Improved brine recycling during nitrate removal using ion exchange

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Abstract

Ion exchange technology is currently the best for removing nitrate from drinking water. However, problems related to the disposal of spent brine from regeneration of exhausted resins must be overcome so that ion exchange can be applied more widely and economically, especially in small communities. For this purpose, a novel spent brine recycling system using combined biological denitrification and sulfate reduction processes was developed for more efficient reuse of brine. A granular activated carbon (GAC) adsorption column was introduced as an additional step to prevent contamination of resins by bio-polymers and dissolved organics present in the bio-reactor effluent. Two upflow sludge blanket reactors (USBRs) were operated in series for 166 days to provide denitrification and sulfate reduction. The denitrification reactor provided a nitrate removal efficiency of 96% at a nitrate-N loading rate of 5.4 g NO₃⁻/N/d. The sulfate reduction efficiency of the sulfate reduction reactor remained ~62% at a sulfate loading rate of 1.8 g SO₄²⁻/d.

Five ion exchange columns containing A520E resins were repeatedly operated in up to 25 cycles of service and regeneration using five kinds of brine: one virgin 3% NaCl and four differently recycled spent brines. Throughput decreased remarkably when the biologically recycled brine was not treated with the GAC column, probably due to the presence of bio-polymers and dissolved organic compounds. The sulfate reduction reactor placed after the denitrification step increased the bicarbonate concentration, which could be used as a co-regenerant with chloride.

The inclusion of the sulfate reduction reactor into the conventional brine recycling system allowed more efficient reuse of brine, resulting in both reduced salt consumption and brine discharge. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Ion exchange; Nitrate removal; Brine reuse; Denitrification reactor; Sulfate reduction reactor; Granular activated carbon (GAC) adsorption; Bio-polymers; Bicarbonate

1. Introduction

The nitrate in groundwater used for drinking in rural areas is becoming an important problem due to its harmful effects. Among several techniques available for the removal of nitrate, such as ion exchange, biological denitrification, chemical reduction, and reverse osmosis or electrodialysis, the ion exchange process seems to be the most suitable for small water suppliers contaminated by nitrate because of its simplicity, effectiveness, and relatively low cost [1].

Although technically and economically effective, the ion exchange process also has two inevitable problems. The first is the trouble caused by sulfate ions. Since conventional anion exchange resins have general selectivity sequences of SO₄²⁻ > NO₃⁻ > Cl⁻ > HCO₃⁻, sulfate ions in the raw water interfere heavily in nitrate removal and results in short service runs. Recently, however, several nitrate-to-sulfate selective (NSS) resins have been developed and successfully applied to groundwater with high sulfate concentrations [2]. The most notable unsolved problem is the disposal of spent brine
produced during regeneration of exhausted resins. To reduce the requirement for salt and later brine disposal, several regeneration procedures and methods for recycling spent brine have been developed. Among these methods, biological denitrification of spent brine was extensively studied by Van der Hoek et al. [3] and Clifford and Liu [4].

Van der Hoek et al. [3] developed the combined ion exchange and biological denitrification process, and verified that the reuse of spent brine was feasible by connecting an upflow sludge blanket denitrification reactor (USBR) with the ion exchange columns. Clifford and Liu [4] chose to use the sequencing batch reactor (SBR) instead of a USBR for the denitrification step, and investigated the effects of salt concentration and the mass ratio of methanol to nitrate-nitrogen on the denitrification rate. Later, Liu and Clifford [5] successfully recycled the spent brine in their pilot-scale test. The salt consumption and the amount of brine to be treated were reduced up to 75% and 95%, respectively, compared with the conventional process without brine reuse.

As mentioned by previous researchers, sulfate accumulates in the biological loop of the brine recycling system. Van der Hoek et al. [6] reported that sulfate accumulated up to 5000 mg/l in their brine recycling system without any negative effect on the nitrate removal efficiency of NSS resins during service mode. After detecting a sulfide odor from a lab-scale denitrification reactor on one occasion, Liu and Clifford [5] investigated the fate of sulfate during long-term brine recycling. After 38 service-regeneration cycles, sulfate in the recycled brine reached a maximum level of ~16,000 mg/l, without serious impact on the nitrate removal capacity of the NSS resins.

In spite of this fact, such a high sulfate build-up seems undesirable from a practical viewpoint. As suggested by Liu [7], sulfate reduction could occur in the denitrification reactor during idle or shutdown periods. The resulting hydrogen sulfide could alter the reaction pathway from denitrification to ammonia formation and potentially cause odor and health problems for operators. To overcome these problems, a sulfate reduction step was added to the conventional biological brine recycling system.

Previous researchers placed sand filters, with or without cartridge filters, between the denitrification reactor and the ion exchange column to remove suspended solids. It is difficult for either the sand or cartridge filters to entrap sufficient amounts of the bio-polymers or dissolved organics present in the reactor effluent. In this connection, a granular activated carbon (GAC) adsorption column was placed as the last step of the brine recycling system. The purpose of this study was to investigate the effects of the sulfate reduction reactor and the GAC adsorption column on the overall performance of the improved brine recycling system.

2. Materials and methods

2.1. Ion exchange column and brine recycling system

Fig. 1 depicts the novel brine recycling system consisting of nitrate exchange columns, biological denitrification and sulfate reduction reactors, a sand filter, a GAC adsorption column, and several regenerated brine vessels (RBVs). Five ion exchange columns, each containing 50 ml of NSS resin, were operated in parallel to compare the efficiency of different regenerants obtained from each step in the brine recycling process. The spent brine from all the exhausted columns was collected in the spent brine vessel, sent through the denitrification reactor, and stored in the intermediate brine storage vessel (IBSV). From the IBSV, four flow paths through the brine recycling system were used.

The first flow path was from IBSV to the sand filter and then to RBV-B. The second flow path was from the IBSV to the sand filter, then to the GAC column, and finally to RBV-D. The third flow path was from the IBSV to the sulfate reduction reactor, then to the sand filter, and finally to RBV-C. The fourth flow path was from the IBSV to the sulfate reduction reactor, then to the sand filter, next to the GAC column, and finally to RBV-E.

After the first cycle, all the exhausted ion exchange columns were regenerated using 3% NaCl solution made with distilled water. However, from the second cycle, column I using virgin 3% NaCl was used as a control, while columns II–V were regenerated with differently recycled brine from RBVs B through E, respectively. Thus, the salt concentrations of the different regenerants varied from the second cycle. For each cycle, the 330 ml of 3% NaCl used for regenerating the control column was the only salt compensation to the spent brine recycling system. The five kinds of regenerants used in this study are summarized in Table 1.

All columns and reactors were made with clear acrylic material. Two USB reactors, each with a working volume of 0.6 l, were used for denitrification and sulfate reduction. The sand filter column (3.0 cm i.d. x 60 cm height) contained 360 ml of sand (uniformity coefficient 1.85; effective size 0.5 mm). The GAC column (2.5 cm i.d. x 60 cm height) contained 160 ml of coconut shell GAC (surface area 1200 m²/g). Both the sand and the GAC used in this study were taken from a nearby drinking water treatment facility and replaced periodically based on the filtering velocity and color of the column effluent, respectively.
2.2. Operation of the ion exchange columns

The macroporous NSS resin, A520E (Purolite Co.), was used in this study because the NSS resins are much more compatible with biological brine recycling than conventional anion exchange resins [2,5]. According to the manufacturer's information, A520E resin has quaternary ammonium as a functional group, with a total ion exchange capacity of 2.8 mEq/g of dry resin. The size of resin particles ranges from ~0.30–1.18 mm (retained by No. 16–50 US standard screen). Synthetic groundwater having an anionic composition of 30 mg/l of nitrate-N, 40 mg/l of sulfate, 60 mg/l of chloride, and 120 mg/l of bicarbonate was pumped downward at a rate of 20 bed volume (BV) per hour until the termination of service. After the nitrate breakthrough, the resins were backwashed, regenerated, and rinsed, following the operation sequence summarized in Table 2. The efficiency of each regenerant was compared after regenerating the exhausted resins for 80 min. All water samples were filtered through 0.45 μm membrane filters prior to analysis. Nitrate, sulfate, and chloride were measured using an ion chromatograph (DX-300, Dionex Co.).

Table 1
Regeneration processes used for each ion exchange column

<table>
<thead>
<tr>
<th>Regenerant</th>
<th>Recycling process</th>
<th>Column</th>
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<tbody>
<tr>
<td>A</td>
<td>3% NaCl</td>
<td>I (control)</td>
</tr>
<tr>
<td>B</td>
<td>DNR(^a) → SF(^b)</td>
<td>II</td>
</tr>
<tr>
<td>C</td>
<td>DNR → SRR(^c) → SF</td>
<td>III</td>
</tr>
<tr>
<td>D</td>
<td>DNR → SF → GAC(^d)</td>
<td>IV</td>
</tr>
<tr>
<td>E</td>
<td>DNR → SRR → SF → GAC</td>
<td>V</td>
</tr>
</tbody>
</table>

\(^a\)DNR: denitrification reactor.  
\(^b\)SF: sand filter column.  
\(^c\)SRR: sulfate reduction reactor.  
\(^d\)GAC: granular activated carbon column.

2.3. Operation of the denitrification and sulfate reduction reactors

For the inoculation of the denitrification reactor, activated sludge taken from a domestic wastewater treatment plant was acclimatized to 3% NaCl, and ~0.51 of acclimatized sludge was seeded. Granular sludge cultivated in an anaerobic baffled reactor treating...
sulfate-rich wastewater, was used as seed sludge for the sulfate reduction reactor. The two USB reactors were operated continuously, and in parallel, for 166 days as summarized in Table 3. Only spent brine collected during the regeneration step was used for preparing the substrate for the two reactors after adding ethanol and trace nutrients [8].

Since the ion exchange part was operated intermittently for only 25 cycles during 166 days, an imbalance occurred between the amount of spent brine generated and the capacity of the reactors. The total amount of spent brine collected from the five ion exchange columns was insufficient to operate the biological brine recycling system continuously. For this reason, spent brine from ion exchange columns being operated in the lab for other purposes, containing both NSS and conventional anion exchange resins, was also collected and added as necessary for making the substrate during operating steps 4–6.

Both reactors were placed in an incubator maintained at 30°C. The denitrification reactor was started with an initial nitrate loading rate (NLR) of 1.9 g NO₃⁻/C₀-N/ld, while the sulfate reduction reactor was started with an initial sulfate loading rate (SLR) of 0.7 g SO₄²⁻/ld. During operating step 1 (days 1–19), the effluent from the denitrification reactor was fed directly into the sulfate reduction reactor. From operation day 20, however, the effluent from the denitrification reactor was collected in the IBSV. Since the pH of the effluent was high (up to 9.0), phosphoric acid was added to the stream after leaving the intermediate vessel to decrease the pH to ~7.0 to protect the sulfate reducing bacteria from the anticipated pH inhibition. Additional carbon (ethanol) needed for the sulfate reducing bacteria was also added to the stream after leaving the intermediate vessel. The loading rates of each reactor were increased stepwise. The amount of gas produced by each reactor was separately measured using displacement of brine solution.

3. Results and discussion

3.1. Performance of the ion exchange column

Fig. 2 shows the typical breakthrough curves for nitrate, sulfate, bicarbonate, and chloride from the A520E resin regenerated with virgin 3% NaCl brine after 25 cycles of service and regeneration. Since the maximum admissible concentration of nitrate is 10 mg NO₃⁻-N/l, this level was used to define the breakthrough for nitrate. As shown in Fig. 2, the nitrate breakthrough occurred after 275 BV of service run, corresponding to ~90% of throughput obtained with the virgin resin during the first service run. This result indicates that there was no significant decrease in the

### Table 2

<table>
<thead>
<tr>
<th>Conditions for each operation mode of the ion exchange columns</th>
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<tbody>
<tr>
<td>Operation mode</td>
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<tr>
<td>Service</td>
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<tr>
<td>Backwash</td>
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<tr>
<td>Regeneration</td>
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<td>Slow rinse</td>
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<td>Fast rinse</td>
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### Table 3

<table>
<thead>
<tr>
<th>Operation steps for the denitrification and the sulfate reduction reactors</th>
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<tbody>
<tr>
<td>Operation step</td>
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<tr>
<td>Flow rate</td>
</tr>
<tr>
<td>1</td>
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<td>2</td>
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[a]: l/d.
[b]: Volumetric loading rate = g COD/l d.
[c]: Nitrate-N loading rate = g NO₃⁻/C₀-N/l d.
[d]: Ratio of COD to nitrate-N = COD/NO₃⁻-N (g/g).
[e]: Sulfate loading rate = g SO₄²⁻/l d.
[f]: Ratio of COD to sulfate = COD/SO₄²⁻ (g/g).
throughput obtained with A520E resin, even though it was intermittently operated during 166 days. The variation of throughput was estimated from the typical breakthrough curves shown in Fig. 7. There was a chromatographic peaking of sulfate before nitrate breakthrough as shown in Fig. 2. This phenomenon is different from that in conventional anion exchange resins in which the nitrate breakthrough occurs earlier than the sulfate breakthrough [9]. In case of NSS resin, sulfate breaks earlier than nitrate in column test due to the preference of NSS resin for nitrate. Thus, when the sulfate ions reach the breakthrough point, NSS resin dumps sulfate ions displaced by nitrate ions, causing chromatographic peaking of sulfate.

The typical elution curves for sulfate and nitrate during regeneration of exhausted resin using 3% NaCl are shown in Fig. 3. Sulfate is the first stripped from the resin, and nearly all the nitrate and sulfate adsorbed on the resin were eluted within the first 20 min of regeneration. This means that 20 min is sufficient for the partial regeneration of the exhausted resin. Although the data were not included here, partial regeneration was more efficient and practical than complete regeneration in terms of the amount of salt required and the production of waste brine, as pointed out by previous researchers [5,9]. However, a longer regeneration time of 80 min was used in this study for a clear comparison of the various regenerants.

3.2. Performance of the denitrification reactor

The operating results of the denitrification reactor are depicted in Fig. 4, in which the changes in COD, nitrate-N, and sulfate are plotted against operation time. Since
the spent brine from the five ion exchange columns was combined in a single vessel, the spent brine used as feed had a NO$_3$-N concentration of 600–1700 mg/l and a SO$_4^{2-}$ concentration of 500–2500 mg/l. Despite the variation of experimental loading rates, both COD and nitrate-N removal efficiencies of the denitrification reactor were fairly stable. The nitrate removal efficiency at each loading rate was calculated based on the averaged influent and effluent concentrations. The denitrification reactor was successfully operated up to 5.4 g NO$_3$-N/l with nitrate-N removal efficiency of 96% as summarized in Table 4, in spite of the variation in the ratio of COD to nitrate-N. In a previous work using methanol as the carbon source [1,4], deviation from the optimum ratio of carbon to nitrate-N was related to a decrease in the performance of the denitrification reactor. When the spent brines from the conventional anion exchange resins were added to the spent brine vessel from the middle of step 3 to the end of step 6, the sulfate concentration in the feed solution increased. During these periods, sulfate reduction occurred to some extent in the denitrification reactor. Otherwise the removal of sulfate in the denitrification reactor was negligible.

### 3.3. Performance of the sulfate reduction reactor

Fig. 5 shows the operating results of the sulfate reduction reactor. Despite that the seed sludge used in the sulfate reduction reactor was not acclimatized to high salt, the sulfate removal efficiency averaged more than 50% from the first step with an SLR of
0.7 g SO$_4^{2-}$/ld. This was probably due to the high activity of the granules used as seed. The nitrate-N entering the sulfate reduction reactor from the denitrification reactor was further reduced by step 2. However, there was nearly no nitrate reduction in the sulfate reduction reactor because the influent nitrate-N concentration was already <50 mg/l after step 2.

Theoretically, the removal efficiency of bio-reactors have to be calculated under steady state conditions. In this study, however, it was not easy to maintain the steady state condition probably due to the variation in the composition of the influent. Because of this limitation, the removal efficiency of each step was instead calculated using averaged influent and effluent concentrations. As shown in Table 4, the sulfate reduction efficiencies obtained were quite low and unstable compared to the nitrate-N removal efficiencies, even though the applied SLRs were much less than the nitrate-N loading rates. The reason for relatively low sulfate reduction efficiency might be due to the inhibition caused by hydrogen sulfide. Recently, Yamaguchi et al. [10] proposed sulfide stripping with ferrous oxide pellets as a measure to increase sulfate reduction efficiency in an anaerobic process. Incorporation of such a step is likely to be beneficial in improving sulfate reduction efficiency, and further investigation is needed.

### 3.4. Gas production from the denitrification and sulfate reduction reactors

Fig. 6 shows the amount of bio-gas produced by the denitrification and sulfate reduction reactors. It was difficult to measure the amount of gas precisely using the water displacement method since the working volume of each reactor was quite small. Gas production from the denitrification reactor increased with the nitrate-N loading rate. More than 93% of the gas collected from the denitrification reactor was nitrogen, with a small amount of carbon dioxide. The hydrogen sulfide was not measured in this study, even though some gas was produced from the sulfate reduction reactor. A gas chromatograph (Varian 3300) equipped with a thermal conductivity detector was used for analyzing gases.

According to the following equation proposed by Hamon and Fustec [11], 0.46 mol of N$_2$ is produced per mole of nitrate-N reduced, when ethanol is used as a carbon source. This amount of N$_2$ gas corresponds to 803 ml N$_2$/g NO$_3^{-}$/N reduced at 30°C. During step 6, $\sim$550 ml of nitrogen gas was produced at a nitrate-N loading rate of 4.2 g NO$_3^{-}$/N/l, equivalent to $\sim$90% of the theoretical amount. Although there was some discrepancy between the amount of nitrogen gas recovered and the nitrate-N loading rate, the amount of nitrogen gas should be a good indicator of denitrification as suggested by Clifford and Liu [1,4]:

$$97\text{NO}_3^- + 50\text{C}_2\text{H}_5\text{OH} \rightarrow 46\text{N}_2 + 5\text{C}_3\text{H}_7\text{O}_2\text{N} + 75\text{CO}_2 + 84\text{H}_2\text{O} + 97\text{OH}^-.$$ (1)

### 3.5. Regeneration efficiency of the different regenerants

Using the five kinds of regenerants listed in Table 1, the exhausted A520E resins were regenerated and the breakthrough curves were plotted for nitrate to compare the regeneration efficiency of each brine. For this purpose, the throughput before reaching an effluent nitrate concentration of 10 mg NO$_3^{-}$/N/l was estimated for each cycle. Fig. 7 shows six representative sets of breakthrough curves selected from among the 25 cycles of service and regeneration completed during 166 days.

When the 3% NaCl was used as a regenerant at 5 BV/h for 80 min, there was nearly no decrease in throughput in spite of intermittent operation. By the 10th cycle, the efficiency of regenerants B and C decreased sharply to no more than 67% and 77% of regenerant A, respectively. When comparing the throughput of regenerant B with D, or regenerant C with E, there was $\sim$70 BV of difference depending on whether the recycled
Fig. 5. Operating results of the sulfate reducing reactor treating the effluent from the denitrification reactor.

Fig. 6. Gas production by the denitrification and the sulfate reduction reactors.
brines were treated by the GAC column or not. This difference might have been caused by the bio-polymeric materials and residual COD present in the effluent of the reactors. It is difficult for the sand filter to remove these organic compounds. Van der Hoek and Klapwijk [12] pointed out that accumulated humic substances could cause organic fouling of the resin in a closed regeneration circuit with a biological process. Thus, installation of the GAC adsorption column between the nitrate ion exchange and the biological brine recycling system could be a viable measure for preventing resin contamination by the bio-polymeric materials and residual dissolved organics.

By the 10th cycle, the efficiency of regenerants B and C were quite low compared to regenerants D and E. For this reason, only regenerants D and E were compared with regenerant A for the remaining cycles. At the 25th cycle, the throughput of regenerant D decreased noticeably to 140 BV (51% of regenerant A), while the regenerant E produced 250 BV of throughput. The only difference between regenerants D and E was whether the sulfate reduction reactor was included in the brine recycling system or not. In order to elucidate how the presence or absence of the sulfate reduction step caused this difference, the concentrations of several anions were measured at each step in the brine recycling process.

Fig. 7. Comparison of throughput obtained with different regenerants (refer to Table 1 for explanation of A to E).
As shown in Table 5, there was a remarkable difference in both bicarbonate and sulfate concentrations of regenerant D (after denitrification) compared to regenerant E (after denitrification and sulfate reduction), but their chloride concentrations were similar. The bicarbonate concentration of regenerant E was higher than regenerant D, while the sulfate concentration was much less than in regenerant D. The surplus amount of bicarbonate in regenerant E was produced by the sulfate reduction reaction. According to the empirical equation (2) for sulfate reduction, 1.04 g of alkalinity is produced from each gram of sulfate reduced [13]. Van der Hoek et al. [6] showed that bicarbonate could be used with NaCl for regenerating exhausted resin. Thus, the higher bicarbonate concentrations of regenerant E clearly show the potential for more efficient regeneration if the sulfate reduction reactor is included:

\[
\text{C}_2\text{H}_5\text{OH} + \text{SO}_4^{2-} + \text{H}_2\text{O} \rightarrow \text{H}_2\text{S} + 2\text{HCO}_3^- + 2\text{H}_2. \quad (2)
\]

It was interesting to find out that the bicarbonate concentration decreased by a relatively constant amount after regeneration of exhausted resin. This occurred despite the collection of spent brine from all five ion exchange columns into a single storage vessel. If the elution of each column had been measured separately, the differences in anion concentration would have been even more apparent.

It was necessary to control the pH of the biologically recycled brine in this study. Since the bicarbonate level increased during denitrification and again during sulfate reduction, the pH of regenerants C and E was often over 9.0. Thus, the pH of regenerants C and E was adjusted using phosphoric acid before being used to regenerate columns III and V, respectively. It was also desirable to maintain the pH of the recycled brines close to that of the virgin NaCl in order to compare the regeneration efficiencies. The addition of the phosphoric acid into the biological brine recycling line did cause some variation in the anion concentrations summarized in Table 5.

As mentioned earlier, only \( \sim 330 \text{ ml (5 BV/h } \times 80 \text{ min) } \) of virgin brine (3% NaCl) was used for regenerating control column A for each cycle. No more salt was added to the brine recycling system from outside to compensate the salt concentration. This is quite different from previous researchers who added some salt after the denitrification step to compensate the salt to a target concentration. Even though it is not yet clear exactly how much the sulfate reduction reactor could reduce the amount of salt for compensation, it is likely that the sulfate reduction reactor, when located after the denitrification step, will eliminate the need for salt compensation.

### 4. Conclusions

For more efficient reuse of spent brine produced during the nitrate ion exchange process, both sulfate reduction and granular activated carbon (GAC) adsorption units were incorporated into a conventional biological brine recycling system consisting of denitrification and filtration processes. Several conclusions were reached after experimental operation of the improved ion exchange and brine recycling system.

1. An ion exchange process using nitrate-to-sulfate selective resin successfully produced about 300 bed volumes (BV) of throughput while treating groundwater containing about 30 mg/l of NO\(_3\)-N and 40 mg/l of SO\(_4\)\(^{2-}\).

2. When the mixed spent brine was fed into the biological brine recycling system, more than 90% of NO\(_3\)-N was removed in the denitrification reactor at the nitrate loading rate of 5.4 g NO\(_3\)-N/l/d. In the sulfate reduction reactor, \( \sim 50\% \) of sulfate remaining in the effluent of the denitrification reactor was reduced at the sulfate loading rate of up to 1.8 g SO\(_4\)\(^{2-}\)/l/d. The loading rates of nitrate-N and sulfate, and the corresponding removal efficiencies observed during operation of the experimental reactors are valuable information for deciding the proper size of each reactor.

3. The regeneration efficiency of biologically recycled brines without GAC adsorption decreased sharply.
Comparison of the regenerating efficiency obtained with recycled brines, with or without GAC adsorption, revealed that there was ~70 BV of differences in the throughput depending on whether the recycled brines were further treated by a GAC column or not. The bio-polymeric materials and residual COD present in the effluent of the reactors might cause this discrepancy.

4. Comparison of two biologically regenerated brines, one with only a denitrification step and the other with both denitrification and sulfate reduction steps, proved that the sulfate reduction reactor might have a role in reducing salt compensation.

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