



Inferences from the vertical distribution of Fe isotopic compositions on pedogenetic processes in soils



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ABSTRACT

The isotopic compositions of major elements in soils can help understand the mechanisms and processes that control the evolution of soils and the nature and dynamics of the soil constituents. In this study, we investigated the variations of the Fe concentrations and isotopic compositions combined with classical soil parameters, such as granulometry, pH, and C and N concentrations. We selected three soils submitted to different hydrodynamic functioning along a toposequence: a well-drained Cambisol and two hydromorphic soils, an Albeluvisol and a Gleysol. In the Cambisol, the isotopic variations were small indicating little redistribution of Fe which we attributed to centimetric-scale exchanges from the Si-bound to the weakly-bound iron pools and insignificant subsurface Fe export. In contrast, the hydromorphic soils showed an overall variation of 0.37‰ for $\delta^{56}\text{Fe}$ ($\delta^{56}\text{Fe}$ (‰) = $[(^{56}\text{Fe} / ^{54}\text{Fe})_{\text{sample}} / (^{56}\text{Fe} / ^{54}\text{Fe})_{\text{IRMM-014}} - 1] \times 1000$) and an inverse correlation between the Fe isotopic compositions and the oxide-bound Fe concentrations. We suggest that, in the uppermost horizon, the mobilisation of oxide-bound Fe was due to the reducing conditions and predominantly involved the light Fe isotopes. Similarly, within the Bt horizon of the Albeluvisol, the fluctuations of the water table level induced changes in the redox conditions and thus Fe dissolution and transport of isotopically light Fe. The Fe isotopic composition profile in the B/C horizon of the Gleysol is dominated by the signature of the parental material. Overall, the variations of the underground water table combined with topography-driven water flow were suggested to be the main mechanisms of Fe translocation in these hydromorphic soils. Finally, the comparison between Fe isotope profiles in worldwide soils allows us to show that Fe isotopic variations can help discriminate between various mechanisms and scales of Fe transfer in soils and, accordingly, provide information on the evolution of soils, when used in combination with pedological, geochemical, geographical, and environmental characterisations.

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1. Introduction

Understanding mechanisms and processes that control soil evolution, the nature and dynamics of major soil constituents and their behaviour in response to natural and anthropogenic changes is among the key scientific challenges in soil research. Iron is one of the major elements in soils. It is released during the alteration of rocks and soils, and participates in mineral neof ormation, thereby playing a crucial role in soil differentiation. Iron concentration in soil varies because of different processes: physico-chemical redistributions without transport and short-distance or long-range transport of Fe. In addition, the fate of Fe influences the chemical cycles of other important elements in soils, such as nutrients (e.g. P, Zn) and pollutants (e.g. As, Cd, Zn) (e.g. Anderson and Christensen, 1988; Jacobs et al., 1970; Ramos et al., 1993). Finally, Fe is an essential micronutrients for plants (Marschner, 1995) and human nutrition (WHO, 2002).

Stable isotopes analyses have proven to be valuable tools to understand biogeochemical processes in soils (e.g. Fry, 2006). Until the end of the 90's, these studies were limited to C, N, O, H, and S. However, significant analytical developments over the past years, notably due to the advent of Multi-Collector Inductively-Coupled Plasma Mass-Spectrometers (MC-ICP-MS), now allow high-precision analyses required to measure the small isotopic variations of the so-called "non-traditional" stable isotopes that include Fe (e.g. Albar de and Beard, 2004; Dauphas and Rouxel, 2006). Iron has four stable isotopes, ^{54}Fe , ^{56}Fe , ^{57}Fe and ^{58}Fe . The δ -notation is commonly used to describe the isotopic fractionation relative to the isotopic reference material IRMM-014: $\delta^X\text{Fe}$ (‰) = $[(^X\text{Fe} / ^{54}\text{Fe})_{\text{sample}} / (^X\text{Fe} / ^{54}\text{Fe})_{\text{IRMM-014}} - 1] \times 1000$, where X = 56, 57 or 58.

In natural environment, $\delta^{56}\text{Fe}$ ranges between -3.5‰ and $+1.5\text{‰}$ (Beard and Johnson, 2004; Beard et al., 2003a; Dauphas and Rouxel, 2006). Igneous rocks represent an isotopically homogeneous terrestrial baseline ($\delta^{56}\text{Fe} = 0.0 \pm 0.05\text{‰}$) (Beard et al., 2003a), whereas isotopic heterogeneities were initially measured mostly in rivers and lakes, rocks and minerals formed at low temperature, hydrogenous ferromanganese

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precipitates, pyrites and hydrothermal systems (e.g. Dauphas and Rouxel, 2006; Fantle and DePaolo, 2004).

Continental weathering involves processes of mechanical and chemical breakdown of rocks and minerals and subsequent soil development. The parent material, i.e., igneous rocks and clastic sediments, show limited isotopic variations: $\delta^{56}\text{Fe} = 0.0 \pm 0.3\%$ (Beard and Johnson, 2004; Fantle and DePaolo, 2004; Johnson et al., 2003). In contrast, soils display a range of variations of the $\delta^{56}\text{Fe}$ from -0.62 to $+0.72\%$ (Emmanuel et al., 2005; Fantle and DePaolo, 2004; Poitrasson et al., 2008; Thompson et al., 2007; Wiederhold et al., 2007a, 2007b) indicating that pedogenic processes, leading to soil formation and evolution, generate Fe isotope fractionations with respect to the parent material. Experimental studies have shown that mineral dissolution results in the preferential liberation of light Fe isotopes to the solution while the residual material becomes accordingly heavier than the parent material (e.g. Beard et al., 1999; Brantley et al., 2004; Wiederhold et al., 2006). In contrast, sorption of Fe(II) onto goethite and mineral ferrihydrite neoformation arising from the abiotic oxidation of aqueous Fe(II) into Fe(III) appears to favour the heavy isotopes (Bullen et al., 2001; Icopini et al., 2004). These experimental results have been confirmed by soil studies that show that, in general, the mobile fraction in soils has lighter Fe isotopic composition (Brantley et al., 2004; Fantle and DePaolo, 2004; Thompson et al., 2007; Wiederhold et al., 2007a, 2007b). Therefore, the variations of the Fe isotopic signatures in soils can help to investigate the behaviour of Fe in the near-surface environment during pedogenesis and to discriminate between potential processes at the origin of soil evolution.

In this study, we investigated both vertical and lateral variations of the Fe concentrations and isotopic compositions combined with the study of classical soil parameters such as granulometry, pH, and C and N concentrations. We used three soil profiles submitted to different hydrodynamic functioning along a toposequence: a well-drained oxic profile and two profiles that are waterlogged during a part of the year. The objectives were to (1) study the expression of the isotopic fractionation of iron in these soils, (2) characterize the mechanisms of Fe transport, (3) compare the results with those obtained in previous studies of the Fe isotopes fractionation in soils and (4) evaluate the potential of Fe isotopes to record information about mechanisms of soil transformations.

2. Material and methods

2.1. Study area, sampling and soil description

The sampling area is located in the Kervidy–Naizin catchment that extends over an area of 4.9 km² in the centre of Brittany, western France and belongs to the Environment Research Observatory (ERO) AgrHyS (response time in Agro-Hydro Systems). The region is characterised by temperate oceanic climate according to the Köppen climate classification, with a mean annual precipitation of 909 mm and a mean monthly temperature ranging from 5.4 °C (January) to 17.4 °C (August). The land-use is dominated by corn and wheat farming, temporary pastures for dairy production and indoor pig-stock breeding.

The soils in this catchment developed from the weathering of the sedimentary Brioverian schist unit (older than 530 Ma) and eolian Quaternary deposits that overlay locally the bedrock (Olivié-Lauquet et al., 2001; Thomas and Le Berre, 2009; Van Vliet-Lanoe et al., 1998; Walter and Curmi, 1998). The soils are organised in a toposequence consisting of three soil types: (1) a well-drained, cropped Cambisol (IUSS Working Group WRB, 2006) on the upper part of the slope, characterised by oxic conditions, (2) an Albeluvisol (IUSS Working Group WRB, 2006) at midslope, which represents a transition zone between the well-drained cropland and the poorly drained lowermost part of the landscape and (3) a Gleysol (IUSS Working Group WRB, 2006) developed next to a small creek that flows at the bottom of the slope. The Albeluvisol and Gleysol are planted with poplar trees. Both soils are

roughly ploughed every two years to avoid weed growth between the trees (Durand et al., 1998). They both undergo seasonal fluctuations of water saturation, with winter–spring corresponding to a period of reducing chemical conditions while summer, or late summer in the case of the Gleysol, is dominated by oxidative chemical conditions (Davranche et al., 2011; Trolard et al., 2002). The water table in these two soils is located close to the topographic surface (Davranche et al., 2011; Olivié-Lauquet et al., 2001; Pauwels et al., 1996). During rainy periods a temporary water table could develop in the Ap-horizon and lateral drainage could occur.

The sampled profiles are located close to sites C, I and F described by Trolard et al. (2002) for the Cambisol, the Albeluvisol and the Gleysol, respectively. We sampled the soils using an auger with one sample collected every 10 cm respecting the horizons boundaries.

In the Cambisol, we sampled the Ap- and B-horizons (Supplementary Table 1). The Ap-ploughing horizon (0 to 20 cm depth) has a silty texture and a dark brown colour (10YR3/1 according to the Munsell chart). The B-horizon (20 to 60 cm depth) has a silty texture and a yellowish-brown colour (10YR5/6). The ratio of 2–20 μm to 20–50 μm fractions is close to one indicating a parent material of eolian origin for the sampled horizons (Walter and Curmi, 1998), likely similar to the loess of northern and/or western Brittany (Haase et al., 2007; Le Calvez, 1979).

In the Albeluvisol, we sampled the Ap- and Bt-horizons (Supplementary Table 1). The Ap-horizon (0 to 20 cm depth) has a silty texture and a dark-brown to greyish colour (10YR3/2 to 10YR4/2). The Bt-horizon (20 to 60 cm depth) is silty with clay concentration increasing with depth. The ratio of 2–20 μm to 20–50 μm fractions in all the horizons is close to 2 indicating that the soil profile is developed from the sedimentary Brioverian schist parent material (Walter and Curmi, 1998).

In the Gleysol, we sampled the Ap- and B/C-horizons (Supplementary Table 1). The Ap-horizon (0 to ~25 cm depth) is dark grey (10YR2.5/1) with a silty-sandy-clayey texture. The illuvial B/C-horizon (~30 to 80 cm depth) is brown to brown-dark yellow (7.5 to 10YR4/6) with silty to slightly clayey texture. The ratio of 2–20 μm to 20–50 μm fractions in all the horizons is close to 2 suggesting that the soil mainly developed from the sedimentary Brioverian schist parent material (Walter and Curmi, 1998).

The Albeluvisol and Gleysol contain redoximorphic features such as mottling and rust accumulations in root spaces. These features are of millimetric to centimetric size, of brown, yellowish to red or pale grey to greenish colour. They are sparse in the upper Ap horizon but frequently present in the deeper Bt and B/C horizons of the Albeluvisol and Gleysol.

2.2. Methods and analyses

Collected samples were dried at 40 °C and sieved to <2 mm. Aliquots were taken for elemental and isotopic analyses.

2.2.1. Fe concentrations: bulk analyses and selective extractions

Total iron concentration of the bulk soils was determined on 0.25 g aliquots. Each aliquot was dissolved in a HF–HClO₄ mixture after calcination of the organic matter (450 °C).

We also used two methods of selective extractions using reducing agents of increasing strength to obtain information about the major pools of iron in the soils studied: (1) the Tamm's extraction in the dark and (2) the citrate–bicarbonate–dithionite (CBD)-extraction.

The Tamm's reagent is a mixture of oxalic acid and ammonium oxalate (Tamm, 1922). The extraction was performed by shaking the sample-solution mixture over 4 h, at 20 °C and in the absence of light with a solid/liquid ratio of 1.25 g/50 mL. This method allows the extraction of weakly bound, poorly crystalline and organic-bound iron (Duchaufour and Souchier, 1966). For the extraction by CBD, the soil sample was exposed to the reactant mixture at 80 °C with a solid/liquid ratio of 0.5 g/25 mL during 30 min. This method extracts the iron

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