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Carbo-Iron as improvement of the nanoiron technology: From laboratory design to the field test

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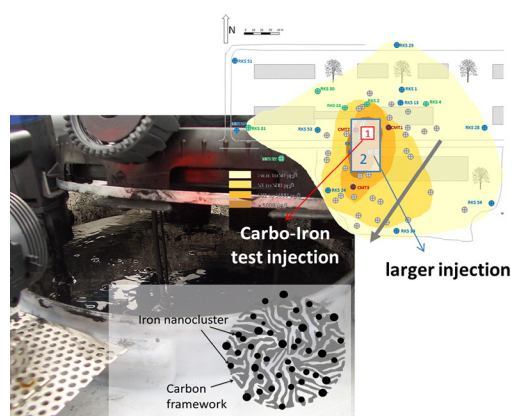
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HIGHLIGHTS

- The Fe–AC composite Carbo-Iron effectively degraded PCE at the field scale.
- Ethane and ethene were main products; no VC was found in 2.5 years.
- Carbo-Iron field performance supports lab results and the intended particle design.
- The material showed transport lengths of several metres in the field.
- The Carbo-Iron injection initiated a microbiological PCE-degradation process.

GRAPHICAL ABSTRACT



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ABSTRACT

In a first pilot-scale field test the use of Carbo-Iron® was successfully demonstrated. Carbo-Iron was developed with the goal to overcome significant shortcomings of nanoscale zero-valent iron (NZVI) for in-situ groundwater remediation. The composite material of colloidal activated carbon and embedded nanoiron structures has been tested for the remediation of a tetrachloroethene (PCE) contaminated field site in Lower Saxony, Germany. The results of the two-step field test confirmed the properties intended by its design and the particle performance achieved in the laboratory experiments. The material showed transport lengths of several metres in the field and fast PCE decomposition with no vinyl chloride formation. Extended longevity of the PCE decrease in the treated area and evidence for microbiological participation were found. Carbo-Iron is now under study in the framework of the EU-project NanoREM where its performance is being further optimized at various scales from laboratory via large-scale tank to field testing. Targeted property adjustment was successful for Carbo-Iron performance in both directions: plume treatment and source attack.

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1. Introduction

The further development of the barrier concept for groundwater treatment using reactive colloidal particles has received much attention over the last decade. Especially nanoiron (NZVI)-based approaches signal the high potential of reactive particles. At the same time, the limited subsurface transport of bare NZVI (Yang et al., 2007; Phenrat et al., 2007) and the unsolved phase treatment problems call for new strategies to fully utilize this high potential (Quinn et al., 2005). Consequently, the lessons learned from the potential and the shortcomings of NZVI led to the design of Carbo-Iron: a material with improved subsurface colloid transport and maintained iron reactivity (Mackenzie et al., 2012). Carbo-Iron is a composite material where the nanoiron structures (mean cluster size around 50 nm) are embedded in activated carbon colloid (ACC) particles. With a Fe(0) content of 20 to 30 wt.%, the porous composite has an effective density in water of about 1.7 to 2 g/cm³. Based on this, one can estimate an optimal particle size for subsurface transport as being in the range of 0.5 to 2 μm (Elimelech et al., 1995; Ryan and Elimelech, 1996).

The ACC carrier can be milled to the desired grain size. In addition, for effective subsurface transport, the electrostatic interactions play a major role. The Carbo-Iron material is not only small enough for subsurface transport; since the iron structures are placed within the carbon grain, the properties of the external ACC surface are maintained and the spacer function of the carbon framework inhibits the effect of the magnetic attraction forces known for nanoscale iron. Fig. 1 shows the “construction” principle of the composite, due to which Carbo-Iron has a much better precondition for subsurface transport with its more negative surface charge stemming from the AC support (Mackenzie et al., 2012).

The repulsion between a particle with a negatively charged colloid surface and the average aquifer sediment surface, which is negatively charged under the near-neutral aquifer conditions, counteracts the immobilization of particles. In addition, the ACC acts like a spacer between the individual nanoiron structures. This impedes the magnetic attraction forces and reduces the aggregation tendency of Carbo-Iron particles significantly in comparison to that of NZVI, leading to a higher sedimentation stability of Carbo-Iron suspensions even for higher particle concentrations. A minimum of suspension stabilizer, such as carboxymethyl cellulose (CMC), is needed for stabilization of the suspension during injection. However, small amounts of CMC have been found to significantly improve the mobility of the particles. Bleyl et al. (submitted for publication) recently found that a fully optimized suspension formulation (e.g. 20 g/L Carbo-Iron stabilized with 4 g/L CMC) is long-term stable. Particles are immobilized in a sand filled column only after exchange of several pore volumes of water, whereas in a semi-stable suspension (e.g. 20 g/L Carbo-Iron with only 1 g/L CMC) particles sediment out within hours, which is still sufficient for a straightforward injection but does not allow long transport distances through sediment layers. Transport indicators $L_{T,50}$ (transport length

reached by 50% of the particle mass) differ in column tests with medium hard water (U.S. EPA, 2002) and original site sediment from $L_{T,50} = 1.3$ m to $L_{T,50} = 0.4$ m for additions of 4 and 1 g/L CMC, respectively. For the $L_{T,99.9}$ (transport length reached by only 0.1% of the particle mass) the difference is even more pronounced: $L_{T,99.9} = 11.2$ m vs. $L_{T,99.9} = 1.2$ m for additions of 4 and 1 g/L CMC, respectively. By varying the added CMC concentration, we now have a tool to address either the in-situ formation of a broad treatment zone (plume control) or a narrow and focused target-specific particle placement (source zone). From a more fundamental point of view it may be of interest which CMC concentration is the relevant mobility parameter, that related to the water amount or that related to the colloid concentration? Based on a reversible adsorption of CMC on the external AC surface the CMC concentration in the suspension should not fall below a certain limit (about 1 g/L).

The laboratory tests for particle mobility, suspension stability and longevity of the particles in aquifer sediments, their ecotoxicological behaviour (Weil et al., 2015) and their chemical reactivity towards the target contaminants tetrachloroethene (PCE) and trichloroethene (TCE) strongly encouraged the field application of this material (Mackenzie et al., 2012). Similar to the use of NZVI, the Carbo-Iron approach is based on injection and immobilization of the colloidal reagent. The field site was chosen for the demonstration of the particle performance – full site remediation was not within the scope of this study.

2. Materials and methods

Prior to the first field application, the up-scaled synthesis of Carbo-Iron from laboratory rotary kiln to a semi-industrial scale had to be performed with the aim of process and product optimization. First of all, efforts were made to stabilize chemically the newly designed colloidal reagent Carbo-Iron in such a way that the pyrophoric properties are quenched but the iron-specific reactivity in water is kept.

The Carbo-Iron production schemes are documented in (Bleyl et al., 2012). The laboratory performance of Carbo-Iron and the characterization are described in (Mackenzie et al., 2012). The laboratory-synthesized material and the particles produced at larger scale were fully characterized and compared for their reactivity and transport behaviour in columns of various lengths and in 2D-cuvettes (Bleyl et al., submitted for publication). Carbo-Iron properties are summarized in Table 1.

For the field application a mobile injection unit was designed and set up, considering the special properties of Carbo-Iron, which ensured the full stability and inertization of the suspension during the injection. The injection into the aquifer was carried out in two steps:

- (1) Test injection with 20 kg Carbo-Iron: The suspension was prepared by dispersion of dry Carbo-Iron into de-oxygenized tap water using a high-speed homogenizer forming a 10 g/L suspension. CMC was used as colloid stabilizer with a concentration of

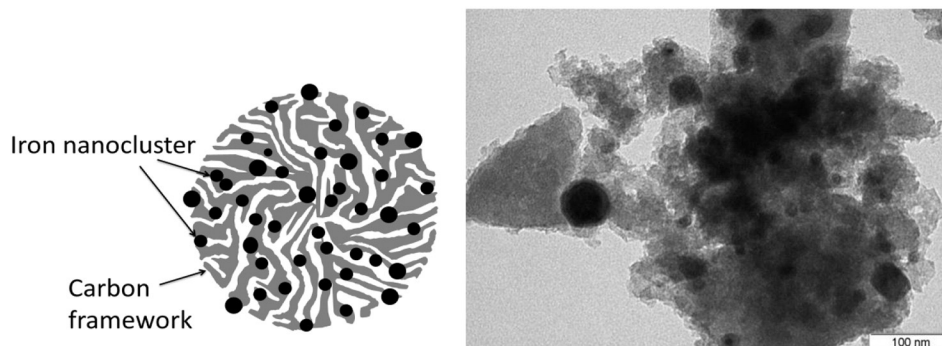


Fig. 1. Sketch (left) and TEM (right) of the Fe-AC composite material Carbo-Iron.

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