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DOI 10.1016/j.scitotenv.2020.142684

Publication date 2020 **Document Version** Final published version

Published in Science of the Total Environment

Citation (APA)

Ochs, P., Martin, B. D., Germain, E., Stephenson, T., van Loosdrecht, M., & Soares, A. (2020). Ammonia removal from thermal hydrolysis dewatering liquors via three different deammonification technologies. Science of the Total Environment, 755, Article 142684. https://doi.org/10.1016/j.scitotenv.2020.142684

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STOTEN-142684; No of Pages 10

ARTICLE IN PRESS

Science of the Total Environment xxx (xxxx) xxx



Contents lists available at ScienceDirect

Science of the Total Environment



journal homepage: www.elsevier.com/locate/scitotenv

Ammonia removal from thermal hydrolysis dewatering liquors via three different deammonification technologies

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HIGHLIGHTS

GRAPHICAL ABSTRACT

- THP/AD dewatering liquors had no impact on deammonification technologies.
- Instrumentation and control had major impact on deammonification performance.
- Free ammonia inhibition was mainly caused by extreme pH values >8.0.



ARTICLE INFO

Article history: Received 27 July 2020 Received in revised form 24 September 2020 Accepted 25 September 2020 Available online xxxx

Editor: Huu Hao Ngo

Keywords: Deammonification Thermal hydrolysis process, THP/AD Sequencing batch reactor Moving bed biofilm reactor Suspended sludge Granular sludge

ABSTRACT

The benefits of deammonification to remove nitrogen from sidestreams, i.e., sludge dewatering liquors, in municipal wastewater treatment plants are well accepted. The ammonia removal from dewatering liquors originated from thermal hydrolysis/anaerobic digestion (THP/AD) are deemed challenging. Many different commercial technologies have been applied to remove ammonia from sidestreams, varying in reactor design, biomass growth form and instrumentation and control strategy. Four technologies were tested (a deammonification suspended sludge sequencing batch reactor (S-SBR), a deammonification moving bed biofilm reactor (MEDIA), a deammonification granular sludge sequencing batch reactor (G-SBR), and a nitrification suspended sludge sequencing batch reactor (N-SBR)). All technologies relied on distinct control strategies that actuated on the feed flow leading to a range of different ammonia loading rates. Periods of poor performance were displayed by all technologies and related to imbalances in the chain of deammonification reactions subsequently effecting both load and removal. The S-SBR was most robust, not presenting these imbalances. The S-SBR and G-SBR presented the highest nitrogen removal rates (NRR) of 0.58 and 0.56 kg N m⁻³ d⁻¹, respectively. The MEDIA and the N-SBR presented an NRR of 0.17 and 0.07 kg N m⁻³ d⁻¹, respectively. This study demonstrated stable ammonia removal from THP/AD dewatering liquors and did not observe toxicity in the nitrogen removal technologies tested. It was identified that instrumentation and control strategy was the main contributor that enabled higher stability and NRR. Overall, this study provides support in selecting a suitable biological nitrogen removal technology for the treatment of sludge dewatering liquors from THP/AD.

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1. Introduction

* Corresponding author. *E-mail address:* a.soares@cranfield.ac.uk (A. Soares). Deammonification is considered an established nitrogen (N) removal technology for the treatment of sidestream dewatering liquors from

https://doi.org/10.1016/j.scitotenv.2020.142684

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Please cite this article as: P. Ochs, B.D. Martin, E. Germain, et al., Ammonia removal from thermal hydrolysis dewatering liquors via three different deammonification tech..., Science of the Total Environment, https://doi.org/10.1016/j.scitotenv.2020.142684

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conventional mesophilic anaerobic digestion (AD) (Lackner et al., 2014). Deammonification consists of two biological reactions: partial nitritation (PN) and anaerobic ammonia oxidization (anammox, A) (Strous et al., 1997). In partial nitritation, the ammonia oxidizing bacteria (AOB) convert ammonia and oxygen into nitrite (Ward, 2018). In the anammox reaction, the anaerobic ammonia oxidizing bacteria (AMX) convert ammonia and nitrite into nitrogen gas (Jetten et al., 1999; Strous et al., 1997). The challenge is to halt nitrification at the partial nitritation step and thus limit the second step, the nitratation (Christensson et al., 2013; Lackner et al., 2014; Vázquez-Padín et al., 2009; Wett, 2007). In the nitratation step, nitrite is converted with oxygen to nitrate by nitrite oxidizing bacteria (NOB) (Ward, 2018). Overgrowth of NOB has been frequently reported to lead to deammonification instability (Lackner et al., 2014).

Different deammonification technologies have been developed over the past decades (Lackner et al., 2014). The technologies differ in reactor design (e.g. sequencing batch reactor (SBR), continuous stirred tank reactor (CSTR) and plug flow reactor (PFR)), instrumentation and control strategy as well as biomass growth form (e.g. suspended sludge, attached biofilm and granular sludge) (Lackner et al., 2014). The major commercially available deammonification technologies are designed as one-stage SBR or CSTR with suspended sludge, biofilm or granular sludge biomass and vary in nitrogen loading rates (NLR) (Christensson et al., 2013; Driessen et al., 2020; Vázquez-Padín et al., 2009; Wett, 2007). The DEMON® process is a one-stage suspended sludge SBR or CSTR with NLR of 0.5 kg N m⁻³d⁻¹ (Wett, 2007). The AnitaTMMox process is a one-stage moving bed biofilm reactor (MBBR) or integrated fixed film activated sludge process (IFAS) with NLR of 0.5–1.2 kg N m $^{\rm 3-}$ d⁻¹ (Christensson et al., 2013; Lackner et al., 2014). The ELAN process has granular sludge biomass in SBR configuration and achieved NLR of $0.5-1.0 \text{ kg N m}^{-3} \text{d}^{-1}$ (Vázquez-Padín et al., 2009). The ANAMMOX® process is a granular sludge CSTR with NLR of 1.0–2.0 kg N $m^{-3}d^{-1}$ (Driessen et al., 2020). The Cleargreen[™] process is a suspended sludge SBR with NLR of 0.5 kg N m⁻³d⁻¹ (Lackner et al., 2014). Besides deammonification technologies, nitrification or nitrification/denitrification (N/DN) have been used for ammonia removal from sludge dewatering liquors in the past. However, the economic benefits of deammonification often outweigh conventional technologies having 60% less energy consumption from aeration, no chemical usage (e.g. alkalinity dosing for nitrification or carbon addition to denitrification) as well as 90% reduction in sludge production (Daigger, 2014; Fux and Siegrist, 2004; Wett, 2007). Yet, it is still unclear how the different nitrogen removal technologies compare side-by-side, with only limited studies available tested under field conditions. In a previous study, Leix et al. (2016) compared side-by-side a suspended sludge deammonification SBR, a deammonification MBBR and two suspended sludge partial nitritation SBRs treating dewatering liquors from AD. The deammonification SBR achieved the highest nitrogen removal rates (NRR) with 0.60 kg N m⁻³d⁻¹ compared to MBBR and partial nitritation SBR with 0.50 and 0.10 kg N m⁻³d⁻¹, respectively. It was identified that higher nitrite to ammonia ratios of >0.2 contributed to the higher NRR in the SBR (Leix et al., 2016). Another study compared a nitrification/denitrification (N/DN) SBR with a deammonification MBBR treating dewatering liquors from AD (Kanders et al., 2019). The two technologies were compared over separate years with the deammonification being an upgrade of the previous N/DN SBR (Kanders et al., 2019). The N/DN SBR achieved higher NRR of 0.17 kg N $m^{-3}d^{-1}$ compared to the deammonification MBBR with a NRR of 0.13 kg N m⁻³d⁻¹ (Kanders et al., 2019). In a deammonification technology survey, Lackner et al. (2014) investigated 14 different full-scale deammonification technologies, identifying the granular sludge technology as the one with the highest NRR of >1.5 kg N m⁻³d⁻¹. Furthermore, common process disturbances were identified as pH-shock, ammonia, nitrite and nitrate accumulation (Feng et al., 2017; Lackner et al., 2014).

Currently, the push for more energy neutral wastewater treatment plants (Ødegaard, 2016) results in an increasing number of wastewater

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treatment plants (WWTP) upgrading the AD process with pretreatment technologies (Barber, 2016). A common pre-treatment technology is the thermal hydrolysis process (THP) which uses steam at 160-180 °C to break down complex macromolecules and solubilize sludge (Barber, 2016; Carrère et al., 2010). Thermal hydrolysis changes the rheology of sludge, allowing greater loading rates to the AD which subsequently lead to increased biogas production (Barber, 2016; Carrère et al., 2010). On the other side, THP/AD increases the ammonia concentration in the dewatering liquors up to 2500 mg N L^{-1} (Winter et al., 2017). If these dewatering liquors are left untreated, they could decrease the capacity of the mainstream wastewater treatment process, making sidestream nitrogen removal technologies more critical. Past studies investigating the application of sidestream deammonification to treat THP/AD dewatering liquors reported inhibition by organic compounds (Figdore et al., 2012; Zhang et al., 2016). In a deammonification pilot scale study with a suspended sludge SBR, Figdore et al. (2012) reported a reduction in NRR to <0.5 kg N m⁻³d⁻¹ when shock-loading the biological reactor. Furthermore, the authors observed a reduction in the volumetric rate of the bacterial activity for AOB and AMX by 40-80%, thus a 1:1 dilution of the feed was proposed (Figdore et al., 2012). In another study two suspended sludge SBRs were compared treating THP/AD dewatering liquors and conventional AD dewatering liquors respectively (Zhang et al., 2016). The authors reported that performance deviation in the THP/AD SBR related to inhibition of AOB and AMX by colloidal and particulate COD (Zhang et al., 2016). Further investigation of upstream process optimisation units (e.g. dewatering, polymer dosing and AD) identified various operational parameters that could be optimised to reduce inhibition towards AOB and AMX (Zhang et al., 2018). Oppositely, Driessen et al. (2020) reported successful treatment of THP/AD dewatering liquors with a granular sludge CSTR achieving NRR on average 1.00 kg N $m^{-3}d^{-1}$ without inhibition by dewatering liquors from THP/AD.

The existing pilot plant comparisons are limited to the application for sludge dewatering liquors from conventional AD, making it unclear how technologies perform with THP/AD dewatering liquors. Limited number of studies discussed a side-by-side comparison of technologies, leaving controversy about which parameters (i.e. instrumentation and control, reactor design and biomass growth form) are most relevant to achieve suitable deammonification. Furthermore, different studies on the treatment of THP/AD dewatering liquors are contradictive on potential inhibition. This study aims to provide a comparison of three major deammonification technologies and one nitrification technology with different biomass growth forms (suspended sludge, granular sludge and biofilm), reactor designs, as well as instrumentation and control strategies. This study investigates robustness, effluent quality and the efficiency of four different biological nitrogen removal technologies (i.e. one nitrification based and three deammonification based) for the treatment of ammonia in dewatering liquors from THP/AD.

2. Materials and methods

2.1. Influent characteristics

The dewatering liquors in this study originated from post THP/AD dewatering at a UK WWTP with a population equivalent of 200,000 (Fig. 1). Sludge was pre-thickened and then dewatered in belt presses before being hydrolysed in the THP. The dewatering liquors from the pre-THP dewatering were returned untreated to the influent of the WWTP. The temperature of the THP was around 160 °C. The AD was fed with a 1:1 mixture of sludge from the THP and recirculated digestate, as reported by the site operators. Post-AD the sludge was dewatered in hydraulic filter presses with a cake dry solid content of 35–40%. The sludge dewatering liquors from the hydraulic filter presses were collected in a 500 m³ balancing (Balancing tank 1, Fig. 2) with a hydraulic retention time of 1 day before being distributed to the different sidestream technologies tested in this study.

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Fig. 1. Schematic of the sludge processing line of a UK wastewater treatment plant including sidestream treatment configuration.

2.2. Technologies tested

Four different biological nitrogen removal technologies were compared side-by-side for their ability to remove ammonia from THP/AD dewatering liquors described above. Three technologies were based on biological nitrogen removal via deammonification reactions and one technology was conventional nitrification. The technologies were operated following the manufacturer's specifications and set points (Table 1).

The suspended sludge SBR (S-SBR) (Fig. 2-A) had a reactor volume of 6 m⁻³ and an average NLR of 0.68 kgN m⁻³d⁻¹ (Table 1). The temperature was maintained at 24 °C using electric heaters. The control system of the S-SBR relied on online measurements for ammonia concentration, pH and dissolved oxygen which actuated on feed and aeration. The reactor was intermittently aerated at a dissolved oxygen (DO) set-point of 0.3 mg L⁻¹. The pH was maintained at 6.8 and the reactor ammonia was set to 150 mg N L⁻¹. The hydraulic retention time (HRT) was maintained at 87 h, in average. Prior to the start of the study the S-SBR was seeded with 3 m³ and a MLVSS concentration of 1300 mg L⁻¹ from a full-scale flocculant deammonification SBR in the UK. The S-SBR was in operation for 12 months prior to this study.

The moving bed biofilm reactor, named MEDIA (Fig. 2-B) had a reactor volume of 1.2 m³ and an average NLR of 0.33 kgN m⁻³d⁻¹ (Table 1). The MEDIA process was fed from a 0.5 m³ balancing tank. The reactor temperature was maintained at 29 °C, using an electric heater. The MEDIA process was controlled by measuring ammonia removal and nitrate production which actuated on aeration and feed flow. The HRT was maintained at 100 h, in average. The reactor was continuously aerated and had a DO set-point of 0.8 mg L⁻¹. The pH was maintained at 7.2 and the reactor ammonia concentration was set to 100 mg N L⁻¹. The MEDIA process was loaded with 650 L of pre-seeded plastic carriers, which reflects a fill ratio of 57%. The specific surface area of the plastic carrier used was 500 $m^2 m^{-3}$. Prior to this study the MEDIA was in stable operation for 12 months.

The SBR with a granular biomass, called G-SBR (Fig. 2-C), had a reactor volume of 0.2 m³ and an NLR of 0.72 kg N m⁻³d⁻¹ (Table 1). The reactor was fed from a 1 m³ balancing tank. The reactor temperature was maintained at 28 °C using an electric heater in the balancing tank. The G-SBR control system measured the conductivity change of the biological deammonification reactions across 1 cycle to actuate on the feed flowrate. The HRT was maintained at 62 h, in average. The reactor was continuously aerated, and the DO was set to 1.2 mg L⁻¹. The pH was maintained at 7.2. The G-SBR was seeded with 50 L of granular biomass with a MLVSS concentration of 1500 mg L⁻¹. Prior to this study the G-SBR was in stable operation for 12 months.

The conventional nitrification full-scale SBR with a flocculant (N-SBR) (Fig. 2-D) volume was 1489 m³ and the NLR was 0.29 kg N m⁻³d⁻¹ (Table 1). The reactor temperature was kept at 29 °C. The N-SBR measured pH and DO to actuate on aeration and feed. The HRT was maintained at 154 h, in average. The pH was maintained with 47% sodium hydroxide solution which was dosed at a fixed rate of 120 L d⁻¹. The pH was set to 7.0, the DO was kept at around 3.5 mg L⁻¹ and the reactor ammonia was 20 mg N L⁻¹. The reactor had a MLVSS concentration of 4300 mg L⁻¹ at the beginning of the study. The N-SBR had a solids retention time (SRT) of 4–6 days.

The dewatering liquor fed to the tested technologies contained ammonia, soluble COD and total suspended solids (TSS) average concentrations of 1301 mg N L⁻¹, 2453 mg L⁻¹ and 339 mg L⁻¹, respectively (Table 1). The average pH and alkalinity were 8.4 and 4750 mgCaCO₃ L⁻¹, respectively. Nitrite and nitrate concentration in the influent dewatering liquors were always below detection range (0.01 and 0.20 mg N L⁻¹). That meant that ammonia was the only contributor to the NLR.

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The four technologies were evaluated after start-up. All technologies relied on different control strategies actuating on the feed flow. This limited each technology to its reactor ammonia concentration leading to a range of ammonia loading rates for each technology.

The S-SBR and G-SBR were operated at NLRs of 0.68 and 0.72 kg N m⁻³d⁻¹, respectively (Table 1). The MEDIA and N-SBR were operated at NLRs of 0.33 and 0.29 kg N m⁻³d⁻¹. The G-SBR was reseeded on day 29 after part of the biomass was washed-out following a controller fault.

The NLR for each technology was compared using statistical analysis. The NLRs for the G-SBR and S-SBR were similar at 0.72 and 0.68 kg N m⁻³d⁻¹, respectively (Table 1). MEDIA and N-SBR operated at similar NLRs of 0.33 and 0.29 kg N m⁻³d⁻¹, respectively (Table 1).

2.3. Technology operation and evaluation

The deammonification technologies tested in this study were operated following their manufacturer's operational guidelines. Stable operation was defined as an operational period without any disruptions. Disruptions were linked to equipment failure or changes in reactor operation due to the instrumentation and control system, that resulted in imbalances of the biological deammonification reactions (Fig. 3). The imbalances were evaluated individually for root causes and mostly related to nitrate or nitrite accumulation. Imbalances that resulted in nitrate accumulation were defined as a nitrate production to ammonia removed ratio exceeding the ideal ratio of 0.08 of the deammonification stoichiometry (Lotti et al., 2014).

Imbalances that resulted in nitrite accumulation were defined as a nitrite to ammonia effluent ratio exceeding the ideal ratio of 0.53 for deammonification stoichiometry (Lotti et al., 2014).

After assessing all imbalances and their causes, the nitrogen removal rate (NRR), nitrogen removal and ammonia removal efficiencies (NRE and ARE, respectively) were analysed and compared for the different technologies.

2.4. Sample collection and analysis

Influent and effluent 24-hour composite samples were collected using Hach Lange Autosamplers, model AS900 (Hach Lange, Loveland, Colorado, USA). Ammonia, nitrite, nitrate, COD, soluble COD, alkalinity as CaCO₃, soluble phosphorus, TSS and VSS were analysed following standard methods (APHA, 2012). The reactor pH was measured using a Hach Lange pH probe, model HQ11D (Hach Lange, Loveland, Colorado, USA). Free ammonia (FA) and free nitrous acid were calculated following methods described in Anthonisen et al. (1976). Once per week, biomass samples were collected from each technology for ex-situ activity tests and solids analysis. MLSS and MLVSS concentrations were analysed following standard methods (APHA, 2012).

Ex-situ batch manometric anammox activity tests were performed with biomass from each deammonification technology once per week. Manometric activity measurements were performed in closed bottles using OxiTOP Control manometric sensors, model AN6 (WTW, Weilheim, Germany). The maximum anammox activity was measured and calculated following the methods described by Lotti et al. (2012).

t-Test, ANOVA test and Tukey's Honestly Significance Difference (HSD) were used for comparisons of groups with sample size >30. Non-parametric tests (Mann-Whitney tests) were used for comparisons of groups with a sample size <30. The statistical tests were selected due to their robustness. The data was analysed in Excel using a statistical analysis add-in (Zaiontz, 2020). Other statistical analysis was

considered but deemed as not appropriate due to sample size and distribution of this experiment.

3. Results and discussion

3.1. Influent characterisation

The influent characteristics for the THP/AD dewatering liquors used in this study are presented in Table 1. The ammonia and the soluble COD concentration were around 1300 mg N L⁻¹ and 2400 mg L⁻¹, respectively. The THP/AD dewatering liquors used in this study were not as high as those reported by other studies, where ammonia concentrations ranged from 1700 to 2500 mg N L⁻¹ (Figdore et al., 2012; Zhang et al., 2016). Other studies also reported ammonia concentrations of 1400 and 2000 mg N L⁻¹ (Driessen et al., 2020; Winter et al., 2017). The COD values reported in other studies were consistent between 2000 and 3000 mg L⁻¹ (Driessen et al., 2020; Zhang et al., 2016). The difference in the reported ammonia can be related to different THP operating temperatures, site specific AD loading rates, selection in dewatering process, and potential sludge blending prior to digestion (Barber, 2016; Winter et al., 2017).

3.2. Technology evaluation

All four biological nitrogen removal technologies achieved ammonia removal from THP/AD dewatering liquors. The NRR of the technologies varied between 0.10 and 0.60 kg N $m^{-3}d^{-1}$. However, all of them had periods with poor effluent quality that were caused by disruptions. The first part of the evaluation focused on assessing the stability of the biological reaction in each technology. Due to the different stoichiometry of the deammonification based technologies, they were compared separately from the nitrification N-SBR. The deammonification technolevaluated by imbalances ogies were promoting stable deammonification in the biological reactor. These imbalances were associated with the instrumentation and control strategies. The technologies were evaluated based on the criteria described in material and methods and considered a promotion of deammonification reactions. Imbalances were considered when the biological reactors exceeded stoichiometric values for nitrate production to ammonia (0.08) removed or nitrite to ammonia ratio (0.53). Deammonification technologies that exceeded these stoichiometric values were the G-SBR and the MEDIA (Fig. 4).

The G-SBR was operated for 30 days and had disruptions related to imbalances caused by nitrate accumulation (on seven occasions) and nitrite accumulation (on eight occasions) (Table 2). The reactor displayed events of ammonia and nitrite accumulation in the biological reactor. (e.g. on days 56, 78 and 81). The imbalances were mainly caused by the instrumentation and control strategy of the G-SBR. This was because it relied on conductivity measurements instead of ammonia. Unreliable ammonia readings in the biological reactor lead to overfeeding of the biological reactor and thus ammonia accumulation of >200 mg N L⁻¹ (e.g. on days 16, 28 and 78...). Additionally, a combined effect of high DO of 1.2 mg L^{-1} and ammonia concentrations >200 mg N L⁻¹ in the G-SBR caused accumulation of nitrite in the reactor of $>50 \text{ mg L}^{-1}$ (e.g. on days 30, 38 and 55...) (Table 2). Nitrite and ammonia accumulation in the biological reactor have been identified in previous studies as a cause of unreliable reactor performance (Feng et al., 2017; Lackner et al., 2014). Another effect related to the overfeeding was an increased pH in the biological reactor of 7.7 to 8.5 (e.g. on days 16, 28 and 54). A combined effect of high pH, ammonia and nitrite concentration led to inhibition by FA and FNA in the biological reactor

Fig. 2. Flowcharts of three deammonification technologies and one nitrification technology with (A) suspended SBR (S-SBR), (B) MBBR (MEDIA), (C) granular SBR (G-SBR) and (D) nitrification SBR (N-SBR).

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Table 1

Influent concentrations and technology characteristics of the four tested technologies.

	G-SBR	MEDIA	S-SBR	N-SBR
Nitrogen loading rate ^a , kg N m ⁻³ d ⁻¹	0.72 ± 0.46	0.33 ± 0.26	0.68 ± 0.27	0.29 ± 0.13
Number of operational days under steady state, d	30	49	44	59
Ammonia (NH ₄ -N), mg N L^{-1}	1299 ± 120	1304 ± 123	1301 ± 128	1300 ± 120
pH value	8.4 ± 0.1	8.4 ± 0.1	8.4 ± 0.1	8.4 ± 0.1
Total suspended solids (TSS), mg L^{-1}	332 ± 257	334 ± 258	355 ± 268	334 ± 254
Soluble COD (sCOD), mg L^{-1}	2424 ± 530	2471 ± 528	2486 ± 562	2432 ± 529
$sCOD/NH_4-N$ ratio, mg mg N^{-1}	1.8 ± 0.3	1.9 ± 0.3	1.9 ± 0.4	1.8 ± 0.3
Alkalinity (CaCO ₃), mg L^{-1}	4726 ± 455	4765 ± 453	4776 ± 484	4733 ± 453
Reactor temperature, °C	28.2 ± 3.5	29.7 ± 1.9	23.9 ± 2.1	29.1 ± 2.3
Soluble phosphorus, mg L^{-1}	14.9 ± 2.9	14.8 ± 3.0	14.7 ± 3.1	14.9 ± 2.9
Dissolved oxygen set-point (as given by the manufacturer), mg L^{-1}	1.2	0.8	0.3	3.5
pH set-point (as given by the manufacturer)	7.5	7.2	6.8	7.0
Reactor ammonia set-point (as given by the manufacturer), mg L^{-1}	150	100	150	20

^a NO₃-N and NO₂-N were measured in the influent and the concentrations was always below 0.20 and 0.01 mg L⁻¹, respectively. Hence ammonia was the only contributor to the influent nitrogen loading rate.

with 71.6 mg N L⁻¹ and 13 μ g N L⁻¹, respectively (Table 2). Free ammonia and FNA concentrations of 20–50 mg N L⁻¹ and 10–200 μ g N L⁻¹, respectively have been associated with inhibition of AMX (Fernández et al., 2012; Jin et al., 2012).

The MEDIA technology presented disruptions caused by imbalances in the deammonification reaction chain, including three occasions associated with nitrate accumulation, and three occasions with nitrite accumulation. The imbalances in the MEDIA reactor related mainly to unreliable ammonia readings by the instrumentation and control system of the biological reactor, which led to overfeeding. Most commercially available ammonia sensors (i.e. ion selective) have a maximum ammonia measurement range of 1000 mg N L⁻¹. High influent ammonia concentrations of 1300 mg N L⁻¹ (Table 1) led to unreliable readings, allowing a continuation of the feed flowrate and resulting in high reactor ammonia of 539.3 mg N L⁻¹ (e.g. on days 48, 66 and 63). The overfeeding caused extreme pH values in the reactor of 7.5–8.5. This led to an increase in aeration and extreme DO concentrations of >0.8 mg L⁻¹ in the MEDIA reactor (e.g. on days 38, 61 and 62) (Table 2). More available DO and ammonia led to greater nitrite production with nitrite concentrations in the reactor >50 mg N L⁻¹ (e.g. on days 45, 55 and 74). Extreme pH values in the MEDIA reactor as well as accumulation of nitrite and ammonia caused inhibition of FA and FNA in the biological reactor.

In the N-SBR, nitrite and nitrate conversion rates were used to evaluate based on nitrification stoichiometry (Ward, 2018), converting ammonia to first nitrite and then nitrate. The ideal nitrite to ammonia and



Fig. 3. Decision making flowchart to determine stable operations for the three deammonification technologies. ¹Excess dissolved oxygen concentration was when the oxygen concentration in the reactor exceeded the reactor set-points. (DO for G-SBR = 1.2 mg L⁻¹, MEDIA = 0.8 mg L⁻¹ and S-SBR = 0.3 mg L⁻¹). ²Ammonia and nitrite accumulation in the reactor were when the ammonia and nitrite concentration exceeded recommended concentrations of NH₄-N = 200 mg N L⁻¹ and No₂-N = 50 mg N L⁻¹. ³Temperature shock was when reactor temperature decreased below 25 °C within one day. ⁴High free ammonia (FA) or free nitrous acid (FNA) were defined as inhibition of AOB or AMX. FA and FNA inhibition ranged for AOB from 8 to 120 mg N L⁻¹ and 0.2 to 2.8 mg N L⁻¹, respectively. FA and FNA inhibition ranged for AMX from 20 to 50 mg N L⁻¹ and 0.01 to 0.2 mg N L⁻¹, respectively.

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Fig. 4. (A) Nitrate production (NO_3 -N/NH₄-N) and (B) nitrite accumulation (NO_2 -N/NH₄-N). Evaluation for deammonification technologies tested (S-SBR, MEDIA, G-SBR). Ideal stoichiometric ratios for deammonification were 0.08 and 0.53 respectively, displayed with a dashed line.

nitrate to ammonia ratio is 1.0 mg N mg N⁻¹ and 1.0 mg N mg N⁻¹, respectively (Ward, 2018). The nitrite to ammonia ratio of the N-SBR was average 12.9 and the nitrate production to ammonia removed ratio was 0.01 (Table 3). This indicated an imbalance in the second reaction of

Table 2

Types	and	numbers	of	operational	issues	that	resulted	in	imbalances	of	the
deammonification reactions for G-SBR, MEDIA and S-SBR.											

	G-SBR	MEDIA	S-SBR					
Nitrogen loading rate, kg N m $^{-3}$ d $^{-1}$	0.72	0.33	0.68					
	\pm 0.46	\pm 0.26	± 0.27					
Imbalances that resulted in nitrate accumulation, $NO_3/NH_4 > 0.08$								
Total number of samples	30	49	44					
Number of samples with $NO_3/NH_4 > 0.08$	7	3	0					
Dissolved oxygen ^a	1	3						
Nitrite or ammonia accumulation in reactor ^b	6							
Imbalances that resulted in nitrite accumulation, $NO_2/NH_4 > 0.53$								
Total number of samples	30	49	44					
Number of samples with NO ₂ /NH ₄	8	3	0					
Temperature shock ^c	2							
High free ammonia or free nitrous acid ^d	6	3						

^a Excess dissolved oxygen concentration was when the oxygen concentration in the reactor exceeded the reactor set-points. (DO for G-SBR = 1.2 mg L^{-1} , MEDIA = 0.8 mg L^{-1} and S-SBR = 0.3 mg L^{-1}).

^b Ammonia and nitrite accumulation in the reactor were when the ammonia and nitrite concentration exceeded recommended concentrations of NH_4 -N = 200 mg N L⁻¹ and NO_2 -N = 50 mg N L⁻¹.

 $^{\rm c}$ Temperature shock was defined as a short term (1–2 day) temperature drop to <25 °C.

^d High free ammonia (FA) or free nitrous acid (FNA) were defined as inhibition of AOB or AMX. FA and FNA inhibition ranged for AOB from 8 to 120 mg N L^{-1} and 0.2–-2.8 mg N L^{-1} , respectively. FA and FNA inhibition ranged for AMX from 20 to 50 mg N L^{-1} and 0.01–0.2 mg N L^{-1} , respectively.

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nitrification favouring the partial nitritation. The imbalance was related to a suppression of NOB caused by inhibitive concentration of FNA of 213.3 μ g N L⁻¹. Inhibition of NOB by FNA was reported to be 60–830 μ g N L⁻¹ (Anthonisen et al., 1976; Yao et al., 2017). The imbalance was not observed in operation prior to this study as per site operators and occurred after the pre-belt dewatering was diverted to the influent of the WWTP (Fig. 1). This caused an increase in the influent concentration. It was identified that the higher pH in THP/AD dewatering liquors of >8 (Winter et al., 2017) caused the pH to shift in the N-SBR leading to FNA inhibition and the undesired operation in partial nitritation mode. Since the whole operational period consisted of imbalanced operation the N-SBR was evaluated as a stable partial nitritation reactor.

The operational disruptions caused by imbalances in the deammonification chain of reactions for the MEDIA and G-SBR can be associated with their instrumentation and control systems. This led to frequent inhibition by FA and FNA due to unreliable sensor readings. The importance in biomass selection was highlighted in the laboratory scale comparison of Wells et al. (2017) identifying biofilm technologies such as the MEDIA as the more stable technology. However, previous studies in field conditions highlighted the relevance of robust instrumentation and control strategies for deammonification technologies. Joss et al. (2011) suggested the adoption of ammonia, nitrite and nitrate online sensors and their relevance as an early detection system for NOB growth. Ammonia, nitrite and nitrate online sensors provide important insights in the stoichiometry of the deammonification chain of reaction during operation. Other reports also highlighted pH-based control systems for greater deammonification stability (Graham and Jolis, 2017; Klaus et al., 2017). This study highlighted the need for robust instrumentation and control strategies to achieve deammonification.

3.3. Stable operation

The tested deammonification technologies achieved NRRs between 0.07 and 0.58 kg N $m^{-3}d^{-1}$ and the nitrification technology achieved 0.07 kg N $m^{-3}d^{-1}$ (Fig. 5-B). The G-SBR achieved an NRR of $0.56 \text{ kg N m}^{-3}\text{d}^{-1}$ (Fig. 5-B). Similar to this, G-SBR technologies treating conventional AD dewatering liquors reported in literature achieved NRRs of 0.55 kg N m⁻³d⁻¹ (Vázquez-Padín et al., 2014, 2009). The MEDIA achieved NRRs of 0.17 kg N m⁻³d⁻¹ (Fig. 5-B). MBBR technologies treating conventional AD dewatering liquors reported NRRs of $0.90 \text{ kg N m}^{-3}\text{d}^{-1}$ (Christensson et al., 2013; Veuillet et al., 2014). The lower performance of the MEDIA could be associated with the high reactor ammonia values of 513 mg N L⁻¹ and lower AMX activity of $0.66 \text{ g N gVSS}^{-1}\text{d}^{-1}$ (Table 3), where previous MBBR studies reported AMX activities of 1.5 g N gVSS⁻¹d⁻¹ (Kanders et al., 2014). The S-SBR achieved an NRR of 0.58 kg N m⁻³d⁻¹ (Fig. 5-B), which when compared to suspended sludge SBRs treating conventional AD dewatering liquors, achieved NRRs of 0.50 kg N m⁻³d⁻¹ (Lackner et al., 2014; Wett, 2007). The N-SBR achieved an NRR of 0.17 kg N $m^{-3}d^{-1}$ (Fig. 5-B). Suspended sludge nitrification technologies treating conventional AD dewatering liquors have been reported to achieve NRRs of 0.08 kg N $m^{-3}d^{-1}$ (Kanders et al., 2019). The N-SBR of this study displayed similar ammonia conversion to the SHARON process with ARE of 85-95% (Shalini and Joseph, 2012; Van Hulle et al., 2010). Overall, the biological sidestream technologies treating THP/AD dewatering tested in this study displayed similar nitrogen removal performances to these treating conventional AD dewatering liquors.

In order to evaluate the potential of the technologies, they were evaluated for stable periods with similar NLRs. The G-SBR and S-SBR had similar NLRs of 0.68 to 0.72 kg N m⁻³d⁻¹ (Table 1), and these also presented similar NRRs of 0.56 to 0.58 kg N m⁻³d⁻¹ (Table 3). Nevertheless, the NRE of the S-SBR was higher with 84% compared to the NRE of the G-SBR (70%) (Fig. 5-A), resulting in better ammonia effluent quality of 163 mg N L⁻¹ in the S-SBR. The nitrite to ammonia ratios in the biological reactors were 0.2 and 0.1 for the S-SBR and G-SBR, respectively (Table 3). Leix et al. (2016) identified a nitrite to ammonia ratio of

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Table 3

Effluent characteristics and performance for stable operation of the compared biological nitrogen removal technologies.

		G-SBR	MEDIA	S-SBR	N-SBR
Nitrogen removal rate (NRR), kg N	Mean	0.56	0.17	0.58	0.07
$m^{-3} d^{-1}$	Min	0.09	0.01	0.02	0.003
	Max	1.48	0.47	0.96	0.33
Ammonia removal efficiency	Mean	74	61	88	91
(ARR), %	Min	10	21	48	71
	Max	96	99	92	99
Nitrogen removal efficiency (NRE),	Mean	70	58	84	21
%	Min	11	26	48	5
	Max	94	96	91	86
Ammonia (NH ₄ -N), mg N L^{-1}	Mean	357	513	163	113
	Min	51	19	89	15
	Max	1187	1064	448	375
Nitrite (NO ₂ -N), mg N L^{-1}	Mean	15.8	34.6	23.8	856.9
	Min	0.01	0.01	0.01	
	Max	82.2	187.0	57.6	1218.0
Nitrate (NO ₃ -N), mg N L^{-1}	Mean	22.7	5.1	13.1	13.7
	Min	0.6	0.1	0.2	0.2
	Max	77.0	40.7	69.6	129.3
NO ₃ -N/NH ₄ -N ratio	Mean	0.02	0.01	0.01	
	Min	0.01	0.01	0.01	0.01
	Max	0.07	0.04	0.06	0.11
NO ₂ -N/NH ₄ -N ratio	Mean	0.1	0.1	0.2	12.9
	Min	0.01	0.01	0.01	0.01
	Max	0.4	0.4	0.4	79.2
pH reactor	Mean	7.8	7.4	6.9	7.5
	Min	7.3	6.4	6.8	7.0
	Max	8.9	8.5	7.3	8.0
Dissolved oxygen (DO), mg L^{-1}	Mean	0.6	0.8	0.2	3.5
	Min	0.2	0.1	0.1	2.0
	Max	1.3	3.0	0.3	4.2
Total suspended solids (TSS), mg	Mean	180	345	405	10,988
L^{-1}	Min	30	34	52	5360
1	Max	1360	3112	6164	18,640
Free ammonia (FA), mg N L^{-1}	Mean	119.4	20.8	0.8	2.9
	Min	1.6	0.2	0.4	0.5
	Max	634.3	115.7	3.7	9.6
Free nitrous acid (FNA), μ g N L $^{-1}$	Mean	2.0	13.0	28.0	213.3
	Min	0.01	0.01	0.01	0.01
	Max	9	59	67	551.80
Mixed liquor volatile suspended	Mean	1098	2754	3633	4295
solids (MLVSS), mg L^{-1}	Min	355	13	1890	2460
	Max	4680	9545	6230	6000
Maximum specific anammox	Mean	0.05	0.66	0.04	,
activity (MSAA), g N gVSS $^{-1}$ d $^{-1}$	Min	0.02	0.10	0.02	N/A
	Max	0.09	1.26	0.06	N/A

0.15 to 0.2 to contribute to greater nitrogen removal performance in a suspended SBR. No reports have been found comparing granular sludge SBR and suspended sludge SBR deammonification systems side by side. In a full-scale deammonification application survey Lackner et al. (2014) reviewed 14 different full-scale deammonification systems. The authors identified that granular sludge based deammonification systems operated at NLRs of 1.50 to 2.00 kg N m⁻³d⁻¹, while suspended sludge deammonification systems operated at NLRs of 0.30 to 0.60 kg N m⁻³d⁻¹ (Lackner et al., 2014). However, the NLR is highly dependent on the instrumentation and control strategy and comparisons for their performance are limited to similar NLRs, making the instrumentation and control strategy one of the key considerations for selecting a technology.

In the comparison between MEDIA and N-SBR (with NLRs of 0.33 and 0.29 kg N m⁻³d⁻¹, respectively) it was found that the NRR of the MEDIA was higher at 0.17 kg N m⁻³d⁻¹ compared to 0.07 kg N m⁻³d⁻¹ for the N-SBR (Fig. 5-D). When evaluating effluent quality, it was found that the N-SBR achieved ammonia concentrations of 113 mg N L⁻¹ and the MEDIA 513 mg N L⁻¹ (Table 3). The lower effluent quality in the MEDIA related to unreliable influent ammonia readings that caused overfeeding of the biological reactor. Limited studies are available comparing side-by-side conventional nitrogen removal technologies

(i.e. nitrification) with deammonification technologies. In a full-scale comparison study, Kanders et al. (2019) compared a suspended sludge SBR nitrification/denitrification (N/DN) to a deammonification MBBR with NLRs of 0.15 to 0.21 kg N m⁻³d⁻¹. The N/DN SBR achieved an NRR of 0.17 kg N m⁻³d⁻¹ and an effluent ammonia concentration of 60 mg N L⁻¹ while the MBBR achieved an NRR of 0.13 kg N m⁻³d⁻¹ and an ammonia concentration of 187 mg N L⁻¹ (Kanders et al., 2019). Previous comparisons vary largely in their applied NLR, which is due to their instrumentation and control strategies.

Overall, all four tested technologies were tested for their ability to remove ammonia from THP/AD dewatering liquors. The technologies performed at similar nitrogen removal performance compared to technologies treating conventional AD dewatering liquors. However, the S-SBR is the technology most suited for the application by achieving an NRR of 0.58 kg N m⁻³d⁻¹. This was related to the S-SBR instrumentation and control strategy using online ammonia and pH measurements in the biological reactor instead of the indirect measurements used in the other technologies. The key indicator to promote ammonia removal performance in the technologies was the instrumentation and control system since it was the main characteristic that influenced the nitrogen load.

3.4. Inhibition of thermal hydrolysis dewatering liquors

All four technologies achieved successful biological nitrogen removal with 0.10 to 0.60 kg N m⁻³d⁻¹ treating dewatering liquors originating from THP/AD. In previous operation with the three deammonification technologies, the nitrogen removal ranged from 0.20 to 0.55 kg N m⁻³d⁻¹, treating ammonia from mixed dewatering liquors (Ochs, 2020, p.X). Previous studies reported on inhibition related to compounds of the THP/AD dewatering liquors such as soluble, particulate or colloidal COD (Figdore et al., 2012; Zhang et al., 2016) were not



Fig. 5. Box plot comparison between G-SBR, MEDIA, S-SBR and N-SBR for (A) nitrogen removal efficiency and (B) nitrogen removal rate for stable operation. The averages for each technology are displayed by X.

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observed in this study. All technologies compared in this study were fed with THP/AD dewatering with soluble COD concentrations of average 2500 mg L⁻¹ (Table 1). In a study with a suspended sludge SBR, it was reported that soluble inert COD concentrations of 2000–2500 mg L⁻¹ led to inhibition of the biological rates of AOB and AMX (40 g NO₂N L⁻¹ h⁻¹ to <10 g NO₂N L⁻¹ h⁻¹) in ex-situ bacterial activity tests under shock-loading conditions. It was recommended that the suspended sludge SBR is operated using a 1:1 feed dilution, remarking that biology in the biological reactor should be given sufficient time to acclimatize to the THP/AD dewatering liquors (Figdore et al., 2012).

In another study, Zhang et al. (2016) operated two suspended deammonification SBRs side-by-side, with one being fed conventional AD dewatering liquors and the other one THP/AD dewatering liquors. It was identified that inhibition by particulate and colloidal COD occurred on days 62, 85 and 93 from the THP/AD dewatering liquors (Zhang et al., 2016). Furthermore, the authors reported that mainly the AOB rate was negatively (reduced by 20%) impacted by constituents in the THP/AD dewatering liquors (Zhang et al., 2016). Nevertheless, the authors reported the THP/AD dewatering liquors as inhibitive, correlating a combined effect of particulate and colloidal COD fraction to suppression of AOB activity (Zhang et al., 2016). Another study assessed different downstream process units improvements (e.g. dewatering process, polymer dosing, AD) and their impact on AOB and AMX activities (Zhang et al., 2018). The authors associated organic compounds in the dewatering liquors with the previously reported inhibition (Zhang et al., 2018). These could be overcome by process improvements to the dewatering process and polymer dosing for AOB inhibition and THP/AD operation for AMX (Zhang et al., 2018). On the contrary, Driessen et al. (2020) reported successful treatment of THP/AD dewatering liquors with a granular sludge continuous stirred tank reactor, achieving NRRs of 0.40 to 1.80 kg N $m^{-3}d^{-1}$. The results from the present study align with these, demonstrating the ability of deammonification technologies to remove ammonia from THP/AD dewatering liquors.

Inhibition from THP/AD dewatering liquors could be associated with shock-loading to the biological reactor, exceeding the design values as reported by Figdore et al. (2012). However, a robust instrumentation and control strategy such as the one of the S-SBR (ammonia and pH) would overcome such issues. Any disruptions displayed in this study could be associated with imbalances in the deammonification chain of reaction in G-SBR and MEDIA caused by the unreliable readings in the instrumentation and control strategy.

Overall, the deammonification technologies were most suited for sidestream ammonia removal due to the economic benefits (e.g. chemical dosing and energy saving for aeration). Based on the results of this study, the S-SBR was deemed as most suited for ammonia removal from THP/AD dewatering liquors due to its high NRR of $0.72 \text{ kg N m}^{-3} \text{d}^{-1}$ and robustness in promoting deammonification reactions.

4. Conclusions

Three different deammonification pilot plants and one nitrification technology were compared for their ability to remove ammonia from THP/AD dewatering liquors. All technologies relied on different control strategies actuating on the feed flow. This led to a range of ammonia loading rates to each technology that sometimes resulted in imbalances in the chain of deammonification reactions that led to poor effluent quality:

- The G-SBR had the highest number of operational disruptions of deammonification. These were predominantly caused by the conductivity-based control strategy resulting in regular overfeeding of the reactions, giving too high ammonia concentrations.
- The inhibition caused by FA and FNA in the G-SBR and MEDIA reactor was related to extreme pH values >8.0, indicating that pH control is one of the main contributors to stable operation in deammonification

technologies.

 A robust instrumentation and control strategy were shown to have a high impact on performance of the biological nitrogen removal technologies.

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Overall, the S-SBR achieved the highest NRR with an average value of 0.58 kg N m⁻³ d⁻¹, whilst being the most robust and not having any disruptions related to imbalances in the deammonification chain of reactions. Furthermore, inhibition caused by THP/AD dewatering liquors was not observed in any of the biological nitrogen removal technologies, contradicting previously published studies that reported a decrease in nitrogen removal performance.

CRediT authorship contribution statement

P.O. completed all the experiments and drafted the manuscript. B.M. advised on the manuscript data collection and provided minor comments on the manuscript. E.G. advised on the manuscript data collection, experiment design and provided minor comments to the manuscript. M.C.M.V.L. acted as advisor on the project and provided comments on the manuscript. T.S. acted as an advisor on the project and provided minor comments on the manuscript. A.S. is the principal investigator, having provided significant input on the data collection interpretation, writing of the manuscript and completed all the revisions.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The authors are grateful for the funding provided by Thames Water Utilities Ltd. and the Engineering and Physical Sciences Research Council (EPSRC) through their funding of the STREAM Industrial Doctoral Centre (IDC) EP/L015412/1. Data underlying this study can be accessed through the Cranfield University repository at https://doi.org/10. 17862/cranfield.rd.13063589.v1. We would like to express our thanks to the Laboratory of Thames Water Utilities Ltd. for analysing the composite samples. We would also like to thank Adrian Steele and Steve Perry for their support with the pilot plants.

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