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Speciation of lipid and humic fractions in soils under pine and eucalyptus forest in northwest Spain and its effect on water repellency

Esther de Blas [a,*], María Rodríguez-Alleres [a], Gonzalo Almendros [b]

[a] Departamento de Biología Vegetal y Ciencia del Suelo, Universidad de Vigo, E-36310, Vigo, Spain
[b] Centro de Ciencias Medioambientales, CSIC, Serrano 115 B, E-28006, Madrid, Spain

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ABSTRACT

Soil organic matter characteristics in extremely water-repellent soils developed under forests of *Pinus pinaster* and *Eucalyptus globulus* in Galicia (Northwestern Spain) were analyzed with special emphasis paid in lipid and humic acid fractions. A total of sixteen soils were studied including Leptic Regosols and Leptic Umbrisols developed on granites and schists, and showing extreme water repellency: up to 6 h using water drop penetration time (WDPT) test, i.e., ranging from strongly to extremely hydrophobic (ethanol percentage test, MED). The experimental design involved the measurement of the water repellency as WDPT after the successive removal of lipid and humic fractions by: (i) direct extraction of the soil free lipid with petroleum ether (40–60 °C), (ii) extraction of the 'fixed' lipid from the soil residue after 2M H_3PO_4 treatment and further recovery from the aqueous phase with petroleum ether and (iii) final extraction of humic substances with alkaline solutions. The results showed significant decrease in the WDPT (from class 6 to classes 4 or 5) after removing free lipid. Nevertheless, removal of 'fixed' lipid resulted into the most substantial decrease of the WDPT, which occurred on almost all soils (to classes 0–3). This fact is to large extent associated to the simultaneous removal of hydrophobic particulate fractions (free organic matter) which — even after extraction of free lipid — resulted extremely water-repellent in laboratory conditions (>1 h WDPT). Finally, the extraction of humic and fulvic acids was required for the total disappearance of the water repellency (class 0). Regarding vegetation types, lipid removal (free + 'fixed' fractions) was significantly more effective in increasing wettability in the case of forest soil samples under pine than under eucalypt. Concerning geological substrate, water repellency in soils under granites remained comparatively more persistent than in soils under schists. After treatment with 2M H_3PO_4, all samples from soils on granite–eucalyptus remained slightly water-repellent, whereas all samples from soils developed on schist–pine samples became wettable. Multivariate data treatments (multiple regression models, variable ordination by multidimensional scaling, and discriminant analysis) were useful to identify the soil characteristics most significantly associated to its water repellency. These treatments suggest that water repellency in the soils under study is a complex emergent property, reflected in specific patterns depending on vegetation type and geological substrate and arising from the interaction between different soil organizational levels (mainly free lipid, 'fixed' lipid, macroscopic particulate organic matter and the concentration and maturity of humic substances).

1. Introduction

Occurrence of soil water repellency has been reported worldwide from different climatic conditions, soil types and vegetation covers (Wallis and Horne, 1992; DeBano, 2000; Jaramillo et al., 2000; Doerr et al., 2000). Despite it is a property with major repercussions for plant growth, surface and subsurface hydrology, and for soil erosion the precise reasons for this phenomenon remain largely unknown. In particular, considerable experimental effort has been carried out in the last decade to identify specific substances with a potential bearing on water repellency (e.g., Walis et al., 1993; McIntosh and Horne, 1994; Hudson et al., 1994; Franco et al., 1994, 1995; Doerr et al., 2005). Classical literature have emphasized the importance of lipid fractions released to soil by plants or microorganisms (fungi), as well as the bearing of specific characteristics of the organic matter, in general associated to moisture regimes, e.g., temporarily waterlogged soils (Fridland, 1982). However, despite the current advances in instrumental techniques for analytical chemistry, explaining the relationship between water repellency and the accumulation of specific substances in soil is far to be established. It has been postulated that soil water repellency is the result of complex organo-mineral interactions where not only the characteristics of the organic matter fractions but also the association patterns — speciation status — of the lipid and humic fractions, as well as the molecular composition of these fractions, could play a substantial role.

Some studies such as those by Ma'shum et al. (1988), Roy et al. (1999), Horne and McIntosh (2000), and Doerr et al. (2005), were

* Corresponding author. Tel.: +34 986812395.
E-mail address: eblas@uvigo.es (E. de Blas).

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concerned in comparing the efficiency of different solvents in achieving total removal of soil water repellency after single-step extraction. This should lead to identify the best solvent for extracting the hydrophobic organic materials considered as responsible for the water repellency in soils. The authors following such an approach (Doerr et al., 2005) aim to isolate most of the hydrophobic material to be further analyzed in order to identify molecules inducing water repellency to the soil. Other authors (Franco et al., 2000) applied eluotropic series of solvents for the sequential removal of progressively polar hydrophobic materials from soil.

Nevertheless, in this study special emphasis has been paid to the role of the speciation status of organic hydrophobic fractions in forest soils of Galicia (NW of Spain). The soils were developed under the same wet–temperate climate but with contrasting vegetation and geological substrate. In these soils, a sequential extraction of different organic fractions, progressively more associated to the clay–humus complex, was carried out in order to determine the remaining water repellency in the soil residues after removing the different organic fractions. For this purpose, free lipids (i.e., substances presumably forming hydrophobic coatings on soil aggregates) were extracted, and the residue was treated with 2M H_3PO_4 with the double purpose of removing particulate organic matter (light organic fraction) and releasing "fixed" lipid (presumably associated to soil matrix by physical or weak chemical interactions). In a final step, the humic substances were isolated with alkaline extractions. According to this approach, it could be postulated that the speciation status of the hydrophobic components sequentially removed from soils would inform not only on the total extent but also about the persistence after wetting of water repellency in soils. For this purpose, the differences between soils where hydrophobic coatings of soil particles are of a recent formation in soils, from those where water repellency has been acquired by the soil organ–mineral matrix, were compared. Conclusions would be inferred from the quantitative effect of the sequential treatments in order to reduce soil water repellency, its persistence, and its different behaviour in terms of forest type and geological substrate.

2. Materials and methods

2.1. Soil sampling

Sixteen soil samples were collected from the two Western Provinces of Galicia (NW of Spain) aiming to minimize the effect of climatic variability, selecting representative geological materials: granites, which are associated to soils with sandy loam texture, and biotite-rich schists, more frequent in soils with loam texture. In each sampling site, soil samples were collected under two forest vegetations (*Pinus pinaster* and *Eucalyptus globulus*), which in the wet–temperate climatic conditions of Galicia lead to soils with high levels of soil organic matter.

The samples belong with extremely repellent topsoils (up to 6 h Water Drop Penetration Time, WDPT) from Leptic Regosols and Leptic Umbrisols (WRB, 2006). The main characteristics of the soil samples are shown in Table 1.

After litter removal, surface (0–5 cm) soil samples (at about 1 kg) were randomly collected with a spade and combined to obtain composite samples for further analyses., The sampling campaigns were carried out in July 2003 after a prolonged dry period in order to reduce the influence of the moisture content in the water repellency of the samples (Rodríguez-Alleres et al., 2007a).

2.2. Soil general analyses

Samples were air-dried at room temperature and sieved to 2 mm to obtain the fine earth fraction. Total C and N were determined in the fine fraction with an elemental analyser; total carbon values coincide with those of organic carbon due to the lack of carbonates in the soils. Particle-size distribution was determined after oxidizing the organic matter with H_2O_2, then sieving the sand fractions and using the pipette method to separate silt (50–2 μm) from clay (<2 μm) fractions (USDA, 2004).

2.3. Determination of soil water repellency

The WDPT test was used to assess the persistence of water repellency both on the initial soil samples and on the extraction residues. This test, described by several researchers (e.g. Van't Woudt, 1959; Letey, 1969; Dekker and Jungerius, 1990; Doerr, 1998) involves placing drops of distilled water (approximately 50 μL) on the surface of the soil samples and recording the time for complete droplet penetration. An amount of about 5 g of each soil sample was placed (in triplicate) in 70-mm diameter Petri dishes and 5 distilled water drops were applied on each plate. Seven classes of repellency were distinguished according to the time required for infiltration drops (Bisdom et al., 1993): class 0, wettable or non-water-repellent (WDPT

Table 1
Main characteristics of the studied soils.

Soil no.	Location[a]	Geological substrate	Vegetation	Geographic coordinates	Soil OC[b]	C/N	Sand[c]	Clay[d]	Texture[e]
1	Fontáns	Granite	Pine	42°28′54″N/08°53′39″W	14.4	31	63	16	SL
2	Fontáns	Granite	Pine		11.8	25	63	16	SL
3	Caldas	Granite	Pine	42°36′58″N/08°38′40″W	8.1	20	67	11	SL
4	Mondaríz	Granite	Pine	42°13′23″N/08°28′05″W	9.5	20	68	14	SL
9	Órdes	Schist	Pine	43°04′52″N/08°22′06″W	13.0	18	32	26	L
10	Órdes	Schist	Pine		14.8	21	32	26	L
11	Arzúa	Schist	Pine	42°55′20″N/08°12′09″W	11.4	21	50	20	L
12	Sigüeiro	Schist	Pine	42°56′33″N/08°26′36″W	11.2	20	53	20	SCL/SL
5	Fontáns	Granite	Eucalypt	42°28′54″N/08°53′39″W	13.6	25	64	16	SL
6	Fontáns	Granite	Eucalypt		11.7	21	64	16	SL
7	Caldas	Granite	Eucalypt	42°36′58″N/08°38′40″W	10.6	24	75	11	SL
8	Mondaríz	Granite	Eucalypt	42°13′23″N/08°28′05″W	10.9	25	72	12	SL
13	Órdes	Schist	Eucalypt	43°04′52″N/08°22′06″W	8.3	15	39	25	L
14	Órdes	Schist	Eucalypt		8.0	15	39	25	L
15	Arzúa	Schist	Eucalypt	42°55′20″N/08°12′09″W	9.7	13	44	19	L
16	Sigüeiro	Schist	Eucalypt	42°56′33″N/08°26′36″W	10.7	17	53	20	SCL/SL

[a] Northwestern Spain.
[b] (g OC 100 g^{-1} soil).
[c] (2–0.05 mm) (g 100 g^{-1}).
[d] (<2 mm) (g 100 g^{-1}).
[e] L = loam; SL = sandy loam; SCL = sandy clay loam.