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Evidence for Magnetite Weathering in a Tropical Soil: an EPR study

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Magnetite as a lithogenic mineral is widespread in soils. Its bulk magnetic properties have been studied in detail and grain size distribution has been used as proxy to reconstruct the weathering environment. During weathering oxidation processes can lead to the formation of ferric oxides (e.g. maghemite) at the magnetite surface. The detection of such secondary ferric phases by rock magnetic methods is difficult, because the bulk properties generally dominate the magnetic signal. In this research we test the use of electron paramagnetic resonance spectroscopy (EPR) to provide evidence for weathered magnetite in a tropical soil. The magnetite grains were dispersed in a soil matrix mainly consisting of diamagnetic quartz and paramagnetic clay minerals. Under the SEM the grain size of more than 10 μm indicates magnetite in the pseudo single domain (PSD) to multidomain (MD) range.

The magnetic experiments were carried out with samples that were dried at 60° C and passed through a 2-mm sieve. Untreated and chemically treated samples were investigated. Chemical treatments included citrate-bicarbonate-dithionite (CBD) extraction, which removes ferric oxides (e.g. maghemite, hematite) by reductive dissolution. In addition a concentrated acid treatment (HCl:H_2SO_4 ratio of 19:1) followed by magnetic separation, was used in order to remove the magnetite.

Hysteresis loops of the untreated (Ap) and CBD treated (Ap_dit) samples at room temperature (RT) exhibit a magnetite with a coercive force (Hc) of 10 mT and a hysteresis closure at 400 mT. The hysteresis of the acid treated sample reveals a closed loop indicating the absence of phases carrying a remanent magnetization. The FORC diagrams of Ap and Ap_dit reveal two features. One is characterised by a Hc distribution with maximum at 10 mT and the other by a vertical spread (bias fields) with Hc around 2mT. The magnetic data suggest that CBD treatment has no significant effect on the magnetite in the soil samples.

X-band EPR spectra are dominated by a broad signal which is characteristic for ferromagnetic resonance. The EPR spectrum of the Ap sample has a g value of 2.11 and a linewidth of 120 mT. For the Ap_dit sample, the g value is 2.25 and the linewidth is 150 mT. At X-band frequency (9.8 GHz), magnetite has a skin depth of about 2 μm, which is smaller than the grain size of magnetite investigated. Therefore the absorption is mainly caused by the near-surface part of the grains. In addition, Ap_dit exhibits a significant angular dependence. The higher g-values in Ap dit is indicative of an increase in the internal field, and is most likely due to changes in the magnetic coupling caused by the reductive dissolution of the surface. The angular dependence provides evidence that this process is more pronounced where oxidation has chemically destabilised the magnetite surfaces.

In conclusion, EPR spectroscopy provides a better insight into the magnetic properties of magnetite surface, and, therefore it can help to elucidate weathering processes in soils.