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1 **What deep-soil profiles can teach us on deep-time pH dynamics after land use change?**

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3 **Short title: Deep-soil profiles teach us deep-time pH dynamics**

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14

15 **Abstract**

16 Soil profiles keep records of the legacies of historical land uses on soil physicochemical
17 properties with deep-soil (3m depth) profiles providing information on centuries-old
18 dynamics. By combining geohistorical archives on past land-uses and management practices
19 together with soil pH data from 19 plots scattered across five study areas in North-France,
20 we analyzed the effect of two contrasting historical land-use change trajectories
21 (afforestation of former arable land vs. cultivation after deforestation) on the acidification
22 and alkalinisation dynamics along 3m depth loessic luvisols profiles. An analysis of
23 covariance (ANCOVA) and a generalized additive model (GAM) were used to test the
24 interaction effect between depth and the two studied chronosequences levels on soil pH.
25 Results show two contrasting alkalinization dynamics for formerly acidic forest soils
26 converted to arable land depending on past management practices. One century of liming
27 practices based on hydrated lime is enough for a complete deprotonation whereas
28 alkalinization dynamic was much slower (2-4 centuries are necessary to neutralize the
29 relictual acidity below 2m) under liming based on chalk-blocks supply. Per contra,

30 afforestation of former arable land cultivated and limed during at least 2-4 centuries keep an
31 alkaline “memory” effect over more than a millennium. The acidification process is
32 progressive through time from the surface to deeper soil horizons. This suggests that soil pH
33 profiles can be used to estimate time since afforestation of former arable land. Linking deep-
34 soil pH profiles with chronosequences is a powerful tool to understand the potential impact
35 of future land-use change trajectories on soil physicochemical properties.

36 **Keywords: Afforestation, deforestation, geohistory, liming, loess, luvisol**

37

38

39

40 **Introduction**

41 It is now widely acknowledged that today’s soils encapsulate the legacy of not only natural
42 processes but also actual and past human activities (Dambrine *et al.*, 2007; Kluiwing *et al.*,
43 2015). Since the Neolithic, farmers and foresters throughout their activities are influencing
44 the pedogenesis and evolution of anthropogenic landscapes (Van Vliet-Lanoe *et al.*, 1992;
45 Langhor, 2001; Emadodin *et al.*, 2009). Imprints of past land uses (e.g., croplands,
46 grasslands, woodlands) are still visible in current biodiversity patterns (Closset-Kopp &
47 Decocq, 2015; Dambrine *et al.*, 2007), molecular biomarkers (Lavrieux *et al.*, 2012) and the
48 chemical composition of soils (Koerner *et al.*, 1999; Cornu *et al.*, 2007; Von Oheimb *et al.*,
49 2008). Among soil physico-chemical parameters, potential hydrogen (pH) is a very good
50 indicator of land-use changes (LUCs) from forest to arable land and *vice versa* (Blake *et al.*,
51 1999; Bossuyt *et al.*, 1999; Verheyen *et al.*, 1999). Soil pH controls pedogenetic processes
52 such as acidolysis and acido-complexolysis when soil horizons become acid, it also controls
53 the bioavailability of nutrients for plant growth and influences bacterial, fungal, vegetal and
54 animal biodiversity (see for example, Chauchan, 2014; Fichtner *et al.*, 2014), hence soil pH is
55 a key parameter in pedogenetic dynamics through time.

56 The dynamic of soil alkalinization results from a negative proton balance which can
57 be triggered by calcic mineral amendment (e.g., limestones, lime, marls) in agricultural
58 regions under temperate humid climate. These agronomic practices, first described during
59 the 1st century BC by Varron (Ed. 1864: book I - chapter VIII) and much more detailed later
60 on by Pliny the Elder (Pliny the Elder, 1877: book XVII - chapter 4), are very old in north-
61 western Europe and varies in nature (Goulding, 2016), amount and frequency of inputs
62 (Tables S1-S2). Liming is required as cereals growth is seriously limited on acid soils of
63 formerly forested lands which were deforested to extend land cultivation. Thus in North
64 France, liming was a very systematic practice in the first years following deforestation
65 sometimes accompanied by grazing (Crescentiis, 1548). First writings on soil liming following
66 deforestation are from the 12th century (Fossier, 1987) (i.e., the “sartier” contracts). Other
67 farming practices than liming such as fertilization, product removal and crop rotations may
68 also affect the proton (H^+) and hydroxyl (OH^-) budgets (Merry, 2009; Schroder *et al.*, 2011)
69 and thus the dynamic of soil alkalinization. Not only the dynamic of soil alkalinization due to
70 agronomic practices is a well-known phenomenon, but also the short-term (few decades)
71 dynamic of soil acidification after afforestation of former cultivated areas has been heavily
72 investigated (de Schrijver *et al.*, 2012). For instance, the impact of LUC on soil pH is known to
73 persist over 30-80 years of forest growth on formerly agricultural lands irrespective to the
74 pH measurement method, i.e., pH_{water} , pH_{KCl} or pH_{CaCl_2} (Ritter *et al.*, 2003; Falkengren-Grerup
75 *et al.*, 2006). Some authors have even demonstrated that the dynamic of soil acidification
76 after afforestation depends on the selected tree species (Hagen-Thorn *et al.*, 2004; de
77 Schrijver *et al.*, 2012), albeit land-use history seems to influence soil properties more than
78 the selected tree species (Ritter *et al.*, 2003).

79 Over longer timescale, the imprint of former agricultural practices on soil properties,
80 including soil pH, is also documented. However, the few studies documenting it focused on
81 the soil surface solely (from 5 and up to 20cm) (Dambrine *et al.*, 2007; Closset-Kopp &

82 Decocq, 2015), which precludes a wide part of the available soil archives. Wigley et al. (2013)
83 nicely emphasized this issue by asking: “*What do ecologists miss by not digging deep*
84 *enough?*”. Drouet *et al.* (2015) provided some answers by showing that deep nutrients could
85 significantly contribute to tree growth. Only a handful of studies, focusing on loessic luvisols
86 exclusively, examined the long-term acidification or alkalinisation dynamics through deep
87 soil profiles. The experimental site of Rothamsted farm (UK) is a good example where soil
88 profiles were sampled up to 90cm depth during a period of 110 years after the afforestation
89 of cultivated chromic luvisols. The $\text{pH}_{\text{CaCl}_2}$ profiles of this experimental site revealed a net
90 surface acidification from 6.2 to 3.8, whereas the deepest horizons of the investigated soil
91 profiles showed a drop of 0.25 pH unit only (Blake *et al.*, 1999). In parallel, Bossuyt et al
92 (1999) used a chronosequence (i.e., a synchronic approach) of afforested loessic luvisols in
93 Belgium and found a very marked decline in pH_{KCl} during the first 30 years after afforestation
94 followed by a slower decline in the next 20 years after which pH_{KCl} was similar to the level
95 observed in an ancient forest. Regarding the long term dynamic of soil alkalinisation, Pécrot
96 (1956) focused on cultivated soils after deforestation in Belgium and concluded that the
97 alkalinization process of the Ap horizon was performed after 50 years, with the Bt horizon
98 being neutral to alkaline after 100 years. After 700 years of agricultural practices, the entire
99 soil profile was neutral to alkaline. More recently, Colinet (2003) measured, within the same
100 region, a slower alkalinisation dynamic with the Bt horizon remaining acid ($\text{pH}_{\text{water}} = 5.3$)
101 after 100 years of cultivation while a neutral (around 6.5) soil profile was observed up to
102 165cm depth after 250 years of cultivation. Hence, deep-time (>100 years) acidification and
103 alkalinization dynamics following LUCs still remain unknown and yet could provide important
104 insights for ecosystem functioning such as productivity.

105 Here we use two chronosequences spanning several centuries (up to 1500 years) of
106 land use legacies and supported by robust and ascertainable geohistorical data (Tables S1-S2
107 & S3) to describe deep-time alkalinization and acidification dynamics. A total of 102 soil

108 pH_{water} profiles on luvisols in North France were examined to answer the following
109 questions: (i) How long are the impacts of past LUCs (e.g., cultivation after deforestation and
110 afforestation after cultivation) still noticeable on soil pH? (ii) What are the vertical and
111 temporal dynamics of soil alkalinization and acidification? (iii) Is soil pH a good proxy to date
112 past LUCs?

113

114 **Materials and methods**

115

116 *Study area*

117 The study area is located on loessic luvisols in North France (Thiérache: 49°48'34"N,
118 4°01'11"E). We sampled 19 plots within a geographical area (Figure 1) comprising 5 ancient
119 forests sites: (1) the Haye of Aubenton (AU; 4 plots); (2) the Val-Saint-Pierre forest (VSP; 4
120 plots); (3) the Andigny forest (AN; 4 plots); (4) the Plomion wood (PL; 6 plots); and (5) the
121 Metz-en-Couture/Havrincourt wood (MC; 1 plot). The local plateau is about 100-150m above
122 sea level and the climate is humid temperate: mean annual air temperature is 8.75°C;
123 precipitations are 750-800mm/year; and ETP Turc is 570mm (SILVAE portal, AgroParisTech-
124 INRA & LERFoB). Forest stands are dominated by common beech (*Fagus sylvatica*), oak
125 (*Quercus robur*) and hornbeam (*Carpinus betulus*), all older than 80 years excepting the
126 recently afforested plots. Every plots are located on the plateau (i.e., very gentle slopes <5%)
127 to avoid soil erosion and spatial heterogeneity in acid neutralizing capacity due to
128 topography (Brahý *et al.*, 2000).

129 The studied soils are developed on periglacial thick loamy deposits of the Western-
130 European loess belt, which is a relatively uniform parent material in North France (Antoine
131 *et al.*, 2003; Jamagne, 1972). This loess belt is lying on Turonian-Santonian clayey marls and
132 white chalks.

133

134 *Geohistorical reconstruction of land uses across the study area*

135 Various kinds of historical data sources were used to reconstruct past land uses, such as: the
136 regional archaeological database (*Patriarche*); local archives; the Napoleonic cadastral maps
137 and the associated register of status of the land (1820); historical Etat-Major maps (1830);
138 aerial photographs from 1930 to 2013 (IGN); and oral interviews.

139 We prepared two chronosequences spanning several centuries of LUCs each. The first
140 one (cf. the afforestation chronosequence) focused on afforestation after cultivation and the
141 second one (cf. the deforestation chronosequence) focused on cultivation after
142 deforestation. For each of the 19 studied plots, LUCs from arable land to forest and *vice*
143 *versa* were controlled by robust and ascertainable geohistorical data (see Figure 2 for
144 example), all summarized in the supporting information (Table S3).

145 The afforestation chronosequence contains six different levels along an ordinal scale
146 ranging from “recently afforested” to “afforested since a very long time”: a medieval strip
147 field afforested 15 years ago (C→F₁₉₉₉); a field afforested 80 years ago (C→F₁₉₃₅); two
148 medieval strip fields afforested 150 years ago (C→F_{1865ck} & C→F₁₈₆₅; with and without Ck
149 loessic lower horizon, respectively); one medieval strip field afforested 500 to 1000 years
150 ago (C→F₁₀₀₀₋₁₅₀₀); and four ancient forests (C→F_{500AU+VSP+PL+AN}). These four ancient forests
151 were already forested during the 12th or 13th century (Table S3) but could have been
152 cultivated during bronze-iron ages and/or antiquity as many west European ancient forests
153 (Dubois, 1990). Gallo-Roman sites such as farms (AU, PL), roads (AU, AN, VSP), buildings (AU,
154 PL, VSP), and cremation graves (AU, AN, PL, VSP) occur within these ancient forests. No
155 Medieval or Modern sites were found suggesting that these areas could have been
156 cultivated during antiquity and afforested as early as the troubled period of the end of the
157 Western Roman Empire during the 5th century AD. The absence of the upper part of
158 albeluvic tonguing on these luvisols (ISSS Working Group RB, 1998) as well as the brownish
159 colour of the E horizon (7.5YR4/4) due to an increased burrowing activity from earthworms

160 and moles enhanced by liming and manuring (Bossuyt *et al.*, 1999; Langhor, 2001) support
161 this hypothesis. Hence, a putative age of 1500 years BP has been attributed to C→F₅₀₀ plots.

162 The deforestation chronosequence contains six different levels along an ordinal scale
163 ranging from “recently deforested” to “deforestation since a very long time”: several
164 formerly ancient forests deforested about 5 (F→C₂₀₀₉); 30 (F→C₁₉₈₅); 140 (F→C₁₈₇₅); 150
165 (F→C₁₈₆₅); and 170 (F→P₁₈₄₅→C₁₉₂₀ & F→C₁₈₄₅; with F→P₁₈₄₅→C₁₉₂₀ being a pasture without
166 liming practices, but manured, during the first 85 years following deforestation and then
167 being limed & cultivated during the next 85 years until now) years ago. In addition to those
168 six levels, four cultivated ancient fields (C→C_{AU+VSP+PL+AN}) whose great age was proven by
169 medieval strip shapes (Sivery, 1990) were used as control plots. All plots from the
170 deforestation chronosequence excepting the control plots were located in sites previously
171 described as medieval forests (F→C₂₀₀₉, F→C₁₉₈₅, F→C₁₈₇₅, F→C₁₈₆₅, F→P₁₈₄₅→C₁₉₂₀ and
172 F→C₁₈₄₅).

173 Following Pliny the Elder (Pline l’ancien, 1877), liming practices were already used by
174 Gallic, Germanic and Britannic farmers for cereals culture during Antiquity. In North France,
175 the use of white chalk (sometimes miscalled marl) is confirmed from the beginning of the
176 high medieval period and marling contracts were recorded for lands after deforestation
177 (Derville, 1999; Fossier, 1987). Note that liming practices (amount and periodicity of inputs)
178 were likely variable over time (Table S1). Actual practices from the farmers who currently
179 own the arable fields where our studied plots are located are usually around 15T/Ha each
180 10-20 years (Table S2). Bolton (1977) observed, on the sandy-clay loam fields of the
181 Rothamsted farm, that CaCO₃ mean loss rate were ranging from 368kg/Ha/yr (for 5T/Ha
182 added initially) to 823 kg/Ha/yr (for 20T/Ha added initially) over 12 years. Thus adding 5T/Ha
183 each 12 years was sufficient to maintain soil pH values over long time periods. Liming in
184 greater amount once and/or more frequently over time can help to increase soil pH values
185 from acidic to neutral conditions for farming. Thus, local liming practices reported across our

186 study sites and from the lower medieval to the modern period were sufficient to maintain
187 and even raise soil pH values (Tables S1 and S2).

188

189 *Soil sampling and measurement methods*

190 Between 2013 and 2016, a total of 6 soil profiles were sampled for each of the 19 studied

191 plots (C→C_{AN}, C→C_{VSP}, C→C_{AU}, C→C_{BP}, C→F_{500AN}, C→F_{500VSP}, C→F_{500AU}, C→F_{500BP}, F→C₂₀₀₉,

192 F→C₁₉₈₅, F→C₁₈₇₅, F→C₁₈₆₅, F→P₁₈₄₅→C₁₉₂₀, F→C₁₈₄₅, C→F₁₉₉₉, C→F₁₉₃₅, C→F_{1865ck}, C→F₁₈₆₅,

193 C→F₁₀₀₀₋₁₅₀₀) excepting C→C_{AN}, C→C_{VSP}, C→C_{AU} and F→C₁₈₇₅, where only 3 soil profiles were

194 sampled, thus totalizing 102 soil profiles across the entire study area (Table S4). By using an

195 Edelman hand auger, we retrieved a deep-soil profile (3m depth) for each of these 102 soil

196 profiles. The soil profiles were distant from each other of about 10 to 100m within a given

197 plot. The soil augering descriptions were carried out following the FAO standard guidelines.

198 Soil horizon depths and textures were noted and Munsell colors were determined on moist

199 samples. For each of the 102 sampled soil profiles, we characterized the humus (Brethes *et*

200 *al.*, 1995) and noted the humus index (Ponge & Chevalier, 2006) as well as the colors of the

201 soil horizons (Table S5). None of the 102 profiles had any archaeological remains,

202 topographic anomalies, natural (windthrow) or anthropogenic disturbances, anomaly of

203 humus thickness (Bossuyt *et al.*, 1999) or a tree trunk that was closer than 2m (Beniamino *et*

204 *al.*, 1991).

205 Soil samples for soil pH analyses were collected at the following depths: 5; 25; 50; 75;

206 100; 125; 150; 175; 200; 250; and 300cm. For each of these 11 depths, 500g of soil material

207 was extracted and sealed in labeled plastic bags and transported to the lab where the soil

208 was air-dried and sieved to 2mm. Soil pH was measured with two glass electrodes in distilled

209 water (ISO 10390:2004) on two sub-samples, before being averaged. Among the 1122 (n =

210 102x11) samples of 500g of soil material, soil pH measurements could be obtained and used

211 for subsequent analyses for a total of 1074 samples (Table S4). For subsequent analyses, soil
212 pH measurements for C→C_{AN}, C→C_{VSP}, C→C_{AU} and C→C_{BP} as well as for C→F_{500AN}, C→F_{500VSP},
213 C→F_{500AU} and C→F_{500BP} were pooled in the C→C and C→F₅₀₀ levels, respectively.

214

215 *Data analyses*

216 To detect an imprint of past LUCs on soil pH profiles and represent the inter- and intra-plot
217 variability in the data (n = 1044), we ran several statistical models testing the impact of
218 different LUC trajectories (the main explanatory variable) on soil pH (the response variable)
219 while controlling for the potential confounding effect of soil depth (a covariate) and its
220 potential interaction with past LUCs. Soil pH and depth were treated as continuous variables
221 while the variable representing the different LUC trajectories of the afforestation and
222 deforestation chronosequences tested here was treated as a factor variable with 12 levels
223 (C→C, C→F₅₀₀, F→C₂₀₀₉, F→C₁₉₈₅, F→C₁₈₆₅, F→P₁₈₄₅→C₁₉₂₀, F→C₁₈₄₅, C→F₁₉₉₉, C→F₁₉₃₅,
224 C→F_{1865ck}, C→F₁₈₆₅, C→F₁₀₀₀₋₁₅₀₀). Note here that the F→C₁₈₇₅ level of LUC trajectory was not
225 considered in any of the statistical models due to low sample size (n = 3 replicates per soil
226 depth for F→C₁₈₇₅). Using ordinary least square (OLS) regression and generalized additive
227 model (GAM), we tested four candidate models: a null model (or intercept model) without
228 any explanatory variable as a control or baseline (m0: pH~1); a model that tests the effect of
229 soil depth on soil pH (m1: pH~Depth); a model that tests the effect of the different LUC
230 trajectories while controlling for soil depth (m2: pH~Depth+LUC); and a model that tests the
231 interaction effect between soil depth and LUC trajectories (m3: pH~Depth*LUC). The OLS
232 regression approach assumes a linear relationship between soil pH and depth while the
233 GAM approach can allow non-linear relationships. As an intermediate type of model, we also
234 ran OLS regression models with a second-order polynomial relationship between soil pH and
235 depth. In total, we ran 3x4 different models (Table 2) and we used the Akaike information
236 criterion (AIC) to select the best model (Burnham & Anderson, 2002), which is the one with

237 the lowest AIC value (AIC_{min}). We also computed the relative support for each model, using
238 the differences in AIC values, $\Delta AIC_i = AIC_i - AIC_{min}$, where AIC_i is the observed AIC value of the
239 focal model i and AIC_{min} is the lowest AIC value from the best model in the set of candidate
240 models (Burnham & Anderson, 2002). Based on the outputs from the best model with the
241 lowest AIC value, we subsequently ran Wilcoxon signed-rank tests to compare the impact of
242 different LUC trajectories on soil pH at a given depth.

243 All statistical analyses were performed within the R software environment (R
244 Development Core Team, 2016), using the “bbmle” and “mgcv” packages. All our models’
245 predictions were associated to a 95% confidence interval ($CI = 1.96 \times SEM$; where SEM means
246 standard error of the mean) around the mean soil pH profile. Residuals from the best model
247 were checked for normality and homoscedasticity (Figure S1).

248

249 **Results**

250

251 *Soils’ description*

252 The 102 soils exhibited a contrasted development of eluvial (E) and illuvial (Bt) horizons. The
253 diffuse transition horizon (BC) and C decarbonated loess material mostly constitute the
254 lower half part of the soil profile whereas calcareous loess material was completely absent
255 except for $C \rightarrow F_{1865ck}$, which exhibited calcareous loess at 220-250cm depth. For afforested
256 soils, the humus index score increased from 1 (i.e., eumull) to 3.6 (i.e., oligomull-dysmull)
257 with increasing afforestation age (Table S5) and the A/E transition appeared between 12cm
258 ($C \rightarrow F_{500}$) and 25cm ($C \rightarrow F_{1999}$) depth. For tilled cultivated soils, the Ap/E transition showed a
259 lesser variation with a range between 26cm ($F \rightarrow C_{1845}$) and 35cm ($F \rightarrow C_{2009}$).

260 The E horizons were bioturbated with some soils sampled in the Aubenton site
261 ($C \rightarrow F_{500AU}$, $C \rightarrow F_{1865}$ and $C \rightarrow F_{1865ck}$) having gleyic colour patterns evidenced by high values

262 and low chroma in the Munsell soil color chart. The E/Bt transition across the study area was
263 found between 33cm (F→C₁₈₆₅) and 60cm (AF) depth. Importantly, the upper parts of the
264 transitional albeluvic tonguing between the E and Bt horizons were systematically truncated
265 and leveled by bioturbation, even on C→F₅₀₀ luvisols which suggests cultures as past land
266 uses. The Bt horizon of most plots showed light grey polygonal crack networks typical of a
267 fragipan. Hence, the soils are classified (IUSS Working Group WRB, 2006) in luvisols but still
268 bear relict patterns of albeluvisols. All soils' horizons textures are very homogeneous, from
269 silt loam in the surface to silty clay loam for the deep part (Figure S2), albeit the sand
270 content of C→F₁₉₉₉ raised from 100cm towards the deepest part.

271

272 *Soil pH profiles of ancient forested (C→F₅₀₀) and ancient cultivated (C→C) soils*

273 We found a strong variation in soil pH values with depth in both the afforestation and
274 deforestation chronosequences (Table 1 and Figure S3). The topsoil of ancient forests (AF)
275 showed the lowest pH values (4.61) while the highest pH values (8.18) were found at 3m
276 depth in the carbonated plot (C→F_{1865ck}). Soil pH in C→F₅₀₀ increased with depth and
277 reached 5.17, 5.54 and 5.71 at 1m, 2m and 3m depth, respectively, whereas the ancient
278 cultivated soil reference (C→C) exhibited a homogeneous soil pH profile around neutral
279 values independently of soil depth.

280 Independently of the modelling approach used, the best candidate models of soil pH
281 included an interaction effect between soil depth and LUC trajectories (Table 2) with soil pH
282 profiles being specific to each of the 12 studied LUC trajectories (Figure 3). The lowest AIC
283 value was obtained with a generalized additive model (GAM) (cf. model "m3g" in Table 2),
284 which explained 91% of the total variation in soil pH.

285

286 *Chronosequences*

287 For the youngest afforested plot (C→F₁₉₉₉), neutro-alkaline pH values were still found below
288 75cm depth but decreased significantly to 6.66 and 6.03 at 50cm and 5cm depth,
289 respectively. Soil pH values in the C→F₁₉₉₉ LUC trajectory significantly differed from the
290 ancient cultivated soils (C→C) reference above 100cm depth and up to the surface (Figure
291 3). Eighty years after afforestation (C→F₁₉₃₅), soil pH values became significantly more acidic
292 than the C→C reference all along the soil profile (Figure 3), with pH dropping to 6.10 at 5cm
293 depth. About 150 years after afforestation, the mean of topsoil pH was 5.2 and 4.8 for the
294 C→F₁₈₆₅ and C→F_{1865ck} LUC trajectories, respectively, with topsoil pH being significantly
295 greater than the C→F₅₀₀ reference only for the C→F₁₈₆₅ LUC trajectory (Wilcoxon signed-rank
296 test: W = 131, p = 0.0012). At 175cm depth, soil pH increased up to 7.07 for the C→F_{1865ck}
297 LUC trajectory, thus being not significantly different from the C→C reference. The oldest
298 afforested plot (C→F₁₀₀₀₋₁₅₀₀) exhibited greater mean pH values than the C→F₅₀₀ reference
299 for all depths, with the difference being significant for the topsoil (Wilcoxon signed-rank
300 test: W = 105, p = 0.0435) but not significant in the deeper part of the soil profile: from
301 250cm depth (Wilcoxon signed-rank test: W = 91, p = 0.0551).

302 Shortly after deforestation and cultivation (F→C₂₀₀₉, F→C₁₉₈₅), soil pH values were
303 near neutral in the topsoil but remained very acid (<5) under ploughed horizons (i.e., 50 to
304 300cm depth) (Table 1 and Figure 3). On F→C₁₈₇₅ and F→C₁₈₆₅ plots, about 150 years after
305 deforestation and cultivation, soil pH values were neutro-alkaline (≥7) from topsoil down to
306 300cm depth. For the F→P₁₈₄₅→C₁₉₂₀ LUC trajectory, the neutro-alkaline pH values observed
307 between the surface and 100cm depth sharply declined to acid values (5.51 at 250cm depth)
308 more or less similar to the C→F₅₀₀ reference. The F→C₁₈₄₅ LUC trajectory showed weakly acid
309 to neutral pH values from topsoil (pH = 6.09 at 5cm depth) to the deep C horizon (pH = 6.33
310 at 250cm depth) and through the Bt horizon (pH = 7.02 at 75cm depth).

311

312 **Discussion**

313

314 *Alkalinization and acidification dynamics*

315 Based on deep-soil profiles, the strong interaction we found between the different land-use
316 changes (LUCs) we tested here and soil depth explained 91% of the observed variation in soil
317 pH across the study area. This clearly suggests different acidification and alkalinization
318 dynamics with soil depth depending on the two LUC trajectories. Regarding the potential
319 drivers for the unexplained variation see Appendix S1.

320 On the deforestation and culture chronosequence, time since deforestation clearly
321 matters to explain the alkalinization kinetic in the soil profile. For recent deforestation
322 events that happened 5-30 years ago (i.e., $F \rightarrow C_{2009}$ and $F \rightarrow C_{1985}$), our results suggest that the
323 alkalinization process is confined within the ploughed horizon (Ap) with a sharp decline in pH
324 values towards initial acidic conditions, reflecting former land use as forest. Following the
325 Rothamsted's model ($\Delta pH = 0.109L - 0.690$; L is lime added as T/Ha $CaCO_3$) (Goulding *et al.*,
326 1986; Goulding, 2016), we found that for a topsoil pH value of 4.6 (equivalent to ancient
327 forest) if we add 30T/Ha of chalk, pH should rise up by 2.6 points in the next years. This
328 situation is likely similar to the one observed for $F \rightarrow C_{2009}$, showing an increase in pH values
329 reaching 7.1 in the Ap horizon (Table 1).

330 Coherently, for older deforestation events that happened 140 to 170 years ago (e.g.,
331 $F \rightarrow C_{1865}$), the alkalinization process migrates (more or less rapidly) towards the deepest soil
332 layers. Interestingly and for LUC trajectories being more or less equivalent in terms of time
333 since deforestation and culture (i.e., $F \rightarrow C_{1875}$, $F \rightarrow C_{1865}$, $F \rightarrow P_{1845} \rightarrow C_{1920}$ and $F \rightarrow C_{1845}$), the
334 migration rate of this alkalinization front likely depends on the former management
335 practices used by farmers. For cultivated soils limed with defecation lime ($F \rightarrow C_{1875}$ and
336 $F \rightarrow C_{1865}$), the alkalisation dynamic operated all along the soil profile and soil pH values are
337 now neutro-alkaline from the surface until 3m depth. However, for cultivated soils limed
338 with chalk ($F \rightarrow P_{1845} \rightarrow C_{1920}$ and $F \rightarrow C_{1845}$), the alkalisation process was much less

339 pronounced and pH values gently decline towards acid values in the deep soil layers. For the
340 F→P₁₈₄₅→C₁₉₂₀ LUC trajectory, for which successive liming practices were restricted to the
341 last 85 years, the migration rate of the alkalization front is more or less intermediate with
342 pH values declining towards initial acidic conditions at 3m depth. These results suggest that
343 the high solubility of Ca(OH)₂ in defecation lime (Table S6) has triggered faster and deeper
344 soil alkalization rates through winter infiltrations of waters (Girard *et al.*, 2005) highly
345 concentrated in OH⁻ ions (strong base). In contrast, the low solubility of coarse chalk
346 fragments limits the basicity of infiltration waters, which bring few CO₃²⁻ (weak base).

347 As expected for the afforestation chronosequence, we found a progressive
348 acidification process over time, from topsoil to deeper soil horizons, with the oldest
349 afforestation events (C→F₁₀₀₀₋₁₅₀₀) showing the largest decrease in soil pH values relative to
350 the ancient culture (AC) reference. The magnitude and rate of this acidification dynamic,
351 relatively to the C→C reference, is more important towards the soil surface where the main
352 sources of acidity are concentrated: litter decomposition (Markewitz *et al.*, 1998; Reich *et*
353 *al.*, 2005); root exudation and respiration (Hinsinger *et al.*, 2003); and atmospheric
354 precipitations (Van Breemen *et al.*, 1983). Below pH = 5.5, soil Al solubilization trigger acidity
355 buffering, which decrease acidification kinetic on the acid forest plots. Hence, depending on
356 the time since afforestation, the imprint of the former cultivation on soil pH can still be
357 visible in the deepest part of the soil profile. For instance, 150 years after afforestation
358 (C→F₁₈₆₅), soil pH values below 200cm depth are more or less similar to the C→C reference.
359 Noteworthy, our results suggest that the alkalisation process after deforestation and
360 culture is faster (F→C₁₈₄₅; chalk) to much faster (F→C₁₈₆₅; defecation lime) than the
361 acidification process after afforestation.

362

363 *Towards a use of pH for dating of LUCs?*

364 Our results of alkalinisation dynamics after deforestation and culture are very consistent
365 with former findings from the East Hesbaye area in Belgium (Pécrot, 1956), where the
366 alkalinisation front propagated until 40cm (B₂₁ horizon) and 125cm (B₃) depths after 50 and
367 100 years of cultivation, respectively. However, pH data from the West Hesbaye area
368 (Colinet, 2003) suggested slower alkalinisation dynamics with slightly acid pH values in the
369 Ap horizon after 100 years of cultivation and such acidity preserved below 150cm depth
370 even after 200 years of cultivation.

371 Owing to the diversity of liming practices (chalk vs. hydrated lime, frequency, amount
372 by Ha, granulometry) there is a high variability of alkalinisation dynamics after deforestation
373 and culture. If farmers use fine-grained hydrated lime, this could trigger a very rapid and
374 deep alkalinisation process whereas if they use coarse chalk blocks this process will be much
375 slower. Because of this variability, it appears difficult to date a deforestation event just from
376 soil pH profiles except if precise historical liming practices are available to infer the most
377 likely alkalinisation dynamics. Therefore, historical archives should always be preferred over
378 inferences from soil pH profiles only to date a deforestation event.

379 For the afforestation chronosequence, we found an age dependent evolution of the
380 acidification dynamic from topsoil to the deeper horizons. In the upper soil layers, pH values
381 rapidly decreased with time and became hardly discriminable from the C→F₅₀₀ reference
382 150 years after afforestation. In contrast, beyond one meter depth, the more recently
383 afforested soils (i.e., 15 years ago) were indistinguishable from the cultivated soils. Similarly
384 and beyond 100cm depth, soil pH values from the C→F₁₉₃₅ & C→F₁₈₆₅ LUC trajectories are
385 indistinguishable.

386 Considering soil pH at 50cm depth, it rapidly decreased from 7.33 to 5.61 during the
387 first 150 years and then slowly decreased to 4.74 after 1500 years of forest land use (Figure
388 4). Our 5 plots from the last 150 years were precisely dated and we compared our results
389 with the pH_{CaCl2} values, converted in pH_{water} (Libohova *et al.*, 2014), from the Rothamsted

390 experimental station (Figure 4, Blake *et al.*, 1999) where the climatic conditions are close to
391 our study area. Both datasets show high consistency and suggest that soil pH at 50cm depth
392 is negatively correlated with afforestation age during the first 150 years (see Figure 4). The
393 slower decay of soil pH values at 50cm depth after 150 years likely reflects the increase in
394 the soil buffering capacity caused by the alteration of aluminosilicates when pH drops below
395 5.5. The basal part of the studied soil profiles (2m to 3m depth) best preserves the initial soil
396 properties before LUCs, like the C→F_{1865ck} LUC trajectory which still show high pH values at
397 3m depth due to loess initial calcium carbonate. As 2-4 centuries of chalk liming practices
398 are necessary to complete deep soil deprotonation and re-saturate exchange complex with
399 Ca²⁺ (Colinet, 2003), only the afforestation plots on ancient fields display deep neutro-
400 alkaline profiles when acidification began.

401

402 **Conclusion**

403 Our findings suggest that one century of liming practices with fine-grained hydrated lime is
404 enough to reach a complete deprotonation of formerly acid forest soils. A much slower
405 alkalization is observed when coarse chalk (or limestone) blocks are used during 170 years
406 of cultivation and liming, with pH values remaining slightly acid (6-6.5) below 2m depth. We
407 assume that two to four centuries of chalk liming practices over loessic luvisols should be
408 necessary to completely neutralize the relictual acidity below 2m depth. Hence, the
409 occurrence of imprints of former forest occupation in the deepest soil layers will mainly
410 depend upon the wide diversity of liming practices through time rather than time elapsed
411 since deforestation and culture. Per contra, imprints of former agricultural lands limed
412 during at least two to four centuries will still be visible in the deepest soil layers more than
413 millennia after afforestation. The acidification dynamic is more regular and progressive from
414 the surface to the deepest soil horizons. Finally, we argue that soil pH at 50cm depth could

415 be used as a proxy to track afforestation events that happened few decades to a millennium
416 ago. Below 2m depth, heterogeneities between soils initial alkalinity levels may still
417 influence the level of acidity of afforested sites. Human exploitation of the landscape deeply
418 transform the pedosphere chemistry through history and studying deep soil pH profiles may
419 help, together with complementary sources, to reconstruct the history of past land uses.

420

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425

426 **Supporting information**

427 Figure S1: Histogram of residuals on the best AIC score model (m3g)

428 Figure S2: Soil textures (Pipette method NF X31-107) ratio values for Clay-Silt-Sand

429 Figure S3: Mean pH of the plots studied in the two chronosequences: a-afforestation on

430 culture and b-deforestation and culture

431 Table S1: Local liming practices from the lower medieval period to the modern period and

432 antique liming practices over Gaul and Britain territories.

433 Table S2: Liming practices on the recent field plots ($F \rightarrow C_{year}$) following oral communications

434 and archives.

435 Table S3: Geohistory and geohistorical arguments of the studied plots over the a)

436 afforestation and b) deforestation chronosequences

437 Table S4: Number of soil samples at each soil depth and for each of the 13 levels

438 characterizing the factor variable of LUC trajectories

439 Table S5: Summary statistics of the average soil profile, in terms of humus type, horizon
440 colors and depth limits, for each of the 13 levels

441 Table S6: Alkaline reagent used on our F→C plots and their principal characteristics

442 Appendix S1: Comments on intra- and inter-plot variability

443

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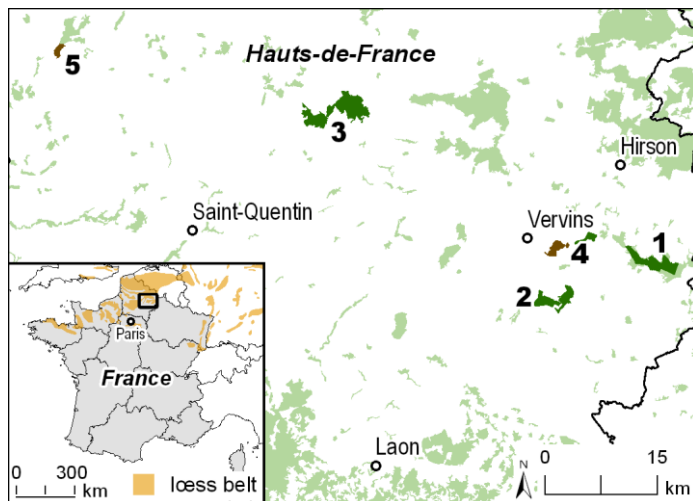
Table 1. Summary statistics (mean and standard deviation) of soil pH values at different soil depths and for each of the 13 modalities characterizing the factor variable of land-use change (LUC) trajectories (cf. the afforestation and deforestation chronosequences). The meaning of each of the levels of the factor variable of LUC trajectories is fully described in the main text of the manuscript (see the Materials and Methods section). The total number of soil profiles (i.e., sample size) per LUC trajectory and per soil depth is provided in Table S4.

Depth (cm)	C→F ₅₀₀		C→F ₁₀₀₀₋₁₅₀₀		C→F _{1865ck}		C→F ₁₈₆₅		C→F ₁₉₃₅		C→F ₁₉₉₉		F→C ₂₀₀₉		F→C ₁₉₈₅		F→C ₁₈₇₅		F→C ₁₈₆₅		F→P ₁₈₄₅ →C ₁₉₂₀		F→C ₁₈₄₅		C→C		
	Mean	σ	Mean	σ	Mean	σ	Mean	σ	Mean	σ	Mean	σ	Mean	σ	Mean	σ	Mean	σ	Mean	σ	Mean	σ	Mean	σ	Mean	σ	Mean
5	4,61	0,34	4,90	0,27	4,78	0,41	5,16	0,26	6,10	0,21	6,03	0,33	7,08	0,61	6,19	0,39	7,88	0,24	7,67	0,18	7,63	0,23	6,09	0,47	7,19	0,37	
25	4,63	0,28	4,90	0,05	4,84	0,36	5,18	0,31	5,99	0,32	6,22	0,25	7,05	0,83	6,18	0,43	7,77	0,28	7,76	0,25	7,65	0,21	6,72	0,10	7,28	0,43	
50	4,74	0,21	5,05	0,18	5,50	0,32	5,61	0,34	6,11	0,12	6,66	0,27	4,68	0,35	4,81	0,18	7,91	0,15	7,70	0,25	7,70	0,15	6,98	0,08	7,33	0,30	
75	4,98	0,21	5,34	0,21	5,92	0,10	5,80	0,29	6,31	0,17	6,90	0,18	4,65	0,18	4,77	0,26	7,86	0,13	7,69	0,26	7,68	0,15	7,02	0,15	7,34	0,22	
100	5,17	0,18	5,60	0,22	6,20	0,27	6,02	0,27	6,43	0,21	7,13	0,15	4,87	0,29	5,07	0,15	7,84	0,11	7,66	0,27	7,39	0,42	6,73	0,34	7,34	0,25	
125	5,34	0,19	5,65	0,18	6,56	0,17	6,44	0,20	6,53	0,28	7,22	0,16	5,12	0,24	4,99	0,21	7,79	0,15	7,49	0,35	6,79	0,69	6,60	0,48	7,24	0,25	
150	5,46	0,26	5,75	0,17	6,84	0,21	6,58	0,29	6,40	0,33	7,29	0,17	5,27	0,26	4,95	0,20	7,72	0,09	7,36	0,40	6,09	0,68	6,61	0,50	7,22	0,28	
175	5,49	0,28	5,83	0,17	7,07	0,23	6,71	0,30	6,33	0,23	7,24	0,17	NA	NA	4,91	0,19	NA	NA	7,36	0,38	NA	NA	6,57	0,48	7,16	0,36	
200	5,54	0,28	5,78	0,20	7,34	0,46	6,78	0,28	6,41	0,23	7,21	0,17	5,33	0,27	4,91	0,18	7,71	0,18	7,27	0,31	5,78	0,72	6,54	0,45	7,15	0,30	
250	5,66	0,47	5,91	0,30	7,91	0,43	6,80	0,30	6,44	0,26	7,24	0,20	5,18	0,14	4,98	0,61	7,71	0,22	7,30	0,28	5,51	0,36	6,33	0,62	6,95	0,29	
300	5,71	0,52	5,91	0,37	8,18	0,05	6,87	0,25	6,74	0,26	7,18	0,24	5,30	0,12	5,07	0,51	7,64	0,15	6,98	0,61	5,44	0,23	6,25	0,60	6,99	0,24	

1 **Table 2.** The 12 tested models clustered in 3 different categories of four candidate models (m0, m1,
 2 m2, m3): ordinary least square (OLS) regression models with a linear relationship between soil pH
 3 and soil depth; OLS regression models with a second-order polynomial (mXp) relationship between
 4 soil pH and soil depth; and generalized additive models (GAMs: mXg) allowing more complex non-
 5 linear relationships. Difference in Akaike Information Criterion (AIC) values between the best model
 6 and the focal model (ΔAIC), Akaike weights (w_i), adjusted R^2 values (R^2_{adj}) as well as degrees of
 7 freedom (Df) are provided for comparison purposes.
 8
 9

Modelling approach	Id	Depth	LUC	Depth x LUC	AIC	ΔAIC	w_i	R^2_{adj}	Df
generalized additive model	m3g	X	X	X	519	0	0	0,91	968
two order polynomial model	m3p	X	X	X	818	299	<0,01	0,88	996
linear model	m3	X	X	X	1026	508	<0,01	0,85	1008
generalized additive model	m2g	X	X		1843	1324	<0,01	0,69	1014
two order polynomial model	m2p	X	X		1843	1324	<0,01	0,69	1018
linear model	m2	X	X		1845	1327	<0,01	0,67	1019
linear model	m1	X			2967	2448	<0,01	0,02	1030
generalized additive model	m1g	X			2967	2448	<0,01	0,04	1026
two order polynomial model	m1p	X			2968	2449	<0,01	0,04	1029
linear model	m0				2987	2468	<0,01	/	1031
two order polynomial model	m0p				2987	2468	<0,01	/	1031
generalized additive model	m0g				2987	2468	<0,01	/	1031

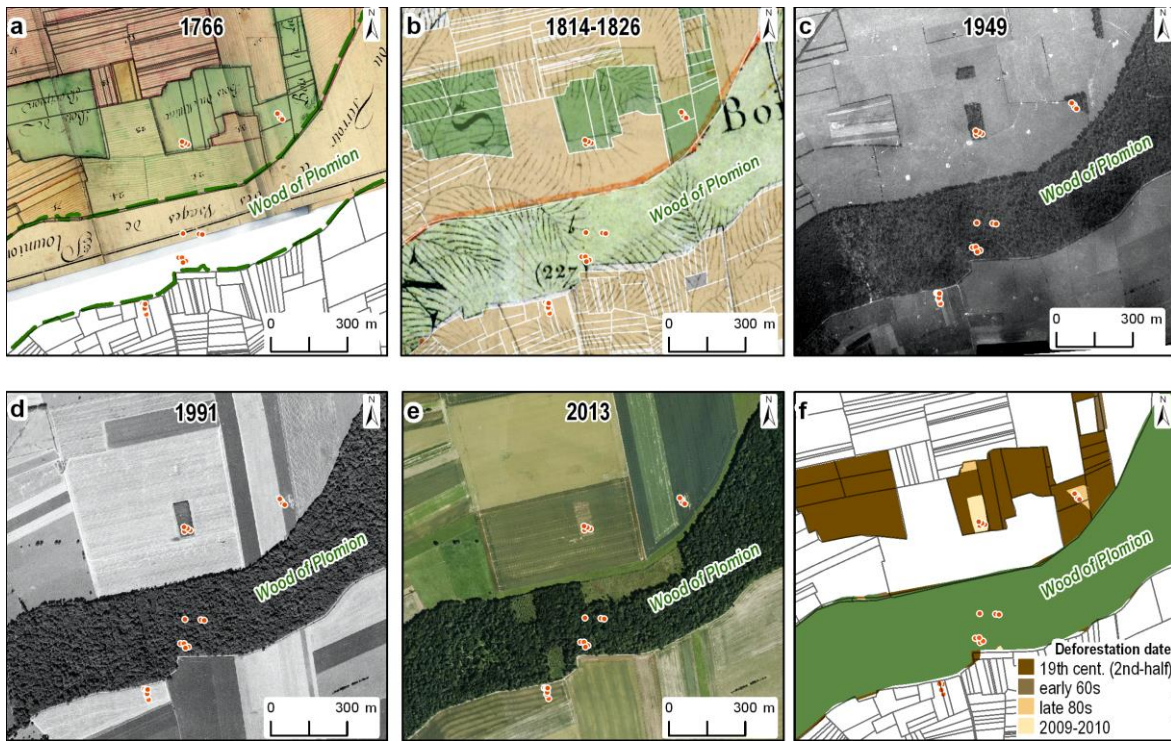
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 22 **Figure 1.** Map of forested areas (light green color; Corinne Land Cover 2006) within the studied
 23 spatial extent located in North France (Aisne; Hauts-de-France) with the 5 focal study sites labelled
 24 from 1 to 5: (1) the Haye of Aubenton; (2) the Val-Saint-Pierre forest; (3) the Andigny forest; (4) the
 25 Plomion wood (East) and the Thenailles ancient forest now deforested (West); and (5) the Metz-en-

26 Couture/Havrincourt ancient forest now partly deforested. The dark green color refers to ancient
27 forests still afforested while the brown color refers to ancient forests now deforested.

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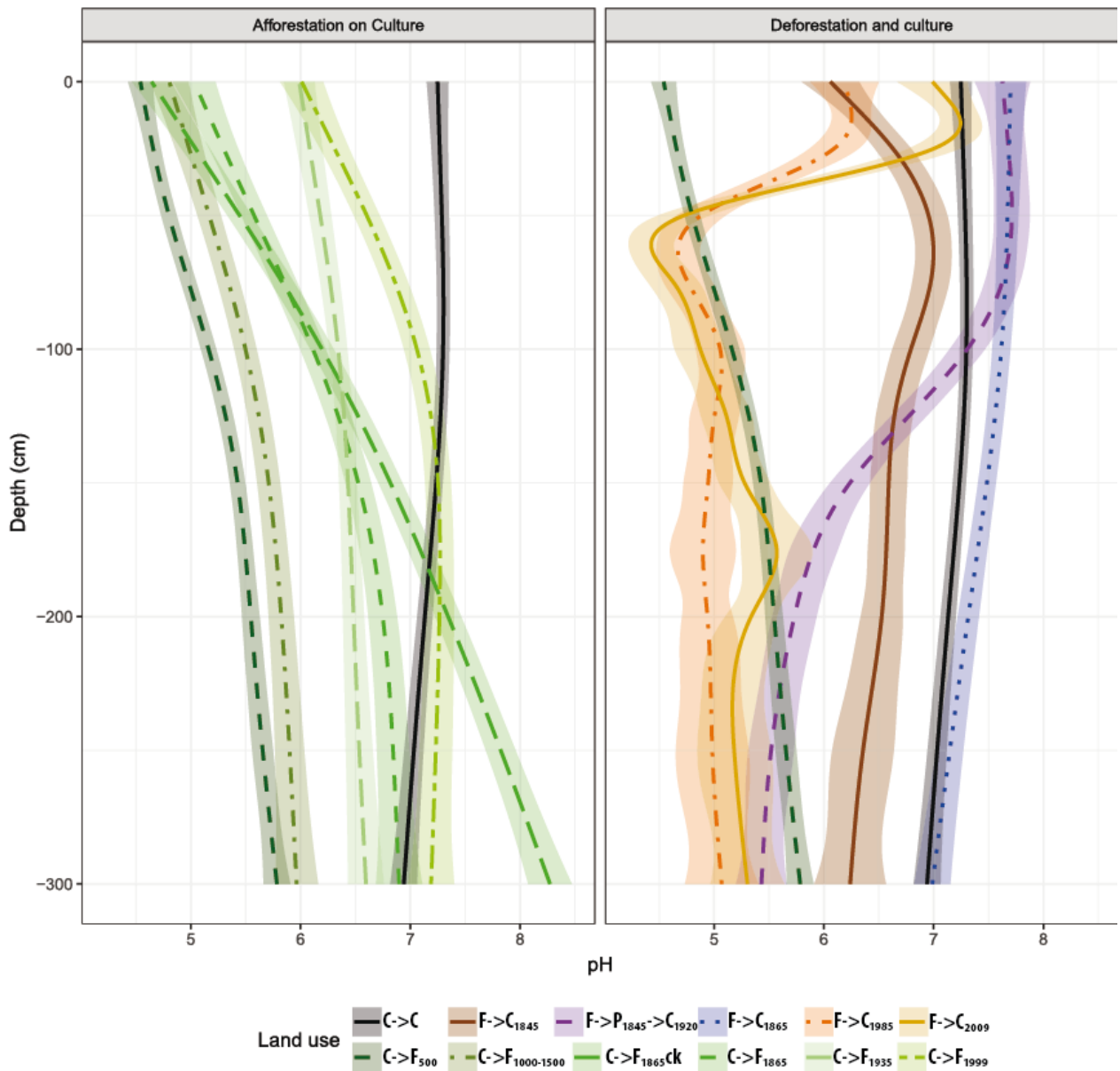
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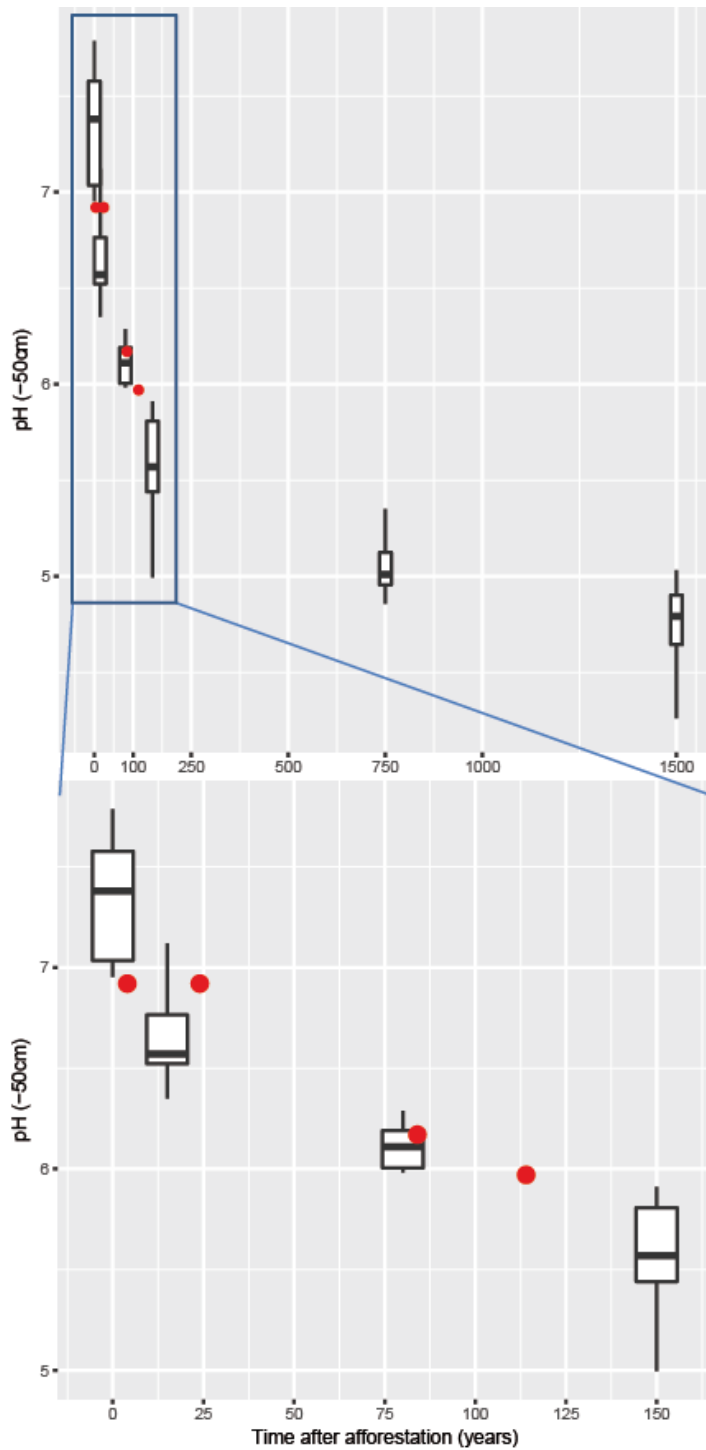
32 **Figure 2.** Geohistorical reconstruction of the Plomion wood area: (a) old map (1766) of the Foigny's
33 abbey domain (AD02 H658b); (b) Napoleonic cadaster with associated land-use status (1814) and
34 Etat-Major map (1826); (c) aerial photograph (1949) (Source: IGN-Geoportail); (d) aerial photograph
35 (1991) (Source: IGN-Geoportail); (e) aerial photograph (2013) (Source: IGN-Geoportail); and (f)
36 geohistorical reconstruction made within a geographic information system (GIS) (Source: ArcGIS-
37 ESRI). Red dots on the maps show the exact locations of the studied soil profiles (n = 24) within the
38 Plomion wood study area with some soil profiles located in the ancient forest of Plomion afforested
39 since a very long time (before 1194) and some located on different deforestation chronosequences
40 ranging from recent (F→C₂₀₀₉) to older (F→C₁₉₈₅) deforestation events.

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44 **Figure 3.** Predicted soil pH values along the soil depth profiles for each of the 12 different levels
 45 characterizing the factor variable of land-use change (LUC) trajectories (cf. the afforestation and
 46 deforestation chronosequences). Predictions are based on the best model (see Table 2) that accounts
 47 for the interaction effect between soil depth and LUC trajectories as well as for complex non-linear
 48 relationships between soil depth and soil pH. Shaded surfaces show the 95% confidence interval
 49 (1.96xSEM; where SEM means standard error of the mean) around the estimated coefficients. The
 50 meaning of each of the levels of the factor variable of LUC trajectories is fully described in the main
 51 text of the manuscript (see the Materials and methods section).



52
 53 **Figure 4.** Variation in soil pH values at 50cm depth for different afforestation chronosequences
 54 depicted by the time elapsed since the afforestation. Soil profiles located on ancient cultivated fields
 55 (C→C) are used as the baseline (cf. age = 0) whereas soil profiles located within ancient forests are
 56 used as the other extreme situation of afforestation since a very long time (cf. age = 1500). For the
 57 C→F₁₀₀₀₋₁₅₀₀ level of the factor variable of land-use change (LUC) trajectories, data are positioned at
 58 the median year after afforestation, i.e., 750 years ago. Box plots show data from this study whereas
 59 red dots show pH_{CaCl2} data from Geescroft site (Blake 1999) converted in pH_{water} (Libohova and others
 60 2014). The lower panel depicts a zooming window for the 0-150 years interval.

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