

## Comparative column testing of three reactive mixtures for the bio-chemical treatment of iron-rich acid mine drainage



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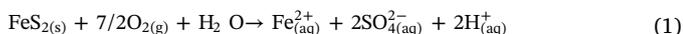
### ABSTRACT

Optimization of the hydraulic properties of reactive mixtures in passive systems can improve treatment efficiency of iron-rich acid mine drainage (Fe-rich AMD). The use of highly permeable and porous substrates could limit clogging and flow-related issues. The efficiency and evolution of hydraulic conductivity ( $k_{sat}$ ) of three reactive mixtures – two types of dispersed alkaline substrate (DAS), composed of wood ash (50% v/v – WA50) or calcite (20% v/v – C20), and one mixture consisting mainly of organic matter (70% w/w), typically used in passive biochemical reactors (PBRs) – were tested in 1.5 L columns.

The reactors were operated with hydraulic retention times (HRTs) of 1 to 5 d over a period of 16–63 days. In the WA-DAS reactors, results showed that a minimum HRT of 2 d was required to remove 33–62% of Fe in AMD with 2500 mg/L Fe. The calcite-DAS showed limited Fe removal (< 10%) in AMD at > 1500 mg/L Fe, but was around 40% at < 1500 mg/L Fe, over a 7-day period, at 2 d of HRT. Slight increase of the PBRs efficiency was found (77% and 91%), at initial Fe concentration of 500 mg/L, when the HRT was doubled (from 2.5 d to 5 d). All reactors removed other metals (37–99.9% for Al, Zn, and Pb; 20–98% for Ni, except in C20) and  $SO_4^{2-}$  (5–37%). Evolution of the hydraulic parameters of all reactors showed insignificant variation of the initial porosity of 0.68–0.74 and  $k_{sat}$  around  $10^{-2}$  cm/s, indicating no evidence of clogging throughout the testing period, even at 5 d of HRT. Nonetheless, Fe removal was HRT-dependent. Therefore, water quality, especially Fe concentration, should be among the design criteria for a long-term satisfactory treatment of Fe-rich AMD.

### 1. Introduction

Besides the variable content in bivalent heavy metals, metalloids (often As), and sulfate ( $SO_4^{2-}$ ), acid mine drainage (AMD) can contain high concentrations of trivalent acidogenic elements, such as Fe and Al. Iron is the most common and often the predominant metal in AMD because it originates from sulfide minerals, such as the ubiquitous pyrite. The oxidation of Fe in AMD occurs as shown in Eq. (1) (Nordstrom and Alpers, 1999).



The Fe-rich AMD is frequently encountered at closed and abandoned mine sites and must be treated to avoid significant environmental impacts to the surrounding ecosystems (Gazea et al., 1996; Diz et al., 2006; Genty et al., 2016). The use of passive treatment is prioritized for such mine sites because of its low cost and little maintenance requirements relative to active treatment (Johnson and Hallberg,

2005a; USEPA, 2014a). The treatment of Fe-rich AMD is particularly challenging because Fe removal is limited by the kinetics of Fe(II) to Fe(III) oxidation and subsequent hydrolysis, with consequences such as pH decrease, alkalinity exhaustion, and oxydoreduction potential (ORP) increase (Kirby et al., 1999; Neculita et al., 2008a). In addition, the rate of Fe(III) oxides/oxyhydroxides formation could be accelerated at higher pH (> 4) and may lead to rapid clogging and armoring, as well as changes in the hydraulic properties of the treatment system itself (R tting et al., 2008a; Neculita et al., 2008a; Zipper et al., 2011; Orakwue et al., 2016). Such a situation was encountered at the abandoned Lorraine mine site (Quebec, Canada), where a three-step treatment system (composed of two passive biochemical reactors [PBRs], separated by one unit of wood ash) was constructed for the treatment of Fe-rich AMD (Genty et al., 2016).

The composition of reactive mixtures used in passive systems influences the overall treatment performance. Generally, the components of mixtures are low-cost neutralizing agents and bio-wastes (Zipper and

*Abbreviations:* AMD, acid mine drainage; DAS, dispersed alkaline substrate; HRT, hydraulic retention time; IRB, iron-reducing bacteria; PBR, passive biochemical reactor; WA-DAS, wood ash–dispersed alkaline substrate; SRB, sulfate-reducing bacteria

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Skousen, 2014; USEPA, 2014a; Skousen et al., 2017). Limestone/calcite are widely employed in conventional passive treatment (e.g., PBR/anoxic limestone drain), but they are not recommended to treat highly acidic Fe-rich waters because their efficiency is significantly limited by the high Fe content (Maree et al., 1992; Potgieter-Vermaak et al., 2006), and they are subjected to coating/armoring by Fe(III) oxides/oxyhydroxides minerals (Hammarstrom et al., 2003; Zipper et al., 2011). Different options were attempted to address this issue: (1) the use of alkaline materials with a slower reactivity and less  $\text{Ca}^{2+}$  release (i.e., dolomite) to retard the clogging by gypsum (Huminicki and Rimstidt, 2008; Genty et al., 2012a; Kagambega et al., 2014); (2) the use of alkaline materials capable of increasing the pH over 8.5 (e.g., MgO, wood ash) to remove simultaneously divalent and trivalent metals (Rötting et al., 2006; Genty et al., 2012b); and (3) a combination of the neutralizing agents listed above with coarse and highly porous materials (e.g., wood chips) also called dispersed alkaline substrate (DAS) to improve hydraulic properties and avoid clogging (Rötting et al., 2008a; Ayora et al., 2013).

Mixtures used in PBRs are composed of a substrate (source of electrons, organic carbon, and nutrients for microorganisms), an inoculum of sulfate-reducing bacteria (SRB), a solid support (structural agents) for microbial attachment and porosity-permeability improvement, and a neutralizing agent for pH and alkalinity increase to prevent bacterial shock in the early start-up (Cocos et al., 2002). Previous work was essentially based on the study of substrate sustainability, efficiency, and permeability (Waybrant et al., 1998; Gibert et al., 2004; Neculita et al., 2007; Vasquez et al., 2016a). Mixed biodegradable materials were more efficient substrates for anaerobic bacteria compared to individual ones (Johnson and Hallberg, 2005a; Zagury et al., 2006). The efficiency of the PBRs is mainly based on their ability to remove  $\text{SO}_4^{2-}$ , of which final concentrations vary according to the mixture components and proportions, as well as water quality (Johnson and Hallberg, 2005b; Neculita et al., 2007; Skousen et al., 2017). In addition, Fe removal is limited when influent water contains more than 500 mg Fe/L (Neculita et al., 2008a). The porosity of mixtures usually ranges between 0.34 and 0.53 (Neculita et al., 2008a; Vasquez et al., 2016b), while saturated hydraulic conductivity ( $k_{\text{sat}}$ ) is  $10^{-4}$ – $10^{-2}$  cm/s and can decrease to as low as  $10^{-9}$  cm/s due to changes in substrate properties caused by suspended solids from AMD, secondary precipitates (oxides/oxyhydroxides, carbonate, and sulfide minerals), biomass and metabolic products generated by bacterial activity (URS, 2003; Neculita et al., 2008a).

At the same time, existing research emphasizes the use of DAS mixtures because their efficiency in Fe removal ranges from 20 to 99.9%, depending on their type (calcite, dolomite, wood ash, or MgO) and the water quality (Caraballo et al., 2009, 2011; Rakotonimaro et al., 2016). Moreover, calcite-DAS and MgO-DAS present better hydraulic properties with porosity of 0.62–0.76 (Rötting et al., 2007; Caraballo et al., 2010) and  $k_{\text{sat}}$  of  $10^{-3}$ – $10^0$  cm/s, with less decrease (down to  $10^{-3}$  cm/s) because of the grains dissolution which increases specific surface (Ss) to allocate metal precipitates, depending on the hydraulic residence time (HRT) (Caraballo et al., 2010; Ayora et al., 2013).

Based on such knowledge, research on the passive treatment of Fe-rich AMD that is frequently encountered at coal and base metal mine sites often recommends the design of materials/mixtures that achieve satisfactory efficiency in removing Fe, other metals/metalloids (if present), and  $\text{SO}_4^{2-}$  while preserving enough permeability to accommodate input flow. However, uncertainty about  $\text{SO}_4^{2-}$  removal using only DAS units has generally led to an additional well-known  $\text{SO}_4^{2-}$  treatment reactor—namely, PBRs.

Thus, the present work aims to comparatively evaluate the efficiency and hydraulic performance of two reactive mixtures used in DAS systems for Fe pre-treatment, as well as a third reactive mixture used in PBR for  $\text{SO}_4^{2-}$  removal.

## 2. Materials and methods

### 2.1. Column design, set-up, and operating conditions

The DAS experiments were performed with six 1.5 L columns (height 22 cm, inner diameter 10 cm) equipped with perforated drain-pipes placed at the bottom (as inlet) and on top (as outlet) (Fig. S1A, Supplementary material). Four reactors were filled with WA50 [50% v/v wood ash, and 50% v/v wood chips] and operated at four different HRTs: 1 d (WA50-1), 2 d (WA50-2), 3 d (WA50-3), and 5 d (WA50-5). Each reactor was in operation for a 30-day period. Two other column reactors were used to evaluate the efficiency of C20 [20% (v/v) calcite and 80% (v/v) wood chips, at two HRTs, of 1 d (C20-1) and 2 d (C20-2), for 16 days. Previous batch testing showed that the calcite-DAS had maximal efficiency only in a very short time (Rakotonimaro et al., 2016). These two types of DAS mixtures (WA50 and C20) were fully characterized prior their use to pre-treat Fe-rich AMD during batch testing (Rakotonimaro et al., 2016), while their efficiency and hydraulic performance was evaluated in continuous flow reactors. In each reactor, glass bead layers and fine-mesh geotextiles (1.66 cm) compressed and sealed off the mixtures at the top and bottom, prior to their covering. Artificial AMD (Table 1) was prepared (weekly/biweekly) in 20 L buckets and fed by peristaltic pumps in upward flow to the columns. This design allows uniform distribution of influent since downward and/or horizontal flow could favor early clogging and preferential flow (Neculita et al., 2007). Notably, the synthetic effluents simulate the typical quality of AMD from hard rock mines in Canada (Aubertin et al., 2002).

Two columns having the same size as the DAS reactors, but equipped with a gas chamber, were used as PBRs (Fig. S1B, Supplementary material). They were filled with reactive mixtures consisting of 70% (w/w, dry weight) organic materials (40% wood chips, 20% chicken manure, 10% leaf compost), and 30% (w/w) inorganic materials (10% sand and 20% calcium carbonate). The set-up was thereafter similar to that of DAS columns, where the mixture was slightly compressed between two layers of glass beads and geotextile at the top and bottom of the reactors, prior to their covering. After the set-up, the reactors were saturated with a Postgate B medium, prepared in distilled water, with the following composition: 3.5 g/L sodium lactate (or 4.7 mL lactate liquid 56.8%); 2.0 g/L  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ; 1.0 g/L  $\text{NH}_4\text{Cl}$ ; 1.27 g/L  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ; 1.0 g/L yeast extract; 0.5 g/L  $\text{KH}_2\text{PO}_4$ ; 0.5 g/L  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ; 0.1 g/L thioglycolic acid, and 0.1 g/L ascorbic acid (Postgate, 1984). Then, the columns were incubated for 3–4 weeks (acclimation period) before starting the continuous flow. This acclimation period allowed SRB to grow and produce enough sulfides and alkalinity to treat the initial input of AMD (Waybrant et al., 2002; Neculita et al., 2008a). Calibrated Masterflex-peristaltic pumps were used to feed in upward flow the AMD in the PBRs. Each PBR was operated for a 63-day period, at 2.5 d (R2.5) and 5 d (R5) of HRT.

**Table 1**  
Quality of Fe-rich AMD used in column type DAS reactors and PBRs.

Parameters	Concentration (mg/L, except for pH)	Source
$\text{Al}^{3+}$	1.6 ± 0.6	$\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$
$\text{Fe}^{2+}$	2500 ± 171 (DAS)	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$
$\text{Fe}^{2+}$	500 ± 33 (PBRs)	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$
$\text{Mg}^{2+}$	33.5 ± 3.8	$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$
$\text{Mn}^{2+}$	8.2 ± 1.0	$\text{MnSO}_4 \cdot \text{H}_2\text{O}$
$\text{Ni}^{2+}$	0.7 ± 0.4	$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$
$\text{Pb}^{2+}$	0.2 ± 0.1	$\text{Pb}(\text{NO}_3)_2$
$\text{Zn}^{2+}$	0.2 ± 3.0	$\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$
$\text{Ca}^{2+}$	430 ± 5	$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$
$\text{SO}_4^{2-}$	5395 ± 988	NA <sup>a</sup>
pH	3–4	NA

<sup>a</sup> Not applicable (NA); Passive biochemical reactors (PBR).

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