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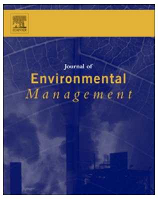

Research article

Pressurized hydrogenotrophic denitrification reactor for small water systems

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ABSTRACT

The implementation of hydrogenotrophic denitrification is limited due to safety concerns, poor H_2 utilization and low solubility of H_2 gas with the resulting low transfer rate. The current paper presents the main research work conducted on a pressurized hydrogenotrophic reactor for denitrification that was recently developed. The reactor is based on a new concept suggesting that a gas-liquid equilibrium is achieved in the closed headspace of denitrifying reactor, further produced N_2 gas is carried out by the effluent and gas purging is not required.

The feasibility of the proposed reactor was shown for two effluent concentrations of 10 and 1 mg NO_3^--N/L. Hydrogen gas utilization efficiencies of 92.8% and 96.9% were measured for the two effluent concentrations, respectively. Reactor modeling predicted high denitrification rates above 4 g NO_3^--N/($L_{reactor}$·d) at reasonable operational conditions. Hydrogen utilization efficiency was improved up to almost 100% by combining the pressurized reactor with a following open-to-atmosphere polishing unit. Also, the potential of the reactor to remove ClO_4^- was shown.

1. Introduction

The benefits of using H_2 gas as the electron donor for biological denitrification of groundwater were discussed previously. Among them, the clean nature and the low cell yield of hydrogenotrophic bacteria are the major advantageous features, resulting in small waste sludge production, minimal reactor clogging and reduced-cost post treatment. Additional advantage is the low price of H_2 gas compared to other electron donors per electron-equivalent delivered for contaminant reduction (two-thirds the price of methanol). Therefore, H_2 gas is an excellent choice for decentralized and small water systems where a simple and reliable technology with minimal manpower control is required. However, supplying H_2 gas at high transfer rates, yet economically and safely, remained the main challenge limiting the use of hydrogenotrophic denitrification (Karanasios et al., 2010). In some cases, a co-contamination of NO_3^- and ClO_4^- is observed and requires an overall solution for removing both ions (Zhao et al., 2014).

A summary of the prior technology available for hydrogenotrophic denitrification was given elsewhere (R. Epsztein et al., 2016a). Among these technologies, the membrane biofilm reactor (MBfR) has gained the most attention due to its safe and economic gas delivery system with close to 100% utilization efficiency of H_2 gas. Membrane fouling and scaling together with difficulties of biomass control are possible drawbacks of a typical MBfR (Karanasios et al., 2010). Additionally, the lower surface area provided by the membrane for biofilm growth compared to the surface area provided by plastic carriers in a packed- or fluidized-bed reactor may result in lower denitrification rates. For comparison, in the original paper presenting the MBfR, the specific total (i.e. clean) surface area reported was 390 m^2/m^3 (Lee and Rittmann, 2000), while Aqwise® plastic carriers, for example, provide a specific total surface area of 900 m^2/m^3. Despite the drawbacks described, the MBfR provides a promising solution for H_2 delivery and is implemented in full-scale in various groundwater treatment plants in California, USA since 2012 for the treatment of various pollutants (Martin and Nerenberg, 2012).

To the best of our knowledge, there are no earlier reports of hydrogenotrophic systems based on pressurized reactors with closed headspace. Most of conventional hydrogenotrophic denitrification reactors based on packed or fluidized bed present a similar H_2 delivery scheme of gas purging to the atmosphere in order to improve H_2 transfer rates and enable discharge of N_2 gas produced during denitrification. This operation, of course, results in

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a significant release of H_2 gas to atmosphere with its related economic and safety concerns.

A novel pressurized hydrogenotrophic denitrification reactor was recently proposed (R. Epsztein et al., 2016a). The reactor is based on a new concept suggesting that N_2 gas pressure reaches a constant level in the closed headspace of denitrifying reactor and therefore purging is not required (see explanation in the next section). The reactor is characterized by high denitrification rates, minimal hydrogen loss and low risk and can serve also for ClO_4^- removal. A following small open-to-atmosphere polishing unit can be used to eliminate residual dissolved H_2 and allow for safe effluent discharge. The simplicity of the new reactor may encourage its full implementation, especially in remote and small water plants, where process monitoring and control should be minimized.

In the following research work, the pressurized reactor's proof of concept and capability to operate at high denitrification rates and high H_2 utilization efficiencies are described. Also, investigation of the combined pressurized reactor-polishing unit treatment scheme and the potential ClO_4^- removal in the pressurized reactor are addressed.

2. Materials and methods

2.1. Explanation of the new concept

The main novelty of the reactor is the operation under a pressurized closed headspace without any gas discharge. The common concern of N_2 gas build-up in a pressurized denitrifying system is addressed by the idea that in continuous operation the effluent water carries excess N_2 gas out of the reactor. The dissolved N_2 concentration in the reactor reaches a constant level according to the concentration of NO_3^--N removed and therefore the partial pressure of N_2 in the reactor must also remain constant and correlate with the dissolved N_2 concentration according to Henry's law. For example, in the case of typical conditions of an inlet NO_3^--N concentration of 25 mg/L and a projected reactor effluent at drinking water regulations of 10 mg NO_3^--N/L, approximately all 15 mg NO_3^--N/L removed are converted to N_2 (assuming low biomass yield). An additional source of N_2 in the effluent is atmospheric N_2 dissolved in the influent water and carried into the reactor (~14 mg N_2/L under conditions of normal air mixture with 0.8 bar of atmospheric N_2 gas and Henry's constant of 17 mg N_2/[L·bar] at 20 °C). Therefore, the effluent water will contain about 29 mg N_2/L. Assuming for simplification that the reactor is completely mixed so the dissolved N_2 concentration is the same at any position in the reactor, this dissolved N_2 concentration will result in the development of a new gas-liquid equilibrium in the closed-headspace reactor with N_2 pressure around 1.7 bar according to Henry's law (29 mg N_2/L divided by 17 mg N_2/[L·bar]). Applying a total pressure of 2 bar, for instance, will leave room for 0.3 bar of H_2 gas at gas-liquid equilibrium. Since N_2 reaches equilibrium and does not further accumulate over time, there is no need for gas discharge and the risky and economic H_2 loss to atmosphere through gas purging of the reactor is prevented. Hydrogen loss is therefore limited only to the dissolved H_2 in the effluent. The operation under low-pressurized headspace consisting uniquely of H_2 and N_2 gases prevents hazardous H_2-O_2 contact and minimizes the risk of explosion in case of failure.

In its original version presented in the current paper, the reactor is operated under an unsaturated flow regime as a trickling filter where water is recirculated and trickled over the biofilm carriers (Fig. 1). Plastic carriers with high surface area are used and together with high mass transfer of H_2 gas due to the unsaturated flow, high denitrification rates are achieved. The reactor is continuously fed with NO_3^--contaminated groundwater. When enough liquid collects at the reactor's bottom and reaches a level switch, a drain valve is opened and effluent water is released (i.e. pulsed discharge).

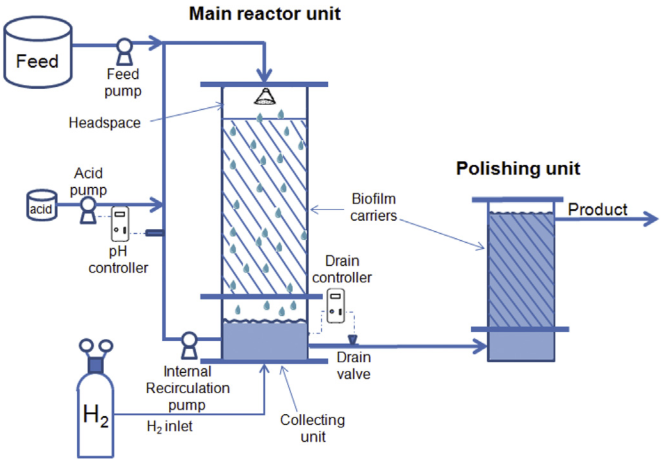

Fig. 1. Schematic diagram of the full experimental system.

An alternative version of the pressurized reactor, using a submerged bed where gas is recirculated from the reactor's headspace to the bottom and bubbled through the submerged bed, was tested in another work (R. Epsztein et al., 2016c).

2.2. Experimental setup

A schematic diagram of the full experimental system is illustrated in Fig. 1.

The full system included the main reactor unit and a following polishing unit to remove the residual H_2 in the reactor effluent. Except for the experiment testing the polishing unit performance (section 3.3), only the main reactor unit was used. The main reactor unit comprised of a clear PVC cylindrical reactor 100 cm in height and 10.5 cm in diameter divided into three unequal parts. The top part of the reactor (height 29 cm) served as an empty headspace, the middle part (height 51 cm) contained plastic biofilm carriers (total surface area of 900 m^2/m^3, *Aqwise*) and was separated by a metal screen from the bottom part (height 20 cm) of the reactor where recirculating water collected. The reactor was connected to a gas supply (H_2 cylinder with pressure regulator), feed pump (Diaphragm pump model 7090-42, Cole-Palmer), recirculation pump (FL-2403, ProPumps) and pH controlling unit (standard pH electrode, pH controller – pH190, Alpha; hydrochloric acid tank and acid pump – gamma/L, ProMinent).

In the experiment described in section 3.3, the main reactor unit was connected in a row to a PVC cylindrical polishing unit 22 cm in height and 10.5 cm in diameter, filled with the same plastic biofilm carriers as in the main reactor unit (see Fig. 1). The effluent water from the main reactor unit was introduced at the bottom of the polishing unit and released at the top part. The polishing unit was operated under a saturated-flow mode and its discharge was open to atmosphere.

For reactor start-up, a 2 L solution consisting of tap water enriched with NO_3^-, bicarbonate (added as 2 g of $NaHCO_3$) and phosphate with 0.5 L bacteria originating from a former hydrogenotrophic reactor was prepared and recirculated through the reactor in a batch mode under a constant H_2 pressure of 2 bar by purging. Water temperature was maintained constant at 25.5 ± 1 °C for the proof of concept trials (section 3.1). Model results (section 3.2) were based on model development experiments performed at

27.5 ± 1 °C. The trials with the polishing unit and ClO_4^- (sections 3.3 and 3.4, respectively) were also performed under constant temperature of 27.5 ± 1 °C. Bulk pH was kept at 7 ± 0.1 by dosing hydrochloric acid. The feed solution was tap water mixed with concentrated stock solutions of $NaNO_3$ and KH_2PO_4. For the experiment with ClO_4^-, the feed solution was mixed with stock solution of $NaClO_4$. Specific operational conditions applied for each experiment (e.g. flow rate, recirculation flow rate, pressure, etc.) are given in the 'Results and discussion'.

In all experiments reactor cleaning by flushing with water was carried out every few days to remove excess biomass growth (the polishing unit never had to be cleaned). The cleaning in sections 3.3 and 3.4 were performed once a week. Cleaning included washing of carriers, column and pipes with tap water.

All rate calculations in the experiments described in sections 3.1 and 3.2 were calculated based on a carriers packing volume of 3.4 L. In section 3.3, the polishing unit was loaded with carriers taken from the main reactor unit so the new packing volumes were 2.5 and 1.9 L for the main reactor unit and the polishing unit, respectively. The packing volume of the carriers used in the experiment with ClO_4^- (section 3.4) was also 2.5 L.

2.3. *Analyses*

Nitrate and perchlorate were determined using a Metrohm 761 ion chromatograph (IC) equipped with a 150 mm Metrosep A Supp 5 column with column guard and suppressor using a CO_3^{-2}/HCO_3^- eluent. Nitrite-N and alkalinity were measured according to Standard Methods (Method 4500 and Method 2320, respectively). H_2 concentration in gas phase was measured by gas chromatography (TCD detector; column: HP-PLOT-Q 30 m; 0.53 mm 40u, Agilent 7890A). Samples for the gas phase analysis by gas chromatography were taken by direct injection of fresh gas mixture from the reactor headspace into a 20 mL sealed serum bottle. The bottle was first flushed with the same gas mixture from the reactor headspace for 1 min with gas flow rate of 250 mL/min to ensure exchange of the entire gas volume in the bottle. The dissolved H_2 concentration in liquid phase was measured by headspace analysis of effluent samples injected into a sealed serum bottle using the same gas chromatograph.

3. Results and discussion

3.1. *Proof of concept — reaching a gas-liquid equilibrium*

Two operational modes with different effluent NO_3^--N concentration were chosen for proving the concept of reaching a gas-liquid equilibrium in the pressurized reactor. Operation (i) simulates the common treatment process aimed to meet the worldwide regulations for NO_3^--N in drinking water. A common alternative practice is to treat a portion of NO_3^--N containing groundwater to very low concentrations (e.g. ~1 mg/L) and to mix the low concentration product water with untreated groundwater to meet the drinking water standard. This type of operation, sometimes called split treatment process, is represented by operation (ii).

The inlet NO_3^--N concentration was 25 mg/L for both operations. The influent flow rates were adjusted to 430 and 130 mL/min for operation mode (i) and (ii), respectively, in order to achieve the desired effluent concentrations (i.e. 10 and 1 mg/L, respectively). The recirculation flow rate was 2500 mL/min for both operations. Each operation started with pressurizing the reactor with H_2 gas to a total pressure of 3 bar (2 bar of H_2 gas were added above the atmospheric pressure). The total pressure was maintained constant throughout the experiment by keeping the H_2 cylinder connected to the reactor through a pressure regulator. The concentration of H_2 gas in the reactor headspace was measured over time and converted to partial pressure. Assuming H_2 and N_2 are the only gases in the reactor (after initial oxygen depletion), the partial pressure of N_2 gas could also be calculated by subtracting the partial pressure of H_2 from the total pressure.

Table 1 presents the main results achieved at gas-liquid equilibrium for the two operation modes.

The gas-liquid equilibriums described in Table 1 were achieved after 23 h and 105 h for operation (i) and (ii), respectively. After these times, gas composition in the reactor's headspace remained constant, proving the reactor's main concept. The longer time to reach a gas-liquid equilibrium in operation (ii) was due to the lower denitrification rate (with the accompanied lower N_2 production rate) and the higher N_2 pressure at gas-liquid equilibrium. The small deviation of the experimental results from the theoretical calculations for the partial pressures of N_2 at gas-liquid equilibrium can be attributed to the fact that the reactor is not a true completely mixed system. This results in higher NO_3^--N concentration at the top of the reactor (i.e. where less NO_3^--N is removed) with the corresponding lower partial pressure of N_2 produced according to the reactor's theory. Alternatively, the deviation may be explained by the density differences of H_2 and N_2 gases. The concentration of H_2, the lighter gas, at gas-liquid equilibrium was a bit higher than expected at the top of the reactor where the gas was sampled. The lower denitrification rate calculated for operation (ii) was due to NO_3^--N limitation with the resulting lower penetration to biofilm. Nitrite concentrations were always below detected levels for both operations. According to the stoichiometry suggested by McCarty (1972), the dissolved H_2 concentrations measured correlate with H_2 utilization efficiencies of 92.8 and 96.9% for operation (i) and (ii), respectively. The high H_2 utilization efficiencies were achieved due to the fact that H_2 gas was not wasted through gas purging. Further improvement of H_2 utilization efficiency is possible using the polishing unit as described in section 3.3.

3.2. *Denitrification rates in the pressurized reactor*

As mentioned above, on top of the inherent advantages of safety and economics in terms of H_2 utilization, the new reactor was designed to ensure high denitrification rates in comparison to existing hydrogenotrophic systems due to the use of high-surface-area carriers and the maintenance of high gas (H_2)-liquid transfer rate by the unsaturated flow. A mathematical model based on simple mass balances for steady-state and completely stirred hydraulic conditions was developed and validated for evaluating the performance of the unsaturated-flow pressurized reactor. Detailed description of model development was given in another publication (R. Epsztein et al., 2016b). Denitrification rates and H_2 utilization efficiencies were evaluated by the model for reasonable operational conditions of effluent NO_3^--N concentration of 10 mg/L, recirculation ratio (Q_R/Q) of 5 and different total pressures. The

Table 1
Main results achieved at gas-liquid equilibrium for the two operation modes under the constant conditions described.

	Operation (i)	Operation (ii)
Inlet NO_3^--N concentration [mg/L]	25	25
Effluent NO_3^--N concentration in reactor [mg/L]	10.1 ± 1.2	1.3 ± 0.6
Denitrification rate [g N/($L_{reactor}$ · d)]	2.1 ± 0.2	1.06 ± 0.06
Total pressure [bar]	3	3
[a]Theoretical N_2 pressure [bar]	1.7	2.3
Measured N_2 pressure [bar]	1.6	2.1
Effluent dissolved H_2 concentration [mg/L]	0.5	0.34
Hydrogen utilization efficiency [%]	92.8	96.9

[a] Based on the theory described in section 3.1.

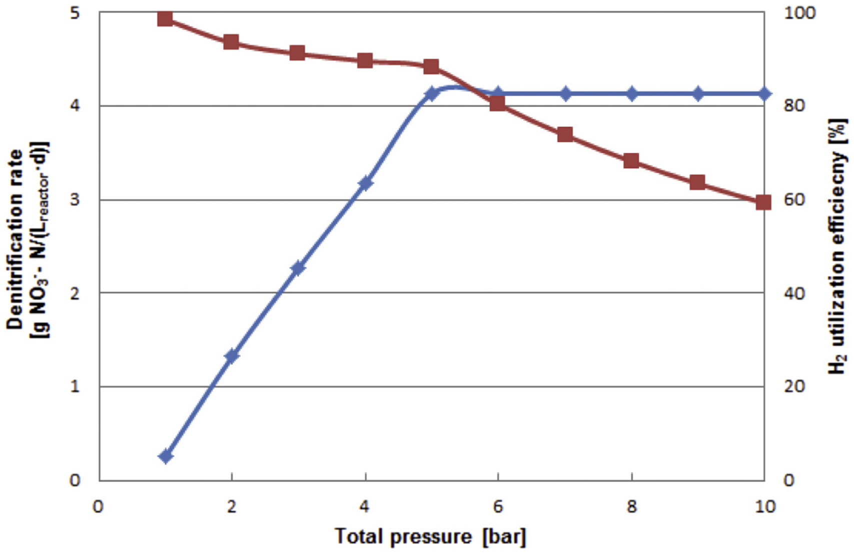

Fig. 2. Model results for denitrification rates (blue diamonds) and H_2 utilization efficiencies (red squares) for conditions of effluent NO_3^--N concentration of 10 mg/L, recirculation ratio (Q_R/Q) of 5 and different total pressures (X axis). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

model results are shown in Fig. 2.

In general, Fig. 2 shows that higher denitrification rates above 4 g N/($L_{reactor}$·d) together with higher utilization efficiencies of H_2 can be achieved in the pressurized reactor as compared to most hydrogenotrophic systems reviewed previously (R. Epsztein et al., 2016a). Higher denitrification rates up to 7.5 g N/($L_{reactor}$·d) were predicted by the model for higher recirculation ratios (R. Epsztein et al., 2016b). For the conditions described in Fig. 2, H_2 limited denitrification rates at low pressures (<4.5 bar). At higher pressures, NO_3^- was found to be the rate-limiting substrate since no improvement in denitrification rate was observed with increase in the pressure. Obviously, higher pressure had a negative effect on H_2 utilization efficiency. At higher pressure, a higher amount of unutilized H_2 gas is dissolved in the liquid phase and released with the effluent. The dip in the curves at total pressure of ~5 bar is a result of the model transition from H_2 limitation to NO_3^- limitation.

3.3. *Hydrogen utilization efficiency using the polishing unit*

The full system, i.e. the main reactor unit plus polishing unit, was operated for 60 days under the following conditions: inlet NO_3^--N concentration of 25 mg/L, flow rate of 150 mL/min, recirculation flow rate of 3800 mL/min and total pressure of 2.5 bar. The main results achieved at gas-liquid equilibrium are summarized in Table 2.

Despite the low NO_3^--N concentration in the reactor, i.e. low penetration to biofilm, the denitrification rates in the main reactor unit were always above 2 g N/($L_{reactor}$·d) due to the high active biofilm surface area achieved by the higher recirculation flow rate with the resulting improved media wetting. Nitrite concentrations were always below detected levels. According to the theory suggested in section 3.1, the N_2 pressure developed at 25 °C and removal of ~25 mg NO_3^--N/L at gas-liquid equilibrium is around 2.2 bar. This leaves room for 0.3 bar of H_2. The H_2 pressure measured at gas-liquid equilibrium was slightly lower (0.23), probably due to asymmetrical distribution of H_2 gas in the column and the accuracy level of the GC measurement. Assuming the dissolved H_2 concentration in the reactor is close to saturation (~1.5 mg H_2/(L·bar) at 25 °C) and using the suggested stoichiometry of hydrogenotrophic denitrification (McCarty, 1972), the dissolved H_2 in the reactor effluent should be 0.345 with the corresponding H_2 utilization efficiency of 96.8%.

In the polishing unit, the submerged-up-flow regime minimizes H_2 discharge to atmosphere and allows for additional H_2 consumption by bacteria. All the residual NO_3^--N in the reactor effluent (0.66 mg/L) was removed using additional 0.283 mg/L of H_2 (according to the stoichiometry), so that the residual H_2 after the polishing unit effluent was decreased from 0.345 to 0.062 mg/L with the corresponding H_2 utilization efficiency of 99.4%. It is important to note that this calculation of H_2 utilization efficiency was based on the effluent dissolved H_2 concentration in reactor at saturation, while the real dissolved H_2 concentration should be

Table 2
Main results achieved at gas-liquid equilibrium for the full scheme under the constant conditions described.

Inlet NO_3^--N concentration [mg/L]	25.8 ± 0.6
Effluent NO_3^--N concentration in reactor [mg/L]	0.66 ± 0.2
Denitrification rate [g N/($L_{reactor}$·d)	2.172 ± 0.15
Total pressure in main reactor unit [bar]	2.5
[a]Theoretical H_2 pressure [bar]	0.3
Measured H_2 pressure [bar]	0.23 ± 0.01
Effluent dissolved H_2 concentration in reactor at saturation [mg/L]	0.345
H_2 utilization efficiency before polishing unit [%]	96.8
Effluent NO_3^--N concentration in polishing unit [mg/L]	0
[b]Effluent dissolved H_2 concentration in polishing unit [mg/L]	0.062
H_2 utilization efficiency after polishing unit [%]	99.4

[a] Based on the theory described in section 3.1.
[b] Based on theoretical calculation (see explanation in text).

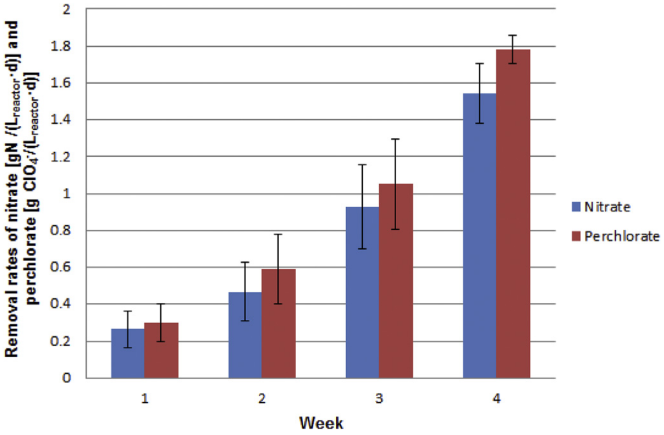

Fig. 3. Average weekly removal rates of NO_3^--N (blue) and ClO_4^- (red) during four weeks of operation. At the beginning of week 1, ClO_4^- was introduced in the reactor for the first time. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

lower with the corresponding even higher H_2 utilization efficiency.

3.4. Potential perchlorate removal in the pressurized reactor

The potential of ClO_4^- removal in the pressurized reactor using the biofilm carriers from the former denitrification experiments was studied for four weeks. Inlet NO_3^--N and ClO_4^- concentrations were kept constant on approximately values of 15 and 20 mg/L, respectively. The flow rate was increased gradually over time from 20 to 200 mL/min. Each increase in the flow rate was performed after effluent ClO_4^- concentration was reduced to below 1 mg/L. The total pressure was 2 bar and the recirculation flow rate was 6600 mL/min. The average weekly removal rates of NO_3^--N and ClO_4^- are shown in Fig. 3.

Fig. 3 shows the potential of the pressurized reactor to simultaneous remove NO_3^- and ClO_4^-. The immediate acclimation of bacteria from the denitrification reactor to reduce ClO_4^- shows that no specialized inoculation is required. Maximal ClO_4^- removal rate of 1.83 g/($L_{reactor}\cdot$d) was observed after four weeks of operation. For comparison, Logan et al. reported a removal rate of 1.16 g/($L_{reactor}\cdot$d) in a non-pressurized unsaturated-flow hydrogenotrophic reactor at a temperature of 23 °C, similar pH (7) and influent ClO_4^- concentration (18 mg/L) without NO_3^- (Logan and LaPoint, 2002). Effluent ClO_4^- concentrations were generally below 1 mg/L, except on days when the loading rate was increased where the effluent concentrations reached 3–4 mg/L ClO_4^-. The effluent NO_3^--N concentration from the pressurized reactor was always below 1 mg/L. Effluent NO_2^- concentrations were always below detection levels.

In a following experiment, reduction of ClO_4^- concentration to permitted trace levels below 7 μg/L together with complete H_2 utilization was achieved using the polishing unit (data not shown). The plug-flow character of the polishing unit is advantageous for reducing ClO_4^- concentrations to such low trace levels for two main reasons: (1) in CSTRs, reaching such low trace concentrations is harder due to the mixing with the inlet high-concentrated stream; (2) better performance of ClO_4^- reduction can be achieved downstream after depletion of NO_3^-.

4. Conclusions

An unsaturated-flow pressurized hydrogenotrophic reactor for denitrification without gas purging was tested. The prevalent notion claiming that N_2 gas accumulates in the closed headspace of denitrification reactor was refuted. Denitrification rates of one order of magnitude higher than most previously reported denitrification rates were achieved by applying the concept of the pressurized reactor in a trickling filter with recirculation. Using an up-flow open-to-atmosphere polishing unit, the residual H_2 from the pressurized could be further consumed, thus increasing H_2 utilization efficiency up to almost 100%. The potential of the pressurized reactor to simultaneously remove of NO_3^- and ClO_4^- was shown with further decrease of ClO_4^- concentration to the permitted trace levels in the polishing unit. Process simplicity and safety may encourage the use of the pressurized reactor in remote and small water treatment plants.

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