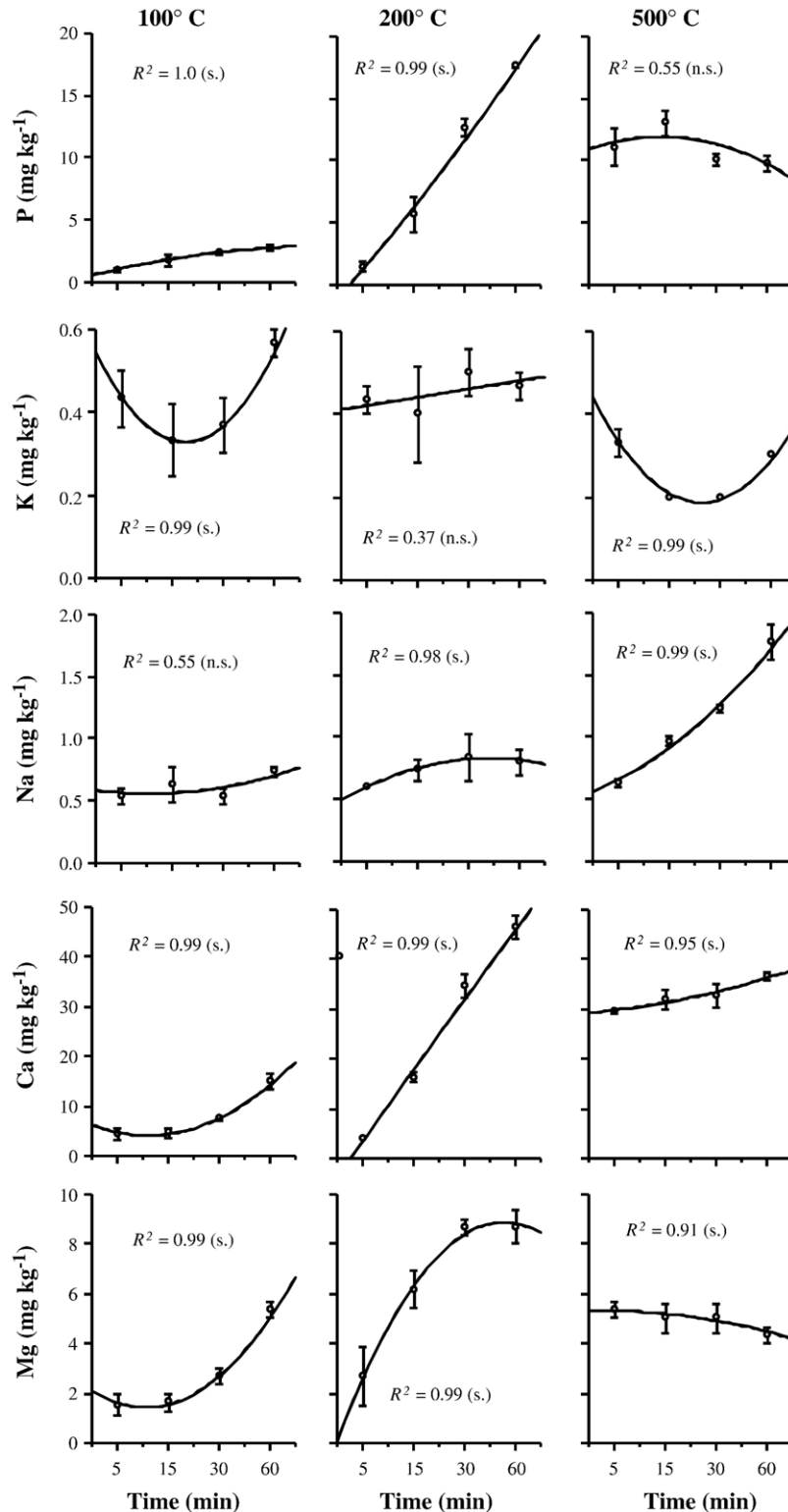

Fig. 3. Changes in available soil nutrients in relation with heating time. Mean values and standard error bars from the three replications of each treatment. Quadratic polynomial curve fitting is also included (s = significant fit, $P<0.05$; n.s. = no significant fit, $P\geq0.05$).

variability and in no case were significant differences detected among the treatments. No significant changes in the WSI were detected. Significant interaction ($P<0.05$) between temperature and heating time was detected for pH, SOM and total N.

The available P content increased significantly at 100 °C ($F=7.0$; $P<0.05$) but the increase was higher at 200 °C ($F=78.3$; $P<0.05$) without a clear tendency at 500 °C (Fig. 3). Soil heating has different effects in the soluble cations. No significant changes were recorded in the soluble K^+ content for any treatment. Soluble Na^+ increases significantly ($F=37.5$; $P<0.05$) at 500 °C. Soluble Ca^{2+} content increased slightly when the soil was heated to 100 °C ($F=21.3$; $P<0.05$), and very significantly at 200 °C ($F=115.1$; $P<0.05$). The increase was also significant for soluble Mg^{2+} at 100 °C ($F=28.2$; $P<0.05$) and 200 °C ($F=12.7$; $P<0.05$). Significant interaction ($P<0.05$) between temperature and heating time was detected for available P and soluble Na^+, Ca^{2+} and Mg^{2+}.

Whilst the different treatments were carried out, soil colour changed. At 200 °C for 60 min and 500 °C for 15 min the soil sample was completely black, whereas at 500 °C for 30 and

60 min a reddish surface layer is observed with the sample being black underneath this.

3.3. Global analysis of the treatments

Fig. 4 shows the PCA carried out for the set of treatments and the variables analysed: pH, organic matter, total N, C/N, CEC, WSI, available P and soluble cations. This analysis identifies three main temperature groups: the first would be represented by soils heated to 100 °C at the four heating times and those heated to 200 °C for 5 min. No appreciable changes in the soil occur in this temperature group and SOM content remains high as do the variables closely associated with it. The second group includes the soils heated to 200 °C for 15, 30 and 60 min and 500 °C for 5 min, where there was a considerable increase in available P and divalent cations. Finally, the third group would consist of the soils heated to 500 °C for 15, 30 and 60 min, where there was a clear increase in soluble Na^+ content and pH, in addition to a drastic decrease in SOM and total N.

4. Discussion and conclusions

4.1. Differential thermal analysis

The two peaks observed in DTA are in accordance with results recorded by other authors. The first, endothermic, peak is due to loss of the water as pointed out Grisi et al. (1998) and Gibbs et al. (2000). But the most interesting transformations in soil occur in the temperature interval from 229 °C to 579 °C, corresponding to SOM combustion, which is pointed out by one exothermic peak also observed by Mitchell and Birnie (1970) and Giovannini et al. (1990) using DTA and Salgado et al. (2004) using DSC. So Plante et al. (2005) also found in a DSC thermogram two exothermic peaks: a sharp one just below 300 °C and a second, broader peak at approximately 350 °C. As the C contents decreased across samples, the height of the first exothermic peak became discernible only as a shoulder to the second peak, similar to that found in the present study. Grisi et al. (1998) also found similar results in one of the studied soils. They explained these results by its very low clay content (8%) which, in turn, would result in less stabilization of organic C and relatively lower SOM. This could agree with our study because SOM content of samples ranged from 7.9 to 9.2 g 100 g^{-1}, which is low such as showed in the thermogravimetric curves, and clay content around 4.4%.

4.2. Soil heating

The highest temperature favours the increase in pH, which could be due to the formation of metal oxides leading to hydroxides and then to basic salts such as carbonates (Ulery et al., 1993; Giovannini, 1994). Fernández et al. (2001) detected an increase of 1 pH unit on heating the same type of soil at 350 °C for 1 h. Sertsu and Sánchez (1978) and Kutiel and Shaviv (1989, 1992) also observed considerable pH increases in other soil types heated to 400 °C and 600 °C. In the opposite, high temperatures decrease SOM content, as showed in the thermal analysis. Various
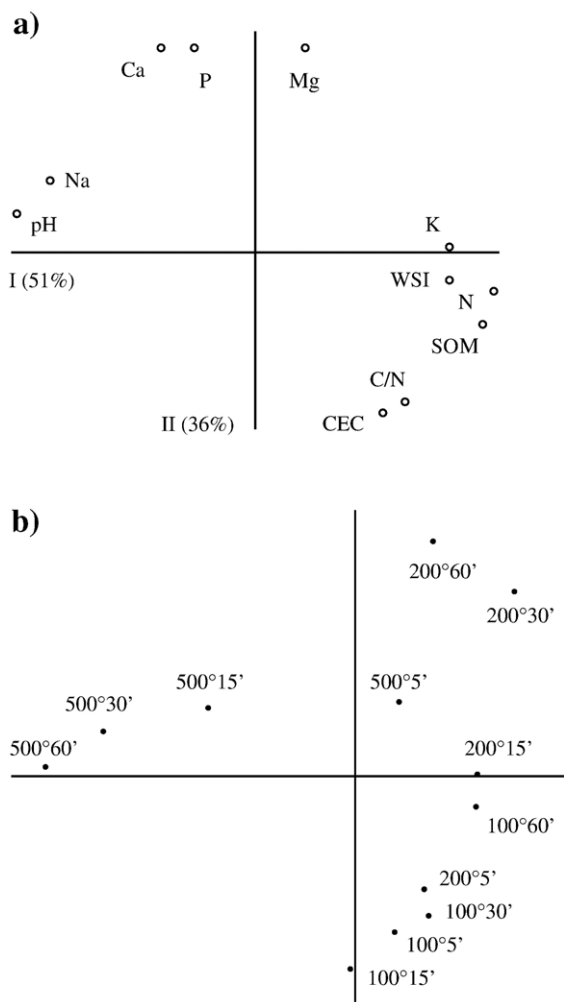

Fig. 4. Situation in the plane defined by the first two axes of the PCA. (a) Variables ordination. (SOM = soil organic matter, CEC = cation exchange capacity, WSI = water stability index). (b) Ordination of sampling data.

authors have recorded losses in the SOM content and total N as the temperature increased. Thus Sertsu and Sánchez (1978), Giovannini et al. (1990) and Fernández et al. (1997) also recorded that a little SOM decrease starts at 200 °C, as this is approximately its ignition temperature (Salgado et al., 1995), and an almost complete destruction at about 400 °C. Organic matter combustion results in a decrease in total N, which is volatilized from temperatures of 300°–400 °C, losing between 50% and 75% of its content (Dimitrakopoulos et al., 1994). The WSI decrease at 500 °C is probably due to the considerable SOM losses because a decrease in organic matter usually goes with a decrease in WSI (Giovannini and Lucchesi, 1984).

The different trends observed in available P depending on the temperature were also pointed out by other authors. Marcos et al. (1993–94) recorded a significant increase at 200 °C for 5 min, Kutiel and Shaviv (1989, 1992) also detected an increase at 250 °C for 60 min and Giovannini et al. (1990) reported an increase up to 460 °C and then a sharp decrease at higher temperatures. However, Sertsu and Sánchez (1978) observed an increase in available P with temperature increases. All these authors agree that the available P increase is due to the mineralization of organic P as a result of the heat applied and that the increase is proportional to SOM content. Heating the soil produces a different response in the water-soluble cations. The temperature range between 200 °C and 500 °C seems to favour an increase in divalent cations also reported by Giovannini et al. (1990) and Marcos et al. (1993–94) for Ca and Mg.

Colour change observed in the soil was explained by Ulery et al. (1993) that found, during an intense fire, the soil surface becomes reddish and then black underneath this layer. The red tone is due to the elimination of dark organic elements by combustion, with a 100% SOM decrease being recorded, whilst the black layer are due to the organic matter which remains. Sertsu and Sánchez (1978) reported that the soil acquires this reddish colour when subjected to 200 °C for 48 h.

4.3. Global analysis of the treatments

The changes produced in soil characteristics by heating depend on the temperature but also, and very significantly, on its duration. As stated by Giovannini (1994), the changes in soil properties really respond to the temperature according to a discrete step model. The changes occur at different temperature thresholds: temperatures up to 220 °C, from 220 °C to 460 °C, from 460 °C to 600 °C and beyond 600 °C.

Based on the results, this study establishes three temperature ranges and durations with different effects on the soil. On the one hand, temperatures of 100 °C or 200 °C at very short heating times do not produce significant changes in soil characteristics. The effects would be similar to those produced by a low intensity fire. Temperatures from 200 °C and from 15 min heating, or 500 °C with short heating times, increase available P and water-soluble cations, similar effects to those recorded in moderate intensity fires (Gimeno-García et al., 2004). Temperatures of 500 °C and heating times of over 15 min are harmful to the soil due to the loss of SOM and total N as well as to alteration of the physical characteristics of the soil. These

effects would be found in high intensity fires (Gimeno-García et al., 2004). In view of these results it can be stated that low temperatures (200 °C) at longer exposure times (30 and 60 min) produce similar effects on the soil as higher temperatures (500 °C) applied for a shorter time (5 min).

Heating in the laboratory gives us an approximate idea of how the temperature and its duration modify soil characteristics, which is fundamental to understand the effects of forest fire on soil. However, if these results are compared to those found for the same soil on which an experimental fire at known temperatures had been carried out (Marcos et al., 1998), differences can be observed, due to the fact that this laboratory work is only on mineral soil and therefore the added effect of burning vegetation is not taken into account. Although laboratory heating experiments help to widen knowledge of the effects of temperature, they must therefore be used with certain reservations when comparing them to forest fires in which the different factors interact in a complex way.

Acknowledgements

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