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Spatial and Temporal Observation of Redox Reactions in Sands by NMR relaxometry

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Using laboratory methods, we investigated the effect of the presence of dissolved paramagnetic ions, such as oxygen and iron, on the proton nuclear magnetic resonance (NMR) relaxation times. Oxygen is the most important electron acceptor that stimulates the activity and growth of aerobic microbes, and iron(III)-ions is the same for anaerobes, which is a key factor for contaminant degradation. We show that both, oxygen as well as iron(III)-ions affect the relaxation times by their paramagnetic properties and thus could be determined inversely by NMR relaxometry in environmentally relevant concentrations. Due to the strong difference of iron(III)- versus iron(II)-ions on relaxation time, we chose the iron(III)–iron(II) redox pair for further studies.

We showed that NMR relaxation measurements are sensitive to small changes in the concentration of iron(III) species. A decrease in the relaxation times (T_1 & T_2) was observed corresponding to an increase in the dissolved iron(III) concentration. This effect was used to monitor relative changes in concentrations of dissolved iron(III) in natural sands. Column experiments were conducted, in which an acid (hydrochloric or sulphuric acid) was applied from the top on iron-bearing sands to dissolute the mineralogical bound iron(III). The relaxation times for different sand fractions with different concentrations of dissolved iron(III) were measured and compared to the relaxation time of water saturated sand without dissolved iron. In further experiments the iron(III) concentration was observed while iron(III) was reduced by reducing agents, e.g. magnesium and oxalic acid, and also experiments in which the pH value was increased by addition of sodium hydroxide to the pore water solution. These processes, the dissolution of iron(III) by an acid, the reduction of iron(III) and the precipitation of iron(III) in natural sands, were monitored with sufficient spatial and temporal resolution.

NMR relaxometry offers a non-invasive and non-destructive measurement method to observe and to visualise changes in the iron(III)-ion concentrations due to its paramagnetic influence on the relaxation times. The reaction front can be captured at pore scale with sufficient temporal resolution.

Iron(III)-ions function as electron acceptors during biodegradation of organic contaminants. Using NMR and iron(III) can provide more insight into the behaviour and mobility of many organic chemicals causing subsurface contaminations.