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# Cost-effective bioregeneration of nitrate-laden ion exchange brine through deliberate bicarbonate incorporation

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## ABSTRACT

Bioregeneration of nitrate-laden ion exchange brine is desired to minimize its environmental impacts, but faces common challenges, i.e., enriching sufficient salt-tolerant denitrifying bacteria and stabilizing brine salinity and alkalinity for stable brine biotreatment and economically removing undesired organics derived in biotreatment. Incorporation of 0.25 M bicarbonate in 0.5 M chloride brine little affected resin regeneration but created a benign alkaline condition to favor bio-based brine regeneration. The first-quarter sulfate-mainly enriched spent brine (SB) was acidified with carbon source acetic acid for using  $\text{CaCl}_2$  at an efficiency >80% to remove sulfate. Residual  $\text{Ca}^{2+}$  was limited below 2 mM by re-mixing the first-quarter and remained SB to favor denitrification. Under  $\text{CO}_2\text{--HCO}_3^-$  system buffered pH condition (8.3–8.8), nitrate was removed at 0.90 gN/L/d by hematite-enriched well-settled activated sludge (SVI 8.5 ml/g) and the biogenic alkalinity was retained as bicarbonate. The biogenic alkalinity met the need of alkalinity in removing residual  $\text{Ca}^{2+}$  after sulfate removal and in  $\text{CaCl}_2$ -induced  $\text{CaCO}_3$  flocculation to remove 63% of soluble organic carbon (SOC) in biotreated brine. Carbon-limited denitrification was also operated after activated sludge acclimation with sulfide to cut SOC formation during denitrification. Overall, this bicarbonate-incorporation approach, stabilizing the brine salinity and alkalinity for stable denitrification and economical removal of undesired SOC, suits long-term cost-effective brine bioregeneration.

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## 1. Introduction

Nitrate pollution in groundwater is increasingly serious worldwide (Burow et al., 2010; Kaushal et al., 2011; Zhang

et al., 1996). Proper treatment of nitrate-contaminated groundwater is in great need to minimize its health risks as drinking water (Fewtrell, 2004; Ginkel et al., 2008). A practical approach has been intensively explored for processing nitrate-contaminated groundwater, i. e., obtaining drinkable

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water through ion exchange and then treating the nitrate-concentrated spent brine (SB) (Bae et al., 2002; Lehman et al., 2008; Yang et al., 2013). It is desirable to reuse SB after removal of nitrate and other undesired substances, especially in water-shortage inland areas, not to worsen soil and aquifer salinization. Direct bioregeneration of nitrate-exhausted resin has also been explored to bypass the need of SB biotreatment, (Meng et al., 2014), but creates the needs of repeated resin unloading/reloading and intensive resin cleaning after bioregeneration.

Various chemical approaches, including photochemical, electrochemical and catalytic approaches, have been explored for nitrate removal in SB (Dortsiou et al., 2009; Yang et al., 2013), but generally face the difficulty in controlling the formation of byproducts nitrite and ammonium (Hirayama et al., 2012; Zhang et al., 2005). Highly concentrated nitrate in SB can also be removed at high efficiencies in downsized bioreactors (Ginkel et al., 2008; Lehman et al., 2008; McAdam and Judd, 2008), although three common challenges exist, i.e., enrichment and maintenance of sufficient salt-tolerant denitrifying bacteria and stabilization of brine salinity and alkalinity for long-term stable brine biotreatment, and economical removal of bacterially derived organics for reliable brine reuse.

Salt-tolerant denitrifying bacteria, typically in form of denitrifying activated sludge (DAS), are often enriched with inocula from saline and/or non-saline environments (Cang et al., 2004; Ginkel et al., 2008; Logan et al., 2001; Okeke et al., 2002). A good settling property is essential for DAS to be less likely lost but well maintained at suitable levels for long-term brine biotreatment (Dan et al., 2002; Lefebvre and Moletta, 2006). The use of chemically inert and reactive substances such as activated carbon and soluble iron may promote the enrichment of well-settled activated sludge (Vlyssides et al., 2009; Yu et al., 1999).

Stabilization of brine salinity and alkalinity also favors stable brine biotreatment (Ginkel et al., 2008). Nevertheless, both sulfate and bicarbonate typically accumulate to cause considerable variations of brine salinity and alkalinity in recycled brine. Even the sulfate level in recycled brine is finally stabilized when nitrate selective resins are used, the stabilized sulfate level may be more than 150 mM, considerably increasing the brine salinity (Lehman et al., 2008). When non-selective resins are used, sulfate continuously accumulates in recycled brine and needs to be lowered below certain levels (e. g., <100 mM) to not affect brine biotreatment and resin regeneration. Sulfate removal via  $\text{CaSO}_4$  precipitation is less effective than via  $\text{BaSO}_4$  precipitation but cheaper and environmentally safer, and is applicable if the residual  $\text{Ca}^{2+}$  level can be well limited (e. g., <2 mM) to lower its negative effect on denitrifying bacteria (Bae et al., 2004; Ismail et al., 2010). Sulfate removal through bioreduction is also possible but increases the cost for electron donor and the need for malodor control (Bae et al., 2002; Cang et al., 2004).

Unlike sulfate, which is enriched from raw water, bicarbonate accumulated in recycled brine is primarily transformed from hydroxide alkalinity generated from denitrification. Since bicarbonate as resin regenerant is much less effective than chloride, very often when its level in recycled brine is already high enough (e. g., >50 mM), extra bicarbonate is typically neutralized so that pH may not continue to increase

to affect the performance of denitrification (Bae et al., 2002). However, with the co-presence of a certain level of dissolved  $\text{CO}_2$  either internally derived from heterotrophic denitrification or externally supplemented, the  $\text{CO}_2\text{--HCO}_3^-$  buffer system can maintain a suitable pH range during the process of denitrification (Huang et al., 2011), although the addition of  $\text{Mg}^{2+}$  is needed to compensate the limitation of  $\text{Ca}^{2+}$  to favor bacterial growth when bicarbonate is at a much higher level (e.g., >200 mM) (Lehman et al., 2008). When sulfate and bicarbonate levels in recycled brine are properly stabilized and chloride is supplemented to compensate its consumption during resin regeneration, then the brine salinity and alkalinity can be basically stabilized to favor long-term stable performance of brine biotreatment.

Cost-effective removal of bacterially derived undesired organics should be one of the biggest practical challenges for brine bioregeneration. A considerable portion of carbon source supports the growth of denitrifying bacteria and finally goes into the pool of undesired organics during brine biotreatment. Thus, without proper post treatment of biotreated brine before reuse, the performance of resins quickly deteriorates (Bae et al., 2002; Clifford and Liu, 1993). The use of activated carbon for removal of undesired organics in biotreated brine is relatively expensive. Isolating denitrifying bacteria from brine with ion exchange membrane during biotreatment may lower the need of post treatment (Ginkel et al., 2008; Shrimali and Singh, 2001). Since a low level of nitrate (e. g., up to 10 mM) in brine little affects practical (partial) regeneration of nitrate-exhausted resins, carbon-limited brine biotreatment may be an optional choice when perchlorate is not enriched in SB to reduce the formation of undesired organics. Nevertheless, nitrite reduction is normally slower than its formation to easily cause nitrite accumulation during denitrification even under carbon sufficient conditions (Chung et al., 2009; Peyton et al., 2001; Show et al., 2013; Tavares et al., 2006; Xiao et al., 2010). To conduct carbon-limited brine biotreatment, special acclimation is likely needed to synchronize bacterial nitrate and nitrite reduction to avoid accumulation of residual nitrite at the end of operation.

To jointly meet the three common challenges above-mentioned in SB bioregeneration, namely, enriching well-settled salt-tolerant DAS, stabilizing brine salinity and alkalinity and cost-effectively removing undesired organics from biotreated brine, bicarbonate incorporation in the range of 100–250 mM in brine is proposed to favor bio-based brine regeneration. In the first step for sulfate removal, the first-quarter sulfate-mainly enriched alkaline SB is acidified with acetic acid (also carbon source for denitrification) for using  $\text{CaCl}_2$  to partly remove sulfate, and re-alkalized with the remained alkaline SB to limit the level of residual  $\text{Ca}^{2+}$  to favor brine biotreatment. In the second step for nitrate bioreduction, hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) is used to enrich well-settled high-density DAS to denitrify nitrate and the biogenic alkalinity is retained in the benign form of bicarbonate under  $\text{CO}_2\text{--HCO}_3^-$  system buffered pH condition. In the third step for removal of undesired organics produced in biotreatment, biogenic alkalinity and  $\text{CaCl}_2$ -induced  $\text{CaCO}_3$  flocculation is used to save the cost of post treatment.

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