#### GASEOUS EMISSIONS FROM THE BIOLOGICAL VIA NITRITE NITROGEN REMOVAL PROCESS TREATING ANAEROBICALLY CO-DIGESTED EFFLUENTS N. Frison<sup>1</sup>, A. Chiumenti<sup>2</sup>, E. Katsou<sup>3</sup>, S. Malamis<sup>3</sup>, D. Bolzonella<sup>3</sup>, F. Fatone<sup>3</sup>

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#### Abstract

In this work the gaseous emissions of nitrous oxide (N<sub>2</sub>O), carbon dioxide (CO<sub>2</sub>) methane (CH<sub>4</sub>), ammonia (NH<sub>3</sub>) and methyl mercaptan (CH<sub>3</sub>SH) produced from the nitritation/denitritation biological nutrient removal (BNR) process were determined in a sequencing batch reactor (SBR). The SBR treated the anaerobic supernatant produced from the anaerobic co-digestion of the organic fraction of municipal solid waste (OFMSW) and waste activated sludge (WAS). Two experimental periods were carried out: period 1 in which a volumetric nitrogen loading rate (vNLR) of 0.81 kgN/m<sup>3</sup>d was applied, using acetic acid as carbon source and period 2 with vNLR=1.09 kgN/m<sup>3</sup>d using fermentation liquid produced from biowaste as carbon source. N<sub>2</sub>O emissions were 0.24% and 1.38% of the influent nitrogen load in periods 1 and 2 respectively. The higher dissolved oxygen (DO= 1.5 mg/L) concentration as well as the lower accumulation of nitrite resulted in significantly lower N<sub>2</sub>O emissions in period 1 compared to 2. NH<sub>3</sub> emissions were very low (<1% of influent nitrogen load), while some methane (5.3% of influent load as COD) was emitted during period 2, as the lower DO (0.95 mg/L) concentration probably created micro-anaerobic conditions within the sludge flocs; this was also favoured by the use of fermentation liquid as a carbon source.

#### Keywords

Gaseous emissions; nitrous oxide emissions, nitritation/denitritation; sequencing batch reactor

#### Introduction

During the operation of a wastewater treatment plant (WWTP) nitrous oxide (N<sub>2</sub>O), carbon dioxide (CO<sub>2</sub>), methane (CH<sub>4</sub>), nitric oxide (NO) as well as other gases can be emitted. N<sub>2</sub>O is of particular environmental concern since it has a global warming potential that is 300 times higher than that of CO<sub>2</sub>. The global production of N<sub>2</sub>O emissions from wastewater treatment was estimated to be 0.22 TgNO<sub>2</sub>-N/yr in 1990, which corresponded to 3.2% of the total estimated anthropogenic N<sub>2</sub>O emissions in the world [1-3]. The contribution of biological nutrient removal (BNR) to N<sub>2</sub>O emissions can be up to 10.2% [4]. The N<sub>2</sub>O emissions from wastewater management are estimated to contribute 26% to the total greenhouse gas emissions of the water chain. The latter includes water supply and treatment, wastewater collection and treatment and final discharge [3, 5]. The IPCC guidelines (2006) decreased the standard N<sub>2</sub>O emission factor from 1% to 0.5% of the nitrogen load of the WWTP influent. In countries having advanced

WWTPs, a low factor is applied by IPCC (2006) for direct emissions from WWTPs; this is 3.2 gN/person/year and corresponds to ~0.035% nitrous oxide emissions of the influent nitrogen load of a WWTP [3]. In terms of CO<sub>2</sub> equivalents nitrous oxide contributes by 7.9% to the total anthropogenic greenhouse gas (GHG) emissions. Methane has global warming potential that is 23 times higher than that of CO<sub>2</sub> and contributes by 14% to the total GHG emissions. Methane is manly emitted from livestock farming activities (166 million tonnes CO<sub>2</sub> equivalent in the EU per year), from landfills, composting units and WWTPs (95 million tonnes CO<sub>2</sub> equivalent in the EU per year) [6, 7].

BNR from wastewater is an effective approach for the prevention of eutrophication of water recipients. However, many practical design and operating decisions in WWTPs (including the BNR processes) have considerable impact on the overall environmental performance, in particular concerning the GHG emissions [8]. The vast majority of nitrous oxide emissions in WWTPs occur in the biological treatment processes. At the level of a BNR treatment plant, the N<sub>2</sub>O impact can reach up to 83% of the operational CO<sub>2</sub> footprint [9]. N<sub>2</sub>O emissions during BNR occur during the biological processes of nitritation and denitritation (Figure 1). During the nitritation process, N<sub>2</sub>O can be formed via two routes: the first pathway is as a by-product of the incomplete oxidation of hydroxylamine (NH<sub>2</sub>OH) to nitrite. Hydroxylamine is formed from the oxidation of ammonium from ammonium oxidizing bacteria (AOB) using the enzyme ammonia monooxygenase (AMO) to catalyze the reaction. NH<sub>2</sub>OH is then oxidized to nitrite using hydroxylamine oxidoreductase (HAO), a biochemical reaction which produces nitrous oxide. The second pathway of N<sub>2</sub>O formation is attributed to the process known as nitrifier denitrification. In this biochemical process nitrite is used as electron acceptor instead of oxygen; this can occur under limiting dissolved oxygen (DO) conditions during nitritation. In this biochemical pathway, nitrite reductase (NIR) and nitric oxide reductase (NOR) catalyze the reduction of NO2<sup>-</sup> to NO and N2O by AOB. The third pathway occurs during the anoxic operation of the reactor. In this process, NO and N2O are produced as process intermediates of NO3<sup>-</sup> reduction to N2. This is also the only stage in which N2O is also consumed as it is reduced to N<sub>2</sub> [10]. N<sub>2</sub>O can be produced in the anoxic phase and subsequently be stripped to the gas phase in an aerated compartment. As N<sub>2</sub>O has a relatively high solubility in water, stripping is therefore not very fast [3].

Desloover et al. [4] performed an overview of the quantified N<sub>2</sub>O emissions from full scale BNR plants that apply the conventional nitrification/denitrification and advanced nitritation/anoxic ammonium oxidation (anammox) and nitritation/denitrification processes; they concluded that nitritation is the bioprocess that mainly contributes to N<sub>2</sub>O emissions. Full-scale measurements also point to nitrite as a factor in N<sub>2</sub>O production [11]. Taking into account that the much higher greenhouse gas impact of N<sub>2</sub>O compared to CO<sub>2</sub>, it is necessary to determine whether nitrogen removal bioprocesses based on transient nitrite accumulation are systematically greater contributors of N<sub>2</sub>O than full nitrification processes [2, 12]. N<sub>2</sub>O emission rates can vary considerably due to the differences in the wastewater composition, the applied treatment process, the operating parameters and the environmental conditions. Figure 2 summarizes the most important parameters that affect the N<sub>2</sub>O emissions: these include the DO concentration which should not be low during nitrification and the nitrite levels in the mixed liquor which should not accumulate to high levels. In this sense the use of SBR technology, particularly when combined

with the treatment of highly nitrogenous effluents can enhance the nitrous oxide emissions due to the accumulation of nitrite in the reactor.



Figure 1: Simplified representation of the biochemical processes which are responsible for nitrous oxide production during nitrification and denitrification: A1 aerobic ammonium oxidation by AOB, A2 aerobic nitrite oxidation by nitrite oxidizing bacteria (NOB), B nitrifier denitrification by AOB and C heterotrophic denitrification [10].

The novel technologies that are introduced should not only remove/recover nutrients at a low cost, but should also be environmentally friendly. Nitritation/denitritation is increasingly being applied, particularly for the treatment of nitrogenous effluents, as it has lower energy requirements for aeration and organic carbon source requirements for denitritation than conventional nitrification/denitrification. However, such processes can result in significant N<sub>2</sub>O emissions [13]. The implementation of strategies to mitigate N<sub>2</sub>O emissions in the via nitrite processes can increase their sustainability. For example, the use of sludge fermentation liquid as carbon source in the via nitrite processes can mitigate N<sub>2</sub>O and NO emissions [14]. Recording and monitoring of the GHG compounds is required to abate their emissions through the implementation of the best available mitigation strategies.

The aim of this work was to determine the gaseous emissions (and particularly nitrous oxide) during the real time operation of a pilot scale nitritation/denitritation process implemented using a sequencing batch reactor (SBR). The SBR treated the anaerobic supernatant produced from the co-digestion of the organic fraction of municipal solid waste (OFMSW) and waste activated sludge (WAS).



Figure 2: Important parameters leading to N<sub>2</sub>O emissions [3]

# Material and methods SBR operation and experimental periods

The pilot scale SBR was installed at the integrated urban municipal waste and wastewater treatment plant (WWTP) of Treviso municipality (Veneto Region), designed to serve a population equivalent of 70,000. The SBR treated up to 6 m<sup>3</sup>/d of anaerobic supernatant which was produced from the co-digestion of WAS and the OFMSW. The anaerobic digestion took place in a mesophilic (37°C) anaerobic digester (reaction volume 2000 m<sup>3</sup>) and was fed with 120 m<sup>3</sup>/d  $\pm$  30% of thickened WAS and with 5.1 tonnes/d  $\pm$  95% of source separated OFMSW in order to obtain an organic loading rate (OLR) of 1.7 kgVS/m<sup>3</sup>·d. The hydraulic retention time (HRT) was 20 d. The anaerobic digestate was dewatered from 4-6% total solids (TS) to 20-25% TS. The liquid stream that was produced (i.e. anaerobic supernatant) was sent to a storage tank and from there it was fed to the pilot scale SBR. The reaction volume of the SBR was 2.8 m<sup>3</sup> [15]. As the anaerobic supernatant was characterized by a very low COD/N ratio an external organic carbon source was applied in order to supply sufficient carbon for denitritation. The external organic carbon was always added during the first 5 minutes of the anaeroic phase.

Agitation was achieved using a Rushton turbine, while three blowers were used for the reactor's aeration. The on/off operation of the blowers was automatically controlled on the basis of the DO and/or pH real-time measurements, properly processed by the programmable logic controller (PLC - Telemecanique, Schneider Electric, Germany). On line submerged probes were used to monitor the SBR operation. These consisted of the DO (LDO Hach Lange), the oxidation reduction potential (ORP, Chemitec and Hach Lange), pH (Hach Lange), conductivity (Hach Lange), NO<sub>x</sub>-N (Nitratax plus Hach Lange coupled with the sample filtration system Filtrax - Hach-Lange), NH4-N (NH4Dsc, Hach-Lange), mixed liquor suspended solids (MLSS, Solitax, Hach-Lange) and temperature. The heating system was set on when the mixed liquor temperature dropped below 15°C. The on line signals were processed by the PLC where the control algorithms were running. During the start up of the process, the via nitrite nitrogen removal

process was accomplished within 20 days. Complete inhibition of the NOB was obtained with nitrite accumulation being 100% [15].

Then two experimental periods were carried out: in period 1 a volumetric nitrogen loading rate (vNLR) of 0.81 kgN/m<sup>3</sup>d was applied and acetic acid was used as carbon source at a ratio of 2.1 kgCOD/kg(NO<sub>2</sub>-N)<sub>denitrified</sub>. The sequence of the SBR cycle was: filling, aerobic reaction, anoxic reaction, settling, decanting, idle time. In period 2, the vNLR increased to 1.08 kgN/m<sup>3</sup>d; the external organic source that was fed consisted of fermentation liquid (FL) produced from source separated organic fraction municipal solid waste (OFMSW) at a ratio of 3.1 kgCOD/kg(NO<sub>2</sub>-N)<sub>denitrified</sub>. Another difference was that the aerobic reaction took place after the anoxic reaction in the SBR cycle.

# **Analytical methods**

TS, total volatile solids (TVS), total and volatile suspended solids (TSS, VSS), MLSS, mixed liquor suspended solids (MLVSS), pH, conductivity, alkalinity, chemical oxygen demand (COD), soluble COD (sCOD), total Kjehldahl nitrogen (TKN), nitrate (NO<sub>3</sub>-N), nitrite (NO<sub>2</sub>-N), phosphorus (TP) and phosphate (PO<sub>4</sub>-P) were determined by Standards Methods [16] and ion chromatography for the anions (Dionex IC-90 with AG14 and AS14 columns)

## **Determination of gaseous emissions**

The determination of the gaseous emissions from the surface of the SBR tank was performed on line by the static chamber method and the Bruel and Kjaer photo-acoustic analyzer (Type 1302, Copenhagen, Denmark) Specifically, a 0.2 m high floating container (chamber) was placed on the surface of the tank and the time variation of the gas concentration inside the chamber was measured on line, in order to determine its emission rate (Figure 3). The gas concentration inside the chamber increased with time as a result of the gaseous emissions from the surface of the reactor [17, 18]. For each gas, a concentration vs. time plot was prepared and an interval of linear increase of the concentration was determined (Figure 4); the emission rate was calculated as follows [19]:

$$E_{\rm R} = \frac{C_{\rm i} - C_{\rm 0}}{t_{\rm i} - t_{\rm 0}} \frac{V_{\rm ch}}{A_{\rm ch}}$$

Where:

 $E_R$  (mg/m<sup>2</sup>h) is the emission rate of the gas

ti (h) and to (h) represent the time edges of the linear portion of the concentration plot

 $C_i$  (mg/m<sup>3</sup>) and  $C_0$  (mg/m<sup>3</sup>) represent the gas concentration at times  $t_i$  and  $t_0$  respectively

 $V_{ch}\xspace$  is the volume of the chamber,  $0.01116\ m^3$  and

Ach is the area of the emitting surface covered by the chamber, 0.07789 m<sup>2</sup>.

This way  $E_R$  was determined for NH<sub>3</sub>, N<sub>2</sub>O, CO<sub>2</sub>, CH<sub>4</sub> and methyl mercaptan (CH<sub>3</sub>SH) at various times of the SBR process, during aeration reaction, anoxic reaction and sedimentation. Therefore, a plot of  $E_R$  versus time was obtained for each gas for an SBR cycle. To determine the total amount of gas emitted during one cycle, the area below this graph was determined using the following formula:

$$G_{\rm M} = \sum (E_{\rm R} A_{\rm SBR} \Delta t)$$

Where:

 $G_M$  (mg/cycle) is the amount of the emitted gas per cycle A<sub>SBR</sub> (m<sup>2</sup>) is the surface area of the pilot SBR,  $1.5 \cdot 1.5 = 2.25 \text{ m}^2$  $\Delta t$  (h) is the time interval during which the gas emissions were recorded



Figure 3: (a) photo of the configuration of the SBR with the floating container for the on line gas measurement, (b) instrument for the on line measurement of gas emissions and (c) cross section and side view of the floating container



Figure 4: Increase of nitrous oxide concentration with time once the floating container is placed on the surface of the bioreactor (corresponding to one measurement in the SBR cycle)

### **Results and discussion**

#### **Operating periods**

The operating characteristics of the two experimental periods are summarized in Table 1 and the physicochemical characteristics of the anaerobic supernatant and the OFMSW FL are shown in Table 2. The vNLR that was applied in period 1 was close to its nitrogen removal capacity. The vNLR applied in period 2 was >30% higher than the system's nitrogen removal capacity.

Parameters	Units	Period 1	Period 2	
vNLR	kgN/m <sup>3</sup> ·d	$0.81\pm0.14$	$1.08\pm0.20$	
sNLR	kgN/kgMLVSS·d	$0.29\pm0.06$	$0.54\pm0.10$	
HRT	d	$0.75\pm0.01$	$0.52 \pm 0.15$	
OLR	kgCOD/m <sup>3</sup> ·d	1.68	2.13	
F/M	kgCOD/kgMLVSS·d	0.60	1.08	
MLSS	g/L	$3.62 \pm 1.26$	$2.65\pm0.47$	
MLVSS	g/L	$2.78 \pm 1.41$	$1.98\pm0.49$	
FA	mgNH <sub>3</sub> -N/L	$3.22 \pm 1.84$	$7.14\pm6.20$	
FNA	mgHNO <sub>2</sub> -N/L	< 0.02	< 0.02	
DO	mgO <sub>2</sub> /L	$1.48\pm0.07$	$0.95 \pm 0.12$	

Table 1: Operating conditions during the experimental period (mean values ± standard deviation)

HRT: hydraulic retention time, OLR: organic loading rate, F/M: food to microorganism ratio, FA: free ammonia, FNA: free nitrous acid

Table 2: Physicochemical characteristics of the anaerobic supernatant and the OFMSW FL for each experimental period (mean value  $\pm$  standard deviation)

Parameter	Units	Period 1	Period 2:	Period 2:
		Anaerobic supernatant	Anaerobic supernatant	OFMSW FL
Operation period	days	21-52	131-150	131-150
pH	-	$7.82 \pm 0.07$	$7.87\pm0.08$	$4.14 \pm 0.08$
TS	g/L	-	-	$25.37 \pm 4.39$
TVS	g/L	-	-	$22.19 \pm 2.43$
Conductivity	mS/cm	$5.78 \pm 0.31$	$4.44 \pm 0.56$	-
Alkalinity	mgCaCO <sub>3</sub> /L	$3346 \pm 375$	$2196 \pm 374$	-
TSS	mg/L	$145 \pm 9$	$33 \pm 1$	-
COD	mg/L	$134 \pm 11$	$31 \pm 5$	$62.22 \pm 5.37$
sCOD	mg/L	$97 \pm 4$	$26 \pm 5$	$31.33\pm3.09$
TKN	mg/L	$637 \pm 30$	$570 \pm 82$	$1128 \pm 319$
NH <sub>4</sub> -N	mg/L	$593\pm28$	$559\pm75$	$315 \pm 62$
NO <sub>3</sub> -N	mg/L	$0.4 \pm 0.1$	$0.8 \pm 0.1$	< 0.2
NO <sub>2</sub> -N	mg/L	$0.5 \pm 0.1$	$0.4 \pm 0.1$	< 0.2
TP	mg/L	$80 \pm 2$	$31 \pm 6$	$540 \pm 70$
PO <sub>4</sub> -P	mg/L	$72 \pm 18$	$30 \pm 1$	$119 \pm 42$

## Nitrous oxide emissions

In the nitritation/denitritation process the formation of nitrate is inhibited. However, the three biochemical pathways that contribute to N<sub>2</sub>O production can still occur since they are not associated with the activity of NOB. Furthermore, the implementation of an SBR is expected to lead to higher nitrous oxide emissions than an activated sludge process due to the fluctuation and higher nitrite concentrations in the mixed liquor that are observed during the reaction phases.

In period 1 the N<sub>2</sub>O emissions from the treatment of the anaerobically co-digested supernatant were much lower than those of period 2 (*i.e.* 0.24% compared to 1.38% as per cent of influent nitrogen load). In period 2, the DO concentration during the aerobic reaction phase was lower. The DO level is a critical parameter affecting the N<sub>2</sub>O emissions during nitritation. Low DO levels favour nitrous oxide emissions due to nitrifier denitrification [4]. Another parameter that is important is the nitrite level in the mixed liquor. In period 2 the higher vNLR and the sequence

change to anoxic/aerobic resulted in significant accumulation of nitrite which can also increase the emissions of  $N_2O$ .

The range of reported N<sub>2</sub>O emissions from full scale WWTP is very wide and ranges from 0-14.6% of the nitrogen load for full scale WWTPs and 0-95% for lab scale investigations [3]. The treatment of sludge reject water is expected to result in higher nitrous oxide emissions than those of municipal wastewater treatment due to the higher nitrogen loads that are applied and potentially high nitrite/nitrate concentrations in the mixed liquor. The OFMSW and WAS codigestion increases the nitrogen concentration in the resulting co-digestate therefore also increasing the potential N<sub>2</sub>O emissions. As a result, the nitrous oxide emissions are higher than those reported in several works concerning the operation of activated sludge processes with BNR for municipal wastewater treatment [20-22]. Table 3 compares the nitrous oxide emissions obtained in this work with previous works for the treatment of sludge reject water. In our work, the strategy of implementing higher DO and process optimization for effective removal of nitrogen can e decrease significantly the nitrous oxide emissions to low values. The use of OFMSW FL in combination with low DO and high vNLR could not mitigate the nitrous oxide emissions.

Wastewater	Process and conditions	N <sub>2</sub> O emissions (% of N load)	Sampling details	Reference
Anaerobic supernatant from WAS & OFMSW co-digestion	Nitritation/ denitritation High DO Low nitrite accumulation	0.24	On line Continuous 1 day	This study
Anaerobic supernatant from WAS & OFMSW co-digestion	Nitritation/ denitritation Low DO High nitrite accumulation	1.38	On line Continuous 1 day	This study
Sludge reject water	2 stage partial nitritation – anammox 1 stage partial	2.3	Off line Continuous 4 days Off line	[3]
Sludge reject water	nitritation – anammox	1.3	Grab samples 15 min/3×8 h	[23]
Sludge reject water	Nitritation	3.8	On line Continuous 12×6 h	[24]

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Table 3: N <sub>2</sub> O emissions	for the treatment	t of sludge reject wa	ater and anaerobic	c co-digestate



Figure 5: Variation of nitrous oxide emissions during the SBR operation for (a) period 1 and (b) period 2 respectively

Significant variation of the N<sub>2</sub>O emissions was observed within the SBR cycle (Figures 5a & 5b). The aerobic reaction phase contributed much more to the N<sub>2</sub>O emissions than the anoxic reaction phase. However, this does not mean that all the N<sub>2</sub>O emitted during the aerobic phase is actually produced during aerobic conditions. It is also produced during the anoxic phase and can be subsequently stripped during the aerobic phase. The solubility of nitrous oxide in water is relatively high, so it may take some time for its stripping. This is characteristically represented with the peak that is observed at the beginning of the aerobic phase in Figure 5b. In period 2, the aerobic phase takes place just after the anoxic one, so the sudden introduction of air bubbles stripes off the N<sub>2</sub>O that is produced during the aerobic zone it is very difficult to determine the individual contribution of nitritation and denitritation towards N<sub>2</sub>O production.

## Carbon dioxide, ammonia, methane and methyl mercaptan emissions

CO<sub>2</sub>, NH<sub>3</sub>, CH<sub>4</sub> and CH<sub>3</sub>SH emissions from the SBR were also determined as a per cent of the influent nitrogen and COD load (Table 4). The CO<sub>2</sub> emissions were much higher during period 1 since the use of acetic acid resulted in lower biomass yield compared to period 2 in which OFMSW FL was added. The ammonia emissions were very low despite the fact that the mixed liquor contained significant free ammonia concentration (>2 mgN/L and often >4 mgN/L). An interesting finding is the significant levels of methane that were detected during period 2. In this period, the relatively low DO levels in the mixed liquor could have created local micro-anaerobic conditions within the sludge flocs, resulting in the production of gaseous emissions that are produced under anaerobic conditions such as methane. Furthermore, in period 2 fermentation liquid derived from OFMSW was added as carbon source rather than acetic acid, increasing the potential for methane emissions under micro-anaerobic conditions.

Gaseous emissions	Gaseous emissions
in Period 1	in Period 2
0.239	1.379
0.0014	0.0173
0.551	5.343
55	31
0.076	0.895
	Gaseous emissions in Period 1 0.239 0.0014 0.551 55 0.076

Table 4: Gaseous emissions measured in the SBR as % of the influent load

### Mitigation strategy

This work showed that the nitrous oxide emissions can be decreased by (i) providing sufficient aeration during the nitritation stage so that the DO is maintained above 1.5 mg/L, (ii) applying a vNLR that is not higher than the system's nitrifying and denitrifying capacity. This way the accumulation of ammonium and nitrite is limited, (iii) apply the aerobic/anoxic sequence.

## Conclusions

The SBR operation at DO= 1.5 mg/L and vNLR=0.81 kgN/m<sup>3</sup>d resulted in much lower nitrous oxide emissions (0.24% of influent nitrogen load) compared to the operation at lower DO (0.95 mg/L) and higher vNLR=1.08 kgN/m<sup>3</sup>d. Furthermore, the aerobic/anoxic SBR sequence resulted in much lower peak values of emissions than the anoxic/aerobic sequence. In the latter case the introduction of air just after the anoxic phase resulted in the stripping of the gaseous compounds formed during the anoxic phase. Operation at low DO and the addition of OFMSW fermentation liquid favoured emissions associated with anaerobic processes as micro-anaerobic conditions may prevail in the interior of sludge flocs.

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