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Characterisation of iron-(hydr)oxides in bulk soils and colloidal fractions using state-of-the-art techniques

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Iron-(hydr)oxide particles in soils have extremely large surface-to-mass ratios and are therefore considered to make up the largest fraction of the soils reactive surface area. Fe-(hydr)oxide particles are only a few nanometer in size when their surface-to-mass ratios are expressed as equivalent particle diameters. This suggests that Fe-(hydr)oxides are predominantly present as nano-sized particles in the soil. However, Fe-(hydr)oxides may aggregate or may be tightly attached to surfaces of larger particles. As a consequence, Fe-(hydr)oxides can have large surface-to-mass ratios without being present in the nanofraction. To understand the role of Fe-(hydr)oxides in aggregation and to assess their potential mobility in soils, it is important to know the size-distribution and setting of the Fe-(hydr)oxide particles. Therefore, the objective of this study is to characterize Fe-(hydr)oxides in the colloidal fraction, which we defined as the fraction smaller than 0.45 μm, and to compare these outcomes with characteristics of Fe-(hydr)oxides in the bulk soil. Two procedures were used to disperse the colloids from the bulk soil: 1) mechanical dispersion by ultrasonic treatment of $NaHCO_3$-soil suspensions and 2) chemical/mechanical dispersion by ultrasonic treatment of pyrophosphate-soil suspensions. The size-distribution and elemental composition of the colloids of seven soils were analysed by Asymmetric Flow Field-Flow Fractionation (AF4) coupled to High-Resolution Inductively Coupled Plasma Mass Spectrometer (HR-ICP-MS). For two soils, Fe speciation of bulk soil and colloidal fractions were determined by extended X-ray absorption fine structure (EXAFS) spectroscopy. In addition, Fe-(hydr)oxides in bulk soils and colloids were visualized by scanning electron microscopy (SEM) with energy dispersive X-ray detection (EDX) for element detection. Results for the bulk soils showed that reactive surface areas of Fe-(hydr)oxides correspond to equivalent particle diameters of a few nanometers only and SEM-images showed that Fe-(hydr)oxides were present as aggregates or were associated with clay and silt particles. Results for the colloidal fractions showed that mechanical dispersion released only small amounts of Fe-(hydr)oxide nanoparticles from the soils. These Fe-(hydr)oxides were mainly associated with clay and only a small fraction was present as single particles (i.e. not associated to other particles). More Fe-(hydr)oxide nanoparticles were dispersed by pyrophosphate, which also extracted between 39 and 66% of the soil-organic-carbon content. For the different soils, between 1 and 36% of the Fe-(hydr)oxide content was dispersible as single nanoparticles, which were 1 to 15 nm in size. Iron-(hydr)oxide nanoparticles dispersed by pyrophosphate were on average smaller than Fe-(hydr)oxides dispersed by ultrasound energy only. Iron speciation analyses showed that amorphous and crystalline Fe-(hydr)oxides were present in the colloidal fractions. Our results show that pyrophosphate can disperse organo-mineral aggregates which results in the mobilization of Fe-(hydr)oxide nanoparticles. In contrast, ultrasound energy is not sufficient to disperse Fe-(hydr)oxides that are incorporated within organo-mineral aggregates. The majority of Fe-(hydr)oxides in soils cannot be dispersed as nanoparticles, which shows that Fe-(hydr)oxides are strongly retained within the soil matrix. Altogether, the combination of AF4-HR-ICP-MS with microscopic- and spectroscopic techniques is a novel approach to study Fe-(hydr)oxides in the soil's colloidal fraction.