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PHYSICOCHEMICAL ANALYSIS OF NEOTROPICAL SOILS

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Directly inspired by soil science approaches, physicochemical analyses applied to archaeology are part of geoarchaeology, as is micromorphology (see Chapter 3). Initially, in the 1930s, soil phosphorus analysis was used as the first indicator of human activity (Holliday 2004). Subsequently, numerous studies redefined the chemical potential of the remains/residues of past human occupations in various cultural and environmental contexts, through multielement analysis (Barba and Ortiz 1992; Costa and Kern 1999; Dore and López Varela 2010; Holliday *et al.* 2010; Middleton 2004; Oonk *et al.* 2009; Wiedner *et al.* 2015). These analyses highlight the 'chemical-anthropogenic' signature recorded by the soil and are an important tool to deduce the use of space by ancient populations in a given place (Wilson *et al.* 2008; Holliday *et al.* 2010). For example, in Amazonian dark earths (ADEs), the physicochemical analyses carried out revealed contents in chemical elements that are significantly higher than those of the surrounding natural soils (such as oxisols), while being quite variable, reflecting different areas of activity within the same site (Costa and Kern 1999; Schmidt *et al.* 2014).

The domestic space of an occupation area can thus be reconstructed thanks to the chemical elements contained and preserved in the soil (e.g. Ca, P, Mg, K, Mn, etc. [Kern *et al.* 2015]). However, physicochemical analyses only lead to a residual quantitative assessment (inheritance) of the different organic or organo-mineral human inputs and do not reflect the original qualitative dimension, which is essential for the characterisation of human activities (Courty and Miskovsky 2002). Instead, coupling the interpretation of the physicochemical properties of soils with micromorphological analyses makes it possible to extend the overall scope of research and illustrate the remarkable heterogeneity that characterises archaeological anthrosols and their value as an archive of past human activities (Arroyo-Kalin 2014; Brancier 2016; Brochier 2002; Macphail and Goldberg 2017). This approach allows us to qualitatively and quantitatively define the specificities and features of anthrosols, possibly to confirm micromorphological observations, but also to propose new interpretations or lines of research. The archaeological application of geochemical analyses is recent in French Guiana, as soil analyses have so far been restricted mainly to pedological studies, and very few have been used to understand past human occupations.

Methodology: from field to lab with a bucket full of soil

Field procedure

Soil sampling should be carried out with a detailed description of the horizons and only after a good understanding of the local geomorphological context has been reached (see Chapter 2). For French Guianan anthrosols, bulk samples of about 500 grams were collected, with the incremental sampling depth depending on the description of the soil profile. In order to couple soil analysis results with micromorphological observations, soil samples were taken at the locations of the micromorphological samples (see Chapter 3).

Laboratory procedure

Samples were oven-dried for a few days at 50 °C maximum, then sieved through a 2-mm mesh and crushed to a grain size of 0.2 mm for subsequent analyses (Table 4.1). The coarse fraction (>2 mm) was conserved for further characterisation of soils and/or anthropogenic features. The choice of analyses to be carried out was based on the literature on *terra preta* (Costa and Kern 1999; Costa *et al.* 2013; Arroyo-Kalin 2014) in order to allow comparisons. The main analytical methods are referred to in Pansu and Gautheyrou (2006).

Analyses and their interpretations

Invisible remains of past human occupations can still be detected using a set of analytical procedures (Figure 4.1, Table 4.2).

Particle size analysis

Particle size analysis allows us to know the precise texture of the soil horizon (or pedostratigraphic unit), especially the distribution of fine mineral particles <2 mm, according to size classes. The results are directly related to deposition processes and soil functions, and may be strongly correlated with other analytical data. This analysis may also help in the interpretation of discriminating micromorphological features and pedogenetic processes (Goldberg and Macphail 2006, Chapter 2).

TABLE 4.1 Analyses carried out on archaeological pre-Columbian anthrosols of French Guiana

Analysis	*Methodology used*	*Reference*	*Global information*
Particle size	8 (sandy soils) or 5 (clayey soils) fractions without decarbonation	NF × 31–107	Distribution of particles in size, comparison of samples, mechanical and physical behaviour of the soil
Free or bound organic matter between 300 °C and 500 °C	Loss of ignition at 550 °C	Pansu and Gautheyrou (2006)	Organic matter (OM): total C and N, free or bound organic matter, biochemical stability of OM, degree of humification
Loss of ignition	Loss of ignition at 1100 °C	Pansu and Gautheyrou (2006)	
C/N	Ratio of organic carbon to total nitrogen evaluated by dry combustion after heating the sample to about 1000 °C	NF ISO 10694 and ISO 13878	
pH (water and KCl)	Suspension of the air-dried soil sample in solution in a volumetric ratio of 1/5	NF ISO10390	Soil acidity: active acidity and exchangeable acidity
Extractable phosphate	Olsen and Olsen modified by Dabin	NF ISO 11263	Soil chemical status: exchangeable major and minor elements distribution in soil, environmental soil conditions Possible past human activities
Cation exchange capacity (CEC)	Cobaltihexamine (spectrocolorimetry) chloride extraction [Co (NH3) 6Cl3] at 50 mmol/L	NF ISO 23470	
Exchangeable cations: Ca, Mg, Na, K, Fe, Mn, Al	Cobaltihexamine (ICP – AES = argon-induced plasma atomic emission/for Na, EAF = atomic emission of flame)	NF ISO 23470	
Total elements: P, Ca, Mg, K, Mn, Al, Fe, Na, Cu, Zn	Aqua regia: combination of nitric acid and hydrochloric acid (HNO_3 + HCl)	ISO 11466/NF ISO 22036	Soil chemical status: may reveal past human activities

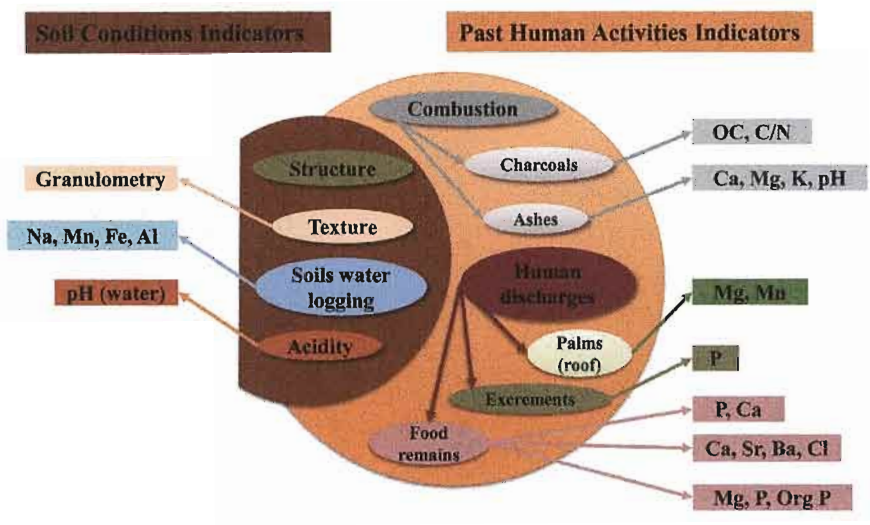

FIGURE 4.1 Useful physicochemical markers for pre-Columbian anthrosols

Source: © J. Brancier.

TABLE 4.2 Sample analytical features and interpretations for several cultural and biophysical contexts

Pedofeatures/ chemical elements	*References*	*Context*		*Interpretation*
		Sandy floodplain	*Clayey highland*	
	Indicators of past sub-surface/surface layers			
Carbon peak **Exchangeable cations peak**	Browne Ribeiro (2014)	X	X	In natural soils, high carbon peaks correspond to the humus zone. Human activities induce the accumulation of various chemical elements on the soil surface. High values can thus correspond to past occupation surface layers.
Exchangeable Fe	Woods (2009)	X	X	Iron reservoirs in soils are (i) ionic and complexed form in solution; (ii) exchangeable; (iii) organically complexed but water insoluble; (iv) insoluble inorganic precipitates; and (v) held in primary minerals. As exchangeable Fe is pH and OM dependent, it can materialise limits between different occupation levels.

(*Coninued*)

TABLE 4.2 (Continued)

Pedofeatures/ chemical elements	*References*	*Context*		*Interpretation*
		Sandy floodplain	*Clayey highland*	
	Soil reworking			
Fe and Al concentrations **Total Fe and Al** **Exchangeable Fe and Al**	Woods (2009)			A stratigraphic inversion (unnatural distribution of concentrations) may indicate human activities such as digging, backfilling and levelling.
	Human food discharges			
Exchangeable and total cations **P and Ca** **Ca-Sr-Ba-Cl, Mg-P, Org P** **Na**	Costa and Kern (1999); Wilson *et al.* (2008); Oonk *et al.* (2009); Couture *et al.* (2016)	X	X	The discharge of food remains, notably bones, results in high concentrations of these elements.
	Soil combustion			
Organic matter (OM)	Certini (2005)			Surface fires cause a decrease in OM, which is completely consumed beyond 300 °C.
Organic carbon (OC) and black carbon (BC) = charcoals (Ch)	Glaser and Birk (2012)			Charcoals result from incomplete combustion of wood at 250 °C–500 °C. OC and BC increase in A-horizon indicate opening area (probably for agriculture). The high contribution of BC to OC (40%) stabilises soil OM.
N **C/N**	Neary *et al.* (1999), Fisher and Binkley (2000)			N is volatilised by fires up to 200 °C–400 °C, also inducing variations in C/N.
Organic P (OP)	Cade-Menun *et al.* (2000)			Fires result in a transient enrichment of available P. Soil combustion turns OP into orthophosphates, better conserved in acidic soils (binding with Al, Fe or Mn oxides).

OP, Ca, Mg, K **pH**	Certini (2005)	Ash deposits on soil surface are rich in these elements, which are rapidly leached. pH significantly increases after fires beyond 450 °C–500 °C.
δ13C	Turney *et al.* (2006)	δ^{13}C depends on the type of vegetation cover (forest/grasses) that has been burnt.
	Environmental soil conditions	
Na, Fe & Al	Glaser and Birk (2012)	High concentrations indicate hydromorphic conditions (soil waterlogging).

pH

Soil acidity is a crucial factor for the preservation of organic constituents, particularly faunal/human remains such as bones and plant residues. The pH quantifies the active (or actual) acidity or alkalinity expressed by the concentration of dissociated protons in the soil solution and exchangeable (or reserve) acidity resulting from protons fixed on the exchangeable complex and likely to move after exchange with neutral salts (KCl). This is an essential routine analysis for the interpretation of other soil properties, such as cation exchange capacity (CEC; see later). Active acidity is a measure of the hydronium (H_3O^+) ion concentration (pH) in a soil solution or in a soil–water suspension; potential acidity is a measure of the sample's ability to produce exchangeable hydronium (H_3O^+) ion by cation exchange with salt solutions (KCl, $CaCl_2$).

Amazonian ferralsols, acrisols and arenosols have a high acidity (about pH 4 in water) that may reflect high levels of exchangeable Al (Glaser and Birk 2012). Intense and/or long human occupations tend to increase the soil pH, i.e. to lower acidity (Holliday 2004; Glaser and Birk 2012). The presence of ashes and charcoal will also increase the pH (Balme and Paterson 2006). Within the same profile, it is possible to find variations in pH related to microlocal soil conditions: for example, leaching events may cause lixiviation of cations from upper to lower horizons, thus increasing pH from top to bottom (Macphail and Goldberg 2017). It is important to keep in mind that the measured pH in water corresponds to the active acidity at the time of the analysis, which may have been very different over time, especially in the case of highly leached tropical soils (van Wambeke 1992).

Cation exchange capacity

The CEC is the total amount of exchangeable cations that soils can absorb. It expresses the potential number of cations needed to neutralise negative soil charges and allows us to estimate the physicochemical state of the soil. For French Guianan archaeological anthrosols, we chose the cobaltihexamine extraction method, the most appropriate for acidic soils, because it is done with a pH close to the *in situ* soil conditions (Pansu and Gautheyrou 2006). This allows us to determine the main exchangeable cations of the soil and to obtain a so-called 'effective' CEC (Ciesielski *et al.* 1997).

Exchangeable nutrients

Naturally occurring ferralsols, acrisols and arenosols contain very low amounts of nutrients, giving them a very low fertility (Glaser and Birk 2012). Two main types of nutrients are distinguished: (i) macronutrients, often absorbed in large quantities by plants, such as nitrogen (N), phosphorus (P), calcium (Ca), magnesium (Mg) and potassium (K), and (ii) micronutrients, which are essential at low doses, such as boron (B), iron (Fe), zinc (Zn), manganese (Mn), copper (Cu) and aluminium (Al). Among these, some can be toxic to plants if they are in excess, such as Zn, Mn, Cu or Al, especially in tropical soils (Duchaufour *et al.* 2018). ADEs are distinguished from natural Amazonian soils by their high CEC and concentrations of P, Ca, K, Mg and, to a lesser extent in some sites, Cu, Mn and Zn (Arroyo-Kalin 2014; Costa *et al.* 2013; Glaser and Birk 2012; Kern *et al.* 2015).

Phosphates

A phosphate ($H_2PO_4^-$, HPO_4^{2-}, PO_4^{3-}) is a chemical derivative of phosphoric acid. The 'Olsen' extractor method is the standard method applicable to a wide range of soils (Fardeau *et al.* 1988). Nevertheless, it is based on measurement at a basic pH (8.5), which does not correspond to the *in situ* conditions of the natural Amazonian soils. Dabin (1980) proposed a modification to Olsen's method that is more suitable for acidic soils. This procedure is a little more aggressive, increasing the extraction time compared to the original Olsen method, but allows one to extract the phosphate ions bound to iron oxides. It is thus a better proxy of the contribution of organic matter to the available phosphorus in soils that are initially poor in this element (Baize 2018).

Total elements

The extraction method with aqua regia is well adapted for tropical soils (Arroyo-Kalin 2014). It extracts only a fraction of the total cations and aluminium, depending on the nature of minerals, i.e. on soil type (see Pansu and Gautheyrou [2006], for the choice of acid to use). Total concentrations are measured by complete extraction of all forms of the elements sought, including those integrated into the crystal networks of primary and secondary minerals (i.e. clay minerals, oxides, etc.) (Baize 2018). The total iron (Fe) content of a soil depends on the parent material and the type of horizon. The lowest values (from 0.05% to 1%) are observed, for example, in sandy and loamy eluvial horizons that are poor in clay and iron. The highest Fe concentrations range from 6% to 19% in clayey horizons derived from particular materials, such as 'ferro-manganic' accumulations (van Wambeke 1992).

Organic matter

Organic matter (OM) plays an important role in many soil functions, such as maintaining structural stability, resistance to erosion, ability to improve water retention, or CEC (Silva *et al.* 2009).

For tropical soils, several parameters can be measured to evaluate organic matter, such as volatile matter at 550 °C and loss of ignition at 1100 °C. These two values reflect the mass lost by calcination, which is an estimate of the amount of OM. In the first case, limiting the temperature to 550 °C does not cause charcoal to burn. This allows us to quantify microcharcoals and/or ashes, hardly distinguishable from each other, even with a polarised microscope (Brochier 2002). At 1100 °C, the losses recorded at 550 °C are added to those resulting from the decomposition of various materials, including carbonates, and from the evaporation of water trapped in some soil components such as clays (Pansu and Gautheyrou 2006).

The C/N ratio (C = organic carbon; N = total nitrogen) makes it possible to judge the degree of evolution of OM, and its determination is useful in non-carbonated soils such as the Amazonian ones. In most national laboratories, a multiplier factor of 1.72 is applied to estimate the organic matter content directly from the organic carbon (OC). However, using such a single multiplier factor is not necessarily appropriate, as the forms of OM vary according to the humification degree (Baize 2018; Pansu and Gautheyrou 2006). Therefore, it seems more conservative to reason on the basis of the measured OC content, rather than on such an estimate of the OM. In a humid tropical context, it is generally accepted that plant matter degrades fairly rapidly under the influence of climate and pedofauna, theoretically inducing a low C/N ratio. However, in the archaeological context, human activities have contributed to increasing the OM, therefore skewing the pedologic interpretation of the C/N ratio, which tends to be higher than in natural soils. This is particularly the case for anthrosols, which are very rich in carbonised OM such as charcoals (Macphail and Goldberg 2017).

$\delta^{13}C$

The $\delta^{13}C$ is an isotopic signature of the palaeovegetation type. In natural environments, enrichment of soil OM comes mainly from vegetation, through litter and roots. In agricultural soils, carbon comes from two major sources: the remains of the old vegetation and the decomposition products of the new crops (Bernoux *et al.* 1999). The OM of soil surface horizons has a $^{13}C/^{12}C$ isotope ratio very close to that of the vegetation from which it comes (Deines 1980). However, the values of $\delta^{13}C$ vary according to three main groups of plant species, differentiated by their carbon uptake pathways: C_3, C_4 and Crassulacean acid metabolism (CAM).

C_4 plants, mainly grasses (*Poaceae*), are significantly less depleted in ^{13}C ($\delta^{13}C$ varying between −9 and −17 ‰ with a mean around −13 ‰) than C_3 plants, a group to which belong most forest species ($\delta^{13}C$ between −22 and −33 ‰, on average −27 ‰) (Boutton 1991; Fernandes *et al.* 2007). By monitoring the $\delta^{13}C$, it is possible to observe spatio-temporal changes in the dynamics of soil OM when two vegetation covers follow one another (e.g. Martin *et al.* 1990; Vitorello *et al.* *1989*). Freycon *et al.* (2010) conducted a robust survey of the $\delta^{13}C$ of soils under French Guianan forests, and Fujisaki *et al.* (2017) measured the C_4 carbon accumulation resulting from a modern change of land use.

Conclusion: routine and flexibility

The list of analytical procedures presented here is not exhaustive. Many soil analysis protocols are internationally standardised, continuously modernised and commonly referred to as 'routine' in soil science. Adapting to the archaeological context requires close discussion with the laboratory for possible methodological adaptations, considering the specificity of anthrosols. The preparation of the bulk sample is the first step that demands special attention to ensure the highest quality of analysis. The conservation of high-quality samples over a long period, in order to allow subsequent analyses with new methodologies and to detect possible analytical drifts, is of utmost importance. Considering that the cost of a set of analysis is lower than that of fieldwork, the establishment of a sample conservatory seems essential.

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