

Total Nutrient Recovery from Urine – Operation of a Pilot-Scale Nitrification Reactor

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ABSTRACT

Nitrification is a suitable pre-treatment to stabilise urine prior to evaporation. In a 120 L pilot scale moving bed biofilm reactor (MBBR) at Eawag, 50 % of the ammonium from stored urine is converted to nitrate without adding alkalinity. The maximum volumetric nitrification rate has been $420 \text{ g N} \cdot \text{m}^{-3} \cdot \text{d}^{-1}$, which corresponds to $1.4 \text{ g N} \cdot \text{m}^{-2} \cdot \text{d}^{-1}$ with regard to biofilm carrier surface (Kaldnes[®] K1). To ensure a stable process, load fluctuations have to be kept to a minimum. A sudden increase in ammonium load boosts activity of ammonium oxidising bacteria (AOB), causing nitrite accumulation, which further inhibits nitrite oxidising bacteria (NOB).

KEYWORDS

Total nutrient recovery, urine nitrification, moving bed biofilm reactor, process control

BACKGROUND

In order to recover all nutrients contained in urine, a two-stage process was developed: First, urine is partially nitrified in a moving bed biofilm reactor (MBBR), and second, the partially nitrified solution is distilled to obtain a concentrated nutrient solution. The preliminary nitrification step is necessary to avoid ammonia losses during distillation. After the process steps had been successfully tested at lab scale (Udert and Wächter, 2012), a pilot-scale installation was designed, constructed and tested at Eawag's main office building (Figure 1). This extended abstract presents details on the start-up strategy and process control of the nitrification reactor.

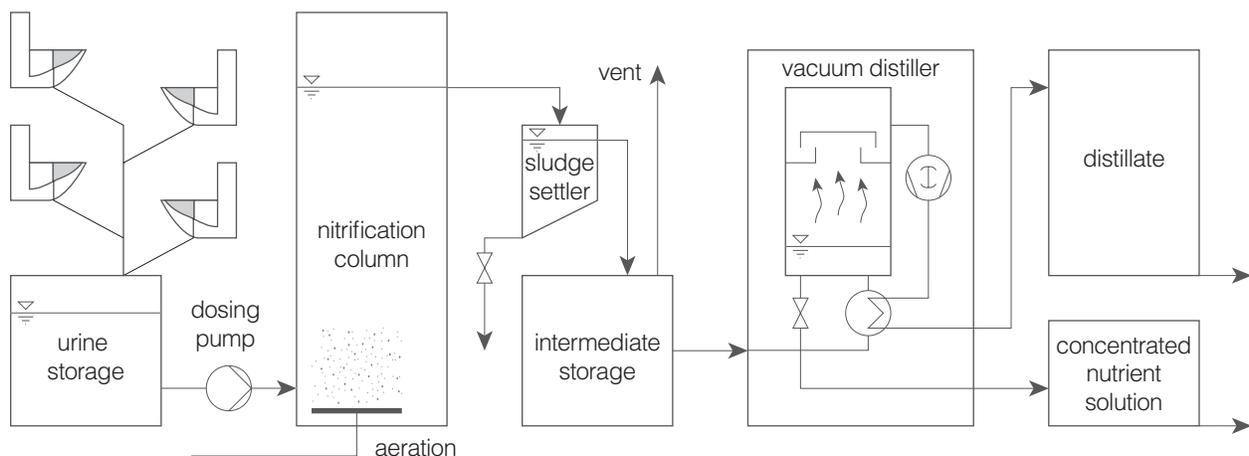


Figure 1: Flow diagram of the reactor set-up for total nutrient recovery from urine.

METHODOLOGY

At Eawag, urine is collected from urine-diversion toilets and waterless urinals. On average, 100 L urine per day is collected. Female and male urine is stored in two separate 1000 L tanks, from where it is pumped into the nitrification reactor. The reactor is operated as a continuous flow stirred tank reactor (CSTR). The influent urine's nutrient concentrations can be found in Table 1. Urine from the women's collection tank is nitrified in a column with 120 L liquid volume. The column contains biofilm carriers (Kaldnes[®] K1, specific surface: 500 m²·m⁻³) with a bulk volume of 60 % of the total reactor volume. Initially, the reactor was filled with 100 L tap water and 20 L activated sludge taken from Eawag's experimental wastewater treatment plant. The aeration rate is set to 1 m³·h⁻¹, which allows good mixing of the biofilm carriers. Dissolved oxygen concentrations in the reactor never dropped below 6 mg·L⁻¹. The reactor is operated in continuous mode and is fed with a membrane dosing pump. In the reactor, pH, dissolved oxygen, and electric conductivity are continuously monitored. Samples are taken twice a week in the influent and in the reactor to measure their chemical composition (Table 1).

Table 1: Concentrations in the influent and in the reactor. The reactor concentrations are reported for conditions, when the nitrification process functioned properly, i.e. nitrite concentration was below 5 mg·L⁻¹.

| <i>Parameter</i> | <i>Unit</i> | Influent | | Reactor | |
|---|----------------------|---------------------|----------|---------------------|----------|
| | | <i>Mean ± Stdev</i> | <i>n</i> | <i>Mean ± Stdev</i> | <i>n</i> |
| NH ₄ ⁺ ammonium | mg N·L ⁻¹ | 1790 ± 180 | 106 | 899 ± 140 | 83 |
| NO ₂ ⁻ nitrite | mg N·L ⁻¹ | – | | 2 ± 1 | 83 |
| NO ₃ ⁻ nitrate | mg N·L ⁻¹ | – | | 914 ± 203 | 78 |
| Cl ⁻ chloride | mg·L ⁻¹ | 1830 ± 320 | 24 | 1780 ± 250 | 11 |
| PO ₄ ³⁻ phosphate | mg P·L ⁻¹ | 108 ± 14 | 14 | | |
| SO ₄ ²⁻ sulphate | mg·L ⁻¹ | 316 ± 14 | 13 | | |
| Na ⁺ sodium | mg·L ⁻¹ | 966 ± 314 | 15 | | |
| K ⁺ potassium | mg·L ⁻¹ | 897 ± 103 | 15 | | |
| Ca ²⁺ calcium | mg·L ⁻¹ | 10 ± 11 | 15 | | |
| Mg ²⁺ magnesium | mg·L ⁻¹ | < 5 | 5 | | |
| TIC total inorganic carbon | mg·L ⁻¹ | 970 ± 109 | 66 | < 4 | 36 |
| TOC total organic carbon | mg·L ⁻¹ | 863 ± 250 | 66 | 77 ± 42 | 37 |
| COD ^a chemical oxygen demand | mg·L ⁻¹ | 2110 ± 390 | 104 | 217 ± 35 | 38 |
| pH | – | 8.9 ± 0.1 | 100 | | |
| EC ^b electric conductivity | mS·cm ⁻¹ | 15.9 ± 2.2 | 40 | | |

^a COD measured as dissolved COD

^b Electric conductivity (EC) temperature compensated (25°C)

RESULTS AND DISCUSSION

The start-up phase took approximately 45 days, until the reactor reached the maximum nitrification rate of 420 g·m⁻³·d⁻¹, which corresponds to 1.4 g·m⁻²·d⁻¹ with regard to biofilm carrier surface (500 m²·m⁻³ specific surface), and a flow rate of 50 L·d⁻¹ urine. At several times during operation, nitrite accumulated in the reactor due to increased activity of AOB. In general,

nitrification processes in urine are prone to nitrite accumulation caused by imbalances between AOB and NOB activity (Sun *et al.*, 2012). Also, the lab-scale reactor had shown to be susceptible to nitrite accumulation caused by suddenly increased loading (Udert and Wächter, 2012).

In order to prevent nitrite accumulation, the inflow rate had to be controlled carefully. The influent flow rate was first controlled with pH set-points: the decrease of the pH value served as a proxy for the ammonium oxidation rate. The influent pump was triggered whenever nitrification lowered the pH value below the set-point. During pumping, the pH increased due to the high alkalinity in the influent urine. When a certain pH interval was exceeded, the pump was shut off. The pH set-points ranged from 5.7 to 6.5 and the pH interval was 0.1 units. Once the AOB activity was sufficiently high, the alkalinity load in the influent was not able to compensate the proton production any more. At this point, the pH in the reactor did not reach the upper set-point anymore even with continuous pumping. Thus, the pump rate had to be increased carefully by hand.

The pump rate was only increased, if the nitrite concentration was negligible to ensure that the NOB activity was sufficient and corresponded to AOB activity. In any case, the flow rate had to be raised in small steps to prevent nitrite from accumulating. The urine volume in the influent storage tank never dropped below 700 L. Hence, a minimum retention time of 14 days at the maximum flow rate of $50 \text{ L}\cdot\text{d}^{-1}$ assured that the fluctuations in concentration were minimised. This can be seen from the standard deviation of the influent ammonia concentrations, which was only 10 % (Table 1) and from the graph depicted in Figure 2.

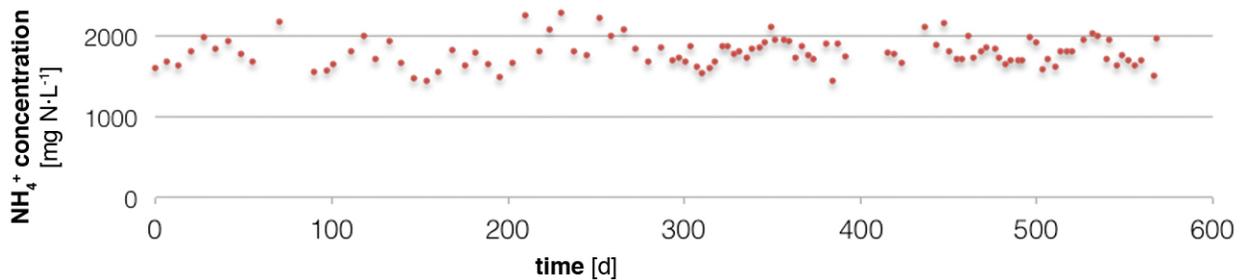


Figure 2: Fluctuating ammonium concentration in the reactor influent tank.

To better understand the growth dynamics of AOB and NOB and retrace the causes for nitrite accumulation, we simulated the processes with a mechanistic computer model implemented in Aquasim (Hug *et al.* 2013). A sudden increase of the influent rate by a factor of 1.2 provided much free ammonia (NH_3) as a substrate for the AOB, causing them to increase their activity abruptly. As NOB activity did not accelerate as quickly as AOB activity, the nitrite produced by the latter started to accumulate. Consequently, NOB became inhibited by their substrate nitrous acid (HNO_2) (Figure 3). The simulations corresponded well with the observations in the reactor and they exemplify that the activity of AOB and NOB in urine is strongly sensitive to the pH value, since it determines the concentration of the actual substrate NH_3 ($\text{pK}_a \text{ NH}_4^+/\text{NH}_3 = 9.25$) and HNO_2 ($\text{pK}_a \text{ HNO}_2/\text{NO}_2^- = 3.35$) respectively (Hug *et al.*, 2013; Udert *et al.*, 2003).

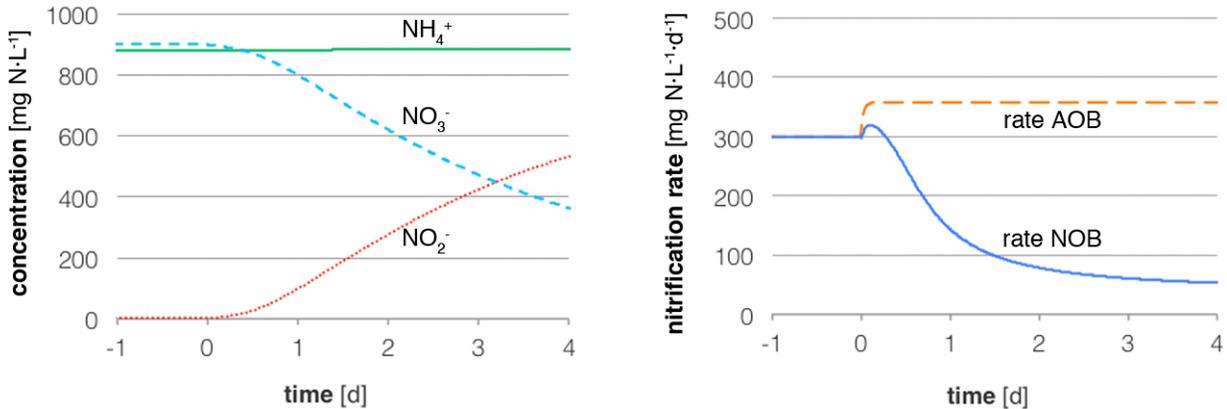


Figure 3: Simulation of nitrite accumulation (left) and corresponding AOB and NOB activity (right) caused by a sudden increase of the influent rate by a factor of 1.2.

CONCLUSION

Scaling up nitrification of urine produced valuable findings with regards to large-scale applications of complete nutrient recovery. Both experiments and simulations showed that maintaining a low and constant free ammonia concentration in the substrate is of pivotal importance to assure process stability, i.e. avoid nitrite accumulation. A sufficiently large storage tank has to precede the nitrification step, in order to attenuate possible fluctuations of the influent ammonium concentration. The combination of simulations with pilot-scale experiments will allow us to further fortify process stability and resilience mechanisms of the system.

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