Mineralogy of reactive iron species: Combining sequential extractions and Mössbauer spectroscopy

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Bioavailable iron minerals are crucial components of global nutrient cycles in marine and terrestrial sediments, influencing carbon transport and sequestration [1]. However, they are difficult to characterise and quantify using standard mineralogical tools (e.g. XRD), since they generally assume amorphous, colloidal, or nanoparticulate forms. Sequential extraction procedures [2] that rely on reagent-specific mineral solubility are currently used to separate reactive iron mineral phases into operationally-defined pools of carbonate-associated iron, iron (oxyhydr)oxides, magnetite, acid volatile sulfide iron, pyrite, and silicate. Although such methods are fundamental where mineral separation is required (e.g. investigating isotopic fractionation), mineral solubility is affected by grain size, complexation, degree of crystallinity and organic matter content, which can lead to the incomplete or premature dissolution of some mineral phases [2-4].

[57]Fe Mössbauer spectroscopy (MBS) only probes the hyperfine interactions between next-nearest neighbouring atomic nuclei in the crystal lattice and is consequently useful for colloidal and nanoparticulate analysis. MBS can also discriminate between individual iron minerals within each operationally-defined pool, and can further determine iron oxidation state, and therefore offers a time-saving, non-destructive, more mineral specific alternative for iron mineral identification, which can also be used in-situ [4]. Here we report the results of a cross-calibration between sequential iron extraction and MBS methods that uses a suite of natural and synthesised iron minerals from each operationally-defined pool, to optimise sequential extraction procedures for more accurate isotopic fractionation studies, and advance the analytical accuracy of quantitative bioavailable iron mineral characterisation.

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