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1 Is Rock-Eval 6 thermal analysis a good indicator of soil organic carbon lability? – A method-
2 comparison study in forest soils

3

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16

17

18 **Abstract**

19 Soil respiration tests and abundance of particulate organic matter (POM) are considered as
20 classical indicators of the labile soil organic carbon (SOC) pool. However, there is still no
21 widely accepted standard method to assess SOC lability and the pertinence of these two time-
22 consuming methods to characterize SOC turnover can be questioned. Alternate ways of
23 determining the labile SOC fraction are thus much needed. Thermal analyses, in particular
24 Rock-Eval 6 (RE6) analysis has shown promising results in the determination of SOC
25 biogeochemical stability.

26 Using a large set of samples ($n = 99$) of French forest soils representing contrasted
27 pedoclimatic conditions, including deep samples (up to 0.8 m depth), we compared three
28 different methods used for SOC lability assessment. We explored whether respired-C isolated
29 by a 10-week laboratory soil respiration test, POM-C isolated by a physical SOC fractionation
30 scheme (particle-size $> 50 \mu\text{m}$ and $d < 1.6 \text{ g}\cdot\text{cm}^{-3}$) and several RE6 parameters were
31 comparable and how they correlated.

32 As expected, respired-C ($\text{mg CO}_2\text{-C}\cdot\text{g}^{-1}$ SOC) and POM-C (% of total SOC) fractions
33 strongly decreased with depth. RE6 parameters showed that SOC from deeper soil layers was
34 also thermally less labile, more oxidized and H-depleted. Indeed, SOC from deeper soil layers
35 had lower proportion of thermally labile SOC, higher $T_{50_HC_PYR}$ (temperature at which 50%
36 of the pyrolysable hydrocarbons were effectively pyrolyzed) and $T_{50_CO2_OX}$ (temperature at
37 which 50% of the CO_2 gas had evolved during the oxidation phase), larger oxygen index, and
38 smaller hydrogen index. Surprisingly, the two classical indicators of the labile SOC pool
39 (respired-C and POM-C) were only marginally correlated ($p = 0.051$) and showed layer-
40 specific correlations. Similarly, respired-C was poorly correlated to RE6 parameters.
41 Conversely, the POM-C fraction showed a strong negative correlation with $T_{50_HC_PYR}$ ($\rho =$
42 -0.73) and good correlations with other RE6 parameters.

43 Our study showed that RE6 parameters were good estimates of the POM-C fraction, which
44 represents a labile SOC pool with a residence time of *ca.* a couple decades that is meaningful
45 regarding SOC stock changes upon modifications in land management. RE6 thermal analysis
46 could therefore be a fast and cost-effective alternative to more time-consuming methods used
47 in SOC pool determination, and may be integrated into soil monitoring networks to provide
48 high-throughput information on SOC dynamics.

49

50 **Keywords:** soil organic carbon kinetic pools; Rock-Eval 6; particulate organic matter; soil
51 basal respiration; deep soil organic carbon; French forest soils;

52

53

54 1. Introduction

55 Soil organic matter (SOM) degradation has multiple consequences on major soil functions
56 like nutrients cycling, soil emissions of greenhouse gases and affects soil carbon sequestration
57 potential. In particular, the labile part of SOM (turnover < 20 years) is associated with
58 biological (microbial) activity and nutrient cycling (Haynes, 2005) and is very relevant to
59 these issues.

60 In that context, information on the temporal trajectories of SOC storage at a fine spatial
61 resolution, in the form of detailed mapping of SOC stock evolutions with time for different
62 land management scenarios, are required. SOC dynamics models are the logical candidates to
63 provide such information, but their predictive performance is not yet satisfying, and they
64 would benefit from an improved initialization using fine-scale information on SOC kinetic
65 pools (Luo et al., 2016). Soil monitoring networks have become more prominent in the last
66 twenty years. However, currently they can only provide information relative to the recent
67 temporal (decadal) evolution of total SOC stocks. To use the full potential of these networks
68 and measure the effects of climate and land-use changes on SOC stocks will require indicators
69 of the size of the different SOC pools.

70 Respiration measurements and particulate organic matter (POM) quantification obtained by
71 various methods of fractionation (particle size only / density only / density + particle-size)
72 (von Lützow et al., 2007) have been used for decades and are now classical estimates of the
73 labile SOC pool.

74 Laboratory incubations are run under optimum temperature and moisture conditions and use
75 the indigenous microflora. They thus represent a maximum potential rate of C mineralization
76 and an index of C availability in the system, integrating the physical, chemical, and
77 microbiological properties of the soil (Haynes, 2005). Incubations are fairly simple to set-up

78 but they require space and are time-consuming. Sieving and rewetting also tend to artificially
79 increase the mineralizable pool (Haynes, 1986).

80 Physical fractionation schemes are easy to implement and do not require expensive equipment
81 although they can become costly when density fractionation is involved. Moreover they are
82 very time-consuming, often requiring multiple and relatively long periods of agitation/settling
83 and drying. The most important limitation is the ability of the fractionation scheme to isolate
84 physical fractions that have homogeneous turnover and thus represent functional non-
85 composite SOC pools (von Lützow et al., 2007).

86 While respired-C and POM-C fractions both represent a labile SOC pool, the former
87 corresponds to a smaller SOC pool with a shorter mean residence time (usually < 1 year for
88 temperate *in-situ* conditions) (Feng et al., 2016) while the latter corresponds to a larger SOC
89 pool with a longer mean residence time (usually < 20 year for temperate *in-situ* conditions)
90 (*e.g.*, Trumbore et al., 1996; Balesdent, 1996). Because these two methods are both very time-
91 consuming, they cannot address the needs of soil monitoring, *i.e.*, a methodology that is
92 informative, high-yield and relatively cheap to implement, to allow for the analysis of
93 numerous samples.

94 Among thermal analyses used to characterize SOM, Rock-Eval 6 (RE6) analysis has shown
95 promising results in the determination of SOM biogeochemical stability (*e.g.*, Barré et al.,
96 2016) and thus appears like a good candidate to fill this methodological gap. Originally
97 developed for oil and gas exploration in sedimentary basins, the method was first applied to
98 study soils with hydrocarbons contamination (Lafargue et al., 1998). RE was also shown to
99 provide useful information on SOM originating from soil profiles worldwide (Disnar et al.,
100 2003) and many studies on SOM characterization have been conducted, sometimes using RE
101 analysis in conjunction with other methods like nuclear magnetic resonance (Albrecht et al.,
102 2015), hydrocarbon analysis by gas chromatography (Di-Giovanni et al., 1998), infrared

103 spectroscopy (Hetényi et al., 2006) or biochemical oxygen demand (Copard et al., 2006) to
104 determine the origin and/or decomposition stage of the organic matter (Hetényi et al., 2005;
105 Sebag et al., 2006).

106 More recently, RE6 results have been compared with respiration test or SOM fractions at the
107 plot (Gregorich et al., 2015) and the small landscape scale (Saenger et al., 2015) but in both
108 cases the analyses were restricted to superficial soil layers. Gillespie et al. (2014) have also
109 related thermal stability assessed by RE6 to respiration test and X-ray absorption near-edge
110 structure spectroscopy for cryosolic soil profiles (up to 75 cm) in Northern Canada but only in
111 four hummocks. Finally, RE6 thermal analysis has been used to look at SOM dynamics in a
112 sample set with a large soil type variability and some deeper horizons (Sebag et al., 2016), but
113 without comparison to other methodologies.

114 The objective of this study was to properly “benchmark” RE6 thermal analysis with two
115 classical yet time-consuming methods for labile SOC pool estimation: a soil respiration test
116 isolating a respired-C fraction under controlled laboratory conditions and a physical SOC
117 fractionation scheme isolating a POM-C fraction. We selected soil samples from the French
118 forests monitoring network RENECOFOR at various depths. To our knowledge, this is the
119 first study considering such a large set of samples (covering a wide pedoclimatic variability),
120 including deep soil layers up to 0.8 m. Our sample set thus included soil samples that
121 presumably contained very different proportions of the labile SOC pool. Because the
122 difference in size of the C pool estimated by the respiration test and the POM fractionation
123 (*e.g.*, Haynes, 2005) and the previously observed correlations between stock of labile SOC
124 estimated by RE6 parameters and the POM fraction (Saenger et al., 2015) on one hand and
125 between cumulative C mineralized and a RE6 parameter (Gregorich et al., 2015) on the other
126 hand, we were expecting that: 1/ the results provided by the two classical methods would
127 differ quantitatively while the results from the three methods would be qualitatively

128 comparable and correlated; 2/ we would be able to derive a significant quantitative
129 relationship between RE6 parameters and the two classical indicators of the labile SOC pool.
130

131 2. Material and methods

132 2.1. Sampling

133 We considered forest soils samples from 53 permanent forest sites of the French national
134 network for the long term monitoring of forest ecosystems (“RENECOFOR”), established in
135 1992 (Ulrich, 1995) by the National Forest Service (ONF; <http://www.onf.fr/renecofor>) as a
136 part of the European ICP-FORESTS (<http://icp-forests.net/>) level 2 network (Fig. 1a). They
137 were representative of a good portion the national variability in terms of climate (with MAP
138 and MAT ranging between 703–1894 mm and 4.8–12.3 °C respectively for the 1971–2000
139 period), soil type (entic Podzol; eutric Cambisol/Calcisol; dystric Cambisol) (IUSS Working
140 Group, 2015) and forest vegetation (coniferous—silver fir; Norway spruce; European larch;
141 Scots pine—and deciduous—beech; oaks *spp*—stands). At each site, samples representing
142 two soil layers were obtained (0–10 cm and 40–80 cm; Fig. 1b). Samples of the top soil layer
143 were composite, at each depth, of 5 × 5 sampling points located over a 5000 m² plot, collected
144 between 2007 and 2012 by digging a 50 cm wide soil profile (Ponette et al., 1997; Jonard et
145 al., 2017). Samples of the deeper soil layer were composite from two soil pits located just
146 outside the central plot and collected in 1994–1995 (Brêthes et al., 1997). The surface and
147 deep samples thus originate from two different sampling campaigns. The deep samples were
148 only collected once, during the first campaign, to limit perturbation on the monitoring plots.
149 Basic soil parameters (pH and texture) were determined by Ponette et al. (1997) and are
150 reported as supplementary information (Table SI-A1).
151 Bulk soils were air-dried and stored in plastic buckets right after sampling. One liter of soil of
152 each layer was retrieved for this study and sieved at 2 mm before analysis.

153

154 2.2. Elemental analysis

155 Bulk < 2 mm-sieved soil samples were ground (< 250 μm ; ultra-centrifugal mill ZM 200,
156 Retsch GmbH) and organic carbon and total nitrogen concentrations were determined by dry
157 combustion with an elemental analyzer (CHN NA 1500, Carlo Elba). Samples with
158 carbonates (total $\text{CaCO}_3 = 3.5\text{--}835 \text{ g}\cdot\text{kg}^{-1}$) were first decarbonated following the same
159 protocol as Harris et al. (2001). Briefly, 30 mg of ground samples were weighed in 5 mm \times 9
160 mm silver boats followed by the addition of 50 μL of distilled water. The boats were put in a
161 glass bell jar, next to a beaker containing 100 mL of concentrated HCl (12 $\text{mol}\cdot\text{L}^{-1}$). The air
162 in the jar was evacuated and samples let to sit in this HCl-saturated atmosphere to allow the
163 acid to dissolve water and hydrolyze the carbonates for 8 h. Then, the decarbonated samples
164 were dried at 60 $^\circ\text{C}$ in the silver boats for at least 48 h. Silver boats were further placed in 10
165 mm \times 10 mm tin boats and analyzed for C and N.

166 POM fractions (see section 2.4.) were ground with a ball mill (mixer mill MM 200, Retsch
167 GmbH) or a mortar and pestle when the sample mass was less than 0.05 g. Carbon
168 concentration was determined as for the bulk soil.

169

170 2.3. Respiration test

171 For each sample, 20 g of 2 mm-sieved soil were transferred in a 120 mL glass-flask and re-
172 wetted at pF 2.5 (-0.033 MPa), which had been previously determined using a 5 Bar pressure
173 plate extractor (#1600, Soilmoisture Equipment Corp.). The flasks were fitted with aluminum
174 seals with PTFE-faced silicone septa to allow for headspace gas sampling and placed inside
175 an incubator (AE240 BIO EXPERT, Froilabo SAS) kept at 20 $^\circ\text{C}$ for 10 weeks following a
176 two-week period pre-incubation to allow the samples microbial activity to stabilize (data not
177 included).

178 Headspace gases were sampled at 1 to 2-week intervals during the 10-week incubation period
179 and CO₂ concentrations were determined using an Agilent 490 micro-gas chromatograph
180 equipped with the OpenLAB Chromatography Data System EZChrom software.
181 When CO₂ concentrations had reached 2.5–3% or was expecting to do so before the next
182 measurement, and/or when the cap had been pierced with the needle four times, flasks were
183 opened and flushed with fresh and moist air to return CO₂ concentrations to ambient levels to
184 avoid anoxia (while maintaining the moisture content), before returning them to the incubator.
185 The CO₂ concentrations measured by the GC were converted in μ CO₂-C using equation 1:
186 $\mu\text{g C-CO}_2 = \text{mmol air} \times \text{ppm CO}_2 (\mu\text{mol C} / \text{mol air}) \times 10^{-3} (\text{mol} / \text{mmol}) \times 12 (\mu\text{g C} / \mu\text{mol C})$
187 (equation 1)
188 where “mmol air” corresponds to the millimoles of air present in the flask and was calculated
189 with the ideal gas law (equation 2):
190 $n = PV / RT = (1 \times 100) / (82.05 \times 293)$ (equation 2)
191 As a result, we multiplied our concentrations of CO₂ expressed in percent by 499.16 to
192 convert them in μg C-CO₂.
193 Finally, the 10-week mineralizable SOC (respired-C) was expressed in mg CO₂-C·g⁻¹ SOC to
194 account for differences in the C content of the various layers and sites.

195

196 2.4. Particle size and density SOC fractionation

197 To isolate the particulate organic matter (POM) fraction, samples were first dried at 50 °C for
198 24 h before weighing 25 g and transferred them in polyethylene (PE) 250 mL flasks. We then
199 added 180 mL of 0.5% sodium hexametaphosphate solution and ten 5 mm-diameter glass
200 beads before shaking the samples overnight (50 rpm; 16 h) on an overhead shaker (Reax 2,
201 Heidolph), in order to breakdown soil aggregates. Samples were thoroughly rinsed over a 50-
202 μm mesh with deionized water. The > 50 μm fraction was then transferred back to a dry PE

203 flask with a sodium polytungstate (SPT) solution of density = $1.6 \pm 0.03 \text{ g}\cdot\text{cm}^{-3}$ (Golchin et
204 al., 1994; Crow et al., 2007) and the solution was added up to *circa* 180 mL. The flasks were
205 shaken overhead by hand 10 times and samples were left overnight to settle down after the
206 cap of the flask was rinsed with the SPT solution. The floating material was collected with a
207 spatula and placed over a 50- μm mesh sieve. If necessary some SPT solution was added back
208 to the flask and the previous step was repeated. This time, samples were placed in a centrifuge
209 for 30 minutes to accelerate the separation (2750 rpm or 1250 g, Six et al., 1998). The floating
210 material was again collected with the spatula or pipetted depending on the amount left. This
211 step was repeated if the light fraction was abundant. If not, samples were left to settle down
212 overnight before one last collection. The POM fraction on the sieve was thoroughly rinsed
213 with deionized water throughout the whole process. The sieves and fractions were then placed
214 in the oven at 50 °C for 24 h before being weighed. To account for differences in the C
215 content of the different samples, we calculated the proportion of OC in the POM fraction
216 (POM-C), expressed in $\text{g POM-C}\cdot\text{g}^{-1}$ total SOC.

217

218 2.5. Thermal analysis: Rock-Eval 6

219 The thermal analysis of the samples was performed with a Rock-Eval 6 turbo device (Vinci
220 Technologies, France). Details about the equipment have been previously published (Behar et
221 al., 2001). We adapted the procedure developed for the analysis of soil organic matter by
222 Disnar et al. (2003). Briefly, about 60 (20.7–62.1 depending on the sample's C content) mg of
223 ground sample were exposed to two consecutive thermal treatments, first in a pyrolysis oven
224 (200–650 °C; thermal ramping rate of $30 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$; under N_2 atmosphere) then in a
225 combustion oven (300–850 °C; thermal ramping rate of $20 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$; under laboratory air
226 atmosphere). At the beginning of the pyrolysis, there was an isothermal step (at 200 °C)
227 during 180 seconds during which the free hydrocarbons (HC) were thermovaporized (S1

228 peak). The pyrolysis effluents (mostly HC) were detected and quantified with flame ionization
229 detection, while CO and CO₂ were quantified by infrared detection during both the pyrolysis
230 and oxidation stages (Fig. SI-A1).

231 Two standard RE6 parameters describing SOC bulk chemistry were determined: the hydrogen
232 and oxygen index values (HI and OI_{RE6}). The HI index corresponds to the amount of
233 hydrocarbons formed during thermal pyrolysis of the sample (HC evolved between 200 and
234 650 °C minus the S1 peak) divided by the total SOC content of the sample and is expressed in
235 mg HC·g⁻¹ SOC. It describes the relative enrichment/depletion of SOC in hydrogen-rich
236 moieties. The OI_{RE6} index describes the relative oxidation status of SOC. It was calculated
237 using the equation proposed by Lafargue et al. (1998):

$$238 \quad OI_{RE6} = 16 / 28 \times OI_{CO} + 32 / 44 \times OI_{CO2} \quad (\text{equation 3})$$

239 Where OI_{CO2} corresponds to the CO₂ yielded during thermal pyrolysis of the sample between
240 200 and 400°C divided by the total SOC of the sample and OI_{CO} corresponds to the CO
241 yielded during thermal pyrolysis between 200 and 400–650°C (wherever a minimum of CO
242 production is observed; in the absence of a minimum, the default upper-limit temperature is
243 set at 550 °C) divided by the total SOC of the sample. Thus OI_{RE6} is expressed in mg O₂·g⁻¹
244 SOC.

245 We derived four additional RE6 parameters describing the thermal stability of SOC: (i)
246 T_{50_HC_PYR}, the temperature at which 50% of the HC resulting from the SOM pyrolysis had
247 evolved (ii) the T_{50_CO2_OX}, the temperature at which 50% of the residual SOM was oxidized
248 to CO₂ during the oxidation phase. Because the signal was noisy at the beginning of the
249 pyrolysis, we started the integration for T_{50_HC_PYR} right after the S1 peak. For T_{50_CO2_OX}, the
250 upper limit temperature for signal integration was set at 611 °C to obtain a total CO₂ signal
251 evolved from pure OM without interference of carbonates. Both these T₅₀ temperature
252 parameters and the HI index have been previously shown as good thermal indicators of SOM

253 biogeochemical stability (Gregorich et al., 2015; Barré et al., 2016). We also included two
254 thermal indices previously used in the literature: the (iii) R-index or $(1 - R_{400})$, which
255 correspond to the integrated area of the HC thermogram above 400 °C over the total area of
256 the HC signal (Disnar et al., 2003; Sebag et al., 2016). The R-index estimates the proportion
257 of thermally stable SOC pool and varies between 0 and 1. We hypothesized that the
258 proportion $(1 - R\text{-index})$ would approximate a thermally labile/intermediate (turnover < 20
259 years) SOC pool. Finally, using equation 4, we calculated the (iv) I-index, which is an
260 indicator of the preservation of thermally labile immature SOM (Sebag et al., 2016):

$$261 \log_{10}((A1 + A2) / A3) \quad (\text{equation 4})$$

262 where $A1 + A2$ corresponds to the integrated area of the HC thermogram below 400 °C and
263 $A3$ the integrated area of the HC thermogram between 400 °C and 460 °C.

264 Signal processing of the RE6 thermograms (signal integration and calculation of the
265 $T_{50_HC_PYR}$, $T_{50_CO2_OX}$, R and I indices) was performed with the R environment software v.3.3
266 (R Core Team, 2016) using the hyperSpec (Beleites and Sergio, 2015) and pracma (Borchers,
267 2015) R packages.

268

269 2.6. Calculations and statistical analyses

270 For RE6 analysis and the respiration test, samples with very low C content (< 0.2%) were not
271 considered as the carbon flux they produced during the incubation or the thermal analysis was
272 too low/too close to the limit of detection for reliable determination. This resulted in the
273 selection of $n = 46$ for the soil layer 40–80 cm (total $n = 99$).

274 The mean values of the variables derived from the SOC respiration test, fractionation and RE6
275 analysis for all layer depths were compared using standard non-parametric statistical methods
276 such as Kruskal Wallis test one-way ANOVA by ranks and Wilcoxon signed-rank test.

277 Relationships between the variables derived from the three methods were estimated using

278 Spearman rank correlation as the data did not meet the assumption of normality. Correlation
279 tests were first performed on the whole dataset ($n = 99$) then within the 0–10 cm and the 40–
280 80 cm layers, the three soil types and the two vegetation types individually. All comparisons
281 were considered significant at an alpha value (α) of 0.05. A principal component of analysis
282 (PCA) was performed to detect linear relations between parameters derived from the 3
283 methods. For that purpose, data were log-transformed, centered and scaled. Because the I-
284 index was negative in some instances, we added the equivalent of the smallest I-index value +
285 0.2 to all the I-index values to run the PCA. To determine the number of principal
286 components to select, we looked at the percentage of the total variance explained and used a
287 scree plot and Kaiser's criterion. To analyze the relationship between RE-based and the two
288 classical indicators of the labile SOC pool, we used a simple linear regression model and
289 relied on the Cook's distance to identify potential outliers. All statistical analyses were
290 performed using R 3.3 (R Core Team, 2016) using the factoextra (Kassambara and Mundt,
291 2016) and Hmisc (Harrell et al., 2016) packages.

292

293 3. Results

294 3.1. Respiration test

295 The 10-week mineralizable SOC (respired-C) was expressed in $\text{mg CO}_2\text{-C}\cdot\text{g}^{-1}\text{ SOC}$ to
296 account for differences in the C content of the various layers and sites. Over the course of the
297 10-week incubation, the surface layer (0–10 cm) samples cumulatively respired on average 17
298 $\pm 7.2 \text{ mg CO}_2\text{-C}\cdot\text{g}^{-1}\text{ SOC}$, while the deeper layer (40–80 cm) samples respired $13.4 \pm 6.9 \text{ mg}$
299 $\text{CO}_2\text{-C}\cdot\text{g}^{-1}\text{ SOC}$ (Table 1). There was a significant decrease in respired-C with depth ($p =$
300 0.003), indicating a smaller size of the labile C pool in the deeper layers of our forest soils.
301 Within each soil layer, the large standard deviation (around $7.0 \text{ mg CO}_2\text{-C}\cdot\text{g}^{-1}\text{ SOC}$)
302 illustrates an important inter-site variability.

303

304 3.2. POM fractionation

305 The POM-C fraction (% of total C) decreased by half between layers 0–10 cm and 40–80 cm
306 with $22.6 \pm 7.3\%$ and $11.0 \pm 6.1\%$ respectively. This indicates a significantly ($p < 0.001$)
307 smaller labile C pool in the deeper (40–80 cm) soil layer. POM-C ranged between 12.1–
308 43.0% and 2.5–33.6% in the 0–10 cm and 40–80 cm layers, respectively, illustrating again an
309 important inter-site variability.

310

311 3.3. RE6 thermal analysis

312 There was a significant effect of depth on all RE6 parameters. Particularly, the two T_{50}
313 parameters increased significantly ($p < 0.001$) with depth: 421 ± 9 °C to 448 ± 10 °C and 399
314 ± 9 °C to 431 ± 18 °C (Table 1), for $T_{50_HC_PYR}$ and $T_{50_CO2_OX}$ respectively, corresponding to
315 an increase in the thermal stability of total SOC (*i.e.* a relative decrease in the labile C pool
316 and increase of the stable C pool). OI_{RE6} showed a similar increasing trend ($p < 0.001$) with
317 depth (225 ± 37 – 439 ± 138 mg $O_2 \cdot g^{-1}$ total SOC; Table 1), reflecting a more oxidized SOC in
318 the deeper layers. Conversely, HI decreased significantly ($p < 0.001$) with depth (276 ± 77 –
319 133 ± 34 mg $HC \cdot g^{-1}$ total SOC; Table 1), suggesting a relative depletion of total SOC in H-
320 rich moieties with increased soil depth. The proportion of thermally stable SOC R-index, also
321 experienced a significant increase ($p < 0.001$) with depth (59–69%; Table 1), while the I-
322 index decreased slightly (0.17–0.11; Table 1).

323

324 3.4. Correlations between methods

325 3.4.1. For all samples

326 There were mainly significant and strong correlations between POM-C and the RE6
327 parameters (Table 2). Notably $T_{50_HC_PYR}$, OI_{RE6} and R-index all had a strong negative

328 correlation with POM-C (Spearman $\rho = -0.73, -0.76$ and -0.69 respectively; Table 2; Fig. 3).
329 $T_{50_CO2_OX}$ and HI moderately correlated with POM-C ($\rho = -0.56$ and 0.67) and the I-index
330 had a weak positive relationship with POM-C ($\rho = 0.35$). I-index, $T_{50_HC_PYR}$ and R-index
331 were the only parameters that were significantly related to respired-C, with a weak correlation
332 ($\rho = 0.32, \rho = -0.26$ and -0.31 respectively; Table 2). The two classical methods of
333 estimation of labile SOC (respired-C and POM-C) were weakly positively ($\rho = 0.20$; Table 2;
334 Fig. SI-B1 a) and indeed only marginally ($p = 0.051$) related.

335 To describe the similarity or dissimilarity in the different indicators of SOC lability, we
336 conducted a principal components analysis (PCA). As shown by the correlation test, T_{50}
337 $_HC_PYR$ and R-index on the one hand and OI_{RE6} and HI on the other hand were highly
338 correlated ($\rho = 0.93$ and -0.92 respectively; Table 2). We thus decided to conduct the PCA
339 using only the 6 following explanatory variables = respired-C; POM-C; HI; $T_{50_CO2_OX}$; T_{50}
340 $_HC_PYR$; I-index). The first two principal components (PC) explained approximately 73% of
341 the total variance, with 53% explained by the first and 20% explained by the second PC,
342 respectively (Fig. 2). PC1 clearly separated surface (0–10 cm) from deeper (40–80 cm) soil
343 samples. Along PC1, POM-C and HI showed moderate negative loadings (-0.47 and -0.46
344 respectively; Table SI-B1) while $T_{50_HC_PYR}$ and $T_{50_CO2_OX}$ had moderate positive loadings
345 (0.53 and 0.45 ; Table SI-B1). Respired-C and the I-index showed strong positive loadings
346 along PC2 (0.55 and 0.69 ; Table SI-B1), while they showed very weak negative loadings
347 along PC1. Samples from layers 0–10 and 40–80 cm did not significantly differ along the
348 second PC.

349

350 3.4.2. For the 0–10 cm and 40–80 cm layer separately

351 These global correlations prompted us into looking at the influence of soil depth on the
352 different parameters. The paired correlations between the 8 parameters differed in surface (0–

353 10 cm) and deep (40–80 cm) layers (Table 2). Specifically, the respired-C in the surface
354 layers was weakly and negatively related to POM-C ($\rho = -0.29$; Table 2). Conversely in the
355 deep layers, respired-C and POM-C were moderately and positively correlated, as it would be
356 expected ($\rho = 0.47$; Table 2; Fig. SI-B1 a). In the surface layers, HI and OI_{RE6} were also
357 moderately (negatively and positively, respectively; Table 2) correlated to respired-C, while
358 in the deep layers we observed again this negative and moderate correlation between
359 $T_{50_HC_PYR}$ and respired-C. For POM-C, we found the same negative correlations with
360 $T_{50_HC_PYR}$ and OI_{RE6} as in the “all samples” comparison but they were less strong ($\rho = -0.35$
361 to -0.42 ; Table 2). In the surface layer, the C/N ratio, pH and clay content had all moderate
362 and significant correlations with respired-C and $T_{50_HC_PYR}$ (Table 2). These correlations were
363 absent in the 40–80 cm layer.

364 We also looked at the evolutions of the correlations as a function of vegetation and soil types,
365 but there were no change as drastic as the ones we observed with depth (Table SI-C1). In both
366 cases the changes affected only the correlations between respired-C and the other parameters.
367 For instance, in coniferous plots, respired-C was weakly to moderately positively correlated to
368 clay content ($\rho = 0.27$) and pH (0.37) while those correlations were absent in deciduous plots
369 (Table SI-C1). For the soil types, POM-C and respired-C were moderately and positively
370 correlated in Podzols (0.42) and eutric Cambisols (0.46) but not in dystric Cambisols.
371 Furthermore, in eutric Cambisols, respired-C was moderately and negatively correlated with
372 $T_{50_CO2_OX}$ (-0.54), R-index (-0.50) and pH (-0.57 ; Table SI-C1).

373

374 4. Discussion

375 4.1. Relationships between respiration test and POM fractionation

376 Unexpectedly the two classical indicators of the labile SOC fraction correlated only weakly
377 and marginally when considering all our samples.

378 POM-C is considered as a labile SOC fraction (Wander, 2004; Haynes, 2005; von Lützow et
379 al., 2007), and we thus expected it would correlate significantly and strongly with the
380 respired-C fraction isolated by the 10-week laboratory respiration test. Indeed, in his review,
381 Haynes (2005) mentioned several studies reporting a positive and usually strong correlation
382 between the respired-C and the POM-C fractions, appearing to support that hypothesis.
383 However when carefully considering these papers (Janzen et al., 1992; Hassink, 1995;
384 Campbell et al., 1999a; Campbell et al., 1999b; Wander and Bidart, 2000) and others (Liang
385 et al., 2003; Hassan et al., 2016; Li et al., 2016), it emerged that the presented data were not
386 normalized by the total SOC concentration of the samples. Without normalization it could be
387 argued that the positive correlation between the POM-C and the respired-C fractions was in
388 fact driven by variations in total SOC concentration and not SOC biogeochemical stability. It
389 also prevented comparisons among studies, given the important difference in SOC
390 concentration.

391 The hypothesis of a positive correlation between the sizes of the labile SOC pool estimated by
392 respiration test and POM fractionation schemes has actually not been properly tested on
393 multiple sites, using SOC normalized data as it has been done in the present study. Indeed, the
394 few studies that have reported moderate to strong positive correlations between the sizes of
395 the labile SOC pool estimated by respiration test and POM fractionation were conducted on
396 similar soils under different management (*e.g.*, Alvarez and Alvarez, 2000) or correlations
397 were made within sites (*e.g.*, Janzen et al., 1992). When combining results from all sites, the
398 correlation appeared to be weaker and it can therefore be hypothesized that in our study the
399 weak and marginally significant correlation between POM-C and respired-C was partially due
400 to the large inter-sites variability of soil properties for our sample set (Table SI-A.1).

401 Finally, the labile SOC pools estimated by the two classical methods were so different in size
402 (*i.e.* the labile SOC pool estimated as respired-C was about an order of magnitude smaller

403 than the one estimated as POM-C; Table 1) that it is not surprising that the correlation did not
404 hold specifically when introducing a lot of inter-sites variability. This constitutes another
405 explanation to the lack of correlation between these two indicators of the size of the labile C
406 pool. The two methods appeared to measure different SOC fractions (*i.e.* different sizes)
407 (Table 2 and Fig. 2) that correspond to different SOC lability (*i.e.* mean residence time).

408

409 4.2. Relationships between RE6 parameters and POM-C and respired-C

410 Our RE6 results agreed with previous observations of thermal indicators of SOC lability. For
411 instance, Sebag et al. (2016) reported a trend of decreasing HI and increasing OI_{RE6} with soil
412 depth. Trends of decreasing HI and increasing $T_{50_CO2_OX}$ were observed with increasing time
413 since beginning of bare fallow experiments, which corresponded with a progressive
414 decomposition of the labile SOC pool (Barré et al., 2016).

415 Our correlations between the RE6 parameters and the POM-C fraction were close to those
416 previously reported by Saenger et al. (2015). They indeed obtained a moderate positive
417 correlation ($R^2 = 0.50$) between the labile SOC pool stocks derived from a SOC fractionation
418 scheme isolating POM-C, and the thermally labile SOC pool stocks derived from RE6
419 indices. We found a similar strong positive correlation between the proportion of labile SOC
420 ($1-R-index$) and POM-C. The strong relationship between $T_{50_HC_PYR}$ and R-index could
421 likely be explained as $T_{50_HC_PYR}$ for our samples were very close to the 400 °C threshold used
422 for the calculation of the R-index. As hypothesized we were able to derive a quantitative
423 relationship between some of our RE6 parameters and POM-C (Fig. SI-B1 b–d). The best
424 model was obtained for $T_{50_HC_PYR}$ ($R^2 = 0.52$; Fig. 3), while HI, R-index and OI_{RE6} were still
425 moderately good predictors of POM-C ($R^2 = 0.42–0.47$ (Fig. SI-B1 b–d)).

426 Nevertheless no strong relationship between respired-C and the other parameters could be
427 established. Our correlations between the RE6 parameters and respired-C were smaller than

428 those previously reported by Gregorich et al. (2015). This could be explained by the fact that
429 their study was, by design, very restricted in terms of its soil properties variability and also
430 only considered surface soils (0–10 cm), in which the C/N ratios were around 10.

431 Previous studies have also demonstrated that RE6 can be used to look at changes in the size of
432 the SOC labile pools with time. For instance, RE6 was able to describe the decrease in the
433 labile SOC pool in long-term bare fallows (Barré et al., 2016). Besides, RE6 captured
434 differences in the size of the labile SOC pools in various land-uses and soil types over a small
435 landscape (Saenger et al., 2015). Our results thus contradict the conclusions from Schiedung
436 et al. (2017) who found no relationship between the thermally labile SOC (200–400 °C) and
437 the C in the POM fractions. The latter (free and occluded POM—obtained by sonication)
438 were indeed more stable at lower oxidation temperatures (300–350 °C) than the mineral-
439 associated fraction. However, their analytical method was different from RE6 protocols: the
440 thermal analysis they used was entirely realized under aerobic conditions (oxidation only),
441 their temperature range was limited (only up to 400 °C) and they used a 50–100 °C
442 temperature step every 15 minutes rather than a constant thermal ramping rate (standard in
443 most thermal studies). For all these reasons, it is likely that their thermal indices differ greatly
444 from our RE6-derived parameters. Moreover their study was based on topsoils (0–10 cm) of
445 only three study sites.

446 The good approximation of the POM-C fraction by RE6 we reported constitutes a very
447 promising result. POM-C mean residence time (< 20 years in temperate conditions in the
448 absence of an important charcoals contribution; *e.g.*, Trumbore and Zheng, 1996; Balesdent,
449 1996; Balesdent et al., 1998; Baisden et al., 2002; Schrumpf and Kaiser, 2015) and its size (11
450 to 23% of total SOC in this study) are much larger than the one of the respired-C fraction, and
451 is thus more meaningful regarding SOC stock evolutions upon changes of land management.

452 This suggests that RE6 could be used to determine the size of the labile SOC pool with a
453 decadal mean residence time.

454

455 4.3. Effects of depth on correlations between the three methods estimating labile SOC
456 Labile SOC content usually decreases with depth (*e.g.*, Lorenz and Lal, 2005; Jenkinson et al.,
457 2008). Such a trend was observed with the three methods used in the present study. Indeed,
458 with depth, we observed a decrease in respired-C (respiration test), POM-C (POM
459 fractionation) and HI alongside with an increase in $T_{50_HC_PYR}$ and R-index that all signified
460 the expected decrease in the size the labile SOC pool. Concurrently, OI_{RE6} increased with
461 depth, confirming the increase in SOC oxidative state with increasing decomposition
462 (Hockaday et al., 2009; von Lützow and Kögel-Knabner, 2010; Hockaday et al., 2015).
463 But more importantly, depth affected the correlations between the methods. The lack of
464 correlation between two classical indicators of the labile SOC fraction previously mentioned
465 appeared to originate from opposite trends in the surface and deep layers. In the 0–10 cm
466 layer we observed a surprising negative (but weak) correlation between respired-C and POM-
467 C while the expected positive and moderate correlation between the two indicators was found
468 only in the deep layers. The differences in the sign of the correlations between respired-C and
469 POM-C in the two considered layers (0–10 cm and 40–80 cm) may be related to pedological
470 factors that can limit SOC mineralization in surface horizons. Indeed, the high C/N ratio
471 found in the surface layer (Table SI-A1) is far from the expected C/N of the microorganisms
472 and this lack of N may limit SOC respiration. Similarly, surface layers are on average more
473 acidic (Table SI-A1) than deep layers which can also reduce SOC respiration. We could
474 hypothesize that respired-C and POM-C correlate only when environmental conditions do not
475 limit SOC mineralization explaining the absence of correlation in the acidic N-poor 0–10 cm
476 layer. The significant correlations observed between respired-C and the C/N ratio, pH and the

477 clay content in the surface layer (Table 2) supports that hypothesis. This opposite behavior in
478 the two layers also affected $T_{50_HC_PYR}$, which was not significantly correlated to respired-C in
479 the surface layer while the two parameters were moderately and negatively correlated in the
480 40–80 cm layer (Table 2). These observations matched those from Peltre et al. (2013) who
481 reported conflicting relationships between the parameter DSC-T50 (temperature at which half
482 of the energy is released in differential scanning calorimetry) and mean soil respiration rates
483 in two sets of high and low SOC content. Their DSC-T50 values were indeed negatively
484 correlated with the respiration values for the low-C soils, whereas there was only a marginal
485 positive correlation between the two parameters for the high-C soils. Their two groups were
486 characterized by soil properties similar to our 0–10 and 40–80 cm layers: their low-C set
487 consisted of samples with a higher pH and lower mean C/N ratio than those of the high-C.
488 Similarly to our 0–10 cm samples, soils in their high-C set had a greater C concentration than
489 those in the low-C set for similar clay contents (Table SI-A1). This would also explain why
490 our results differ from those of Gregorich et al. (2015). In the deep layer, in which the C/N
491 ratios are closer to those reported by Gregorich et al. (2015), we observed the same positive
492 correlation they reported albeit less strong.

493 Vegetation and soil types did not seem to have affected the correlations between the three
494 methods we tested as much as depth did. However, these environmental factors are likely
495 drivers of the size labile SOC pool as they have been shown to significantly influence RE6
496 parameters (*e.g.*, Disnar et al., 2003; Sebag et al., 2006).

497

498 4.4. Towards high-throughput information on SOC biogeochemical stability using RE6 499 analysis

500 Respiration tests and POM fractionation schemes are both time consuming, thus limiting the
501 number of samples and/or replicates that are analyzed. With the RE6 set-up used in this study,

502 about 20 samples per day can be analyzed, and it requires only limited operator interventions
503 (soil weighing and routine supervision of the RE6 analyzer).

504 The lack of normalization in many studies using respiration tests and POM fractionation is an
505 important issue and it should be recommended for further studies to include normalized data
506 (% of TOC) when presenting their results. Moreover, despite the fact that POM-C and
507 respired-C are considered as standard estimates of the labile SOC pool, the temperature and/or
508 duration of incubations often varied from one study to the other. Similarly for the POM-C
509 fraction, the density of the solution used for the flotation may drastically differ among studies.
510 This makes data comparison almost impossible. In that regard, while the harmonization of
511 RE6 programs would probably be much easier to implement than respiration tests or POM
512 fractionation protocols as the number of users is still limited, protocol standardization is an
513 important and pressing goal to achieve and this rather quickly as the method starts to gain
514 interest.

515 RE6 analysis is thus a rapid technique that captures differences in the labile SOC pool as well
516 as other classical techniques. While the understanding of the underlying processes linking
517 SOC thermal stability observed with RE6 and the laboratory or *in-situ* biogeochemical
518 stability of SOC is not fully uncovered and further studies are needed, RE6 analysis appears
519 like a very promising method to provide quick and inexpensive information on the labile SOC
520 pool. Hence, it could constitute a standard method to complement C stock measurements in
521 soil monitoring programs.

522

523

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532 **Reference list**

533 Albrecht, R., Sebag, D., Verrecchia, E., 2015. Organic matter decomposition: Bridging the
534 gap between Rock-Eval pyrolysis and chemical characterization (CPMAS ¹³C NMR).

535 *Biogeochemistry* 122, 101–111.

536 Alvarez, R., Alvarez, C.R., 2000. Soil organic matter pools and their associations with carbon
537 mineralization kinetics. *Soil Science Society of America Journal* 64, 184–189.

538 Baisden, W.T., Amundson, R., Cook, A.C., Brenner, D.L., 2002. Turnover and storage of C
539 and N in five density fractions from California annual grassland surface soils. *Global*
540 *Biogeochemical Cycles* 16, 64-61; 64–16.

541 Balesdent, J., 1996. The significance of organic separates to carbon dynamics and its
542 modelling in some cultivated soils. *European Journal of Soil Science* 47, 485–494.

543 Balesdent, J., Besnard, E., Arrouays, D., Chenu, C., 1998. The dynamics of carbon in particle-
544 size fractions of soil in a forest-cultivation sequence. *Plant and Soil* 201, 49–57.

545 Barré, P., Plante, A.F., Cécillon, L., Lutfalla, S., Baudin, F., Christensen, B.T., Eglin, T.,
546 Fernandez, J.M., Houot, S., Kätterer, T., Le Guillou, C., Macdonald, A., van Oort, F.,
547 Chenu, C., 2016. The energetic and chemical signatures of persistent soil organic matter.
548 *Biogeochemistry* 130, 1–12.

549 Behar, F., Beaumont, V., Penteadó, D.B., 2001. Rock-Eval 6 technology: Performances and
550 developments. *Oil & Gas Science and Technology - Rev.IFP* 56, 111–134.

551 Beleites, C., Sergio, V., 2015. hyperSpec: A Package to Handle Hyperspectral Data Sets in R.

552 Borchers, H.W., 2015. *Pracma: Practical numerical math functions*.

553 Brêthes, A., Ulrich, E., Lanier, M., 1997. *RENECOFOR : Caractéristiques Pédologiques Des*
554 *102 Peuplements Du Réseau : Observations De 1994/95*. Office national des forêts,
555 Département des recherches techniques, Fontainebleau, France, 573 pp.

556 Campbell, C.A., Biederbeck, V.O., Wen, G., Zentner, R.P., Schoenau, J., Hahn, D., 1999a.
557 Seasonal trends in selected soil biochemical attributes: Effects of crop rotation in the
558 semiarid prairie. *Canadian Journal of Soil Science* 79, 73–84.

559 Campbell, C.A., Lafond, G.P., Biederbeck, V.O., Wen, G., Schoenau, J., Hahn, D., 1999b.
560 Seasonal trends in soil biochemical attributes: Effects of crop management on a black
561 chernozem. *Canadian Journal of Soil Science* 79, 85–97.

562 Copard, Y., Di-Giovanni, C., Martaud, T., Albéric, P., Olivier, J., 2006. Using Rock-Eval 6
563 pyrolysis for tracking fossil organic carbon in modern environments: Implications for the
564 roles of erosion and weathering. *Earth Surface Processes and Landforms* 31, 135–153.

565 Crow, S.E., Swanston, C.W., Lajtha, K., Brooks, J.R., Keirstead, H., 2007. Density
566 fractionation of forest soils: Methodological questions and interpretation of incubation
567 results and turnover time in an ecosystem context. *Biogeochemistry* 85, 69–90.

568 Di-Giovanni, C., Disnar, J.R., Bichet, V., Campy, M., Guillet, B., 1998. Geochemical
569 characterization of soil organic matter and variability of a postglacial detrital organic supply
570 (Chaillexon lake, France). *Earth Surface Processes and Landforms* 23, 1057–1069.

571 Disnar, J.-R., Guillet, B., Keravis, D., Di-Giovanni, C., Sebag, D., 2003. Soil organic matter
572 (SOM) characterization by Rock-Eval pyrolysis: Scope and limitations. *Organic*
573 *Geochemistry* 34, 327–343.

574 Feng, W., Shi, Z., Jiang, J., Xia, J., Liang, J., Zhou, J., Luo, Y., 2016. Methodological
575 uncertainty in estimating carbon turnover times of soil fractions. *Soil Biology and*
576 *Biochemistry* 100, 118–124.

577 Gillespie, A.W., Sanei, H., Diochon, A., Ellert, B.H., Regier, T.Z., Chevrier, D., Dynes, J.J.,
578 Tarnocai, C., Gregorich, E.G., 2014. Perennially and annually frozen soil carbon differ in
579 their susceptibility to decomposition: Analysis of subarctic earth hummocks by bioassay,
580 XANES and pyrolysis. *Soil Biology and Biochemistry* 68, 106–116.

581 Golchin, A., Oades, J.M., Skjemstad, J.O., Clarke, P., 1994. Study of free and occluded
582 particulate organic matter in soils by solid state ^{13}C CP/MAS NMR spectroscopy and
583 scanning electron microscopy. *Australian Journal of Soil Research* 32, 285–309.

584 Gregorich, E.G., Gillespie, A.W., Beare, M.H., Curtin, D., Sanei, H., Yanni, S.F., 2015.
585 Evaluating biodegradability of soil organic matter by its thermal stability and chemical
586 composition. *Soil Biology and Biochemistry* 91, 182–191.

587 Harrell, F.E.J., Dupont, C., and others, 2016. Hmisc: Harrell miscellaneous.

588 Harris, D., Horwath, W.R., van Kessel, C., 2001. Acid fumigation of soils to remove
589 carbonates prior to total organic carbon or CARBON-13 isotopic analysis. *Soil Science*
590 *Society of America Journal* 65, 1853–1856.

591 Hassan, W., Bashir, S., Ahmed, N., Tanveer, M., Shah, A.N., Bano, R., David, J., 2016.
592 Labile organic carbon fractions, regulator of CO₂ emission: Effect of plant residues and
593 water regimes. *CLEAN - Soil, Air, Water* 44, 1358–1367.

594 Hassink, J., 1995. Density fractions of soil macroorganic matter and microbial biomass as
595 predictors of C and N mineralization. *Soil Biology and Biochemistry* 27, 1099–1108.

596 Haynes, R.J., 1986. Chapter 2 - the Decomposition Process: Mineralization, Immobilization,
597 Humus Formation, and Degradation. In: Haynes, R.J. (Ed.), *Mineral Nitrogen in the Plant–*
598 *Soil System*. Academic Press, pp. 52–126.

599 Haynes, R.J., 2005. Labile organic matter fractions as central components of the quality of
600 agricultural soils: An overview. *Advances in Agronomy* 85, 221–268.

601 Hetényi, M., Nyilas, T., Sajgó, C., Brukner-Wein, A., 2006. Heterogeneous organic matter
602 from the surface horizon of a temperate zone marsh. *Organic Geochemistry* 37, 1931–1942.

603 Hetényi, M., Nyilas, T., Tóth, T.M., 2005. Stepwise Rock-Eval pyrolysis as a tool for typing
604 heterogeneous organic matter in soils. *Journal of Analytical and Applied Pyrolysis* 74, 45–
605 54.

606 Hockaday, W.C., Gallagher, M.E., Masiello, C.A., Baldock, J.A., Iversen, C.M., Norby, R.J.,
607 2015. Forest soil carbon oxidation state and oxidative ratio responses to elevated CO₂.
608 *Journal of Geophysical Research: Biogeosciences* 120, 1797–1811.

609 Hockaday, W.C., Masiello, C.A., Randerson, J.T., Smernik, R.J., Baldock, J.A., Chadwick,
610 O.A., Harden, J.W., 2009. Measurement of soil carbon oxidation state and oxidative ratio by
611 ¹³C nuclear magnetic resonance. *Journal of Geophysical Research* 114.

612 IUSS Working Group, 2015. World reference base for soil resources 2014 (update 2015),
613 international soil classification system for naming soils and creating legends for soil maps.
614 *World Soil Resources Reports No. 106*. FAO, Rome.

615 Janzen, H.H., Campbell, C.A., Brandt, S.A., Lafond, G.P., Townley-Smith, L., 1992. Light-
616 fraction organic matter in soils from long-term crop rotations. *Soil Science Society of*
617 *America Journal* 56, 1799–1806.

618 Jenkinson, D.S., Poulton, P.R., Bryant, C., 2008. The turnover of organic carbon in subsoils.
619 Part 1. Natural and bomb radiocarbon in soil profiles from the Rothamsted long-term field
620 experiments. *European Journal of Soil Science* 59, 391–399.

621 Jonard, M., Nicolas, M., Coomes, D.A., Caignet, I., Saenger, A., Ponette, Q., 2017. Forest
622 soils in France are sequestering substantial amounts of carbon. *Science of The Total*
623 *Environment* 574, 616–628.

624 Kassambara, A., Mundt, F., 2016. Factoextra: Extract and visualize the results of multivariate
625 data analyses.

626 Lafargue, E., Marquis, F., Pillot, D., 1998. Rock-Eval 6 applications in hydrocarbon
627 exploration, production, and soil contamination studies. *Oil & Gas Science and Technology*
628 - *Revue IFP* 53, 421–437.

629 Li, X.J., Li, X.R., Wang, X.P., Yang, H.T., 2016. Changes in soil organic carbon fractions
630 after afforestation with xerophytic shrubs in the tengger desert, northern china. *European*
631 *Journal of Soil Science* 67, 184–195.

632 Liang, B.C., McConkey, B.G., Schoenau, J., Curtin, D., Campbell, C.A., Moulin, A.P.,
633 Lafond, G.P., Brandt, S.A., Wang, H., 2003. Effect of tillage and crop rotations on the light
634 fraction organic carbon and carbon mineralization in chernozemic soils of Saskatchewan.
635 *Canadian Journal of Soil Science* 83, 65–72.

636 Lorenz, K., Lal, R., 2005. The depth distribution of soil organic carbon in relation to land use
637 and management and the potential of carbon sequestration in subsoil horizons. *Advances in*
638 *Agronomy* 88, 35–66.

639 Luo, Y., Ahlström, A., Allison, S.D., Batjes, N.H., Brovkin, V., Carvalhais, N., Chappell, A.,
640 Ciais, P., Davidson, E.A., Finzi, A., Georgiou, K., Guenet, B., Hararuk, O., Harden, J.W.,
641 He, Y., Hopkins, F., Jiang, L., Koven, C., Jackson, R.B., Jones, C.D., Lara, M.J., Liang, J.,
642 McGuire, A.D., Parton, W., Peng, C., Randerson, J.T., Salazar, A., Sierra, C.A., Smith, M.J.,
643 Tian, H., Todd-Brown, K.E.O., Torn, M., van Groenigen, K.J., Wang, Y.P., West, T.O.,
644 Wei, Y., Wieder, W.R., Xia, J., Xu, X., Xu, X., Zhou, T., 2016. Toward more realistic
645 projections of soil carbon dynamics by earth system models. *Global Biogeochemical Cycles*
646 30, 40–56.

647 Peltre, C., Fernández, J.M., Craine, J.M., Plante, A.F., 2013. Relationships between biological
648 and thermal indices of soil organic matter stability differ with soil organic carbon level.
649 *SSSAJ* 77, 2020–2028.

650 Ponette, Q., Ulrich, E., Brêthes, A., Bonneau, M., Lanier, M., 1997. RENECOFOR - Chimie
651 Des Sols Dans Les 102 Peuplements Du Réseau : Campagne De Mesures 1993-95. ONF,
652 Département des recherches techniques, Fontainebleau, France, 427 pp.

653 R Core Team, 2016. R: A language and environment for statistical computing.

654 Saenger, A., Cécillon, L., Poulencard, J., Bureau, F., De Daniéli, S., Gonzalez, J., Brun, J.,
655 2015. Surveying the carbon pools of mountain soils: A comparison of physical fractionation
656 and Rock-Eval pyrolysis. *Geoderma* 241–242, 279–288.

657 Schiedung, M., Don, A., Wordell-Dietrich, P., Alcántara, V., Kuner, P., Guggenberger, G.,
658 2017. Thermal oxidation does not fractionate soil organic carbon with differing biological
659 stabilities. *Journal of Plant Nutrition and Soil Science* 180, 18–26.

660 Schrumpf, M., Kaiser, K., 2015. Large differences in estimates of soil organic carbon
661 turnover in density fractions by using single and repeated radiocarbon inventories.
662 *Geoderma* 239–240, 168–178.

663 Sebag, D., Disnar, J.R., Guillet, B., Di Giovanni, C., Verrecchia, E.P., Durand, A., 2006.
664 Monitoring organic matter dynamics in soil profiles by ‘Rock-Eval pyrolysis’: Bulk
665 characterization and quantification of degradation. *European Journal of Soil Science* 57,
666 344–355.

667 Sebag, D., Verrecchia, E.P., Cécillon, L., Adatte, T., Albrecht, R., Aubert, M., Bureau, F.,
668 Cailleau, G., Copard, Y., Decaens, T., Disnar, J.-R., Hetényi, M., Nyilas, T., Trombino, L.,
669 2016. Dynamics of soil organic matter based on new Rock-Eval indices. *Geoderma* 284,
670 185–203.

671 Six, J., Elliott, E.T., Paustian, K., Doran, J.W., 1998. Aggregation and soil organic matter
672 accumulation in cultivated and native grassland soils. *Soil Science Society of America*
673 *Journal* 62, 1367–1377.

674 Trumbore, S.E., Zheng, S., 1996. Comparison of fractionation methods for soil organic matter
675 ¹⁴C analysis. *Radiocarbon* 38, 219–229.

676 Trumbore, S.E., Chadwick, O.A., Amundson, R., 1996. Rapid exchange between soil carbon
677 and atmospheric carbon dioxide driven by temperature change. *Science* 272, 393–396.

678 Ulrich, E., 1995. Le réseau RENECOFOR : Objectifs et réalisation. *Revue forestière française*
679 47, 107–124.

680 von Lützow, M., Kögel-Knabner, I., 2010. Response to the concept paper: 'What is
681 recalcitrant soil organic matter?' by Markus Kleber. *Environmental Chemistry* 7, 333–335.

682 von Lützow, M., Kögel-Knabner, I., Ekschmitt, K., Flessa, H., Guggenberger, G., Matzner,
683 E., Marschner, B., 2007. SOM fractionation methods: Relevance to functional pools and to
684 stabilization mechanisms. *Soil Biology and Biochemistry* 39, 2183–2207.

685 Wander, M.M., Bidart, M.G., 2000. Tillage practice influences on the physical protection,
686 bioavailability and composition of particulate organic matter. *Biology and Fertility of Soils*
687 32, 360–367.

688 Wander, M., 2004. Soil Organic Matter Fractions and their Relevance to Soil Function. In:
689 Magdoff, F., Weil, R.R. (Eds.), *Soil Organic Matter in Sustainable Agriculture*. CRC Press,
690 pp. 67–102.

691

692 **Figure captions**

693 **Fig. 1.** (a) Location of the 53 study sites front the French national network for the long term
694 monitoring of forest ecosystems (RENECOFOR); (b) Number of samples by depths and
695 analyses realized. Plot locations are also available via the Interactive Map Viewer.

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697 **Fig. 2.** Biplot of a principal components analysis (PCA) showing the loadings of the 6
698 parameters estimating the labile SOC (red arrows) and the 99 soil samples for the two layers
699 (0–10 cm, n = 53; 40–80 cm, n = 46) along the first two principal component axes (PC1 and
700 PC2). The 95% ellipses for both soil layers were added for information; the circle in the
701 center corresponds to the circle of correlations.

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703 **Fig. 3.** The proportion of OC in the POM fraction (POM-C) as a function of $T_{50_HC_PYR}$ (the
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Table 1

Table 1. Mean (and minimum; maximum; standard deviation) of the RE6 (HI, OI_{RE6} , $T_{50_HC_PYR}$, $T_{50_CO2_OX}$, R-index, I-index), respiration test (10-week mineralizable C, respired-C) and POM fractionation (POM-C) parameters, as well as the bulk SOC content for each soil layer (0–10 and 40–80 cm) of the 53 RENECOFOR plots.

	n	HI (mg HC / g TOC)	OI_{RE6} (mg O_2 / g TOC)	$T_{50_HC_PYR}$ (°C)	$T_{50_CO2_OX}$ (°C)	R-index (% SOC)	I-index	Respired-C (mg CO_2 -C / g SOC)	POM-C (% SOC)	SOC (%) (bulk soil)
0–10 cm	53	276 (161; 443; 77)	225 (161; 288; 37)	421 (400; 439; 9)	399 (382; 422; 9)	59 (50; 68; 4)	0.17 (0.00; 0.32; 0.07)	17.0 (4.4; 33.7; 7.2)	22.6 (12.1; 43.0; 7.3)	5.1 (1.2; 15.1; 2.7)
40–80 cm	46	133 (75; 202; 34)	439 (236; 875; 138)	448 (421; 480; 10)	431 (390; 470; 18)	69 (59; 79; 5)	0.11 (-0.18; 0.39; 0.14)	13.4 (3.6; 32.2; 6.9)	11.0 (2.5; 33.6; 6.1)	0.9 (0.2; 3.9; 0.8)

Table 2

Table 2. Spearman correlation coefficients between 10-week mineralizable SOC (respired-C), the proportion of OC in the POM fraction (POM-C), the RE6 parameters and the C/N ratio, pH and clay content of the bulk soil, for both the 0–10 cm (n = 53) and 40–80 cm (n = 46) layers and each layer individually. Significance is indicated as follows: ***: $p < 0.001$; **: $p < 0.01$; *: $p < 0.05$. The very high correlations are marked in bold.

All (n = 99)	respired-C	POM-C	T _{50_HC_PYR}	T _{50_CO2_OX}	HI	OI _{RE6}	I-index	R-index	C/N	pH
POM-C	0.20									
T _{50_HC_PYR}	-0.26**	-0.73***								
T _{50_CO2_OX}	-0.16	-0.56***	0.76***							
HI	0.06	0.67***	-0.78***	-0.66***						
OI _{RE6}	-0.02	-0.76***	0.78***	0.63***	-0.92***					
I-index	0.32**	0.35***	-0.48***	-0.17	0.10	0.06				
R-index	-0.31**	-0.69***	0.93***	0.64***	-0.64***	0.67***	-0.74***			
C/N	-0.13	0.63***	-0.55***	-0.52***	0.67***	-0.78***	0.16	-0.50***		
pH	0.23*	-0.55***	0.49***	0.44***	-0.57***	0.66***	-0.27**	0.49***	-0.64***	
clay content	0.20	-0.19	0.04	-0.06	-0.17	0.31**	-0.22**	0.13	-0.49***	0.43***
0–10 cm	respired-C	POM-C	T _{50_HC_PYR}	T _{50_CO2_OX}	HI	OI _{RE6}	I-index	R-index	C/N	pH
POM-C	-0.29*									
T _{50_HC_PYR}	0.12	-0.44***								
T _{50_CO2_OX}	0.13	-0.19	0.45***							
HI	-0.43**	0.32*	-0.30*	0.07						
OI _{RE6}	0.52***	-0.41**	0.40**	-0.06	-0.91***					
I-index	0.11	0.37**	-0.87***	-0.43**	0.04	-0.17				
R-index	0.07	-0.44**	0.99***	0.44***	-0.24	-0.35*	-0.92***			
C/N	-0.51***	0.56***	-0.55***	-0.19	0.67***	-0.80***	0.37**	-0.52***		
pH	0.62***	-0.35**	0.46***	0.36**	-0.61***	0.70***	-0.25	0.42**	-0.70***	
clay content	0.43**	-0.29*	0.44**	-0.03	-0.71***	0.75***	-0.31*	0.43**	-0.70***	0.60***

40–80 cm	respired-C	POM-C	T _{50_HC_PYR}	T _{50_CO2_OX}	HI	OI _{RE6}	I-index	R-index	C/N	pH
POM-C	0.47***									
T _{50_HC_PYR}	-0.41**	-0.35*								
T _{50_CO2_OX}	-0.01	-0.01	0.26							
HI	-0.03	0.06	-0.24	-0.24						
OI _{RE6}	0.13	-0.42**	0.10	0.22	-0.47***					
I-index	0.41**	0.13	-0.19	0.43**	-0.49***	0.40**				
R-index	-0.52***	-0.28	0.64***	-0.19	0.20	-0.20	-0.85***			
C/N	-0.17	0.30*	0.10	-0.18	0.25	-0.65***	0.30*	0.23		
pH	0.03	-0.34*	-0.03	-0.18	0.11	0.25	-0.07	0.06	-0.27	
clay content	-0.08	-0.42**	-0.01	0.11	-0.07	0.62***	-0.15	0.17	-0.59***	0.35*

Figure 1
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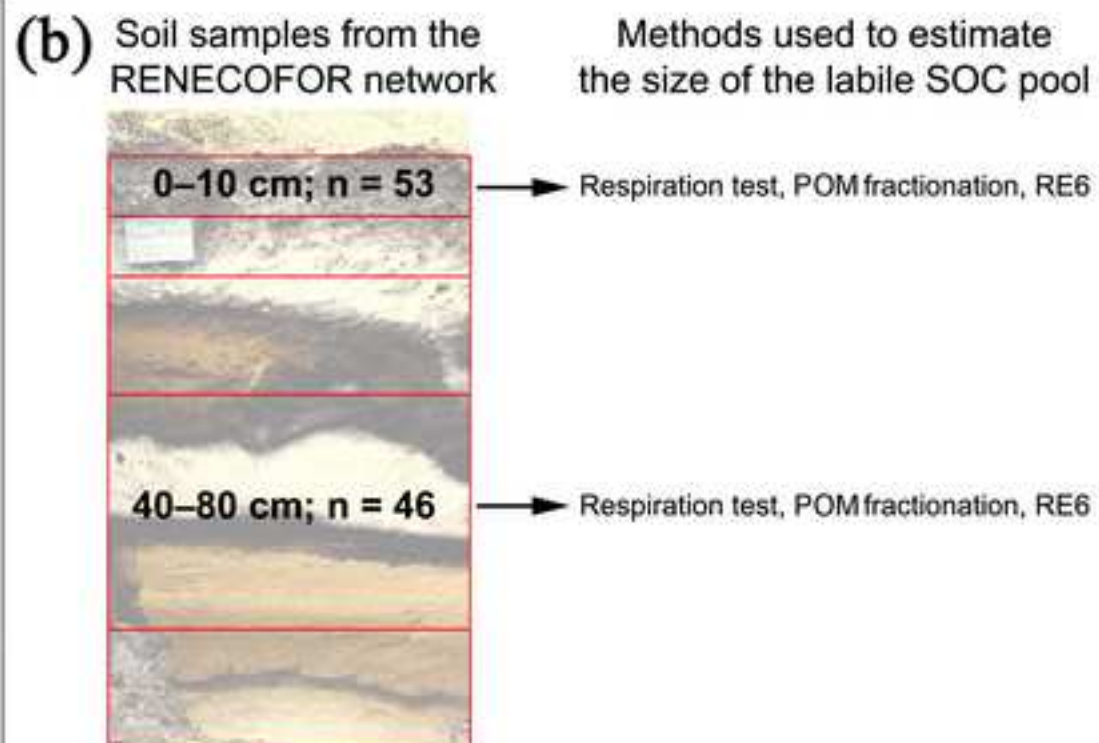
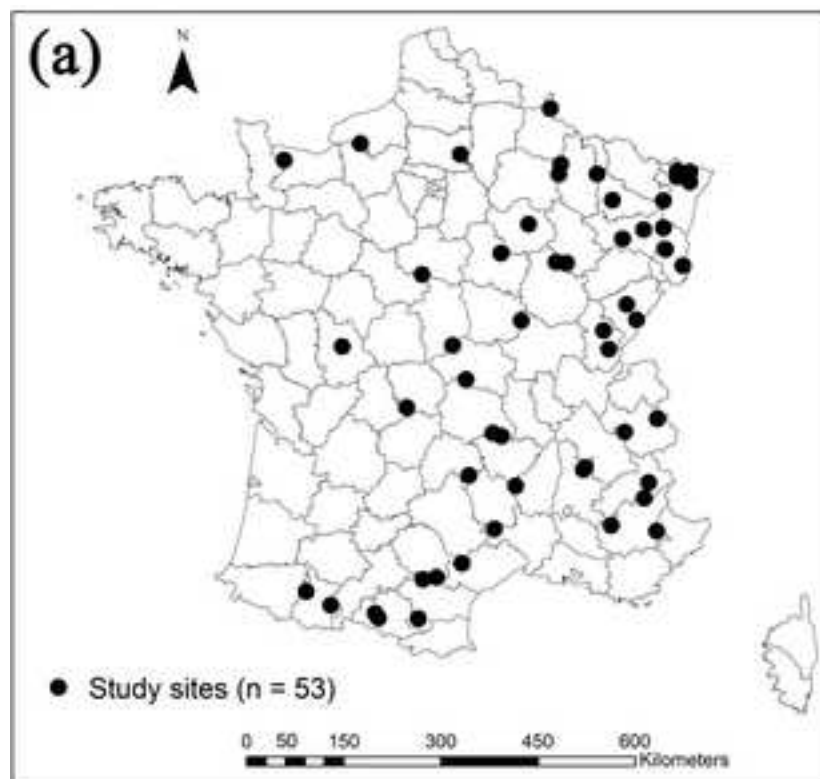


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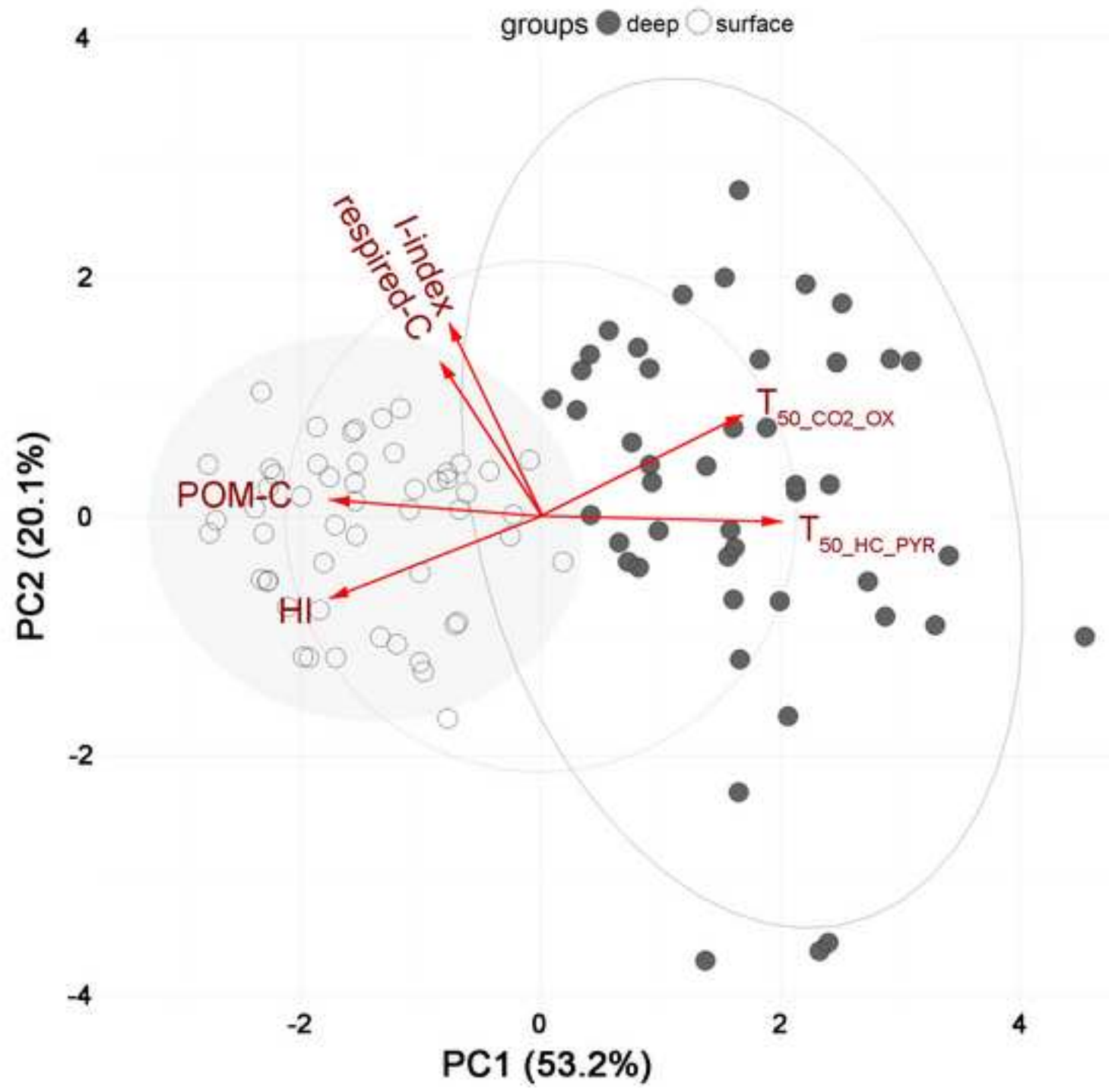
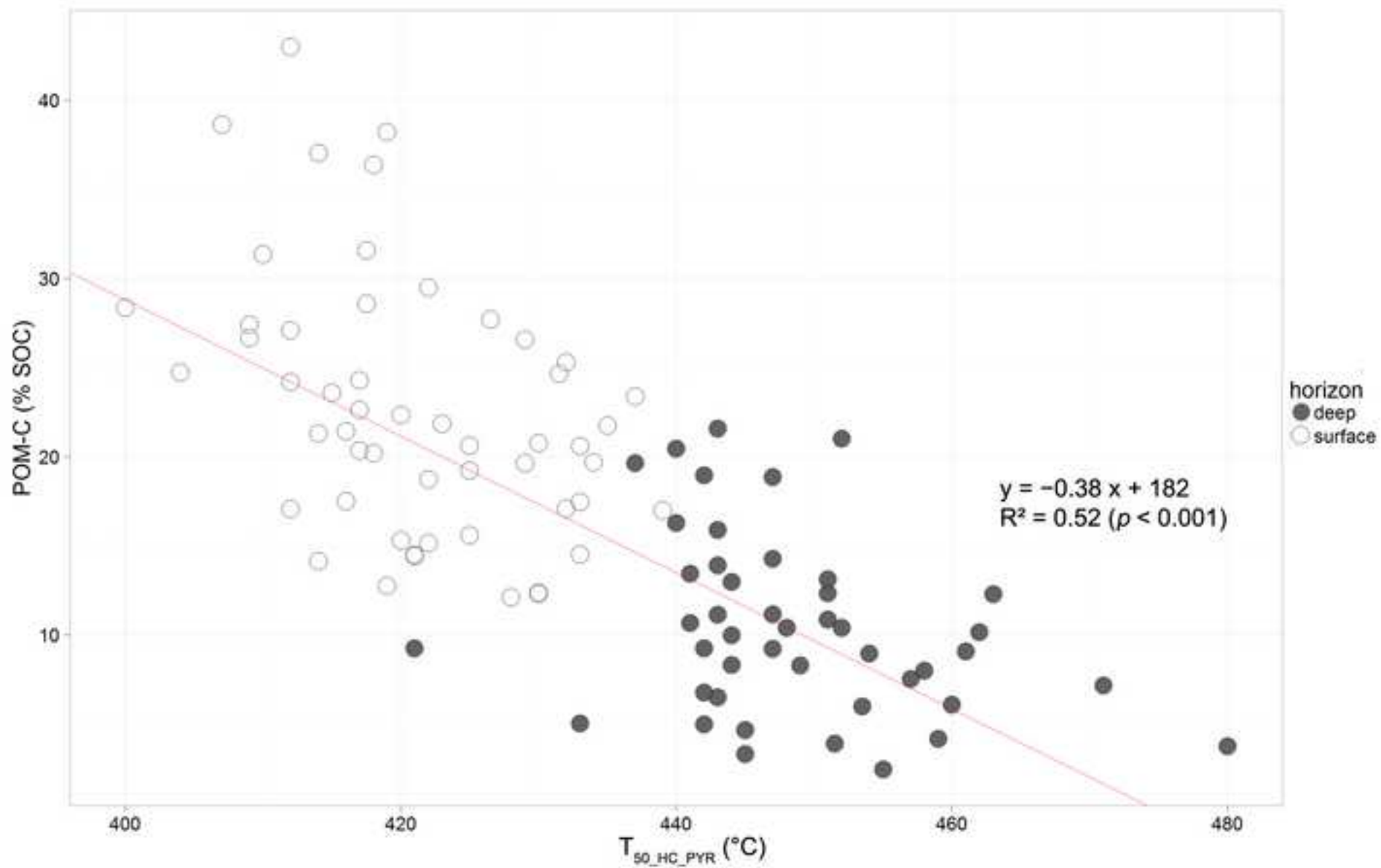


Figure 3
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Is Rock-Eval 6 thermal analysis a good indicator of soil organic carbon lability? – A method-comparison study in forest soils

Laure Soucémariadin^{1,*}, Lauric Cécillon², Claire Chenu³, François Baudin⁴, Manuel Nicolas⁵, Cyril Girardin³ and Pierre Barré¹

Supporting Information

Table SI-A.1. Mean (+ standard deviation) particle-size distribution, pH and C/N ratio of the studied samples.

Table SI-B1. Percentage of variance explained and loadings of the first three principal components (PC) after Box-Cox transformation to correct for skewness for the PCA of all (0–10 cm and 40–80 cm) samples (n = 99). Values in bold indicate the variables with loading greater than the mean of the absolute loading in each PC.

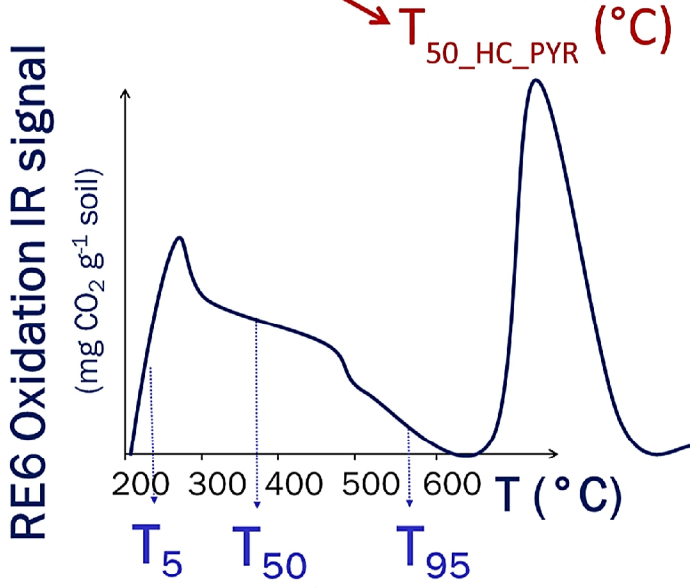
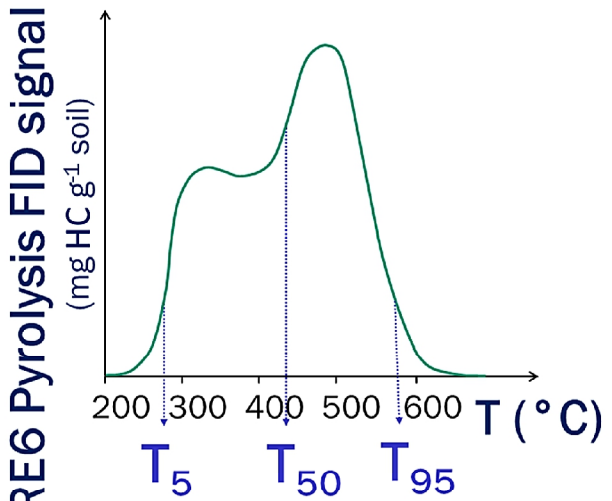
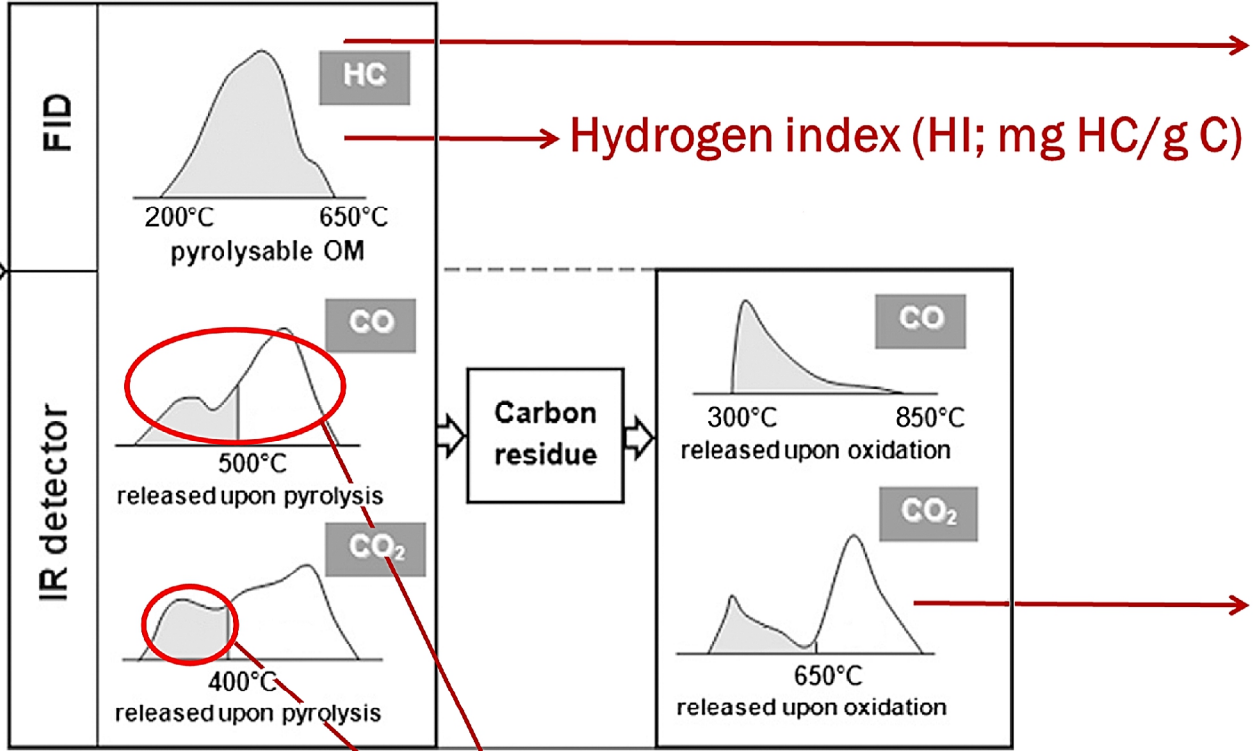
Table SI-C1. Spearman correlation coefficients between 10-week mineralizable SOC (respired-C), the proportion of OC in the POM fraction (POM-C), the RE6 parameters and the C/N ratio of the bulk soil, for the three soil types and the two vegetation types. Significance is indicated as follows: ***: $p < 0.001$; **: $p < 0.01$; *: $p < 0.05$. The very high (> 0.9) correlations are marked in bold.

Fig. SI-A1. Description of the Rock-Eval 6 thermal analysis (adapted from Saenger et al., 2013) and calculation of four RE6-derived parameters (Hydrogen index; Oxygen index; $T_{50_HC_PYR}$, the temperature at which 50% of the HC resulting from the SOM pyrolysis had

evolved; $T_{50_CO2_OX}$, the temperature at which 50% of the residual SOM was oxidized to CO_2 during the oxidation phase).

Fig. SI-B1. The proportion of OC in the POM fraction (POM-C) as a function of (a) respired-C (the proportion of total SOC mineralizable during a 10-week laboratory incubation); (b) OI_{RE6} (the oxygen index); (c) HI (the hydrogen index); (d) R-index (the proportion of thermally stable SOC pool) for all samples ($n = 99$; surface = 0–10 cm and deep = 40–80 cm).

30-100 mg soil sample



Oxygen index (OI_{RE6} ; mg O₂/gC)

T_{50_HC_PYR} (°C)

T_{50_CO2_OX} (°C)

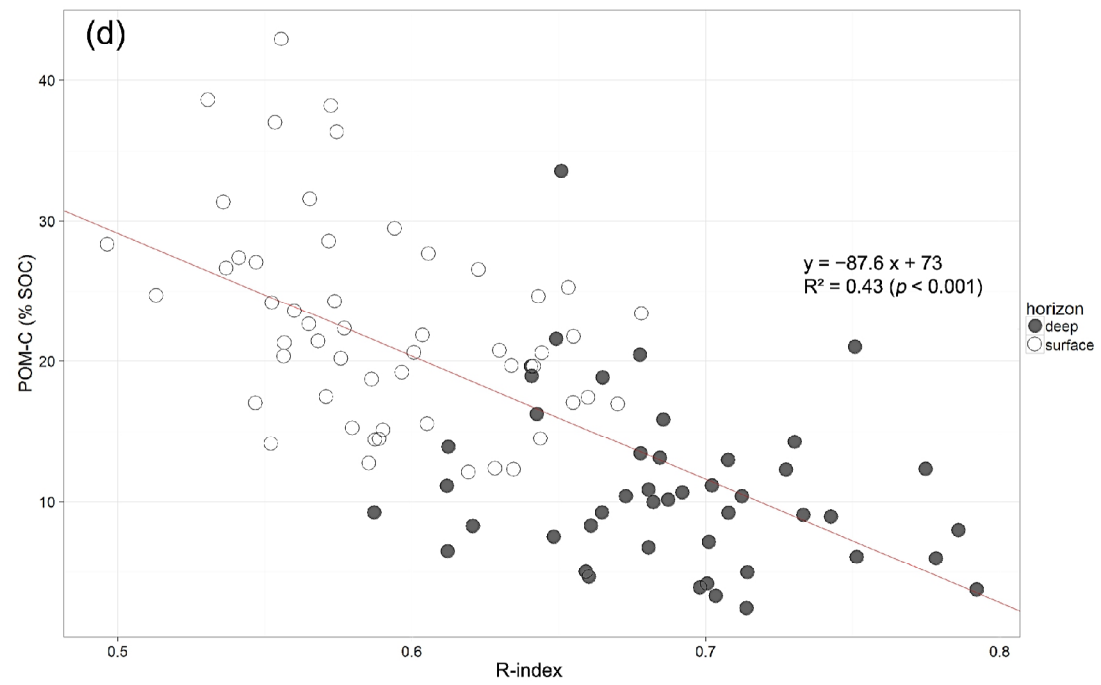
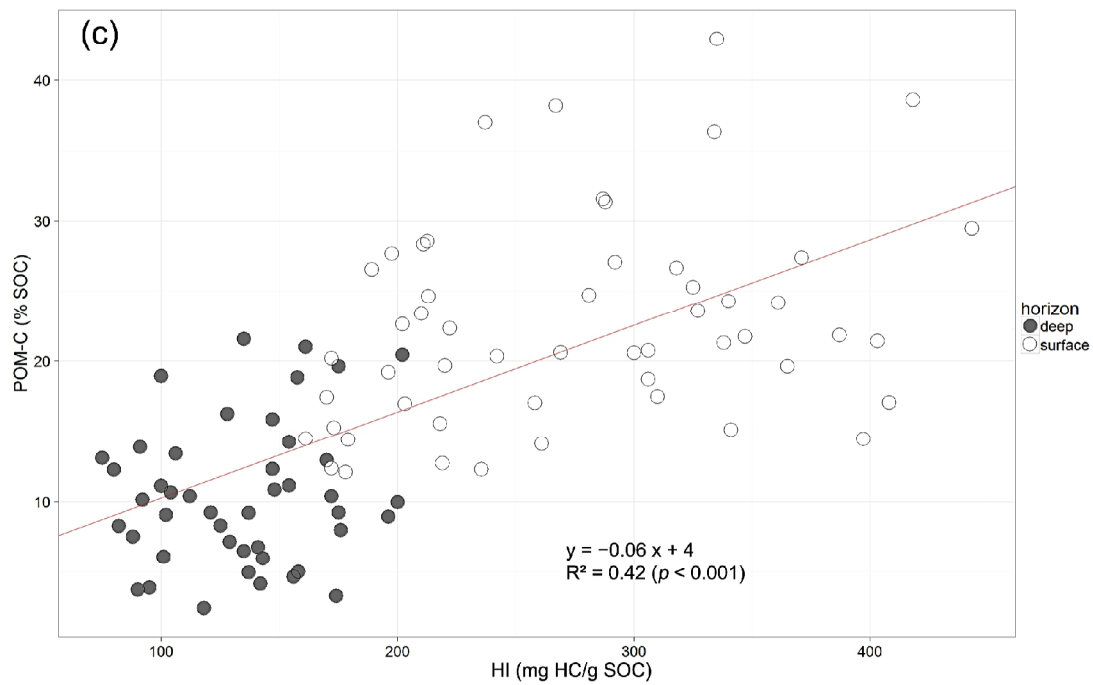
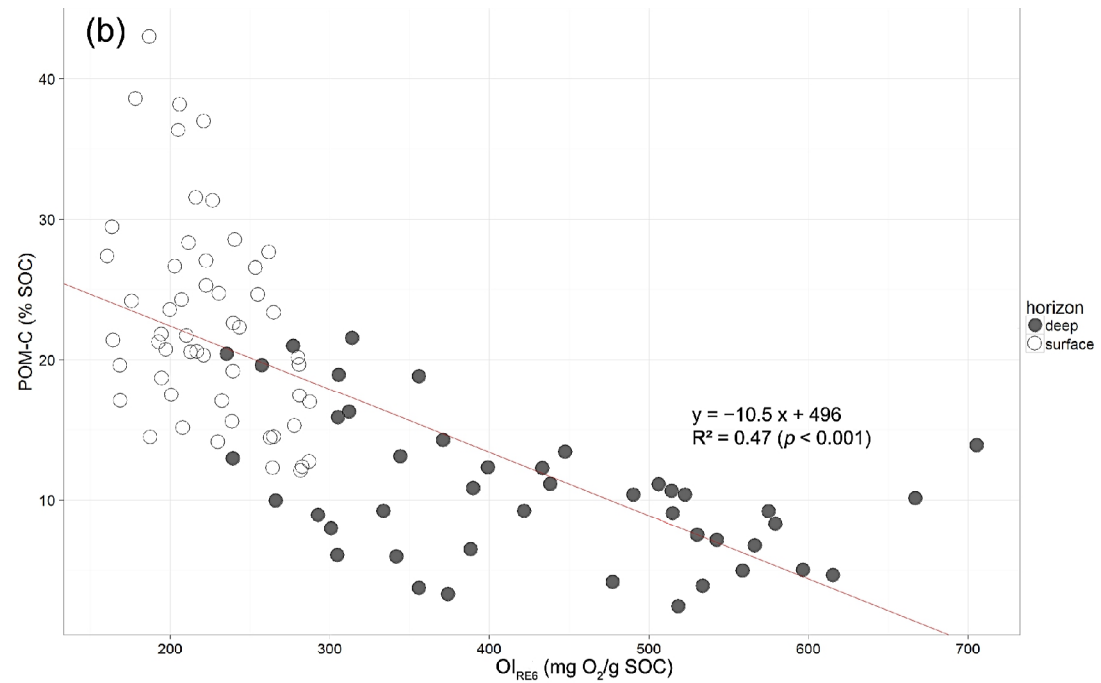
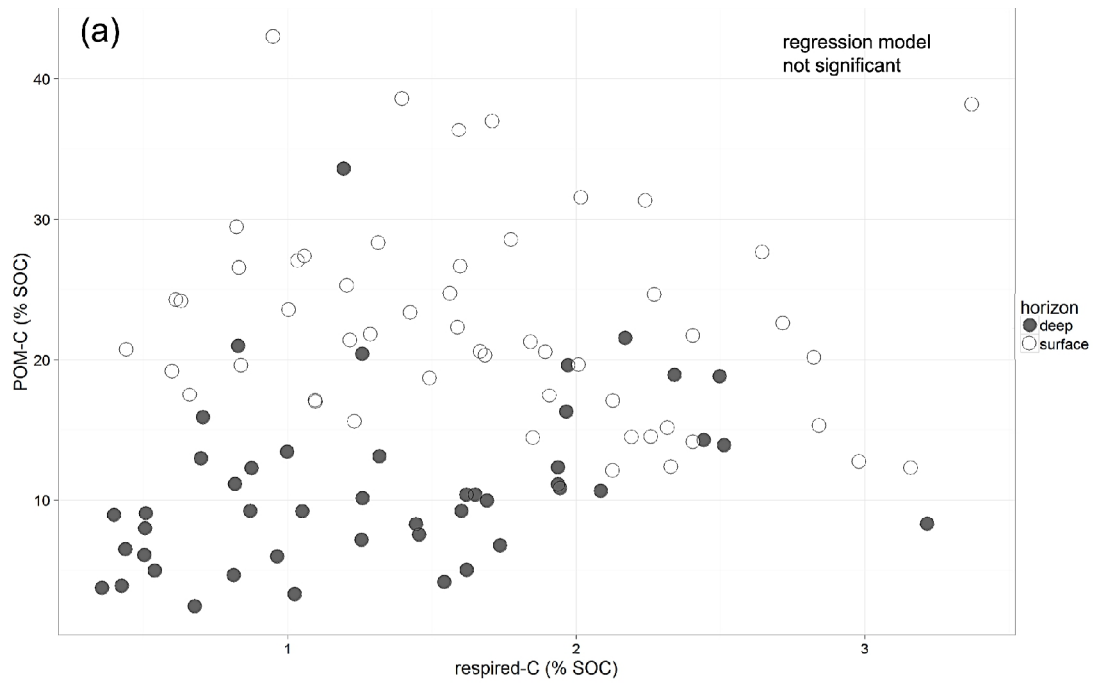


Table SI-A.1. Mean (+ standard deviation) particle-size distribution, pH and C/N ratio of the studied samples in each layer of the 53 plots.

depth (cm)	n	clay (%)	silt (%)	sand (%)	pH _{water}	C/N bulk soil
0–10	53	22.5 (13.6)	35.5 (18.0)	42.0 (28.8)	4.9 (1.0)	16.9 (4.5)
40–80	46	21.0 (15.4)	32.8 (16.2)	46.2 (26.7)	5.9 (1.5)	11.8 (3.8)

Table SI-B1. Percentage of variance explained and loadings of the first three principal components (PC) after Box-Cox transformation to correct for skewness for the PCA of all samples (n = 99). Values in bold indicate the variables with loading greater than the mean of the absolute loading in each PC.

PC	PC1	PC2	PC3
% variance explained	53.2	20.1	13.1
respired-C	-0.22	0.55	0.77
POM-C	-0.47	0.06	0.00
T _{50_HC_PYR}	0.53	-0.02	0.11
T _{50_CO2_OX}	0.45	0.36	-0.18
HI	-0.46	-0.29	-0.15
I-index	-0.20	0.69	-0.58

Table SI-C1. Spearman correlation coefficients between 10-week mineralizable SOC (respired-C), the proportion of OC in the POM fraction (POM-C), the RE6 parameters and the C/N ratio of the bulk soil, for the three soil types and the two vegetation types. Significance is indicated as follows: ***: $p < 0.001$; **: $p < 0.01$; *: $p < 0.05$. The very high (> 0.9) correlations are marked in bold.

		SOIL TYPE									
		respired-C	POM-C	T _{50_HC_PYR}	T _{50_CO2_OX}	HI	I-index	OI _{RE6}	R-index	C/N	pH
dystric Cambisol	POM-C	0.15									
	T _{50_HC_PYR}	-0.36*	-0.68***								
	T _{50_CO2_OX}	-0.18	-0.67***	0.77***							
	HI	0.17	0.62***	-0.81***	-0.65***						
	I-index	0.47**	-0.01	0.20	0.05	-0.17					
	OI _{RE6}	-0.15	-0.73***	0.82***	0.73***	-0.89***	0.15				
	R-index	-0.50**	-0.52**	0.90***	0.61***	-0.62***	-0.58***	0.63***			
	C/N	0.11	0.68***	-0.67***	-0.66***	0.58***	0.14	-0.75***	-0.64***		
	pH	0.14	-0.60***	0.58***	0.51**	-0.61***	0.08	0.60***	0.42*	-0.38*	
	clay content	-0.30	-0.16	0.15	-0.02	-0.03	-0.45**	0.23	0.32	-0.27	-0.02
eutric Cambisol	POM-C	0.46*									
	T _{50_HC_PYR}	-0.45*	-0.68***								
	T _{50_CO2_OX}	-0.54**	-0.53**	0.77***							
	HI	0.31	0.72***	-0.52**	-0.48**						
	I-index	0.39*	0.37*	-0.58***	-0.36	0.21					
	OI _{RE6}	-0.42*	-0.88***	0.68***	0.55**	-0.83***	-0.39*				
	R-index	-0.50**	-0.68***	0.94***	0.72***	-0.55**	-0.78***	0.70***			
	C/N	0.41*	0.84***	-0.60***	-0.49**	0.71***	0.17	-0.82***	-0.54**		
	pH	-0.57***	-0.56**	0.69***	0.71***	-0.54**	-0.20	0.62***	0.62***	-0.56**	
	clay content	-0.03	-0.13	-0.08	-0.08	-0.03	-0.14	-0.05	0.01	0.01	-0.25
entic Podzol	POM-C	0.42*									
	T _{50_HC_PYR}	-0.35*	-0.86***								
	T _{50_CO2_OX}	-0.24	-0.52**	0.69***							
	HI	0.18	0.71***	-0.75***	-0.57***						
	I-index	0.31	0.55***	-0.69***	-0.30	0.27					
	OI _{RE6}	-0.15	-0.71***	0.75***	0.48**	-0.96***	-0.26				
	R-index	-0.32	-0.83***	0.97***	0.62***	-0.68***	-0.81***	0.68***			
	C/N	0.08	0.54**	-0.55***	-0.44*	0.74***	0.13	-0.70***	-0.46**		
	pH	-0.29	-0.69***	0.83***	0.72***	-0.71***	-0.48**	0.72***	0.79***	-0.55***	
	clay content	-0.01	-0.10	-0.06	-0.29	-0.15	0.05	0.23	-0.06	-0.39*	0.02

		VEGETATION TYPE									
		respired-C	POM-C	T _{50_HC_PYR}	T _{50_CO2_OX}	HI	I-index	OI _{RE6}	R-index	C/N	pH
coniferous	POM-C	0.20									
	T _{50_HC_PYR}	-0.25	-0.67***								
	T _{50_CO2_OX}	-0.24	-0.52***	0.72***							
	HI	0.05	0.70***	-0.82***	-0.67***						
	I-index	0.29*	0.38**	-0.52***	-0.16	0.21					
	OI _{RE6}	0.00	-0.75***	0.76***	0.58***	-0.92***	-0.27				
	R-index	-0.25	-0.68***	0.95***	0.63***	-0.75***	-0.74***	0.74***			
	C/N	-0.13	0.60***	-0.46***	-0.33*	0.73***	0.19	-0.80***	-0.47***		
	pH	0.37**	-0.54***	0.42**	0.37**	-0.64***	-0.18	0.70***	0.45***	-0.66***	
	clay content	0.27*	-0.20	-0.11	-0.32*	-0.15	-0.09	0.33*	-0.01	-0.51***	0.50***
deciduous	POM-C	0.21									
	T _{50_HC_PYR}	-0.29	-0.80***								
	T _{50_CO2_OX}	-0.07	-0.56***	0.70***							
	HI	0.10	0.69***	-0.83***	-0.73***						
	I-index	0.40**	0.17	-0.24	0.15	-0.04					
	OI _{RE6}	-0.05	-0.77***	0.84***	0.69***	-0.92***	0.03				
	R-index	-0.44**	-0.73***	0.92***	0.52***	-0.66***	-0.57***	0.67***			
	C/N	-0.16	0.67***	-0.66***	-0.66***	0.81***	-0.20	-0.86***	-0.46**		
	pH	0.07	-0.55***	0.52***	0.42**	-0.53***	-0.19	0.60***	0.52***	-0.61***	
	clay content	0.09	-0.12	0.16	0.15	-0.26	-0.25	0.27	0.24	-0.34*	0.35*