



Cementing agents involved in the macro- and microaggregation of a Mediterranean shrubland soil under laboratory heating



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ABSTRACT

The heating effect on aggregation and cementing agents of a Mediterranean soil was studied in two fractions (macro- and microaggregates) in different environments: under *Quercus coccifera* soil (UQ), under *Rosmarinus officinalis* soil (UR) and between plants or bare soil (BS). Samples were heated under laboratory conditions at different temperatures (220, 380, 500, 750 and 1000 °C) to establish their effects on the studied soil properties by comparison with unheated control samples (25 °C). Increasing temperature treatments caused significant changes in most soil variables. In both soil fractions, the presence of vegetation contributed to increase of the soil organic matter (SOM) content, which, in general, tended to decrease as temperature increased in all soils, reducing also their soil macroaggregate stability (SMS). Reduction in the stability of microaggregates (MSA) was not significant, possibly because the organic matter only controlled the stability of some of their fractions. Up to 500 °C, the mean weight diameter (MWD), calcium carbonate (CaCO₃) and extractable iron contents of macroaggregates presented different tendencies in all soils, but at 750 °C relative maxima were observed in these three parameters, indicating a possible relationship between the potential re-aggregation of medium size macroaggregates into larger ones, and the increasing CaCO₃ and extractable Fe contents. However, these newly formed “aggregates” would show low resistance to erosion as the decrease in SMS values suggested. The CaCO₃ and extractable Fe contents of microaggregates in all soils tended to increase up to 750 °C while their mean diameter (MMD) and stability hardly changed or tended to decrease. At 1000 °C, the cementing agents showed their lowest values in each soil, while the macroaggregate stability tended to increase, and the mean diameter and stability of microaggregates reached their maximum values. This fact could be due to the possible re-aggregation of small fractions into large ones and some “hardening” processes that could have occurred. In general, the macro- and microaggregate stability appeared to depend on the organic matter content in all studied soils but, particularly, in those under canopy. The mean size of both fractions seemed to be controlled mainly by the calcium carbonate and iron contents.

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1. Introduction

Different authors have investigated the effect of temperature on soil structure. The results of these studies vary depending on whether the research is conducted in the field, using experimental fires (Campo et al., 2008a,b; Mataix-Solera, 1999; Virto et al., 2007), or in the laboratory (Guerrero et al., 2001; Llovet et al., 2008; Zavala et al., 2010), by controlled heating programmes. The results of field studies indicate that increased temperatures may promote the stability of aggregates (Arcenegui et al., 2008; Llovet et al., 2009; Mataix-Solera and Doerr, 2004), produce a slight decrease (Badía and Martí, 2003; Jordán et al., 2011; Kavdir et al., 2005), or cause no change, as in the

case of moderate and low severity fires (Arcenegui et al., 2008; Varela et al., 2010). Some authors have also observed aggregate size reductions as a result of increased temperatures caused by fires (Benito et al., 2010; Cerdá et al., 2009), while others have stated that the soil aggregate size distribution is not affected (García-Corona et al., 2004; Virto et al., 2007).

Accordingly, the results vary widely because fire effects depend on the interaction among energy released, amount and degree of combustion of vegetation load, vegetation type, weather conditions during fire, topography, soil properties, extent of burned area, etc. (Certini, 2005; Robichaud et al., 2000). Since the effects of these factors are concomitant, and make it very difficult to identify individual causes of changes in soil properties, laboratory tests are commonly used to assess the effects of individual factors, such as temperature.

There have been many laboratory studies (Badía and Martí, 2003; Guerrero et al., 2001; Terefe et al., 2008; Zavala et al., 2010) about the

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changes caused by increasing temperatures on the aggregation and other soil properties. These researches have been mainly based on the studies carried out by Giovannini (1994) and Giovannini and Lucchesi (1997), which concluded that changes in soil properties respond according to a discrete step model. The most important changes occur at different temperature thresholds (Certini, 2005; DeBano, 2000; Doerr et al., 2000; Pardini et al., 2004; Six et al., 2004): (a) 25–100 °C: sample dehydration; (b) 170–220 °C: hydrophobicity increasing and dehydration of the gel forms; (c) 220–460 °C: combustion of organic matter, re-aggregation of particles into sand-sized particles and hydrophobicity disappearance; (d) 550–700 °C: loss of OH groups of clays, and (e) 700–900 °C: carbonate decomposition.

The effect of high temperatures in soils, which aggregation depends almost entirely on organic matter, produces the destabilization of aggregates (Cerdá et al., 2009; García-Corona et al., 2004; Giovannini, 1994; Mataix-Solera et al., 2011). However, some authors have also stated that changes caused by heating in soil aggregation may be due to variations in inorganic compounds like iron and aluminium oxides, or to carbonate decomposition (Andreu et al., 2001; Cerdá et al., 2009; Ketterings et al., 2000; Mataix-Solera et al., 2010). Changes in the content and mineralogy of the clay fraction, or even soil water repellency may also favour an aggregate stability increase (Blanco-Canqui et al., 2007; García-Corona et al., 2004; Mataix-Solera and Doerr, 2004).

Most of these results have been obtained only for the macroaggregates fraction ($\emptyset > 0.25$ mm) or the entire soil pool. However, it is well known that fires and high temperatures, can affect soil structure at both macroaggregate (Cerdá et al., 2009; García-Oliva et al., 1999) and microaggregate levels (Andreu et al., 2001; Mataix-Solera et al., 2002). Based on this, and on the fact that the role of iron oxides and carbonates in the aggregation of Mediterranean soils is rarely studied, this research aims: (a) to assess the impact of increasing temperatures, under a laboratory heating treatment, on the stability and mean diameter of the macro- and microaggregate fractions of a Mediterranean forest soil with different vegetation covers; (b) to study the effects of this heating treatment on organic matter, calcium carbonate, and extractable iron contents of the different size fractions and vegetation covers of the studied soil; and (c) to identify relationships between changes in the content of these cementing agents, organic and inorganic, and the changes undergone by the aggregation variables at the different heating temperatures.

2. Materials and methods

2.1. Study area and sampling

A Mediterranean forest soil located in La Concordia, Sierra La Calderona, (Valencia province, Eastern Spain: 39°45' N, 0°43' W) was selected for this study. The forested area, belonging to the coastal foothills of the Iberian Mountain System, is situated on a lightly concave hillside with SSE aspect, a slope of 22° and an altitude around 575 m a.s.l. It suffered a wildfire in 1978. The soil is a Rendzic Leptosol (FAO-UNESCO, 2006) with variable depth, smaller than 40 cm, developed on Jurassic limestone, with 45% gravel content and a sandy loam to silty loam texture.

La Concordia forest area is covered by Mediterranean vegetation dominated by *Pinus halepensis*, *Quercus rotundifolia* and shrubland with *Quercus coccifera*, *Rosmarinus officinalis*, *Ulex parviflorus*, *Rhamnus lycioides*, *Stipa tenacissima*, *Globularia alypum* and *Thymus vulgaris* as dominant species (Gimeno-García et al., 2007).

The climate of the area is Mediterranean (mean annual precipitation of 394 mm), with a maximum precipitation in autumn (52 mm in October) and a second, but less rainy period in spring (34 mm in April). The dry period (April–September), in which usually forest fires occur, presents a maximum temperature of 38 °C. The mean annual temperature is 17 °C.

Soil samples were taken in three environments: soil under *Quercus coccifera* (UQ), soil under *Rosmarinus officinalis* (UR), considered to be the most dominant species in the area (Gimeno-García et al., 2011), and on bare plant inter-spaces (bare soil: BS). Soil was sampled from the first 5 cm, in the summer of 2008, by taking three independent sub-samples within an area of approximately 20 m² (trees were at a distance of at least 80 cm), and both the litter layer (± 3 cm) as well as rock fragments > 1 cm were excluded and removed. The sub-samples from each environment were transported in sealed plastic containers, mixed together, homogenized, and next dried at room temperature and passed through a sieve with a 2 mm mesh (fine earth fraction).

2.2. Laboratory tests and analyses

The soil samples were heated in a rapid heating chamber furnace (Carbolite®) that allowed for heating steps of 3 °C min⁻¹, to prevent sudden combustion, and 2 min of heating at the selected temperature (Guerrero et al., 2001). Soil sample was placed in a porcelain flat dish, and a sample amount of less than 1 cm thickness was used to avoid a temperature gradient in the sample. Six heating steps were applied according to known thresholds in which significant changes can occur in both mineral and organic soil components: (a) 25 °C: control (room temperature), (b) 220 °C, (c) 380 °C, (d) 500 °C, (e) 750 °C and (f) 1000 °C. Once heated, samples were separated by gentle sieving in macroaggregates ($\emptyset > 0.25$ mm) and microaggregates ($\emptyset < 0.25$ mm) (Edward and Bremner, 1967).

Standard laboratory analyses were performed, in duplicate (2), to both soil macro- and microaggregates (2) for each of the three environments (3), and at six different temperatures (6) ($n = 72$ total). Soil organic matter content was determined using the oxidation method by potassium dichromate, following Walkley–Black procedure (Jackson, 1958). The factor used to convert C into organic matter was 1.724 (MAPA, 1986). SOM contents were related to the dry soil weight. Total carbonates were measured using the Bernard calcimeter method (MAPA, 1986). The extractable iron content determination was based on the dithionite–citrate–bicarbonate extraction of Mehra and Jackson (1960). To assess soil aggregate stability, a wet-sieving (0.25 mm mesh) procedure modified from the Henin and Feodoroff (1958) method was used (Primo-Yufer and Carrasco, 1973). The soil was sieved over a set of sieves with different mesh sizes, to separate the different size fractions, in which weight was used to calculate the mean weight diameter (MWD) index (Chaney and Swift, 1984). Hydrophobicity was determined by the water drop penetration time (WDPT) procedure (Letey, 1969) in two different fractions (< 2 mm and < 0.25 mm).

The study of the mean size and stability of microaggregates was conducted in a SediGraph 5100 of Micromeritics®, but only in the fraction of < 0.05 mm in agreement with Chan et al. (1994) and Naidu et al. (1996) to avoid obstruction in the measuring device. Initially, the sample was introduced in distilled water without applying any further energy. A first run with the SediGraph 5100 was performed obtaining a first particle size distribution. Afterward, the same sample was dispersed with sodium pyrophosphate 0.1 M (1 mL) and ultrasound at an energy level of 1800 J for one minute, in order to break existing bonds between the aggregated particles. From this treatment, a second size distribution of primary particles was obtained. The total difference between the particle size distributions (the one resulted from the first run and the one resulted from the second run) was used as an indicator of the microaggregate stability (MSA) similar to the method proposed by Cammeraat and Imeson (1998).

A General Linear Model (GLM) uni-variate procedure (95% significance level) was performed (SPSS 15®) to study possible differences in the soil characteristics with regard to environment, aggregate fractions and temperatures reached during heating. Standard statistical bivariate and partial correlation analyses were applied, at 95 and 99% significance levels, between the cementing agents (SOM,

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