



# Improving secondary sludge biodegradability using free nitrous acid treatment

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## ABSTRACT

This study presents a novel strategy based on free nitrous acid (FNA) treatment to improve the biodegradability of secondary sludge. Several experiments were conducted to demonstrate the biocidal effect of FNA on activated sludge. The viable fraction as well as the biological activity of the biomass decreased significantly after 8–48 h treatment with FNA. The biodegradability of the FNA treated sludge was compared to that of the same sludge without FNA treatment by aerobically digesting these sludges with a full-scale activated sludge for 14 and 6 days respectively. Ninety percent of the FNA treated biomass was consumed during the 14-day aerobic digestion compared to 41% achieved with the untreated biomass. During the 6-day aerobic digestion, 50% of the FNA-treated sludge was degraded. The results indicate that FNA treatment substantially increases sludge biodegradability.

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## 1. Introduction

The activated sludge based processes are widely used for biological wastewater treatment. They involve the transformation of dissolved and suspended organic contaminants into biomass and gases. One of the major drawbacks of these treatment processes is their high production of sludge that needs to be treated and disposed. The cost associated with sludge treatment and disposal can be up to 60% of the overall costs in wastewater treatment plants (WWTPs) (Canales et al., 1994). Sludge reduction has been one of the critical challenges for the wastewater industry.

Various strategies for solids reduction have been developed, which can be implemented either in the sludge return line in the main wastewater treatment system, or in the sludge handling train as a pretreatment step prior to the anaerobic or aerobic digestion process (Mahmood and Elliott, 2006; Foladori et al., 2010; Carrère et al., 2010).

The most commonly used techniques for sludge treatment in the sludge return line include thermal, mechanical, electrical and chemical (with the addition of acid, base or oxidant) treatment, with the treated sludge recirculated to the bioreactor for further biodegradation (Saby et al., 2002; Heinz, 2007; Foladori et al., 2010). These treatments increase cell lysis with subsequent release of extracellular and intracellular constituents, which become substrate available for biodegradation (i.e., cell lysis-cryptic growth) (van Loosdrecht

and Henze, 1999; Hao et al., 2010). These technologies reduce sludge production and also enhance denitrification (Boehler and Siegrist, 2004; Dytczak et al., 2007). As examples, Heinz (2007) obtained 45% sludge reduction by treating part of the return sludge using the pulsed electric field technology with a specific energy of 100,000 kJ/m<sup>3</sup>. Saby et al. (2002) reported that 65% sludge reduction was achieved by recirculating chlorine-treated sludge to the bioreactor.

Aerobic and anaerobic sludge digestion is widely used to reduce sludge production and to stabilize sludge prior to disposal. Anaerobic sludge digestion also recovers energy from sludge. However, aerobic and anaerobic sludge digestion is generally limited by the poor degradability of secondary sludge (Eastman and Ferguson, 1981; Malina and Pohland, 1992). Therefore, techniques similar to those applied in the sludge return line have been developed and integrated in the sludge handling line to improve the sludge biodegradability (Muller et al., 1998; Bougrier et al., 2006; Onyiche, 2007; Eskicioglu et al., 2008; Foladori et al., 2010; Ge et al., 2011; Chang et al., 2011; Yang et al., 2011). Onyiche (2007) observed a 30% increase in biogas production and a 23% reduction in sludge volume by treating excess sludge at 150 bar with a high pressure homogenizer (mechanical pretreatment) before anaerobic digestion. Ge et al. (2011) found that the destruction of volatile solids (VSs) in thermophilic–mesophilic temperature phased anaerobic digestion (TPAD) increased from 34% to 48% with thermophilic pretreatment at 50–60 °C. The destruction was 11–30% higher than that achieved with mesophilic–mesophilic TPAD (mesophilic sludge treatment at 35 °C). Yang et al. (2011) found an increase

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on the methane yield and solids reduction (from 57% to 65%) by combining aerobic thermophilic irradiation pretreatment and intermittent irradiation anaerobic digestion.

However, all of the above mentioned approaches require either intensive energy inputs (high pressure and/or high temperature) or large consumption of chemicals such as chlorine, ozone, or alkali, both with substantial economic and environmental costs. A cost-effective and environmentally friendly method for improving the biodegradability of activated sludge is needed.

Free nitrous acid (FNA or  $\text{HNO}_2$ ) has been proven to be inhibitory, at parts per billion (ppb) levels, to several groups of bacteria involved in biological nutrient removal (Vadivelu et al., 2006a,b; Zhou et al., 2007, 2008; Pijuan et al., 2010; Ye et al., 2010). These bacteria can, however, recover from inhibition after FNA is removed. In a recent study, Jiang et al. (2011a) showed that FNA can be a strong biocidal agent towards microorganisms residing in anaerobic sewer biofilms, when used at parts per million (ppm) levels. They found that the viable fraction of microorganisms in the sewer biofilm studied decreased by approximately 80% after 24 h treatment at FNA levels of 0.2–0.3 mg  $\text{HNO}_2$ -N/L, and proposed this strategy as a cost-effective solution to control detrimental biofilm formation in sewer networks (Jiang et al., 2011b). In a pure culture study, Yoon et al. (2006) observed that mucoid, mutants of *Pseudomonas aeruginosa* that are refractory to phagocytosis and antibiotics, could be killed at an FNA concentration of around 0.17 mg  $\text{HNO}_2$ -N/L.

We hypothesise that the biocidal effect of FNA can be used to deactivate and lyse activated sludge, thereby improving its biodegradability. To verify this hypothesis, a denitrifying sludge was treated at different FNA concentrations (0–2.02 mg  $\text{HNO}_2$ -N/L). The biocidal effect of FNA on the sludge was assessed by determining the cell viability and the loss and recovery of sludge activity after FNA treatment. The improvement to the biodegradability of the FNA treated sludge was also determined through aerobic digestion using a full-scale sludge.

## 2. Methods

### 2.1. Sludge sources

Two different types of sludge were used to carry out the experiments.

Sludge 1 was withdrawn from a denitrifying sequencing batch reactor (SBR) treating synthetic wastewater. The reactor, operated with a cycle time of 6 h, was fed with methanol (800 mg COD/L) as the only carbon source and nitrate (200 mg N/L) as the sole electron acceptor. The reactor was operated with a sludge retention time of 10 days and a hydraulic retention time of 24 h. The reactor was in steady state achieving full removal of methanol and nitrate at the time of this study. This sludge was used to test the effect of FNA treatment on cell viability, and the loss and recovery of sludge activity after the treatment. A sludge specialized in one carbon source (methanol) was chosen in these tests because it is relatively easy to measure its recovery due to its carbon preference.

Sludge 2 was collected from a local biological nutrient removal WWTP in Queensland, Australia, receiving primarily domestic wastewater. The plant achieves high-levels of COD and nitrogen removal with an effluent total nitrogen level consistently below 8 mg N/L. This sludge was used as the digesting sludge in the tests to assess the biodegradability of Sludge 1 before and after FNA treatment.

### 2.2. Effect of FNA treatment on activated sludge

Five sets of batch tests were carried out using Sludge 1 to assess the effect of FNA treatment on sludge viability. In Sets 1, 2, 4 and 5,

2 L of mixed liquor was removed from the methanol-fed denitrifying SBR at the end of the cycle and distributed between 4 batch reactors (0.5 L each) operated in parallel. In Set 3, 1.5 L of the same mixed liquor was used and distributed between 3 batch reactors. A nitrite stock solution was added to the batch reactors in different volumes at the beginning of each experiment, which resulted in initial concentrations of nitrite varying between 0 and 900 mg N/L, as summarized in Table 1. The maximum concentration of 900 mg N/L was used because it can be produced on-site at a WWTP by implementing a partial nitrification system to treat the anaerobic sludge digestion liquor (Bartroli et al., 2010). Each anoxic test lasted for 48 h, during which pH was controlled via a program logic controller (PLC) at a pre-designed set-point ( $\pm 0.05$ ) (see Table 1), through automatically adding 0.5 M HCl or 0.2 M NaOH. The concentration of FNA ranged between 0 and 2.02 mg  $\text{HNO}_2$ -N/L (Table 1), which was achieved by varying the nitrite concentration and pH. The FNA concentration was calculated using the formula  $S_{N-NO_2^-}/K_a \times 10^{pH}$  with the  $K_a$  value determined using the formula  $K_a = e^{-2300/(273+T)}$  for a given temperature  $T$  ( $^{\circ}\text{C}$ ) (Anthonisen et al., 1976).

Samples for the live/dead staining analysis of cells, for the analysis of ammonia, nitrite, nitrate, mixed liquor suspended solids (MLSS) and volatile MLSS (MLVSS) (methods described below) were taken at 0, 8, 24, 48 h for Sets 1, 2, 4 and 5 and at 24 h and 48 h for Set 3.

The rate of ammonia production (expected to be caused by cell lysis during FNA treatment) in all the tests was calculated by linear regression analysis of the measured ammonium concentration. Each set of experiments included a blank (no nitrite addition, Table 1), which served as a reference.

### 2.3. Recovery tests

In selected batch tests (T2 in Sets 1–3 with FNA being 0.67, 1.12 and 2.02 mg  $\text{HNO}_2$ -N/L, respectively), the denitrification rate of the sludge during and after FNA treatment (8, 24 and 48 h) was measured to determine the loss of biomass activity and its recovery over a 72 h period. This rate was chosen because denitrification was the main process occurring in the reactor where Sludge 1 was collected.

After each pre-designed exposure time (8, 24 and 48 h), the sludge was washed and re-suspended within 0.5 L nitrite-free water (autoclaved effluent from the SBR where Sludge 1 was sourced) to remove nitrite. Nitrate, methanol, ammonia and phosphate were then added at 50 mg N/L, 200 mg COD/L, 10 mg N/L and 5 mg P/L respectively at the beginning of the recovery study. During the recovery period, nitrate and methanol were monitored regularly and then added once they became limiting. pH was automatically controlled via a PLC at 7.5 ( $\pm 0.05$ ), which was the operating pH in the SBR where Sludge 1 was withdrawn, with the addition of 0.2 M NaOH and 0.5 M HCl solutions. The biomass activity was then measured at 0, 24, 48 and 72 h after FNA removal, as described below.

For the measurement of the biomass activity at 0 h after FNA removal, mixed liquor samples were taken every 15 min for 1 h and immediately filtered through disposable Millipore filter units (0.22  $\mu\text{m}$  pore size) using a syringe for the analyses of ammonia, nitrate, nitrite, phosphate and methanol. The denitrification rate was calculated using linear regression analysis of the nitrate data. For the measurement of the biomass activity at 24, 48 and 72 h after FNA removal, mixed liquor samples were taken every 24 h and the denitrification rate calculated in the same manner. The 24 h sampling frequency in these tests was applied due to the very low denitrification rate detected in the sludge after FNA treatment.

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