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Ninon Delcourt, Anne Marie Farnet da Silva, Catherine Rébufa, Lisa Foli, Nathalie Dupuy. Land use legacy footprint in Mediterranean forest soils: An infrared spectroscopy approach. *Geoderma*, 2023, 430, pp.116299. 10.1016/j.geoderma.2022.116299 . hal-03990704

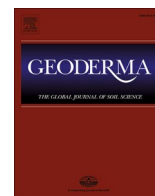
HAL Id: hal-03990704

<https://hal.science/hal-03990704v1>

Submitted on 15 Feb 2023

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Land use legacy footprint in Mediterranean forest soils: An infrared spectroscopy approach

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ARTICLE INFO

Handling Editor: Karen Vancampenhout

Keywords:

Agricultural abandonment
Mediterranean climate
Soil chemical properties
AComDim
Afforestation

ABSTRACT

Chemical properties of forest soils can strongly influence this compartment vulnerability to climate change effects. Past human activities can play a major role in structuring soil chemical properties. In the Mediterranean region, abandonment of terrace agriculture since 1860 induced the coexistence of forests with different ages. Here, soil chemical signature (SCS) was compared between very recent, recent and ancient forests using Fourier Transform InfraRed (FTIR) spectroscopy and other chemical properties (total C and N, CaCO₃ percentages). Land Use Legacy (LUL) effects were analysed depending on soil depths and on contrasted climate conditions (humid vs subhumid Mediterranean climates). Statistic treatments, Linear Mixed-Effect and Anova COMMon DIMension models, were used to highlight the influence of depth's soil, climate and LUL effects. Interestingly, the soil depth did not influence LUL effect on soil spectra. The effect of climate, LUL and their interactions were observed on the SCS. Higher available N was observed in soils from ancient forests than in soils from very recent forests. Moreover, mineral fraction discriminated soils with different LUL (very recent vs recent and ancient forests) and this depended on climate. Under subhumid climate, soils from very recent forests were indeed negatively correlated with the mineral fraction, while under humid climate, they were positively correlated to it. Finally, a recovery of soils chemical properties was observed in recent forests under humid climate, while under subhumid climate, SCS of post agricultural forests was different from that of ancient forests.

1. Introduction

In the context of global environmental change, understanding and forecasting the impact of human activities on terrestrial ecosystems is one of the biggest challenges in environmental sciences (Peñuelas et al., 2017; Morán-Ordóñez et al., 2020). Previous studies have shown that past human activities, notably agricultural activities, have strongly impacted current soil properties and functioning of ecosystems (de la Peña et al., 2016; Krause et al., 2016; Raciti et al., 2011; Zornoza et al., 2009). Considering the great diversity of soil types, the influence of land use on ecological functions varies both spatially and temporally. Indeed, the vulnerability of soils to degradation is not constant for a given landscape and can greatly vary according to the physical characteristics of each soil type (Foster et al., 2003). Interactions have been demonstrated between time, land management and land use change (Falkengren-Grerup et al., 2006) and a comparison between actual and reference areas is necessary to provide a valuable insight into the land use effect (Devátý et al., 2019).

The long history of intense cultivation and a combination of relief, parent material and climate conditions make Mediterranean soils and soil patterns very different from those in other regions in the world. Mediterranean climate stress (high temperature, drought) change soil properties by increasing content of recalcitrant compounds in plant tissue (e.g lignin) and thus in soil composition (Suseela and Tharayil, 2018). Humidity and temperature also influence microorganisms' activities involved in organic matter transformation that ultimately affect soils chemical properties (Allison and Treseder, 2008; Borsali et al., 2021; Kheir et al., 2021). Consequently, in the Mediterranean basin, current soil-loss rates drastically exceed soil formation rates (Raclot et al., 2006).

Mediterranean forests are often confined on superficial soils: the rock is close to the surface and forms part of the substrate exploited by the roots. The Mediterranean climate context and long-lasting human activities lead to specific characteristics of forest soils: low thickness, high load of coarse elements, poorly differentiated horizons and low contents in organic matter (Quézel and Médail, 2003; Verheyne and de la Rosa,

Abbreviations: SCS, soil chemical signature; FTIR, Fourier Transform InfraRed; LUL, land use legacy; AComDim, Anova COMMon DIMension.

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<https://doi.org/10.1016/j.geoderma.2022.116299>

Received 12 March 2021; Received in revised form 19 July 2022; Accepted 29 November 2022

Available online 10 December 2022

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2006). Moreover, farming practices and land development (terraces, stone removal, ploughing, manuring and grazing) have durably modified the environment. Finally, though Mediterranean forests have been described as a hotspot of forest species and genetic diversities, they are also considered as heavily degraded areas due to local agro-sylvo-pastoral practices and to local managements leading to deforestation, forest degradation and desertification (Gauquelin et al., 2018; FAO and Plan Bleu. 2018).

Among the few studies related to the effect of land-use history on Mediterranean forest soils, it was shown that (i) quantitative changes in soil organic matter (lipids and humic acid fractions) between undisturbed Mediterranean forests and neighbouring deforested sites (Tinoco et al., 2010), (ii) vegetation thinning treatments, more frequent in Mediterranean forests, modified physical and microbiological properties in comparison with soil quality of unmanaged and undisturbed forest areas (Andrés-Abellán et al., 2019; Lull et al., 2020; Wic Baena et al., 2013), (iii) different land uses and agricultural practices were discriminated by the aromatic and carbonate compounds in the soils (Delcourt et al., 2019; Du et al., 2008; Mendham et al., 2002), (iv) soils keep, at different depths, the marks of past uses in their chemical composition and their spectral signature appears like an “historical footprint” of their natural or anthropogenic conversion over time (Ertlen et al., 2015; Vysloužilová et al., 2015), (v) different chemical signatures of forest soils were observed under different bioclimatic stages (Delcourt et al., 2019).

Differences of soil physico-chemical properties due to agricultural activities can endure for many years even after use, but few studies have used spectral technique to follow recolonization by native vegetation of abandoned farmed sites, though spectroscopy was recognized to be a promising analytical method for investigating soil sciences (Tinti et al., 2015; Aranda et al., 2014). Spectroscopy was used to detect changes in soil quality, plant cover and to predict soil classification according to different land-use categories (Delcourt et al., 2019; Ertlen et al., 2015, 2010; Paz-Kagan et al., 2014; Du and Zhou 2009).

In this study, we investigated how legacy effects of past agricultural management affected the chemical composition of Mediterranean forest soils. We have chosen a French eastern Mediterranean region, the Parc Naturel Régional du Luberon (PNRL), with forests (*Q. pubescens* as dominant species) at different ages and past land use history (ancient, recent and very recent forests) under two bioclimatic stages (humid and subhumid) (Abadie et al., 2017). Three main questions were addressed in this work: (i) How land use legacy (LUL) effect influences the soil chemical signatures (SCS) depending on soil depth? (ii) Does LUL effect vary depending on the date of land abandonment (recent or very recent forests)? (iii) Does climate influence LUL effect on soil properties? To identify the influence of these environmental factors and of their interactions on soil chemical composition, an Anova COMMON DIMENSION (AcomDim) approach and Linear Mixed Effect (LME) models were performed on soil infrared spectroscopic signatures and individual physico-chemical properties respectively.

2. Material and methods

2.1. Site description and soil sampling

The study was conducted in the Parc Naturel Régional du Luberon (PNRL), located in South-eastern Mediterranean France (Provence Alpes Côte d’Azur region). This regional parkland straddles Vaucluse and Alpes de Haute-Provence (43°39’N–44°02’N, 4°58’E–5°55’E) and covers 195,413 ha (Supplementary material Fig. S1). The study area was chosen for the extensive information on climate and forest stands available from the previous study by Abadie et al. (2017), which identified land use and cover since 1830. Pedoclimatic conditions are typically Mediterranean, characterised by humid winter and intense summer drought, and the soil is mainly calcareous. The most frequent soil types under the forest are carbonate soils, covering 65 % of the production forest area

(Calcosols-Rendosols) and of the remaining soils, 19 % are Calcosols-Rendosols. (WRB IUSS Working Group, 2006). All cartographic data (stands, climate, LUL...) were treated with ArcGIS software (10.3).

(i) Climate

Two distinct bioclimates were considered (Table 1): subhumid and humid, corresponding to elevation-driven arrangements of vegetation type *i.e.* Meso Mediterranean and Supra Mediterranean levels respectively (Quézel and Médail, 2003). While several numerical indices have been proposed to measure aridity (Stadler, 2005), annual precipitation remains the simplest aridity index used by the Intergovernmental Panel on Climate Change (IPCC, 2007; Maliva and Missimer, 2012): 772 mm of annual precipitation in humid climate vs 695 mm in subhumid climate confirms the latter’s drier and warmer conditions.

(ii) Forest stand.

As described by Abadie et al. (2018), 55 % of the area is covered by forests containing three main dominant species: holm oak (*Quercus ilex* L.), downy oak (*Quercus pubescens*) and Aleppo pine (*Pinus halepensis* Miller). Most of the forests do not receive any specific silvicultural treatments. In this study, only forest patches with *Q. pubescens* as dominant species were selected since they were equally represented in the two categories of climate and historical land use.

(iii) Land use legacy.

Based on Abadie et al. (2018, 2020), two historical sources were used to assess past land use for each forest patch:

- 1860: The “État-Major” map (EM map; 1858–1861 in the PNRL; 1:40,000), digitised by Salvaudon et al., (2012) following Favre et al., (2013), with a median position error of 26 m after georeferencing and correction;
- 1958: Historical aerial photographs taken between 1953 and 1958, photo-interpreted for each vegetation plot.

Forests were classified as ancient (A) if present in 1860 and 1958; as recent (R) if they developed between 1860 and 1958 and as very recent (VR) if they developed after 1958. Following Abadie et al., (2020), we considered that no turnover in land cover occurred between the dates of the two maps *i.e.* the “État-Major” map 1860 and the historical aerial photographs (1958).

Additionally, the recent and very recent forests selected were characterised by visible terrace remnants attesting to the presence of agriculture in the past. Terraces were most probably used for olive growing, olive trees traditionally being the main form of terrace cultivation in the French Mediterranean region (de Réparaz, 2000).

For each climate (subhumid and humid), 5 pairs of A and R forests and 5 pairs of A and VR forests were selected based on their geographical proximity. This meant that each post-agricultural forest could be linked to an ancient forest taken as a reference model. Thus, a total of 40 forest patches of a minimum area of two hectares were explored (Supplementary material Fig. S1). These plots were considered to be true replicates as the distance between them exceeded 2 km.

Table 1

Bioclimatic variables from Wordclim data (<https://www.worldclim.org>) calculated using temperature and rainfall values (data for 1970–2000) for both climates considered (humid and subhumid).

	Subhumid	Humid
Annual precipitation (mm)	695	772
Precipitation of driest month (mm)	25	32
Precipitation of wettest month (mm)	86	88
Annual mean temperature (°C)	12	11
Min temperature of coldest month (°C) ^a	0	−1
Max temperature of warmest month (°C) ^b	27	25
Temperature annual range (a - b) (°C)	27	26
Isothermality (%)	35	35

Isothermality: (Mean of monthly (max temp - min temp))/ temperature annual range × 100.

(iv) Soil sampling.

A total of 40 forest patches were selected and soils were collected in February 2019. For each plot, 4 trenches of about 30 cm-deep were dug. In each trench, 3 samples were collected following 3 depths: 0–10 cm, 10–20 cm, 20–30 cm for a total of 480 soil samples. Before the chemical analyses, each sample was sieved to 2 mm and ambient air-dried for 7 days. Residual debris (roots, leaf) and stones (>1mm) were removed. Then, soil sample were ground to obtain a powder using a bullet blender Retsch MM40 (Fisher Scientific, France) and powders were dried again at 40 °C for 72 h. Moreover, from these 480 powdered soils, aliquots of sample coming from the same forest patches and collected at the same depth were pooled to give 120 composite soil samples.

2.2. Chemical characterization of soils by FTIR-ATR (Fourier Transformed infrared – Attenuated total Reflectance)

The 480 powdered soils were directly deposited on the attenuated total reflectance (ATR) accessory (Bruker “Golden Gate”) equipped with a diamond crystal prism (brazed in only one tungsten carbide part), four mirrors and two ZnSe focusing lenses in to reflect the optical path. Soil sample was pressed on the crystal area with the pressure arm and positioned over the crystal/sample area. The recording of FTIR-ATR spectra was performed with a Thermo Nicolet IS10 spectrometer equipped with a MCT detector, an Ever-Glo source and a KBr/Ge beam-splitter, at room temperature. Data acquisition was done in absorbance mode from 4000 to 650 cm^{-1} with a 4 cm^{-1} nominal resolution. For each spectrum, 100 scans were co-added. A background scan in air (in the same resolution and scanning conditions used for the samples) was carried out before the acquisition. The ATR crystal was carefully cleaned with ethanol to remove any residual trace of the previous sample. Three spectra were recorded for each sample. Spectral zone between 4000 and 1800 cm^{-1} was withdrawn to eliminate the broad band representative of O–H, N–H vibrations (4000–3000 cm^{-1}) and the absorption bands of CO_2 and diamond (2400–1800 cm^{-1}). For each sample, the three spectra recorded were average and then, spectra of samples coming from the same forest patch and collected at the same depth were average to obtain 120 soil spectra. Before AComDim treatment, a derivation of spectral data (Savitsky-Golay first-derivate, 3 points smoothing) was used to reduce noise and baseline.

2.3. Additional soil chemical measurements

For each of the 120 composite samples, total C (%) and N (%) contents (respectively C_{tot} and N_{tot}) and their ratio (C/N) were measured using a C/N elementary analyzer (Flash EA 1112 Thermo Scientific series). Determination of calcium carbonate (CaCO_3 (%)) in the 120 composite samples was based on the volumetric analysis of carbon dioxide CO_2 , which was released during the application of hydrochloric acid solution HCl (6 mol.L^{-1}). It was performed using a calcimeter (FOGII Digital Soil Calcimeter). Carbon proportion of CaCO_3 was then calculated using the molar mass of C (Eq. 1). Then organic carbon (C_{org} (%)) was calculated by the difference between total C_{tot} content and C_{CaCO_3} content (Eq. 2).

$$C_{\text{CaCO}_3}(\%) = \frac{12}{100} \times \text{CaCO}_3(\%) \quad (1)$$

$$C_{\text{org}}(\%) = C_{\text{tot}}(\%) - C_{\text{CaCO}_3}(\%) \quad (2)$$

2.4. Statistical analysis

Two different statistical approaches were used according to the type of data. AComDim treatment performed on spectral data allowed to access the identification and assignation of IR bands without the unit of their intensity (arbitrary unit) being an obstacle to the spectral interpretation. Nevertheless, to analyse the value of chemical data and not

those mathematically transformed as when AComDim was used, a LME model was preferred to compare the influence of studied factors using the intact experimental values of chemical data. The advantage of these tools was to retain all the variance in the data for each factor or interaction studied before prioritising them. Particularly, AComDim is a novel and interesting approach to contribute to the recognition of land use history and its legacies and to understand its interconnectedness with climate and to test whether any resilience of Mediterranean forest soils to agricultural practices occurred.

• Anova COMmon DIMension

The AComDim method, a multi-block analysis, was used to examine the variance of spectral data and find if studied factors (LUL, depth and climate) influenced soil spectral profile. The outputs of the AComDim method indicate whether variations in the data from different values relative to the change between two levels of a factor are significantly greater than the residual variability, and thus meaningful. This method is based on the same concept as ANOVA-PCA (also called APCA), and its description can be found in the papers by Rébua et al. (2021) and Bouveresse et al. (2011). The AComDim method decomposes the spectral data matrix into successive matrices (also called mean matrices) containing the average at each level for each factor or interaction. The residual matrix remaining after successive subtractions of all mean matrices is added back to each of them to obtain means plus residual matrices (called blocks). Then, a multi-block PCA analysis of all matrices is performed to extract its first principal component, or “Common Components” (CCs). Each block provides a specific contribution (*i.e* a specific weight), called salience, to the definition of each common component. Since all blocks contain a contribution from the residual matrix, the first Common Component CC1 (with higher saliences) contains mainly noise. The block significance is afterward estimated with a Fisher test (student Fisher *F*-test) applied on the *F*-values (F_i) calculated as per Equation 3:

$$F_i = \frac{\lambda_{\text{res}}}{\lambda_i} \quad (3)$$

where λ_{res} is the salience of the residual block on CC1 and λ_i is the salience of the i^{th} block on CC1. The *F*-test is performed by selecting $n - 1$ degrees of freedom, where n is the number of blocks. The significance level, so-called alpha level, is set equal to 0.01 to be considered statistically significant. The blocks for which F_i is greater than the critical value (F_c) of the Fisher table, are considered as being related to influential factors or interactions. By examining the calculated saliences, it is possible to determine which CC is related to which factor or interaction. For the salience, the probability value (p-value) is also calculated for each *F*-test. It represents the probability that the variability is only due to noise. The more the p-value is close to zero and the more the block is associated with a significant factor. The relative importance of a factor compared to the residual noise is related to its salience value in the CC. To estimate the effect of the factor, it is possible to plot the sample scores on the informative CCs vs CC1. Loadings show how data values vary along a CC and are used to understand the meaning of the scores. Loadings can have negative or positive values; so, can scores. If the loading of a variable and the score of a sample on a particular CC have the same sign, they are related. The larger the scores and loadings, the stronger is that relation. The reading of the CC graph is therefore opposite to the salience graph: the higher the score, the higher the parameter influence and the lower the salience.

The different studied factors were LUL, climate, soil depth and two additional factors, the soil calcareous and siliceous contents ($\text{CaCO}_3\%$ and SiO_2 intensity). These compounds, abundantly present in the mineral fraction of Mediterranean soils, greatly impact the FTIR spectral profile of studied soils. Thus, two additional factors, integrating CaCO_3 and SiO_2 contents, were considered to facilitate the convergence of the

model. The Calcimeter results were used to define the levels of the $\text{CaCO}_3\%$ factor. For SiO_2 intensity factor, the maximum of the characteristic SiO_2 IR band (located between 1097 and 925 cm^{-1} depending on the soil sample considered) was used to define an intensity of IR band (Szymanski, 2017; Krivoshein et al., 2020). Thus, a category corresponding to the maximum intensity of SiO_2 band was allocated to each soil sample.

Thus, 3 levels were considered for LUL (ancient, recent, and very recent forests), 2 levels for climate (humid and sub-humid), 3 levels for soil depth (0–10, 10–20 and 20–30 cm), 3 levels for $\text{CaCO}_3\%$ (0–10 %, 10 %–30 % and 30–60 %) and 2 levels for SiO_2 band intensity (IR band lower to 0.5 or upper 0.5 (au)).

The spectral interpretation of infrared characteristic group frequencies appearing on the loading graphs was realized from Socrates tables (Socrates, 2007) and from literature data referenced in Table 2. Only spectral bands with an intensity > 0.005 (au) were selected for the interpretation of loadings, to remove spectral bands induced by residual noise.

A matrix of 120 soil derivative spectra and 596 variables (wavenumber between 1800 and 650 cm^{-1}) was used for AComDim algorithm.

- Linear mixed effect models

Based on previous studies with similar sampling design (Santonja et al., 2018; Feng et al., 2019), the statistical analyses of the data were realized using linear mixed-effect models (LME) with significance levels indicated as * for $P < 0.05$, ** for $P < 0.01$ and *** for $P < 0.001$, followed by Tukey HSD tests for post hoc comparisons ($P < 0.05$). Linear mixed effect models allow the analysis of complex datasets with hierarchical structures, avoiding the shortcomings of ANOVAs and multiple regression. They consider the correlation of observations contained in a dataset and allow to effectively partition overall variation of the dependent variable, into components corresponding to different levels of data hierarchy. They are called “mixed” because they simultaneously model fixed and random effects. A mixed model allows mixing qualitative explanatory variables (factors) of two types: fixed and random effects. The random effects are here because there are groups of non-independent observations in our data: sites on which several soil samples have been sampled.

Here, LME models were performed to examine the effect of land use legacy (ancient forest, recent forest, very recent forest), climate (humid vs subhumid), soil depth (0–10 cm, 10–20 cm, 20–30 cm) and their interactions on chemical properties (C_{tot} , N_{tot} , $\text{CaCO}_3(\%)$). Land use legacy, climate and season were considered as fixed factors, and plot geography as random factor (following R syntax “random= ~ 1 |plot/mesocosm”). The random part of the model allowed us to account for the geographic proximity of forest couples (ancient forests vs recent forests or ancient forests vs very recent forests). The normality and homogeneity of the variances were determined on the residuals from the regression model with the Shapiro–Wilk and Levene tests, respectively. Data were transformed to common logarithms, \log_{10} , when necessary to meet the requirements of normality and homogeneity of variance.

- Softwares

The pre-treatments of spectral data were performed using CAMO (Computer Aided Modelling, Trondheim, Norway) software UN-SCRAMBLER 9.2. The AComDim procedure was adapted from the ComDim function in the free toolbox SAISIR (Cordella and Bertrand, 2014) and was performed with Matlab 7.14 (R2012a). LME models were performed with R version 3.6.2 (R Core Team 2019), using nlme package.

3. Results

3.1. Characterization of soil FTIR spectra

A selection of 21 FTIR-ATR spectral profiles representative of PNRL soil database at different soil depths are shown before and after a Savitzky-Golay first derivative pre-treatment on Fig. 1.a and b respectively. Assignment of characteristic bands are summarized in Table 2 where a correspondence was done between an IR original band and its two derivative bands (called *a* and *b* in Fig. 1.b and Table 2). Given the limestone accumulation horizons of Mediterranean soils, due to insufficiently rainy climate and a geology dominated by calcareous rocks, IR spectra of studied soils showed intense bands assigned to silicate and aluminosilicate (bands at 1160, 1030, 980, 910, 845, 795, 775, 730 and 690 cm^{-1}) and carbonate (bands at 1400, 870, 875, 775, 725 and 710 cm^{-1}), key components of soils on a calcareous parent material. A double assignation of these bands was also possible because of their overlapping with those from the organic matter represented by polysaccharides, aromatic and aliphatic compounds. Wavenumber shifts and intensity variations could be observed depending on soil depth, climate and forest age for which the significant effect will be really confirmed after chemometric treatment (AComDim) as shown below.

3.2. FTIR spectroscopy highlighted environmental factors effects on the SCS

FTIR-ATR derivative spectra were analysed via AComDim which decomposed FTIR database into 30 blocks: the first corresponding to the five factors (climate, land use legacy, soil depth, $\text{CaCO}_3\%$ and SiO_2 band intensity and their multiple interactions (from binary to quintuple ones), up to, and the residual noise.

(i) significativity of the different environmental factors

According to Fisher test with an alpha level of 0.05 by considering 120 degrees of freedom, a critical F-value (F_c) of 1.43 was obtained and was used to highlight the significant blocks (Supplementary material, Fig. S1). Thus, the F-values (computed from the saliences on CC1) of block 1 (“climate”), block 2 (“land use legacy”), block 4 (“ $\text{CaCO}_3\%$ ”) and block 6 (“climate-land use legacy interaction”) were higher than the critical F-value, while the F-value of the other blocks (“soil depth”, and “ SiO_2 band intensity”) was not significant. The saliences plot (not shown here) confirmed the selection of significant factors and interactions. The score and loading plots of block 4 was not described below because it classed soil samples according their CaCO_3 percent as expected. On each presented loading plot, a derivative form of an averaged spectrum (mean of 120 soil derivative spectra) was overlaid to define which IR bands of loading were correlated to the bands of this average spectrum. In reference to the initial spectrum (Fig. 1.b), all bands pointed on loadings (presented below) will be considered positively correlated when letters “a” or “b” will be placed on the same side, otherwise they will be negatively correlated.

(ii) effect of climate

The effect of climate on FTIR-ATR spectra is shown by the projection associated to CC10 (related to block 1 “climate”). This score plot clearly shows a discrimination between soil samples depending on climate (humid vs subhumid) (Fig. 2.a). From the loading plot (Fig. 2.b), soil samples exposed to humid climate (positive part) were characterized by intense IR signals assigned to organic matter such as polysaccharides, aromatic compounds and the presence of long alkyl chains (*i.e.* bands 4a, 4b, 6a, 8a, 8b, 10b, 11a and 11b pointed at 1010, 960, 880, 850, 840, 770, 730 and 720 cm^{-1}). Soil samples exposed to subhumid climate (negative part) were explained by IR signals assigned to the mineral fraction, particularly CaCO_3 (*i.e.* bands 7a, 12a and 12b pointed at 875, 715 and 710 cm^{-1}).

(iii) effect of LUL

The effect of LUL on FTIR-ATR spectra is shown by the projection associated to CC7 (related to block 2 “land use legacy”). This score plot

Table 2

Assignments of the main bands observed in the FTIR spectra of the organic and inorganic components of soils samples according to the bibliography. Letters a and b indicate the maximum and minimum peaks on FTIR spectra after a Savitzky–Golay derivative treatment.

Band number	Maximum peak (cm ⁻¹)	Spectral region (cm ⁻¹)	Assignments			
			Organic bands		Inorganic bands	
1	1400	1540–1260	O–H deformation, C–O stretching of phenolic OH and C–H deformation of CH ₂ wagging and CH ₃ groups (polysaccharides, cellulose, pectin); C–OH bending vibration of carboxylic acid, alcohol; symmetric stretching vibration COO– of amino acid and fatty acids, C–N stretching and deformation, amide III band (proteins)	Ferrari et al., 2011; Vergnoux et al. 2011	CaCO ₃ (Asymmetric CO ₃ ²⁻ stretching)	Tatzberg et al., 2010; Bruckman and Wriessnig, 2013; Gunasekaran et al., 2006
1a	1435					
1b	1370					
2	1160	1180–1150	C–O, CO–O–C and C–O–C stretching of polysaccharides, C–OH deformation of aliphatic alcohols, carbohydrates	Madari et al., 2006; Tinti et al., 2015; Bornemann et al. 2010; Zaccheo et al. 2002	Si–O of silicate impurities (various clay minerals)	Madari et al., 2006; Müller et al., 2014; Krivoshein, et al. 2020
2a	1170					
2b	1160					
3	1030	1080–940	C–O, C–C stretch of cellulose	Bornemann et al. 2010		
3a	1035					
3b	1020					
4	980		O–CH ₃ in methoxy groups, ring C–O, C–C stretching vibration in Benzoic acid, pyranose ring (carbohydrates), cellulose (ring breathing),	Tinti et al., 2015; Zaccheo et al. 2002		
4a	1010					
4b	960					
5	910	920–890			Al–OH–Al bending vibration (Quartz)	Müller et al., 2014; Krivoshein, et al. 2020
5a	915					
5b	900					
6	875	890–850	aromatic C–H and C=C deformation	Bornemann et al. 2010; Tinti et al., 2015	CaCO ₃ (Asymmetric CO ₃ deformation)	Nguyen et al. 1991; Bruckman and Wriessnig, 2013; Gunasekaran et al., 2006; Tinti et al., 2015
6a	880					
7	870					
7a	875					
7b	865					
8	845	850–840	Aromatic CH out of plane bending (Lignin)	Madari et al., 2006; Tinti et al., 2015	Si–O of silicate impurities (various clay minerals), Al–OH–Mg vibration	Müller et al., 2014; Parikh et al., 2014; Krivoshein, et al. 2020
8a	850					
8b	840					
9	795	800–790	C–H bending (non-aromatic)	Krivoshein, et al. 2020	Inorganic materials (clay and quartz minerals), carbonate, kaolinite	Tatzber et al., 2010; Krivoshein, et al. 2020
9a	800					
9b	790					
10	775	780–770	Aromatic CH out of plane bending	Madari et al., 2006		
10a	780					
10b	770					
11	725	740–715	Long chain alkanes (CH ₂ wag)	Artz et al., 2008; Tinti et al., 2015	CaMg(CO ₃) ₂ (symmetric CO ₃); SiO ₂	Bruckman and Wriessnig, 2013; Gunasekaran et al., 2006
11a	730					
11b	720					
12	710	715–700			CaCO ₃ (Symmetric CO ₃ ²⁻ deformation)	Bruckman and Wriessnig, 2013; Gunasekaran et al., 2006
12a	715					
12b	710					
13	690	710–670			O–Si–O stretching and bending	Nguyen et al. 1991; Tinti et al., 2015; Krivoshein, et al. 2020
13a	695					

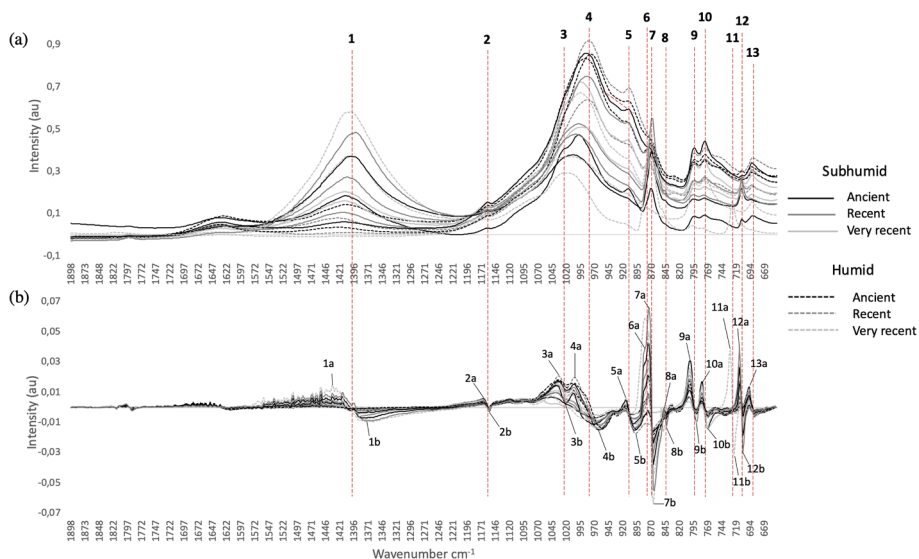


Fig. 1. Selection of 21 FTIR-ATR spectral profiles representative of PNRL soil database, at different soil depths, before (a) and after a Savitzky–Golay derivative treatment (b).

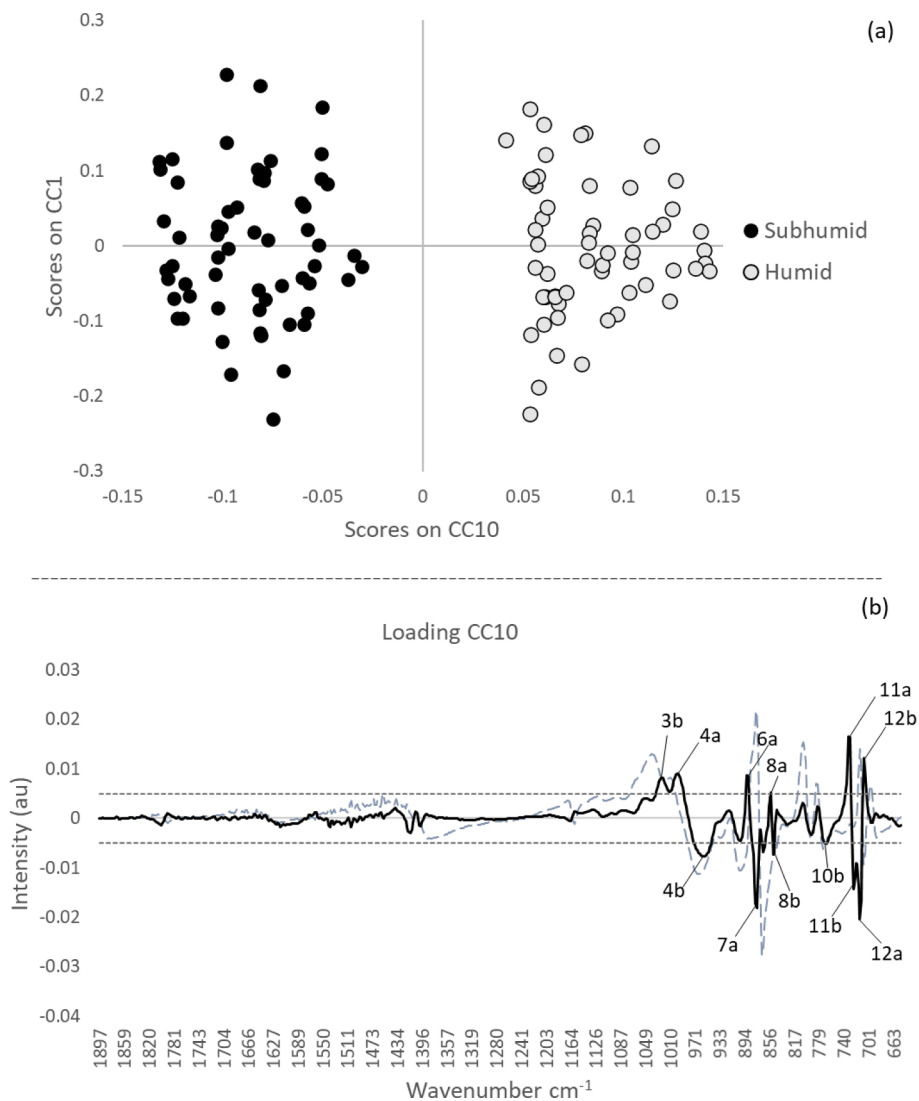


Fig. 2. AComDim of spectral soil data (a) CC1 vs CC10 score plot, (b) loading on CC10 corresponding to climate (black line) and average spectrum (dotted line, N = 120).

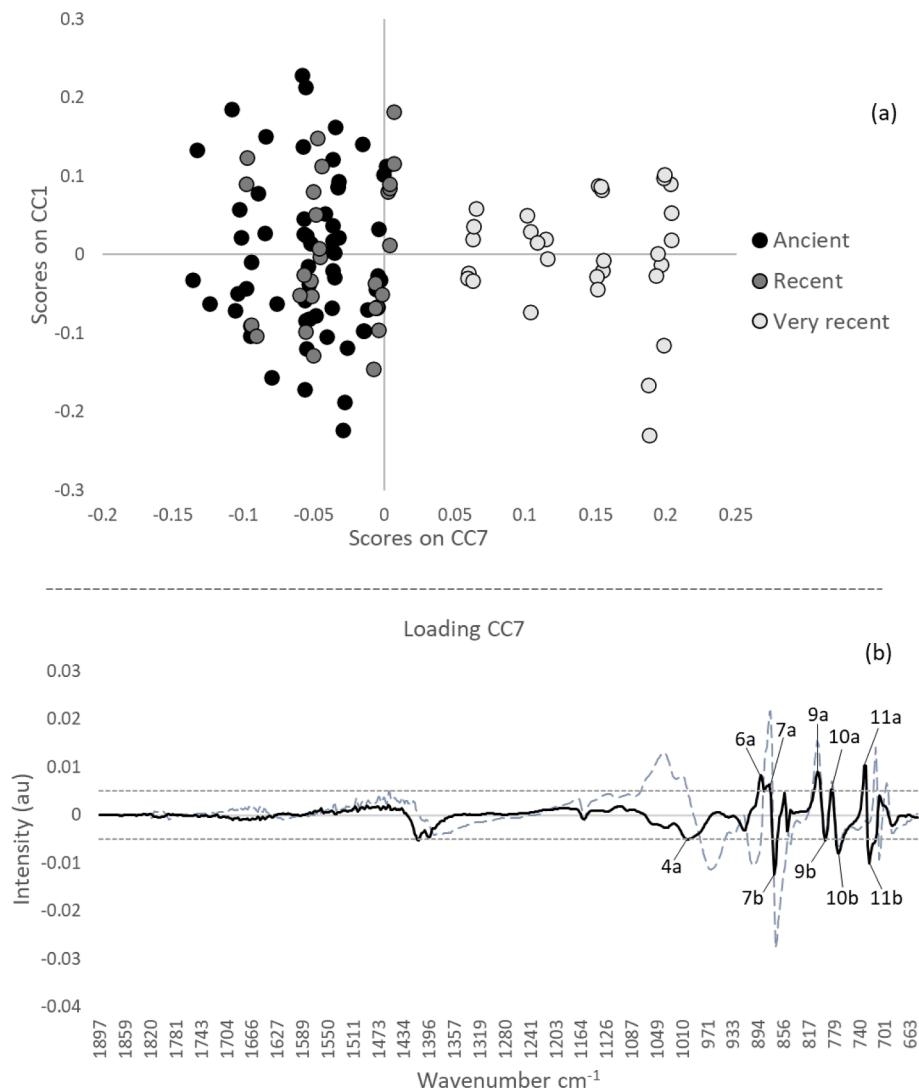


Fig. 3. AComDim of spectral soil data (a) CC1 vs CC7 score plot, (b) loading on CC7 corresponding to land use legacy (black line) and average spectrum (dotted line, $N = 120$).

(Fig. 3.a) clearly shows a discrimination between soil samples depending on LUL (ancient and recent forests vs very recent forests). From the loading plot (Fig. 3.b), soil samples from very recent forests (positive part) were explained by IR signals assigned to recalcitrant organic matter such as aromatic compounds and to the mineral fraction whose IR signal was exalted by the lack of organic matter (*i.e.* bands 6a, 7a, 7b, 9a, 9b, 10a, 10b, 11a and 11b pointed at 880, 875, 865, 800, 780, 770, 730 and 720 cm^{-1}). Soil samples from ancient and recent forests were grouped together in the negative part. Their position was explained by their negative correlation with an IR signal assigned to carbohydrates or silicate (*i.e.* band 4a pointed at 1010 cm^{-1}) since a clear identification was uncertain from one single band.

(iv) effect of interaction between climate and LUL

The effect of the interaction between climate and LUL on FTIR-ATR spectra was shown by the projection associated to CC17 related to block 6 “interaction climate-land use legacy”. This score plot was presented on Fig. 4.a and 4.b: soil samples under humid climate or under subhumid climate are highlighted in Fig. 4.a and 4.b respectively. These figures showed that the SCS of ancient and recent forests (grouped on the positive part) were similar under a humid climate, while a differentiation of soils from recent and very recent forests was possible under subhumid climate. The central position of soils from ancient forests expressed no differentiation for them. The loading plot (Fig. 4.c) showed

IR bands, all anti-correlated to those described on the reference soil spectra (Fig. 2b). These IR signals were assigned to alkyl chains and mineral fraction (*i.e.* bands 1a, 7a, 7b, 11a, 11b, 12a and 12b pointed at 1435, 875, 865, 730, 720, 715 and 710 cm^{-1}) characterizing soils from very recent forests under humid climate and soils of recent forests under subhumid climate.

3.3. Variations in other soil chemical properties

Results of full linear mixed-effect models on soil physico-chemical properties were presented according to three factors (LUL, climate and soil depth) and their interactions (Table 3). The mean value for each chemical property considered in this LME approach (N_{tot} , C_{tot} , CaCO_3 , C_{org} and $C_{\text{tot}}/N_{\text{tot}}$) was reported according to the different levels of factors in Table 4.

(i) effect of LUL

LUL factor influenced all chemical soil properties with different significance degrees, N_{tot} (%) having the higher p-value. CaCO_3 (%) and $C_{\text{tot}}/N_{\text{tot}}$ values of very recent forest soils appeared significantly higher than those measured for ancient and recent forests. C_{tot} (%) and N_{tot} (%) of very recent forest soils were significantly different of ancient forest soils only (with a higher C_{tot} (%) but a lower N_{tot} (%), while no impact of LUL was detected through C_{org} (%). Climate influenced only $C_{\text{tot}}/N_{\text{tot}}$

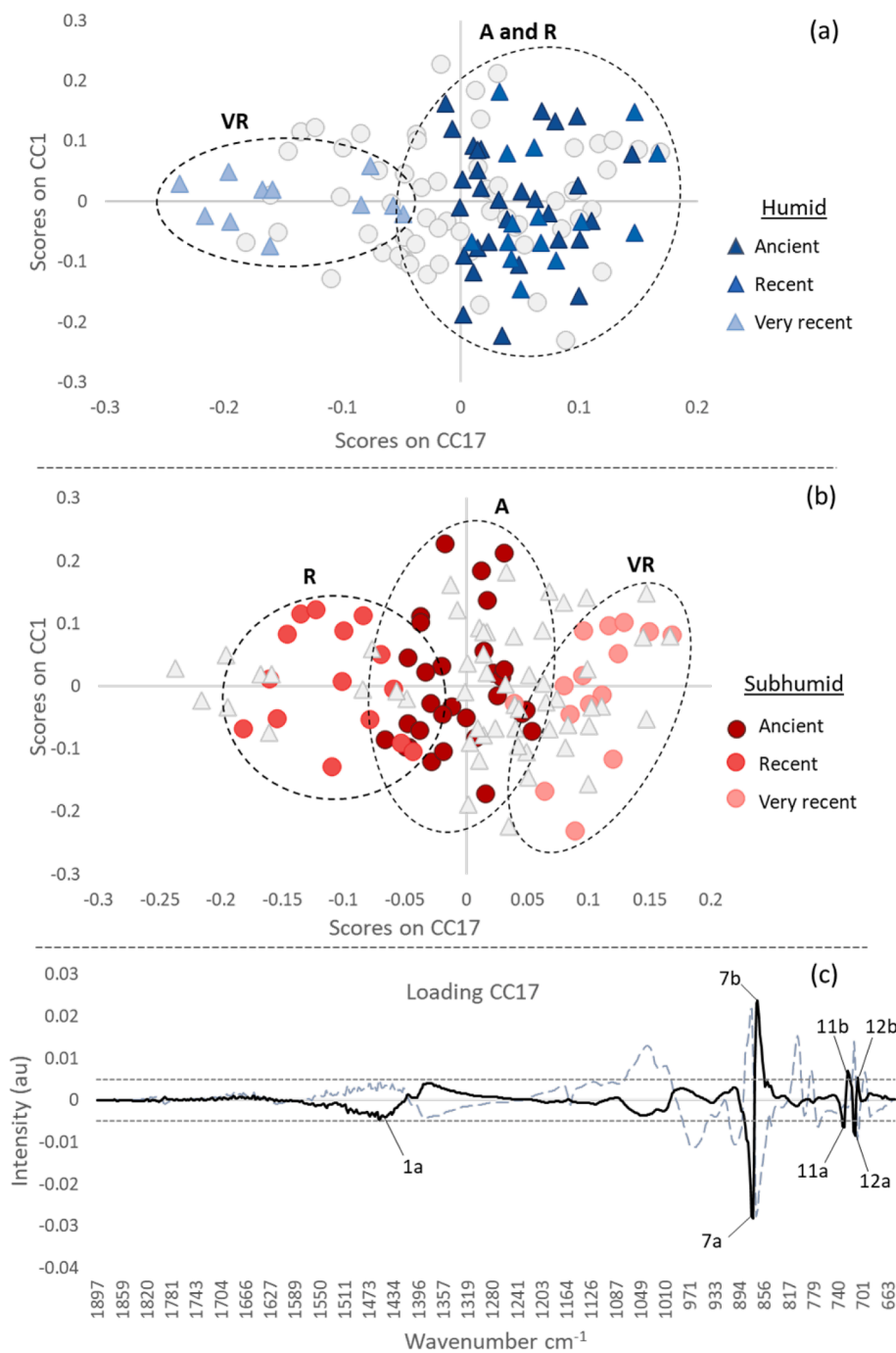


Fig. 4. AComDim of spectral soil data (a) CC1 vs CC17 score plot of soils from humid climate (blue triangles) and subhumid climate (grey circle) and (b) CC1 vs CC17 score plot of soils from humid climate (grey triangles) and subhumid climate (red rings), (c) loading on CC17 corresponding to interaction “climate – land use legacy” (black line) and average spectrum (dotted line, N = 120). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 3

Results of full linear mixed-effect model analyses on soil physico-chemical properties depending on factor land use legacy (LUL), climate (C) and soil depth (D) and their interactions.

Effects	d.f.	N _{tot} (%)		C _{tot} (%)		CaCO ₃ (%)		C _{org} (%)		C _{tot} /N _{tot}	
		χ ²	p-Value	χ ²	p-Value	χ ²	p-Value	χ ²	p-Value	χ ²	p-Value
LUL	2	6.47	*	14.64	***	36.06	***	2.05	ns	30.42	***
C	1	0.73	ns	2.50	ns	3.13	ns	1.12	ns	6.24	*
D	2	309	***	128	***	33	***	244	***	20	***
LUL × C	2	1.78	ns	18.76	***	26.23	***	0.06	ns	23.62	***
LUL × D	4	1.20	ns	5.28	ns	1.50	ns	1.59	ns	3.61	ns
C × D	2	0.15	ns	5.39	ns	5.88	ns	0.09	ns	12.72	**
LUL × C × D	4	7.09	ns	0.97	ns	0.67	ns	4.17	ns	1.60	ns

C: climate; D: soil depth; LUL: Land use legacy; ns: no significant; * p < 0.05; ** p < 0.01; *** p < 0.001.

Table 4

Chemical properties from soils depending on land use legacy (ancient (A), recent (R) and very recent (VR) forests), climate (humid vs subhumid) and soil depth (A:0–10 cm, B:10–20 cm, C:20–30 cm). Means (\pm standard error, N = 120) with different letters are significantly different ($a < b < c$ and for HSD test, $P < 0.05$).

	Land use legacy (LUL)			Climate (C)		Soil depth (D)		
	A	R	VR	Subhumid	Humid	0–10 cm	10–20 cm	20–30 cm
N_{tot} (%)	0.25 ± 0.02^b	0.24 ± 0.01^{ab}	0.24 ± 0.02^a	0.26 ± 0.02^a	0.24 ± 0.02^a	0.33 ± 0.02^c	0.22 ± 0.01^b	0.19 ± 0.01^a
C_{tot} (%)	6.2 ± 0.5^a	5.8 ± 0.4^{ab}	7.6 ± 0.5^b	7.3 ± 0.4^a	5.7 ± 0.5^a	7.8 ± 0.5^c	6.1 ± 0.4^b	5.6 ± 0.4^a
$CaCO_3$ (%)	14.3 ± 2.4^a	14.6 ± 2.0^a	25.0 ± 2.1^b	21.5 ± 2.3^a	12.5 ± 2.1^a	12.8 ± 1.7^a	18.1 ± 2.4^b	20.1 ± 2.7^b
C_{org} (%)	4.5 ± 0.3^a	4.0 ± 0.3^a	4.6 ± 0.4^a	4.7 ± 0.3^a	4.1 ± 0.3^a	6.2 ± 0.4^c	3.8 ± 0.2^b	3.1 ± 0.2^a
C_{tot}/N_{tot}	24.5 ± 1.1^a	21.5 ± 1.4^a	27.3 ± 1.5^b	28.9 ± 1.3^b	22.2 ± 1.2^a	23.5 ± 0.9^a	26.0 ± 1.3^b	27.5 ± 1.7^b

ratio which was lower under humid climate. All the forests (whatever their age) did not present significant variations in the remaining chemical parameters according to the climate. Factor “soil depth” was highly significant for all chemical analyses (p -value < 0.005). The mean values of N_{tot} (%), C_{tot} (%) and C_{org} (%) decreased with soil depth. Conversely, $CaCO_3$ (%) and C_{tot}/N_{tot} ratio were lower at the top layer (0–10 cm) of soil than in the two other soil depths for which the mean value was similar.

(ii) effect interaction between climate and LUL

LME models showed that C_{tot} (%), $CaCO_3$ (%) and C_{tot}/N_{tot} were significantly influenced by the LUL-climate interaction. For soils from humid climate, higher $CaCO_3$ (%) and C_{tot}/N_{tot} ratio were observed in soils from very recent forests than in soils from ancient forests and recent forests, while no effect of LUL was observed for soils from subhumid climate (Fig. 5). An interaction between climate and soil depth was significant for the C_{tot}/N_{tot} ratio. In soils from subhumid climate, higher C_{tot}/N_{tot} ratio was observed in the deepest layer (20–30 cm) than in the upper layer (0–10 cm), while no significant effect of soil depth was observed in soils from humid climate (Fig. 6).

4. Discussion

Spectroscopic signatures highlighted the effect of LUL and climate on the chemical composition of forest soils, while the measurement of some physico-chemical properties did fail in this way. The originality of this approach was to highlight the effects of interactions between the spatio-temporal factors tested (*i.e.* soil depth, climate and date of the abandonment) on SCS.

4.1. Specific pedoclimatic conditions of the Mediterranean modify SCS

Mediterranean area is characterised by a specific pedoclimatic context (Yaalon, 1997; Torrent, 2005), soils are indeed sparsely developed and contain little organic matter. Carbonaceous rocks are the most important parent material in Mediterranean areas. Though they are dominated by a relatively soluble $CaCO_3$ fraction, soils developed on calcareous rocks may well differ due to differences in coherence, permeability, and resistance to weathering of the substratum (Verheyne and de la Rosa, 2006). Here, many mineral bands were observed in soil spectra (*i.e.* bands from carbonate and silicate), highlighting the importance of the mineral fraction in Mediterranean SCS. FTIR spectroscopy revealed that the relative proportion of the mineral fraction is indeed very sensitive to environmental factors *e.g.* current land use and climate (Delcourt et al. 2019; Torrent 2005). The more drastic climate conditions observed in subhumid climate (higher temperatures and less abundant precipitations) resulted in a lower aboveground plant biomass production, which probably led to less-developed soils more exposed to erosion than what observed in humid climate (Ackermann, 2019; Avni et al., 2006; Sarah, 2006). Thus, climate plays a crucial role on the SCS, directly through erosion and leaching and indirectly by influencing soil organic matter inputs and transformation. (Allison and Treseder, 2008; Torrent, 2005; Suseela and Tharayil 2018).

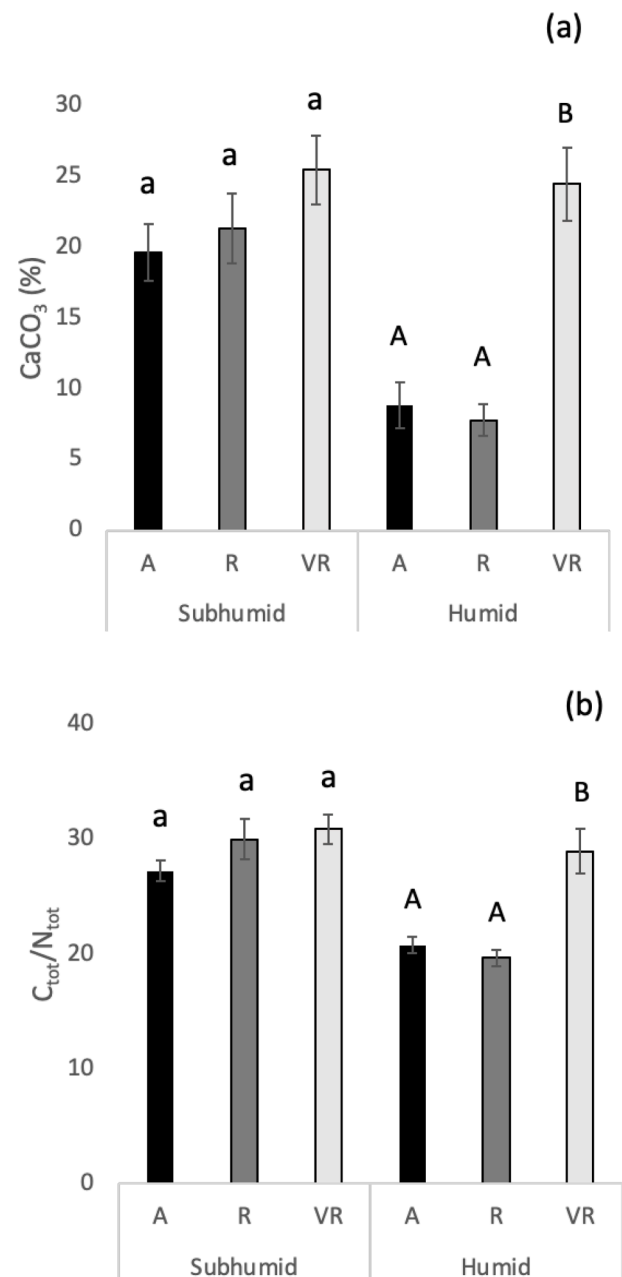


Fig. 5. $CaCO_3$ (%) (a) and C_{tot}/N_{tot} (b) in soils from ancient forests (A), recent forests (R) and very recent forests (VR) under humid and subhumid climate. Means (\pm standard error, N = 120) with the same letters are not significantly different (HSD test, $P < 0.05$).

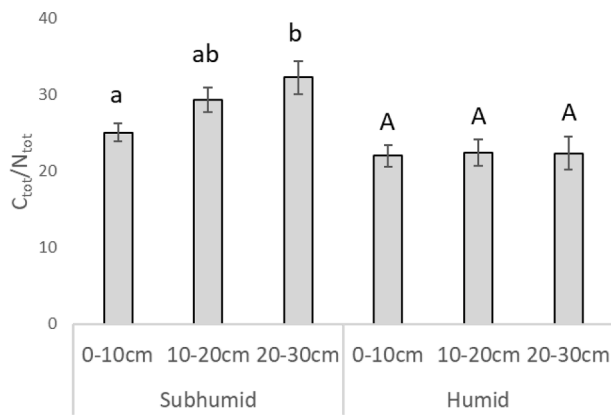


Fig. 6. C_{tot}/N_{tot} in the 0–10 cm, 10–20 cm and 20–30 cm of forest soils under humid and subhumid climate. Means (\pm standard error, $N = 120$) with the same letters are not significantly different (HSD test, $P < 0.05$).

4.2. LUL effect on SCS depending on the date of LUL

As expected, LUL influenced the SCS and the majority of the chemical properties measured as a sole factor. Higher N_{tot} percentage was found in soils from ancient forests than in soils from very recent forests, showing that LUL influenced the quality of soil organic matter (higher available N_{tot} % in soils from ancient forests). According to the results of Zornoza et al. (2009), in ancient forests, the long-term accumulation of organic matter has probably led to more developed soils with a more complex composition and a better organic matter transformation compared to very recent forests. Moreover, in their study, Gao et al. (2016) and Segura et al. (2021) showed that afforestation improved soil quality (e.g total nitrogen and total organic carbon).

With the help of statistical approaches considering interaction effects, our results revealed that LUL impacted the SCS depending on the climate considered (humid vs subhumid climate). In humid climate, soils of very recent forests (associated with a FTIR profile poor in organic matter) were discriminated from those of ancient and recent forests. The importance of the mineral fraction in the chemical composition of the very recent forest soils can be explained by the terrace abandonment, leading to highly vulnerable soils (Tarolli et al., 2019). Thus, soils are exposed to erosion and leaching via rainfalls, which are more frequent and abundant in humid climate than in subhumid climate, leading to accelerated erosion (Lesschen et al., 2008; Stanchi et al. 2012). Moreover, after land abandonment, herbaceous species present during the first stage of the secondary succession, failed to protect soil against erosion (Nunes et al., 2010). Conversely, under humid climate, a recovery of the SCS was observed in our study >60 years after reforestation, leading to similar chemical properties between recent and ancient forests. In this case wettest conditions have probably enhanced the establishment of a new forest as has already been demonstrated (Anderson-Teixeira et al., 2013). Previous studies indeed showed that vegetation cover plays an important role in protecting soils against erosion and leaching, by enhancing organic matter content, soil structure and infiltration rate (Kosmas et al., 2000; Rodrigo-Comino et al., 2018; Stanchi et al., 2012).

In subhumid climate, FTIR data showed a discrimination between the SCS from recent and very recent forests based on the representativeness of mineral fraction. Weaker annual precipitations under subhumid climate have probably limited erosion and organic matter leaching induced by terraced culture but the vegetation was subjected to drier conditions and afforestation may have taken more time. Thus, under subhumid climate, erosion had probably occurred for a longer period in recent forests leading to a marked influence of the mineral fraction on the SCS. Similar patterns were found by Ackermann et al. (2019) who studied the natural vegetation regeneration and erosion

intensity after the abandonment of agricultural terraces along a climate gradient from subhumid to arid conditions. They observed that wetter conditions promoted natural vegetation recovering and thus soil stabilisation. On the contrary, soils under more arid conditions were deprived of dense vegetation and more exposed to erosion. By controlling vegetation cover, climate factor had a major influence on erosion processes after the abandonment of terraces (Durán Zuazo and Rodríguez Pleguezuelo, 2008; Valentin et al., 2005). Moreover, Kosmas et al. (2000) highlighted that the recovery by natural vegetation was lower in soils of lesser depth resulting in very active erosional processes and thus to a degradation of soils. They also showed that without a perennial vegetation, soils were quickly removed by wind and water erosion. In our results, under subhumid climate, neither soils from recent forests nor soils from very recent forests had similar SCS to that of ancient forest. The recovery of soils properties after the abandonment of agricultural terraces indeed seems to need more time in subhumid climate than in humid climate. Moreover, there is no guarantee that soils of post agricultural forests will return to the same state as those of forests that had never been cultivated in the past. Previous studies indeed described degradation and desertification of abandoned lands as an irreversible process under arid conditions (Ackermann et al., 2019; Kosmas et al., 2000). Thus, the impact of climate change may affect the resilience capacity of soils and LUL factor should be considered to assess soil vulnerability to climate change in Mediterranean region.

4.3. LUL effect on SCS depending on soil depth

Though, as expected, a gradient of amount of organic matter was found depending on soil depth, no interaction between LUL and soil depth was detected from spectral and chemical data. Previous studies showed that vegetation changes throughout different soil depths can be detected with spectroscopy methods (Ertlen et al., 2015; Vysloužilová et al., 2015). However, these studies were focused on deeper soils and considered longer-term dynamics (millennia). Conversely, here, our study dealt with Mediterranean soils, known to be very poorly developed (Verheye and de la Rosa, 2006). Studied soils indeed did not exceed 30 cm depth before finding the bedrock. Shallow soils in Mediterranean area thus could explain why here, LUL did not vary depending on soil depth. Thus, the fact that chemical signature resulting from the history of land use in Mediterranean forests was the same regardless of soil depth could facilitate the integration of a spectroscopic approach to include LUL as a proxy in soil studies.

5. Conclusion

In this study, FTIR-ATR spectroscopy combined to AComDim revealed LUL effect on the chemical signature of Mediterranean soils depending on climate conditions and soil depth. It was observed that the effect of agricultural terraces persisted over time on the SCS regardless of the soil depth considered. Additionally, it appeared that agricultural practices under humid climate promoted the recovery of SCS by natural vegetation (*Q. pubescens* as dominant species in our case), which protected soils against erosion. On the contrary, the less developed soils and the more drastic climate conditions under subhumid climate have slowed afforestation, which encouraged erosion. Mediterranean region is one of the most vulnerable terrestrial biomes to climate change. Thus, regarding climate change, our results highlighted that LUL could influence soil vulnerability and should be considered as an important ecological proxy with the help of a spectroscopic approach.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Acknowledgments

We thank Jean-Philippe Orts, Dr Yoann Pinguet and Pr Thierry Tatoni for their contribution to the field work, Martine Martinez for her technical assistance during soil sample pre-treatments and Dr Mathieu Santonja for his advice on LME analyses. Ninon Delcourt was funded by a doctoral scholarship from the French Ministry of Higher Education, Research and Innovation (École Doctorale des Sciences Chimiques, Aix Marseille Université).

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.geoderma.2022.116299>.

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