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Speciation of organic matter in sandy soil size fractions as revealed by analytical pyrolysis (Py-GC/MS) and FT-IR spectroscopy

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This research deals with the assessment of organic matter structural differences in soil physical fractions before and after lipid extractions. Soil samples were collected in sandy soils, Arenosols (WRB 2006) from the Doñana National Park (SW Spain) under different vegetation cover: cork oak (Quercus suber, QS), eagle fern (Pteridium aquilinum, PA), pine (Pinus pinea, PP) and rockrose (Halimium halimifolium, HH). Two size fractions; coarse (C: 1-2 mm) and fine (F: 0.05-0.25 mm) were studied from each soil. . In addition, the two fractions from each soil were exhaustively Soxhlet extracted with a Dichlorometane-Methanol (3:1) mixture to obtain the lipid-free fractions (LF) from each size fraction (LFC and LFF).

The composition of the organic matter at a molecular level in the different soil fractions was approached by analytical pyrolysis (Py-GC/MS) and FT-IR spectroscopy. These techniques are complementary and have been found suitable for the structural characterization of complex organic matrices (Moldoveanu, 1998; Piccolo and Stevenson, 1982); whereas Py-GC/MS provides detailed structural information of individual compounds present and a finger-printing of soil organic matter, FT-IR is informative about major functional groups present. The advantages of these techniques are well known: no need for pretreatment are fast to perform, highly reproducible and only small amount of samples are needed.

Soil size fractions show contrasting differences in organic matter content (C 4-7 % and F > 40 %) and conspicuous differences were found in the pyrolysis products released by the fractions studied. The main families of pyrolysis compounds have well defined macromolecular precursors, such as lignin, polypeptides, polysaccharides and lipids (González-Vila et al., 2001).

The C fractions yield higher relative abundance of lignin and polysaccharide derived pyrolysis compounds. Regarding the differences in the soil organic matter as affected by the different vegetation covers, the C fraction from the PA soil presented a higher abundance of lignin derived pyrolysis products than the soils under the other vegetation. This is somehow unexpected since PA is a pteridophyte, not arboreal vegetation, i.e. low lignin content and, these lignin moieties probably remain in the soil from past vegetation or originate from surrounding woody arboreal vegetation. In contrast the F fractions released mainly lipids and aromatic compound of unspecific origin. Series of alkane/alkene pairs were present in all the pyrograms with varying abundance and composition. Lignin and polysaccharide derived pyrolysis compounds were scarce in the F fractions in all the cases, in fact, no sugar derived compounds were found in the HH sample.

Regarding the composition of the LF soil fractions, the pyrolytic behavior of the LFC fractions was quite similar to the not extracted corresponding C soil fraction, showing a high proportion of lignin and sugar derived pyrolysis compounds. The LFF fractions also showed the same behavior as the C fraction, but with no lipid derived compounds which effectively indicates the occurrence of a selective and efficient removal of soil free lipids.

Agreement was found between analytical pyrolysis results and FT-IR spectral features highlighting functional differences between fractions i.e. a decrease of OH- groups and an increase in aliphatics in the F fraction. With respect to the LF fractions, FT-IR spectra analysis was also consistent with the pyrolysis results with a slight increase in the lignin signals for LFF soil fractions under PA, PP and HH. For the soil under QS no differences were found between the LFF fractions and the whole organic matter in the F fraction, probably due to the high amount of organic matter in this fraction.

In conclusion, despite the “a priori” low organic complexity of the collection of soils studied here, ostensible differences were found in the organic matter present in C and F soil size fractions under different vegetation covers, and not only in its intimate chemical composition, but also in its functionality. Whereas the C soil size fractions are composed mainly by relatively well recognized lignocellulosic plant residues, the F soil size fractions have a clearly distinct type of organic matter, more mature/evolved that clearly resembles the characteristics of customary wet extracted humic materials as characterized elsewhere (Stevenson, 1994; González-Pérez et al., 2013).

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