



Mitigation of contamination in effluents by metallic iron: The role of iron corrosion products



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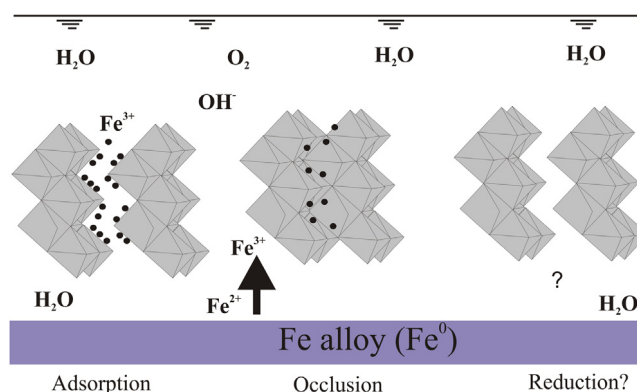
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HIGHLIGHTS

- Granular metallic iron (Fe^0) is a powerful material for water treatment.
- Methylene blue (MB) discoloration eases Fe^0 testing at lab scale.
- Methyl Orange (MO) discoloration optimizes discussion of MB results.
- In-situ generated iron corrosion products enhance both MB and MO discoloration.
- The experimental design is recommended for real world investigations.

GRAPHICAL ABSTRACT



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ABSTRACT

The influence of iron corrosion products (FeCPs) on the efficiency of metallic iron (Fe^0) for aqueous contaminant removal is characterized. Quiescent batch experiments were conducted for four weeks with solutions containing about $30 \mu\text{M}$ of either methylene blue (MB) or methyl orange (MO) at room temperature. Before dye (MB or MO) addition, used Fe^0 (iron filings) was allowed to equilibrate with demineralized water for 0 to 45 days (pre-corrosion). This operation enabled the in-situ generation of various amounts FeCPs. Results clearly demonstrated the crucial importance of freshly precipitated FeCPs for the decontamination process. The differential behavior of MB and MO confirmed the ion-selective nature of $\text{Fe}^0/\text{H}_2\text{O}$ systems. A significant pH increase was observed in the MO system relative to the MB system. This was attributed to increased iron corrosion by virtue

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Zero-valent iron

of formation of soluble Fe^{2+} -MO complexes. The overall result questions the usefulness of pre-treating (e.g. acid washing) Fe^0 materials before testing in short term experiments and reiterates the importance of longer lasting experiments with Fe^0 materials.

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1. Introduction

Metallic iron (Fe^0) has been extensively investigated and employed as reactive medium for environmental remediation and water treatment during the past two decades (Henderson and Demond, 2007; Ghauch et al., 2011; Gheju, 2011; Ghauch, 2015; Guan et al., 2015; Naidu and Birke, 2015; Noubactep, 2015; Naim and Ghauch, 2016; Raman and Kanmani, 2016; Gwenzi et al., 2017). Fe^0 is an affordable, reactive, readily available and environment-friendly material (Wu et al., 2015; Tomizawa et al., 2016). There are over 200 Fe^0 reactive walls working satisfactorily throughout the world (Naidu and Birke, 2015). Despite such large scale application, the science of the environmental $\text{Fe}^0/\text{H}_2\text{O}$ system is far from been established (Domga et al., 2015; Noubactep, 2016a, b, c). In particular, it is still hard to accept that, at the pH values of natural waters (pH > 4.5) Fe^0 is a generator of transforming precipitates which acts as contaminant collectors (Sun et al., 2016; Yang et al., 2017).

Controversy exists on almost all aspects of contamination removal in $\text{Fe}^0/\text{H}_2\text{O}$ systems (Ghauch, 2015; Guan et al., 2015; Noubactep, 2015; Gheju et al., 2016; Noubactep, 2016a, b, c). In particular, the role of iron corrosion products (FeCPs) on the process of decontamination is not understood. Fe^0 is widely considered as a reducing agent for the reductive transformation of pollutants (Guan et al., 2015). However, the omnipresence of FeCPs is a disturbing factor for contaminant reduction as the oxide scale shielding the Fe^0 surface is multi-layered and electronic non conductive as a rule (Triszcz et al., 2009; Guan et al., 2015; Gheju et al., 2016, 2017). Fe^0 is also widely used as a generator of FeCPs for the adsorptive removal of selected contaminants including arsenic (Triszcz et al., 2009; Trois and Cibati, 2015), methylene blue (Noubactep, 2010), selenium (Yang et al., 2017) and triazoles (Jia et al., 2007). The ubiquitous nature of FeCPs in $\text{Fe}^0/\text{H}_2\text{O}$ systems implies that adsorption and reduction potentially occur in all $\text{Fe}^0/\text{H}_2\text{O}$ systems (Furukawa et al., 2002; Noubactep, 2010, 2016a, b, c). These in-situ generated FeCPs have been demonstrated the major cause of permeability loss in Fe^0 filtration systems (Caré et al., 2013; Domga et al., 2015; Ghauch, 2015).

The Fe^0 surface is always coated with layers of FeCPs (oxide scale) (Sikora and Macdonald, 2000; Miyajima and Noubactep, 2013; Wan et al., 2014). In Fe^0 filters, the initial amount of FeCPs is increased by some typical pre-treatments like acid washing, salting or soaking (Guo et al., 2015; Sun et al., 2016; Yang et al., 2017). Thus, the Fe^0 employed in some field applications has been subjected to pre-corrosion under different conditions. However, there is evidence, that controversy still exists about the impact of FeCPs on the efficiency of $\text{Fe}^0/\text{H}_2\text{O}$ systems (including filters) towards contaminant removal (Gheju, 2011; Ghauch, 2015; Guan et al., 2015; Guo et al., 2015; Miyajima and Noubactep, 2015; Sun et al., 2016; Yang et al., 2017; Gheju et al., 2017)). In particular, it is still widely reported that FeCPs selectively act as (i) an adsorbent, (ii) an electron shuttle, or (iii) a physical barrier to mass transfer of contaminant towards or away from the Fe^0 surface (Guan et al., 2015 and refs. cited therein). Progress in understanding the $\text{Fe}^0/\text{H}_2\text{O}$ system will not be achieved before the role of FeCPs on its operating mode is clarified (Noubactep, 2015).

Recently, the suitability of methylene blue (MB) as operational tracer for the investigation of the $\text{Fe}^0/\text{H}_2\text{O}$ system has been established (MB method, see Btatkeu-K. et al., 2016). The MB method exploits the low affinity of positively charged MB to the surface of FeCPs (Mitchell et al., 1955) to shorten the experimental duration. Herein, the MB method is used to characterize the impact of in-situ generated FeCPs on the efficiency of the $\text{Fe}^0/\text{H}_2\text{O}$ system in batch experiments for 4 weeks (28 days). FeCPs were in-situ generated by equilibrating Fe^0 (5 g L^{-1}) in water for 0 to 45 days prior to MB addition (pre-corrosion). Parallel experiments with negatively charged methyl orange (MO) were performed to optimize the discussion. Beside pure Fe^0 systems, Fe^0 /sand mixtures were investigated as well. Achieved results deepen the understanding of the relationship between availability of FeCPs and the efficiency of Fe^0 -based systems for water treatment.

2. Materials and methods

2.1. Solutions

2.1.1. Dyes

Methylene blue (MB) and methyl orange (MO) are widely used as model contaminant to characterize the suitability of various systems for water treatment (Mitchell et al., 1955; Gong et al., 2013). Both dyes were of analytical grade. They are selected due to (i) similitude in their molecular size (Fig. 1 and Table 1) and (ii) differences in their affinity to iron oxides (Table 1). Used initial dye concentration was 10 mg L^{-1} ($31.5 \text{ }\mu\text{M}$ for MB and $30.7 \text{ }\mu\text{M}$ for MO). The working solutions were prepared by diluting a 50 times more concentrated stock solution ($3150 \text{ }\mu\text{M}$ for MB) using the demineralized water. The pH value of the initial solution was 7.6 ± 0.1 . The used initial concentration (10 mg L^{-1}) was selected to approach the concentration range of natural waters (MB and MO as model micro-pollutants) (Miyajima and Noubactep, 2015).

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