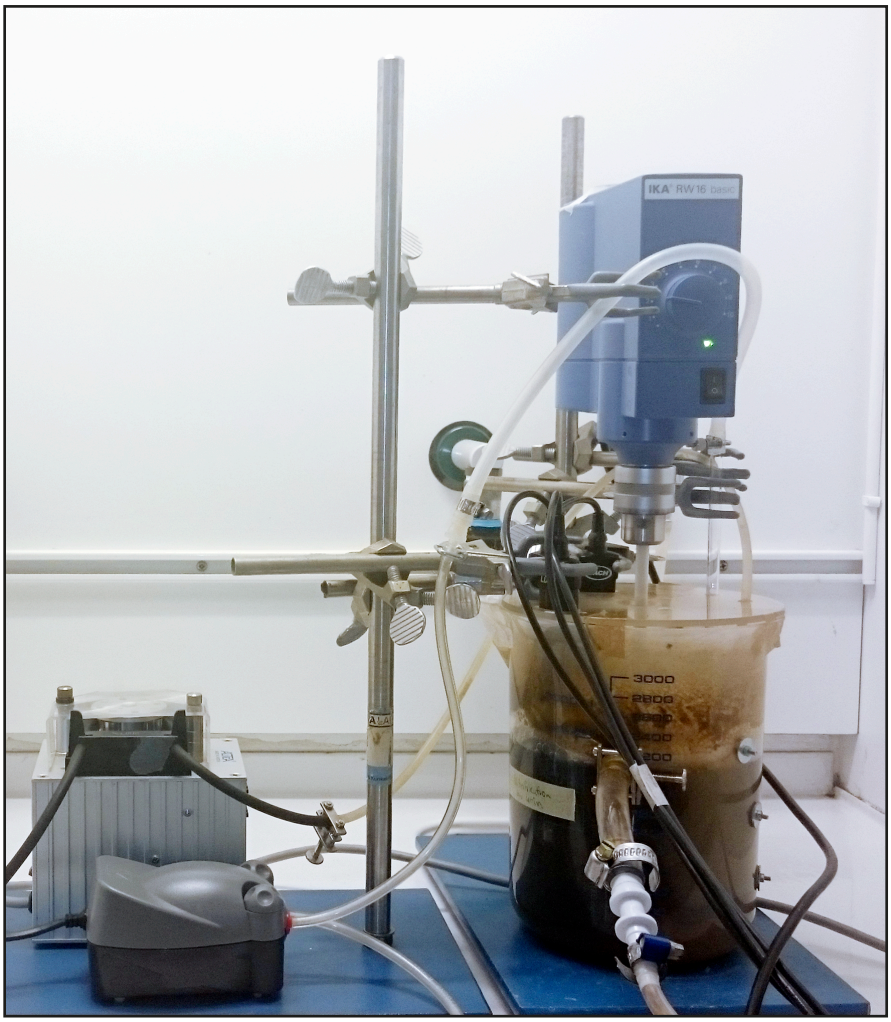
Stabilization of urine by nitrification in a Moving Bed Biofilm Reactor



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Water and Environmental Engineering
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by

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Picture on front page: Nitrification reactor. Photo by Marianne Olofsson.

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Preface

This Master's thesis has been performed in collaboration with Sweden Water Research in order to study the start-up process of a nitrification reactor for stabilization of source separated urine. The work has been performed at Water and Environmental Engineering at the Department of Chemical Engineering, Lund University, in the spring of 2016.

I want to thank the people working at the department for welcoming me and supporting me during this work. I want to say a special thanks to Gertrud Persson for your support during my work in the laboratory and to my supervisor Karin Jönsson for all your advice during this work.

I also want to say a special thanks to David Gustavsson, my supervisor at Sweden Water Research. Thank you for letting me do this work and for all your advice during this period. I have learnt so much from you.

Family and friends, thank you so much for always being there for me. I hope you will hear more about this subject in the future.

Lund, June 2016

Marianne Olofsson

Summary

Municipal wastewater contains a lot of compounds that could be seen as resources. Nitrogen, phosphorus and potassium are common ingredients in fertilizers and can all be found in wastewater. Nowadays only a small part of the nutrients found in wastewater are used on arable land. Urine contains the most of the nitrogen, phosphorus and potassium secreted by humans. Source separation of urine is a way to facilitate recovery of nutrients in wastewater and to lower the load on existing wastewater treatment plants.

Fresh urine contains urea which may undergo ureolysis and form ammonia during storage. In order to prevent ammonia volatilization during further treatment such as volume reduction, urine needs to be stabilized. One method for stabilization of urine is nitrification. Nitrification is a biological process performed in two steps. In the first step, ammonia is oxidized to nitrous acid by ammonia oxidizing bacteria. This oxidation lowers the pH, which prevents ammonia from vaporizing. The second step is performed by nitrite oxidizing bacteria which oxidize nitrite to nitrate. Since ammonia and not ammonium is the substrate for the ammonia oxidizing bacteria only a part of the ammonia will be oxidized as pH drops. Ammonia and nitrate are common compounds in fertilizers.

In 2025, the housing area Sege Park in the city of Malmö should function as an example for sustainable city development. One goal is that the area should accommodate at least one test facility for source separation of urine. Before the construction of the facility can begin VA SYD, the regional water and sewer organisation, wants to investigate different volume reduction methods. Because ammonia may vaporize during volume reduction, they also want to investigate different stabilization methods.

The purpose of this work was to study the start-up process of a nitrification reactor for stabilization of urine. A bench-scale moving bed biofilm reactor was run for 57 days. The highest nitrification rate measured was 0.97 g N m^{-2} d^{-1}. Nitrite accumulation, which is very problematic for a nitrification reactor, occurred two times. The first nitrite accumulation was handled by increased air flow and decreased load. To get rid of the second nitrite accumulation the solution in the reactor was diluted until the nitrite concentration was not measurable (< 0.6 mg L^{-1}). The dilution resulted in a second start-up of the reactor. The two nitrite accumulations occurred at inlet nitrogen concentrations of 1,220 mg N L^{-1} and 1,320 mg N L^{-1} respectively. The load was 2.10 g N m^{-2} d^{-1} before the first nitrite accumulation and 1.54 g N m^{-2} d^{-1} before the second nitrite accumulation. After the second start-up the reactor ran stably and the inlet nitrogen concentration was increased to a maximum of 1,450 mg N L^{-1}.

In order to find signs of process instabilities early and to prevent nitrite accumulations it is important to measure pH and dissolved oxygen in the reactor. Both parameters may indicate if something happens with the process. If any or both of the parameters are regulated, flow changes can probably be studied instead. It may also help to regularly measure the nitrite concentration in the reactor in order to be able to prevent nitrite accumulation from inhibiting the nitrification process.

Keywords: city development, moving bed biofilm reactor, MBBR, nitrification, nitrogen recovery, source separation, urine, urine stabilization

Sammanfattning

Kommunalt avloppsvatten innehåller kväve, fosfor, och kalium, ämnen som kan användas som gödningsmedel. Idag tas endast en liten andel av dessa ämnen tillvara. Av det mänskliga avfallet är det urinen som innehåller det mesta av kvävet, fosforn och kaliumet. Genom att källsortera urin kan dessa näringsämnen lättare tas om hand och återanvändas och belastningen på befintliga reningsverk kan minskas.

Färsk urin innehåller urea, en kväveförening som kan hydrolyseras till ammoniak om urinen lagras. Ammoniak är en flyktig gas och för att förhindra avdunstning, vid till exempel volymreducering, behöver urinen stabiliseras. En stabiliseringsmetod är nitrifikation. Nitrifikation är en biologisk process som sker i två steg. I det första steget oxideras ammoniak till salpetersyrlighet av ammoniakoxiderande bakterier och i det andra steget oxideras nitrit till nitrat av nitritoxiderande bakterier. Under ammoniakoxidationen sänks pH och urinen stabiliseras. Eftersom pH sjunker och ammoniak, inte ammonium, är substrat för de ammoniakoxiderande bakterierna oxideras endast en del av ammoniaken. Om nitrit ackumuleras kan bakterierna inhiberas, vilket är ett problem för den här typen av process.

Malmö stad har för avsikt att göra området Sege Park i Malmö till ett föregångsområde inom hållbar stadsutveckling till år 2025. Ett mål med projektet är att minst en byggnad ska ha ett system för källsortering av urin. Det kommunala vattentjänstföretaget VA SYD har fått ansvaret för anläggninen. Innan byggandet kan börja vill företaget skaffa sig mer kunskap om olika metoder för att volymreducera urin. Eftersom ammoniak kan avdunsta vill de också skaffa sig mer kunskap om olika metoder för att stabilisera urin.

Syftet med det här arbetet var att studera uppstarten av en nitrifikationsreaktor för stabilisering av urin. För att göra det studerades uppstarten av en reaktor med rörlig biofilmsbädd (MBBR) i labbskala i 57 dagar. Den högsta nitrifikationshalten som uppmättes var 0,97 g N m^{-2} d^{-1}. Nitritackumulering, vilket är problematiskt för nitrifikationsprocesser, skedde två gånger. Den första nitritackumuleringen avhjälptes genom att belastningen sänktes och luftflödet ökades. För att få bukt med den andra nitritackumuleringen späddes lösningen i reaktorn tills nitrithalten var under den mätbara nivån (< 0,6 mg L^{-1}). Utspädningen innebar att en ny uppstart fick genomföras. Nitritackumuleringarna skedde vid kvävekoncentrationer på 1220 mg N L^{-1} respektive 1320 mg N L^{-1} i inflödet. Belastningen var 2,10 g N m^{-2} d^{-1} före den första nitritackumuleringen och 1,54 g N m^{-2} d^{-1} före den andra nitritackumuleringen. Efter den andra uppstarten kunde reaktorn köras stabilt ända upp till en kvävekoncentration på 1450 mg N L^{-1} i inflödet.

Nitrifikationsprocessen var stabilare under den andra uppstarten än under den första. Detta berodde troligtvis på att bakterierna då fått mer tid på sig att vänja sig vid höga kvävekoncentrationer. Genom att kontinuerlig mäta och följa pH och syrehalt i reaktorn kan processavvikelser upptäckas och nitritackumuleringar förebyggas eller åtgärdas på ett tidigt stadium. Om pH och syrehalt istället regleras och därmed hålls stabila kan troligtvis variationer i luftflöde och inflöde av urin studeras istället. Att mäta nitrithalten i reaktorn kontinuerligt kan också vara bra för att kunna undvika nitritackumulering.

Nyckelord: hållbar stadsutveckling, kväveåtervinning, MBBR, nitrifikation, urin

List of abbreviations and designations

AOB	Ammonia Oxidizing Bacteria
COD	Chemical Oxygen Demand
D	Diameter
DO	Dissolved Oxygen
INXX	Inlet batch number XX
L	Length
MABR	Membrane Aerated Biofilm reactor
MBBR	Moving Bed Biofilm Reactor
NH4-N	Ammonia Nitrogen
Nit.	Nitrification
NO2-N	Nitrite Nitrogen
NO3-N	Nitrate Nitrogen
NOB	Nitrite Oxidizing Bacteria
N-tot	Total Nitrogen
OUTXX	Outlet batch number XX
P-tot	Total Phosphorus
TOC	Total Organic Compounds
WT	Wall Thickness
WWTP	Wastewater Treatment Plant

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1 Introduction

Nitrogen and phosphorus compounds are two of the main type of compounds treated in wastewater treatment plants (WWTPs) in Europe (European Environment Agency, 2015). The two type of compounds are common ingredients in fertilizers (Dittmar *et al.*, 2009) but the reuse from wastewater are not fully exploited (Statistiska centralbyrån, 2014). Most of the nitrogen, phosphorus and potassium secreted by humans is found in the urine. To implement source separation of urine can therefore be a way to both lower the load on existing WWTP and to recycle the nutrients (Rose *et al.*, 2015).

In 2025, the housing area Sege Park, in Malmö, shall be a predecessor when it comes to city development (Malmö Stad, 2015b). One of the goals for the area is that at least one building in the area shall accommodate a testbed for source separation of urine. VA SYD, the regional water and sewage organization, is responsible for development of the testbed (Malmö Stad, 2015a). VA SYD is one of three owners of the research and development company Sweden Water Research, which has a project called *SuNha* (Sustainable urban Nitrogen handling) *or later…*. The aim of the project is to study how improvements of the nitrogen cycle in the urban environment can be made.

In stored urine, urea is converted to ammonia (Udert *et al.*, 2003a). Ammonia can evaporate, and in order to prevent vaporization, and thereby loss of nitrogen, urine needs to be stabilized. One way to stabilize the urine is to use nitrification (Udert *et al.*, 2003b). Udert and Wächter (2012, p. 462) concluded that "biological nitrification with consecutive distillation of the effluent can be a stable and efficient process for the concentration and recovery of nutrients from urine". At Eawag (research institute), in Dübendorf, Switzerland, a nitrification and distillation pilot plant for nutrient recovery from urine has been run for more than three years. The greatest challenge with the plant was the start-up of the nitrification reactor (Fumasoli *et al.*, 2016).

1.1 Aim

The main aim of this project was to study the start-up of a nitrification process for treatment of urine. Another aim was to design a pilot plant reactor for nitrification of urine. A third aim was to contribute with knowledge about nitrification, which may be useful if nitrification is chosen as stabilization method in the test facility for source separation of urine in Malmö. Questions to be answered within the work were:

- What kind of problems can arise during the start-up?
- How can the problems be avoided and handled?
- How should the nitrification process preferably be controlled during the start-up?

1.2 Limitations

The process was controlled manually and the urine used in the experiment came from only one household. The time available for the experiment were nine weeks. During that time, two nitrite accumulations occurred and a second start-up of the reactor was necessary. Therefore, the concentration of urine was only increased to 14%. When the experimental period was over, the operation of the reactor was continued by Sweden Water Research.

2 Nutrient recovery from wastewater

In the European Union the requirements for urban wastewater treatment depend on the size of agglomerations and on the sensitiveness of discharge areas (European Environment Agency, 2015). The WWTPs that operate under the highest requirements remove suspended solids, organic matter, nitrogen and phosphorus. It is often necessary to have treatment for removal of both nitrogen and phosphorus compounds to prevent eutrophication (Conley *et al.*, 2009).

Nitrogen, phosphorus and potassium compounds are essential nutrients for plants and all three types of compounds are common ingredients in fertilizers. The most widely used nitrogen compound in fertilizers is urea, but ammonium nitrate, ammonia and ammonium phosphates are also common nitrogen compounds in fertilizers (International Fertilizer Industry Association, 2013). The nitrogen part in NPK (Nitrogen Phosphorus Potassium) fertilizers often consists of ammonium and nitrate in an approximately 1:1 ratio (Dittmar *et al.*, 2009).

2.1 Wastewater treatment of today

Most of the wastewater treated at municipal WWTPs in Sweden goes through three types of treatment; mechanical, biological and chemical (Statistiska centralbyrån, 2014; Svenskt Vatten, 2014). Mechanical treatment can consist of grids for separation of large matter, sand traps and primary sedimentation. The aim of the mechanical treatment is to remove large particles. Municipal wastewater contains biological substances which may be digested by microorganisms. This is used in biological treatment, where some of the nitrogen and approximately 90% of biological substances are removed. The chemical treatment is mainly used for phosphorus removal, which is made by adding chemicals that form precipitates with phosphorus compounds. Some WWTPs have additional treatment for enhanced nitrogen removal. To receive an even better effluent quality, some WWTPs also have a filtration step.

2.1.1 Removal of suspended solids and organic matter

The biological treatment is conventionally an activated sludge treatment process which removes suspended solids and organic matter. The activated sludge consists of bacteria and other microorganisms that digest organic matter. The activated sludge is active in aerated basins where it meets wastewater. After the treatment the activated sludge is separated from the water by sedimentation and then recirculated to the aerated basins. Since the bacteria are growing on matter in the wastewater the amount of sludge (biomass) is increasing. Most of the sludge is returned to the activated sludge basin, but an amount corresponding to the bacterial growth is taken out as waste sludge (von Sperling, 2007). Figure 2.1 shows a schematic picture of an activated sludge facility.

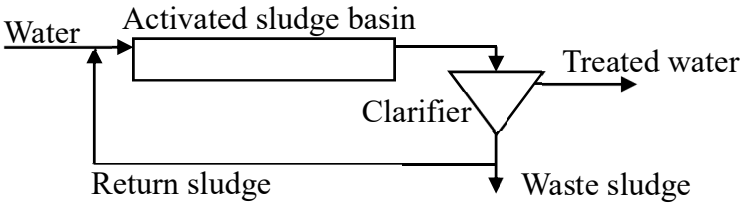

Figure 2.1. Activated sludge facility.

2.1.2 Removal of nitrogen and phosphorus compounds

Nitrogen compounds can either be removed in the activated sludge processes by assimilation or in enhanced nitrogen removal processes. One way to remove the nitrogen compounds in the enhanced nitrogen removal step is to use nitrification combined with denitrification (Koops *et al.*, 2006). In the nitrification step ammonia is first oxidized to nitrite and nitrite is then oxidized to nitrate. In the denitrification step nitrate is removed by anaerobic denitrifying bacteria. An upcoming way for nitrogen removal is to use an anaerobic ammonium oxidizing bacteria (anammox bacteria), which uses ammonium and nitrite to produce nitrogen gas and water (Kartal, Kuenen and van Loosdrecht, 2010). The process that uses that kind of bacteria does not require any addition of an organic compound.

Phosphorus removal can either be achieved through precipitation after addition of chemicals or by biological treatment (Yeoman *et al.*, 1988).

2.2 Potential resources in wastewater

Municipal wastewater contains nutrients that can be used as fertilizers (Statistiska centralbyrån, 2014). Table 2.1 shows values for the amount of nutrients in wastewater and sludge and the amount of fertilizers used in Sweden 2012. The values regarding wastewater only include values from WWTPs that treat large amounts of water, i.e. amounts that correspond to more than 2,000 personnel equivalents. The total amount of produced sludge in 2012, by WWTPs that treat amounts that correspond to more than 2,000 personnel equivalents, was 207,460 tonnes. Of the total amount of produced sludge 83% contained no hazardous substance in an amount that exceeded its threshold value, but only 23% of the sludge was spread on arable land (Statistiska centralbyrån, 2014).

Table 2.1. Amounts of nutrients in wastewater and sludge, and amount of fertilizers used in Sweden 2012. ([1]: Statistiska centralbyrån, 2014, [2]: Eurostat, 2015a, [3]: Eurostat, 2015b, [4]: Eurostat, 2015c, [5]: Eurostat, 2015d)

	Total nitrogen	**Ammonia nitrogen**	**Total phosphorus**	**Potassium**
Content in wastewater influent	41,967 tonnes [1]	-	5,307 tonnes [1]	-
Content in wastewater effluent	17,120 tonnes [1]	9,297 tonnes [1]	275 tonnes [1]	-
Content in sludge	43,020 mg/kg (avg.) [1]	-	26,400 mg/kg (avg.) [1]	-
Estimated consumption of manufactured fertilizer	164,000 tonnes [2]	-	12,000 tonnes [3]	23,000 tonnes [4]
Consumption of inorganic fertilizers	148,100 tonnes [5]	-	10,400 tonnes [5]	-

Urine contains most of the nitrogen, phosphorus and potassium secreted by humans and can be used as a fertilizer (Rose *et al.*, 2015; Andersson, 2015). Source separation of urine is a way to recover the nutrients and lower the loading on WWTP (Rose *et al.*, 2015).

2.3 Source separation of wastewater

Blackwater is water from toilets, i.e. urine, faeces, flush water and toilet paper. Greywater is water from other household sewage and may contain water used for dishwashing, washing, cooking and bathing. Yellow water is urine mixed with flushing water. If the different waters are separated instead of mixed it may be possible to treat them differently and perhaps more effective. There are already toilets for separation of urine on the market. They can have different looks but they all have the same function. von Münch and Winker have written a review ("Technology review of urine diversion components") which comprises descriptions of different toilets and information about how source separated urine should be handled (von Münch and Winker, 2011).

2.3.1 Source separation of urine

Source separation of urine has a lot of advantages. It is resource efficient, high nitrogen removal can be achieved and removal of pharmaceuticals may be easy to implement (Larsen *et al.*, 2009). Wilsenach and Loosdrecht (2006) simulated a scenario in which 50% of the urine, usually going to a WWTP, was collected and treated separately from the rest of the wastewater. They compared the process with urine separation with a process without urine separation and found that separate treatment of the urine can lead to savings in energy. The same result, that urine separation can lead to savings in energy compared to enhanced treatment, was found by Spångberg, Tidåker and Jönsson (2014).

2.3.2 Composition of urine

A human secretes around 1.4 L urine per day (Rose *et al.*, 2015). The value depends on body size, sweating and fluid consumption. Children tend to secrete less urine than adults. The diet, especially the intake of protein, has a large impact on the concentration of nitrogen in the urine. The concentration of urea in fresh urine varies in the interval 9.3 g L^{-1} to 23.3 g L^{-1} and makes up the largest fraction of the compounds containing nitrogen in fresh urine (Rose *et al.*, 2015). The pH value in fresh urine is normally around 5.5 to 7.0, but can also vary with the diet (Rose *et al.*, 2015). Table 2.2 shows concentrations of different compounds in urine.

Table 2.2. Concentration of compounds in fresh and stored urine. The values after ± signs are standard deviations. [a]The ammonia concentration was low due to ammonia volatilization (Udert and Wächter, 2012).

	Fresh urine		Stored urine	
Reference	Rose *et al.*, 2015	Liu *et al.*, 2016	Fumasoli *et al.*, 2016	Udert and Wächter, 2012
Additional info	Data from different studies	Urine from laboratory members	Urine from men, only	Urine from men, only
pH	5.5–7.0	6.0 ± 0.3	9.0 ± 0.1	8.69 ± 0.11
Ammonia-N (mg L^{-1})	125–600	1,125 ± 147	4,140 ± 870	2,390 ± 250[a]
Urea (mg L^{-1})	9,300–23,300			
N-tot (mg N L^{-1})	4,000–13,900	7,523 ± 1,097		
P-tot (mg P L^{-1})	350–2,500	448 ± 56		
Total phosphate-P (mg L^{-1})	205–760		242 ± 23	208 ± 49
TOC (mg L^{-1})		5,298 ± 792		
COD (mg O_2 L^{-1})	6,270–17,500			
Dissolved COD (mg O_2 L^{-1})			3,860 ± 870	4,500 ± 910
Conductivity (mS cm^{-1})	160–270	14.95 ± 1.87		
Calcium (mg L^{-1})	32–230			16 ± 3
Magnesium (mg L^{-1})	70–120			< 5
Potassium (mg L^{-1})	750–2,610		1,470 ± 130	1,410 ± 320

2.3.3 Pathogens, pharmaceuticals and heavy metals

Source separated urine that has been contaminated may constitute a health risk if reused as a fertilizer (Schönning, Leeming and Stenström, 2002). Karak and Bhattacharyya (2011) addressed the need of more research regarding the use of human urine as a fertilizer and the possible risks of spreading pharmaceuticals, pathogens and heavy metals. Pronk *et al.*, (2007) showed that a combination of electrodialysis and ozonation can lower the oestrogenic activity

in urine. Nitrification alone can decrease the amount of active bacterial pathogens, but it is probably not enough to inactivate all types of viruses in urine (Bischel *et al.*, 2015).

2.3.4 Ureolysis, precipitation and ammonia volatilization

The enzyme urease converts urea and water to ammonia and carbamate (Equation 1). The carbamate molecule then undergoes spontaneously hydroxylation to ammonia and carbonic acid (Equation 2). The formation of ammonia causes an increase in pH (Equation 3) (Mobley and Hausinger, 1989).

$$CO(NH_2)_2 + H_2O \rightarrow NH_3 + H_2NCOOH \quad (1)$$

$$H_2NCOOH + H_2O \rightarrow NH_3 + H_2CO_3 \quad (2)$$

$$NH_3 + H_2O \rightarrow NH_4^+ + OH^- \quad (3)$$

Udert *et al.* (2003a) investigated urease activity in a urine collecting system and found that ureolysis was caused by both urease active bacteria and dissolved urease. They also concluded that the urease activity was independent of the pH in the system (Udert *et al.*, 2003a).

Ureolysis due to urease activity makes calcium phosphate and magnesium phosphate form precipitates (Udert *et al.*, 2003a). The precipitation of calcium phosphate and magnesium phosphate depend on pH (Recillas *et al.*, 2012; Jaffer *et al.*, 2002). The magnesium phosphate precipitate is in the form of struvite and the struvite precipitation starts at pH 7.2. The calcium phosphate precipitation does not have a similar distinct starting point. Precipitation is a serious issue since it can cause blockage in urine collecting systems (Udert *et al.*, 2003a). Boyer *et al.* (2013) showed that one way to control precipitation in urine is to use cation exchange resin. They concluded that the use of a cation exchange resin can increase the amount of phosphate that can be recovered (Boyer *et al.*, 2013). The decomposition rate of urea in a storage container can be followed by measurements of pH (Hellström, Johansson and Grennberg, 1999).

Urine contains urea which may undergo ureolysis and form ammonia. Ammonia can both evaporate and form ammonium. The formation of ammonium causes an increase in pH (Mobley and Hausinger, 1989). Ammonia volatilization implies that less nitrogen can be recovered and that an odour may be spread. Because of the spontaneous vaporization urine needs to be used directly or stabilized in order to make recovery of nitrogen possible.

2.3.5 Experiences and challenges with source separation of urine

In the 1990s some implementations of source separation of urine were conducted in Sweden (Mels, van Betuw and Braadbaart, 2007). The benefits of the source separating systems have been shown to be reduction of emissions and recovery of resources. The main problem regarding functionality has been pipe clogging due to precipitation (Mels, van Betuw and Braadbaart, 2007). Another common problem with systems for source separation of urine was odour (Jönsson and Vinnerås, 2007). Jönsson and Vinnerås (2007) stated that it is these two problems, precipitation in pipes and odour, that constitutes the main challenges when designing systems for source separation of urine.

3 Methods for recovery of source separated urine

In today's society urine is often seen as a waste product that requires a lot of energy to handle, but in fact urine contains a lot of substances and could be seen as a resource. Many ways of recovering nutrients from urine have been investigated; microalgae growth (Zhang, S. *et al.*, 2014), precipitation by adding magnesite and zeolite to hydrolysed urine (Xu *et al.*, 2015), precipitation by adding magnesium chloride (Ronteltap *et al.*, 2010), electrolysis with graphite electrodes (Zöllig *et al.*, 2015), forward osmosis dewatering (Zhang, J. *et al.*, 2014) and forward osmosis combined with membrane distillation (Liu *et al.*, 2016). Methods for direct use as fertilizers have also been investigated; sorption of urea from urine on biochars produced of corn cobs (Zhang, Li and Mahmood, 2015) and adsorption of ammonia on zeolite clinoptilolite (Beler-Baykal, Allar and Bayram, 2011). Urine can also be used directly on arable land (Andersson, 2015).

The treatment methods mentioned above are just some examples of treatment methods. Maurer, Pronk and Larsen, (2006) presented seven different purposes to treat source separated urine and related methods that can be used for each purpose. They concluded that no process alone can achieve all seven purposes, which were hygienization, volume reduction, stabilization, phosphorus recovery, nitrogen recovery, nutrient removal and removal of micropollutants (Maurer, Pronk and Larsen, 2006).

In order to reduce the energy required for transportation, volume reduction has to be performed. Udert and Wächter (2012) compared results from different studies evaluating different methods for water removal from urine and found that a combination of reverse osmosis and distillation would be a relatively low energy consuming method, especially if energy recovering is used. Another kind of low energy requiring process has been investigated in pilot scale in Vietnam. The process used solar thermal evaporation of urine to treat the urine and produce a fertilizer (Antonini *et al.*, 2012).

3.1 Stabilization of urine

Several methods for stabilization of urine have been investigated. Chemical oxidation by hydrogen peroxide or ozone can prevent urea hydrolysis and thereby ammonia production. When no or very little ammonia is produced the urine will be stable (Zhang, Y. *et al.*, 2013). Another method for prevention of urea hydrolysis is addition of acid (Hellström, Johansson and Grennberg, 1999). If the ammonia already is produced, graphite electrodes can be used to oxidize it electrochemically (Zöllig *et al.*, 2015). Different biological methods have also been investigated; nitritation combined with autotrophic denitrification, which gives nitrogen gas, (Udert *et al.*, 2003b) and nitrification (Udert and Wächter, 2012; Udert *et al.*, 2003b).

4 Nitrification

Nitrification is a biological process in which bacteria converts ammonium to nitrate. The oxidation is performed by two types of bacteria, ammonia oxidizing bacteria (AOB) and nitrite oxidizing bacteria (NOB) (Koops *et al.*, 2006). AOB oxidize ammonia to hydroxylamine (Equation 4) and hydroxylamine to nitrous acid (Equation 5). Equation 6 shows the overall reaction of both oxidation steps. The formation of nitrous acid results in a decrease in pH. The produced nitrous acid is in equilibrium with nitrite. NOB oxidize nitrite to nitrate (Equation 7). AOB use oxygen and water for their oxidation reactions and NOB use water for their oxidation reaction. Nevertheless, NOB need oxygen in the overall oxidation process (Bock and Wagner, 2013). The nitrification rate increases with pH (Villaverde, García-Encina and Fdz-Polanco, 1997; Udert and Wächter, 2012).

$$NH_3 + O_2 + 2\ H^+ + 2\ e^- \rightarrow NH_2OH + H_2O \quad (4)$$

$$NH_2OH + 1.5\ O_2 \rightarrow HNO_2 + 2\ e^- + 2\ H^+ \quad (5)$$

$$NH_3 + 1.5\ O_2 \rightarrow HNO_2 + H_2O \qquad \Delta G_0 = -235\ kJ\ mol^{-1} \quad (6)$$

$$NO_2^- + 0.5\ O_2 \rightarrow NO_3^- \qquad \Delta G_0 = -54\ kJ\ mol^{-1} \quad (7)$$

4.1 Nitrifying bacteria

The substrate for AOB is free ammonia (Koops *et al.*, 2006) and the substrate for NOB is nitrite (Bock and Wagner, 2013). Free ammonia and nitrous acid can inhibit both types of bacteria. (Anthonisen *et al.*, 1976; Vadivelu, Keller and Yuan, 2007). Temperature and pH determine the relationship between ammonium and ammonia (Equation 8) and between nitrite and nitrous acid (Equation 9).

$$NH_4^+ + OH^- \rightleftharpoons NH_3 + H_2O \quad (8)$$

$$H^+ + NO_2^- \rightleftharpoons HNO_2 \quad (9)$$

Both AOB and NOB have an inefficient cell growth and are therefore slow-growing bacteria. The inefficient cell growth is due to the many oxidization reactions the bacteria need to perform in order to generate enough energy to assimilate carbon dioxide. The two types of bacteria can live in environments like soil, water, rocks and wastewater. The optimal pH for nitrifying bacteria is 7.6–7.8, but they can survive in places where the pH is either lower or higher than that. (Bock and Wagner, 2013). Neither AOB nor NOB can perform their oxidation processes at high salt concentrations. No AOB has been found growing at salt concentrations above 150 g L^{-1} and no NOB has been found growing at salt concentrations above 50 g L^{-1} (Oren, 2011).

When AOB and NOB living in biofilms are studied, active AOB are mainly found in the outer parts of the biofilms and active NOB are mainly found further into the biofilm but close to the AOB population (Schramm *et al.*, 1996; Okabe, Satoh and Watanabe, 1999; Okabe *et al.*, 2002; Kouba *et al.*, 2014).

4.1.1 Ammonia oxidizing bacteria

The AOB is divided into five different genera; *Nitrosomonas*, *Nitrosococcus*, *Nitrosospira*, *Nitrosolobus* and *Nitrosovibrio*. The most common genera of AOB in many WWTP are

Nitrosomonas (Koops *et al.*, 2006). The AOB *Nitrosomonas* is a chemolithotrophic proteobacteria (Kersters *et al.*, 2006). In a pilot-scale reactor for nitrification of urine the AOB was shown to belong to the *Nitrosomonas europea* lineage (Fumasoli *et al.*, 2016).

AOB can produce N_2O. To what extent the production occurs depend on the concentration of dissolved oxygen (DO) concentration and on the nitrite concentration (Kampschreur *et al.*, 2009). In order to minimize N_2O production from nitrifiers the DO concentration should be kept high (Peng *et al.*, 2015).

4.1.2 Nitrite oxidizing bacteria

The NOB is divided into four different genera; *Nitrobacter*, *Nitrococcus*, *Nitrospina* and *Nitrospira* (Bock and Wagner, 2013). The most common NOB in WWTPs are *Nitrospira* and *Nitrobacter*. *Nitrospira* constitutes the main part of the NOB in WWTPs (Daims, 2014). *Nitrospira* and *Nitrobacter* favours different nitrite concentration. At low nitrite concentration *Nitrospira* constitutes the main part of the NOB population and at high nitrite concentration *Nitrobacter* is the most common NOB (Kim and Kim, 2006, Nogueira and Melo, 2006). *Nitrobacter* grow faster but have a lower affinity for oxygen than *Nitrospira* (Nowka, Dalms and Spleck, 2015). *Nitrospira* are chemolithoautotrophic aerobic bacteria (Daims, 2014). *Nitrobacter* are lithoautotrophic or chemoorganotrophic aerobic bacteria. At the right conditions, NOB can survive in anaerobic environments (de Souza *et al.*, 2014).

4.1.3 Conditions for nitrification

When nitrification should be performed, optimal levels for both AOB and NOB are desirable. A lot of research has been performed in order to find the optimal run conditions for partial nitrification, i.e. nitritation (Zhu *et al.*, 2008). In partial nitrification only the oxidation of ammonia to nitrite is performed and the NOB are inhibited. When performing nitrification, these conditions inhibiting the NOB should be avoided. The inhibition can be performed by adjusting parameters such as temperature and DO concentration to favour AOB and disfavour NOB (Ruiz, Jeison and Chamy, 2003; Blackburne, Yuan and Keller, 2008).

Nitrobacter has lower affinity for oxygen than *Nitrosomonas* (Blackburne, Yuan and Keller, 2008). This means that *Nitrobacter* can be outcompeted at low DO concentrations. In a study on partial nitrification in activated sludge nitrite accumulations occurred at DO concentrations below 1.7 mg L^{-1} (pH was 7.85) (Ruiz, Jeison and Chamy, 2003). In the same study, it was shown that complete nitrification could be performed between pH values between 6.45 and 8.95 when DO concentration was 5.5 mg L^{-1}.

The bacterial activity varies with temperature. At temperatures above 20 ˚C AOB grow faster than NOB and at temperatures below 15 ˚C the reverse is true (Zhu *et al.*, 2008).

4.2 Nitrification for stabilization of urine

Nitrification can be used to stabilize source separated urine and experiments with different types of reactors have been performed (Udert *et al.*, 2003b; Udert and Wächter, 2012; Fumasoli *et al.*, 2016). Experimental data and results from four different experiments can be seen in Table 4.1. Since nitrous acid can inhibit both AOB and NOB (Anthonisen *et al.*, 1976), nitrite accumulation is a problem if it occurs during nitrification. Nitrite accumulation may occur if AOB activity increases more rapidly than NOB activity. If this problem arises it may be possible to regain stability by switching of air supply and feed flow and by adding acetate to remove nitrite. If the pH value is too high, acid also has to be added (Udert and Wächter, 2012).

Table 4.1. Experimental data and results from nitrification experiments. The values after ± signs are standard deviations.

Reference	Fumasoli *et al.*, 2016 (Pilot scale)	Fumasoli *et al.*, 2016 (Bench scale)	Udert and Wächter, 2012 (Bench scale)	Udert *et al.*, 2003b (Bench scale)
Reactor volume	120 L	7 L	2.6 L	2.8 L
Reactor type	MBBR	Suspended biomass	MABR	MBBR
Carrier	Kaldnes® K1 biofilm carriers	-	Silicon tubing L = 21.7 m D = 4 mm WT = 0.5 mm	Kaldnes® biofilm carriers L = 8 mm D = 10 mm
Filling degree	60% of total reactor volume	0.7 L activated sludge		50% of total reactor volume
Specific surface	500 m^2 m^{-3}		149 m^2 m^{-3}	460 m^2 m^{-3}
Aeration rate	2 m^3 h^{-1} (humidified air)	1 L min^{-1}	450 mL min^{-1}	
Oxygen concentration in reactor	>7 mg L^{-1}	>7 mg L^{-1}	3.0–5.5 mg L^{-1} (avg.)	3–5.2 mg L^{-1}
Stirring	Aeration	500 rpm Magnetically	-	-
Temperature	27.0 ºC At max nit. rate	25 ºC	23 ± 2 ºC	25.3 ± 0.5 ºC
Maximum nit. rate	3.1 g N m^{-2} d^{-1}	-	1.8 ± 0.3 g N m^{-2} d^{-1}	1.7 g N m^{-2} d^{-1}
Influent ammonia concentration at max nit. rate	-	-	2,390 ± 250 mg N L^{-1}	~2,000–7,100 mg N L^{-1}
Final ammonia load	-	-	-	750 ± 50 mg N m^{-3} d^{-1}

In a reactor for nitrification of urine, the nitrification rate increases with pH and can be controlled by dosing urine. At high nitrification rates, which occur at high pH values, a significant nitrogen loss can take place in an MABR (Udert and Wächter, 2012). Udert and

Wächter (2012) stated that nitrogen loss in an MABR can be a disadvantage of the reactor setup and suggested that heterotrophic denitrification can cause the nitrogen loss. They gave two explanations for the denitrification, high COD to ammonia ratio and accumulation of biomass. They compared an experiment performed in an MABR with an earlier experiment performed in an MBBR (Udert *et al.*, 2003b) and concluded that the MBBR probably is a better choice for nitrification. The possible strong mixing in the MBBR prevents the biofilm from becoming thick (Udert and Wächter, 2012). Degradation of TOC and COD are independent of pH during nitrification (Udert and Wächter, 2012).

The degree to which nitrification occurs depends on pH and the concentration of DO (Feng, Wu and Xu, 2008). If no correction of pH takes place the resulting ratio between nitrate and ammonia becomes 1:1 (Udert *et al.*, 2003b). This ratio is the same as a common ratio of the same compounds in NPK fertilizers (Dittmar *et al.*, 2009).

4.2.1 Pilot plants for nitrification of source separated urine

During more than three years, a pilot plant for treatment of source separated urine has been run at Eawag (research institute) in Switzerland (Fumasoli *et al.*, 2016). The pilot plant process consisted of a nitrification part and a distillation part. The nitrification part was conducted in two columns, each with a liquid volume of 120 L. During the first 36 months the urine was collected from women and diluted with flush water. During the last four months the urine was collected from men and not diluted. An average nitrification rate of 2.1 ± 0.5 g N m^{-2} d^{-1} was received during month 29–30, at which time the average concentration of ammonia in the influent was 1,790 ± 50 mg N L^{-1}. The reactor received higher concentration of ammonia (4,100 ± 450 mg N L^{-1}) during month 36-40. During that time, the nitrification rate was 0.4 ± 0.1 g N m^{-2} d^{-1}. The AOB on the carriers in the reactor was from the *Nitrosomonas europea* lineage and the NOB belonged to the genera *Nitrobacter*.

Another pilot plant for nitrification of urine has been run in eThekwini in South Africa. The highest measured nitrification rate in the reactor in that plant was 1.4 g N m^2 d^{-1} (Udert *et al.*, 2015).

5 Materials and methods

A bench-scale nitrification experiment was performed in order to study the start-up process of a nitrification reactor. Figure 5.1 shows a flow sheet over the experimental set-up. All equipment except the outlet container was placed in a fume hood. The outlet container was placed on the floor under the fume hood. The experiment was conducted during 57 days.

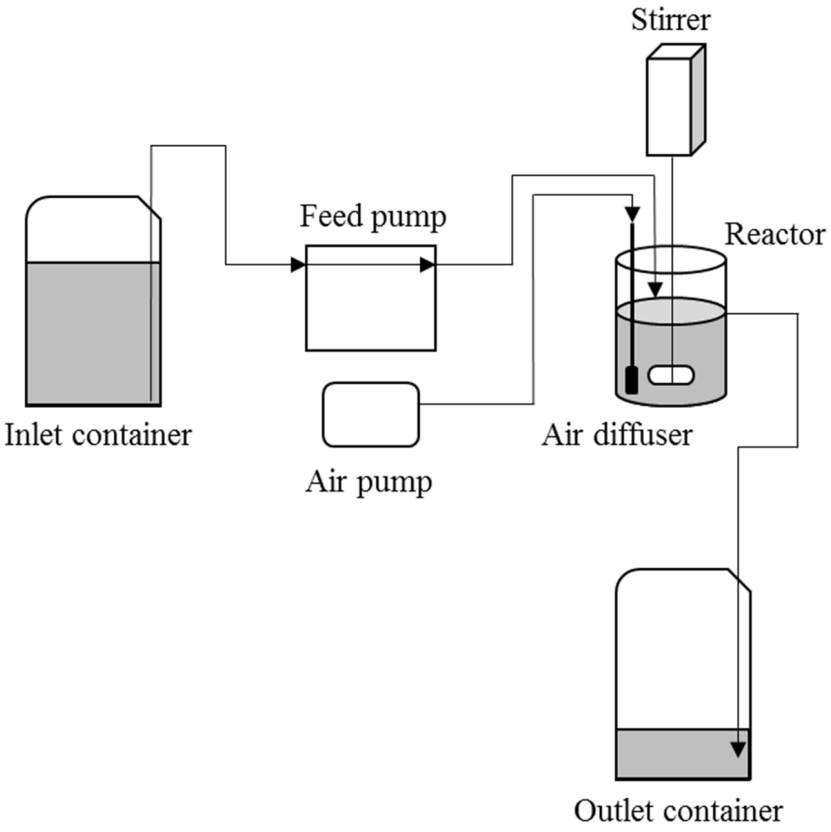

Figure 5.1. Flow sheet of the bench-scale process.

5.1 Equipment and process design

The nitrification was performed in a reactor constituting of a 3 L plastic beaker from VWR. The feed was pumped from an inlet container by an U1-XV (S. NO. L961063) pump from Alitea AB Sweden. Figure 5.2 shows a picture of the process equipment.

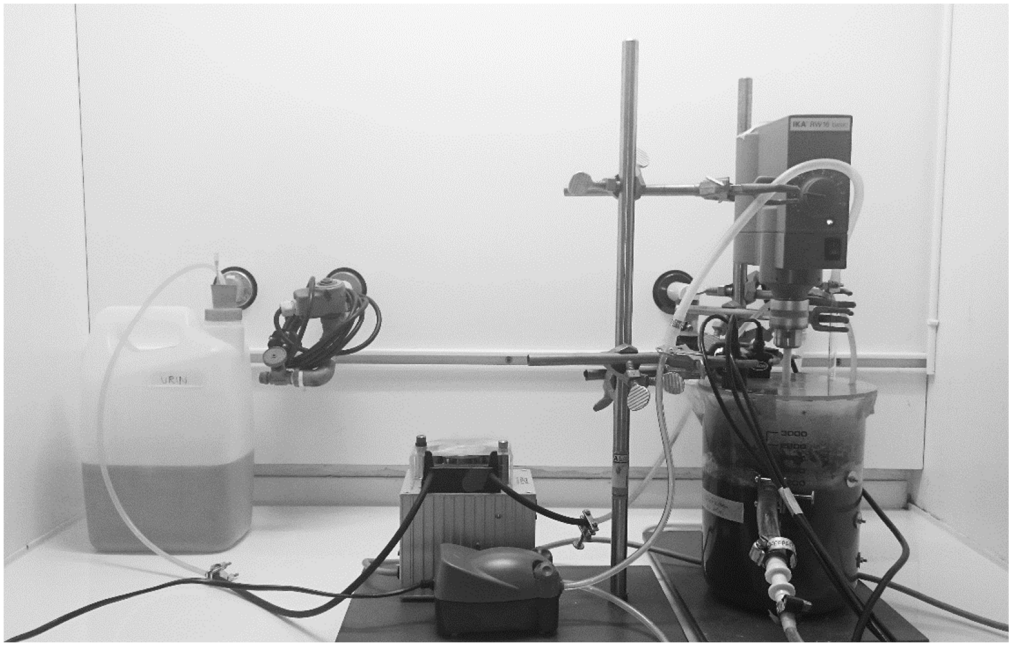

Figure 5.2. Picture of inlet container, feed pump, air pump, stirring equipment, reactor and equipment for continuous measurements.

The reactor had an opening at a level corresponding to 2 L. The opening was connected to a tube through which the liquid could fall by gravity making the liquid volume in the reactor remain constant throughout the experiment (Figure 5.3). The tube connected to the opening led to an outlet container, which was placed on the floor. Both the inlet and the outlet container could hold 5 L. The containers were closed by rubber plugs to prevent vaporization. A tube and a needle ran through each rubber plug (Figure 5.4). The needles were there to equalize the pressure. The outlet of the tube from the inlet container was placed around 2 cm above the surface of the liquid in the reactor.

Figure 5.3. Reactor outlet.

Figure 5.4. Rubber plug with tube and needle.

An IKA® RW16 basic was used for stirring. Aeration of the reactor was performed by a NEWAIR 33 air pump. The flow from the air pump could be adjusted but not fixed to specific flows. The maximum flow the air pump could generate was 190 L h^{-1}. The type of stirring blade and air diffuser used can be seen in Figure 5.5. The reactor was filled with 300 Z-400 carriers from Veolia Water Technologies AB. A baffle was attached to the reactor wall to facilitate mixing of the carriers. The aeration also facilitated the mixing of the carriers.

Measurements of dissolved oxygen (DO) concentration, conductivity, pH and temperature were performed every 30 minutes during the experimental period. DO was measured with a HACH LANGE LDO101, pH was measured with a HACH LANGE pHC101 and conductivity was measured with a HACH LANGE CDC401 (Figure 5.6). The three probes were coupled to two HACH HQ40d multi. The temperature was measured by all three probes. The probes were cleaned with distilled water once a week.

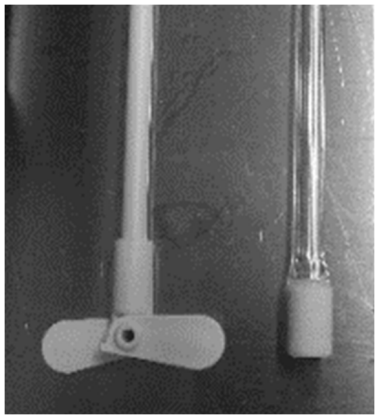

Figure 5.5. Stirring blade (left) and air diffuser (right).

Figure 5.6. Probes for continuous measurements.

5.1.1 Carriers and bacteria

The Z-400 carriers were chosen because they prevent the biofilm from becoming thicker than the height of the grid (Piculell *et al.*, 2016). The relatively low grid of these carriers is preferable in this case since the low grid prevents growth of heterotrophic bacteria. The carriers were taken from a pilot plant run by Veolia Water Technologies AB at Sjölunda WWTP in Malmo. The carriers had received effluent, from a high-loaded activated sludge facility, containing 10–40 mg NH_4^+-N L^{-1}. Each Z-400 carrier has a projected surface area of 1,277 mm^2.

5.1.2 Continuous measurements

Changes in DO concentration can be used to follow the changes in bacterial activity, as long as air flow and temperature are constant. The pH value in the reactor depends on the load and on the activity of the AOB and affects equilibrium concentrations of compounds and bacterial activity. The conductivity can be used to estimate the total amount of dissolved solids in a liquid by multiplying with a factor. The factor is different for different compounds. For high salinity NaCl solutions 0.64 is a common factor, i.e. 100 mS cm^{-1} corresponds to 64 g L^{-1} NaCl (HACH, 2012). In this case, the conductivity values were used to follow the change of total nitrogen concentration in the reactor, since it was assumed that the total nitrogen concentration was proportional to the concentration of total dissolved solids. It is important to measure the temperature since both the bacterial activity and the solubility of compounds depend on it.

5.1.3 Run conditions and changed parameters

During the experiment, concentrations of compounds in the feed, feed flow and air flow were changed in order to create favourable conditions for the bacteria. The goal with the changes was to increase the total nitrogen concentration in the inlet as much as possible without affecting the bacteria negatively. The air flow was only changed one time. It was increased at noon the 22nd day. The feed flow was changed in order to prevent too high loads to occur when the inlet concentration was increased. See Figure 5.7 for total nitrogen concentration, feed flow and nitrogen loads. During the first five load changes the feed pump was turned off for some minutes.

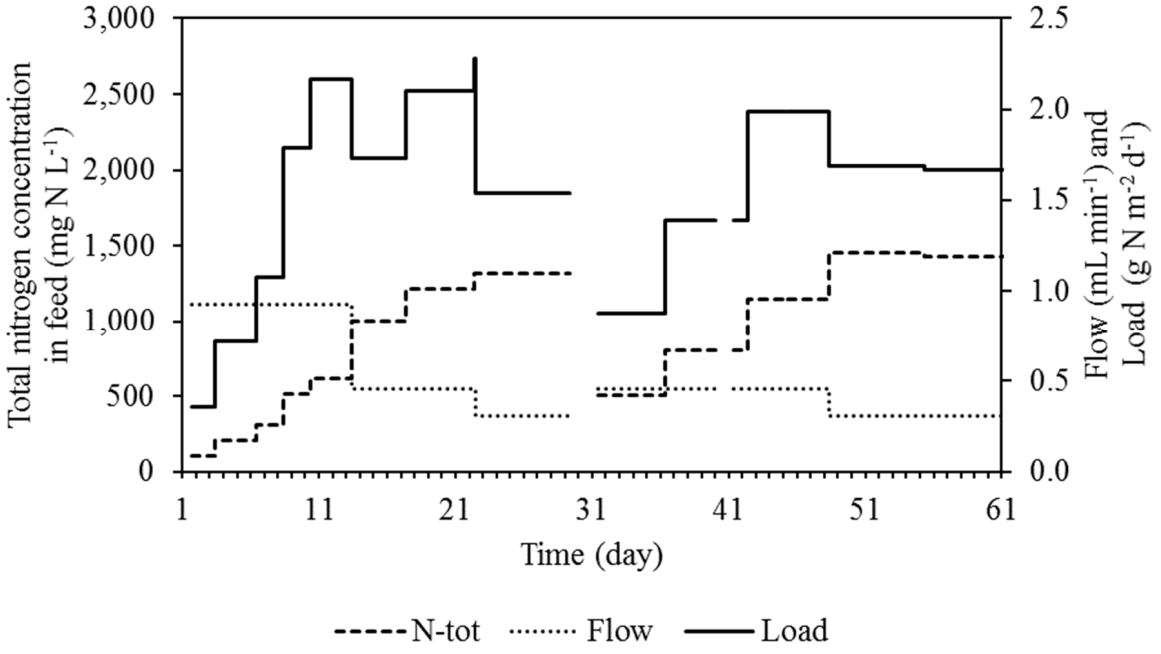

Figure 5.7. Total nitrogen concentration in feed, feed flow and nitrogen load.

The average temperature was 19.3 ˚C (std. dev. 0.4 ˚C), see Figure 5.8 for the temperature curve. The temperature variations were due to the degree of opening of the door to the fume hood and to how much the doors to the laboratory was opened. At days when analyses were performed the doors were opened more and the temperature rose.

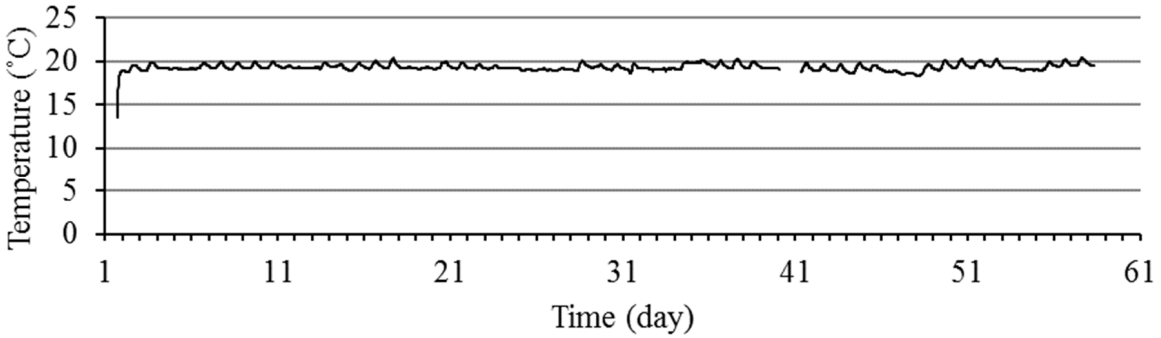

Figure 5.8. Temperature in the reactor.

5.2 Analytical methods

The concentration of ammonium, nitrite, nitrate, total nitrogen, phosphate, total phosphorus and COD was measured in the inlet, the reactor and the outlet. The concentrations were measured with test cuvettes from HACH, see Table 5.1 for the specific tests used. All analyses were made on unfiltered samples. How this may have affected the results, compared to filtered samples, see Appendix II. Three of the test cuvettes are shown in Figure 5.9. A WTW pH 320 was used for pH measurements.

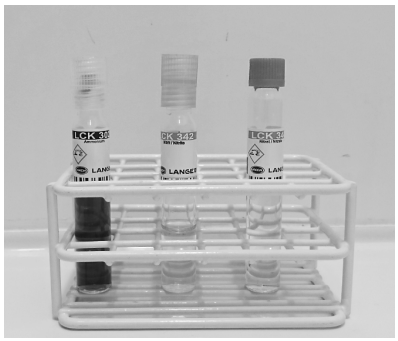

Figure 5.9. Test cuvettes from HACH.

Table 5.1. Test cuvettes from HACH used. [a]*Received from quality certificates from HACH.*

Test	Name	Interval	Method standard deviation[a]
NH_4^+-N	LCK303	2–47 mg NH_4^+-N L^{-1}	0.3 mg NH_4^+-N L^{-1}
NH_4^+-N	LCK302	47–130 mg NH_4^+-N L^{-1}	1.5 mg NH_4^+-N L^{-1}
NO_2^--N	LCK342	0.6–6.0 mg NO_2^--N L^{-1}	0.171 mg NO_2^--N L^{-1}
NO_3^--N	LCK340	5–35 mg NO_3^--N L^{-1}	1.2 mg NO_3^--N L^{-1}
N-tot	LCK338	20–100 mg TN L^{-1}	0.87 mg TN L^{-1}
PO_4^{3-}-P	LCK350	2–20 mg PO_4^{3-}-P L^{-1}	0.091 mg PO_4^{3-}-P L^{-1}
P-tot	LCK350	2–20 mg PO_4^{3-}-P L^{-1}	0.091 mg PO_4^{3-}-P L^{-1}
COD	LCK414	5–60 mg O_2 L^{-1}	0.30 mg O_2 L^{-1}
COD	LCK714	100–600 mg O_2 L^{-1}	1.9 mg O_2 L^{-1}

5.3 Calculations

A few assumptions were made in order to make the calculations. It was assumed that water vaporization only occurred in the reactor, and nowhere else. The total nitrogen concentration was assumed to be proportional to the concentration of total dissolved solids, since the urine used in the diluted urine solutions came from the same source. The liquid volume in the reactor was estimated to 1.8 L. The time available for start-up during the first day of the experiment was limited. There was no time for an exact measurement of the liquid volume in the reactor. The liquid volume was estimated by watching the liquid level in the reactor when the carriers were removed from the reactor and counted.

5.3.1 Nitrification rate

The nitrification rate was calculated as the amount of nitrate produced per carrier area and day, see Equation 10. In Equation 10, F is the feed flow, C is the average nitrate concentration in the outlet during a certain period of time and A is the total carrier area, which was 0.3831 m^2. The nitrification rate is designated NR.

$$NR = \frac{F \cdot C}{A} \tag{10}$$

In another study (Udert and Wächter, 2012), the nitrification rate was calculated from the difference in ammonia load between the inlet and the outlet. The ammonia consumption was not used for calculation in this study since the ammonia concentration in the inlet container increased over time, see Figure 6.1 in section 6.1.

5.3.2 Simulation

The total nitrogen concentration in the reactor was simulated. The nitrification reactor was modelled as an ideal continuous stirred tank reactor (CSTR). The results from the simulation were used to predict the concentrations of substances in the reactor. The simulated data was compared with both conductivity measurements and measurements of total nitrogen concentration in the reactor, to see if the model could describe the concentration in the reactor. The liquid volume was assumed to be 1.8 L. Input data to the model was total nitrogen concentration and feed flow, see Figure 5.7 for data. Two values per hour were calculated. The tube volume (13 mL) between the inlet container and the reactor was only accounted for at the second start-up, when it had to be refilled. For more information about the simulation and the comparison with conductivity data see Appendix III. The model used for the simulation can be seen in Equation 11, in which C is the total nitrogen concentration in the reactor, C_0 is the total nitrogen concentration in the reactor at the moment of a batch change, C_{inlet} is the total nitrogen concentration in the inlet, F is the feed flow and V is the liquid volume in the reactor.

$$C = \frac{C_0 - C_{inlet} \cdot (1 - e^{\frac{F}{V} \cdot t})}{e^{\frac{F}{V} \cdot t}} \tag{11}$$

5.3.3 Flows and vaporization

Three different feed flows were used during the experiment. The flows were set by giving the percentage of the maximal routes per minute the feed pump could work at. The percent number used were 30, 15 and 10. The initial flow generated by the feed pump was measured before the experiment started. The two other flows used were measured during the experiment, by measuring the volume difference in the inlet container during the time the feed pump was turned on. All volumes that were used after the 17th day were measured and every sample from the reactor was noted and included in calculations. For information about the measured values see Appendix I Table AI.3. The flow generated by the feed pump was either 0.93, 0.46 or 0.31 mL min^{-1}, during the experimental period. These flows give hydraulic retention times of 1.3, 2.7 and 4.1 days respectively.

The amount of water vaporization was calculated from the 17th day until the 55th day of the experiment. The amount was calculated by measuring all ingoing and outgoing volumes from the reactor. See Appendix I Table AI.3 for measured volumes. In addition, measured values of the total nitrogen concentration in the reactor was compared with simulated values to estimate

the amount of water vaporization. Since no volume measurements were performed before the 17th day, the amount of vaporization before that day was not calculated. Though, the difference between simulated values of the concentration of the total nitrogen concentration in the reactor and the measured values differed less than 1% during that period. Figure 5.10 shows calculated water vaporization based on volume measurements and based on difference between simulated and measured total nitrogen concentration in the reactor. The total nitrogen concentration, both simulated and measured, are also shown in the figure. The high nitrogen concentration giving the high water vaporization value (9%) on day 30 is probably due to a feed flow stop occurring at that time.

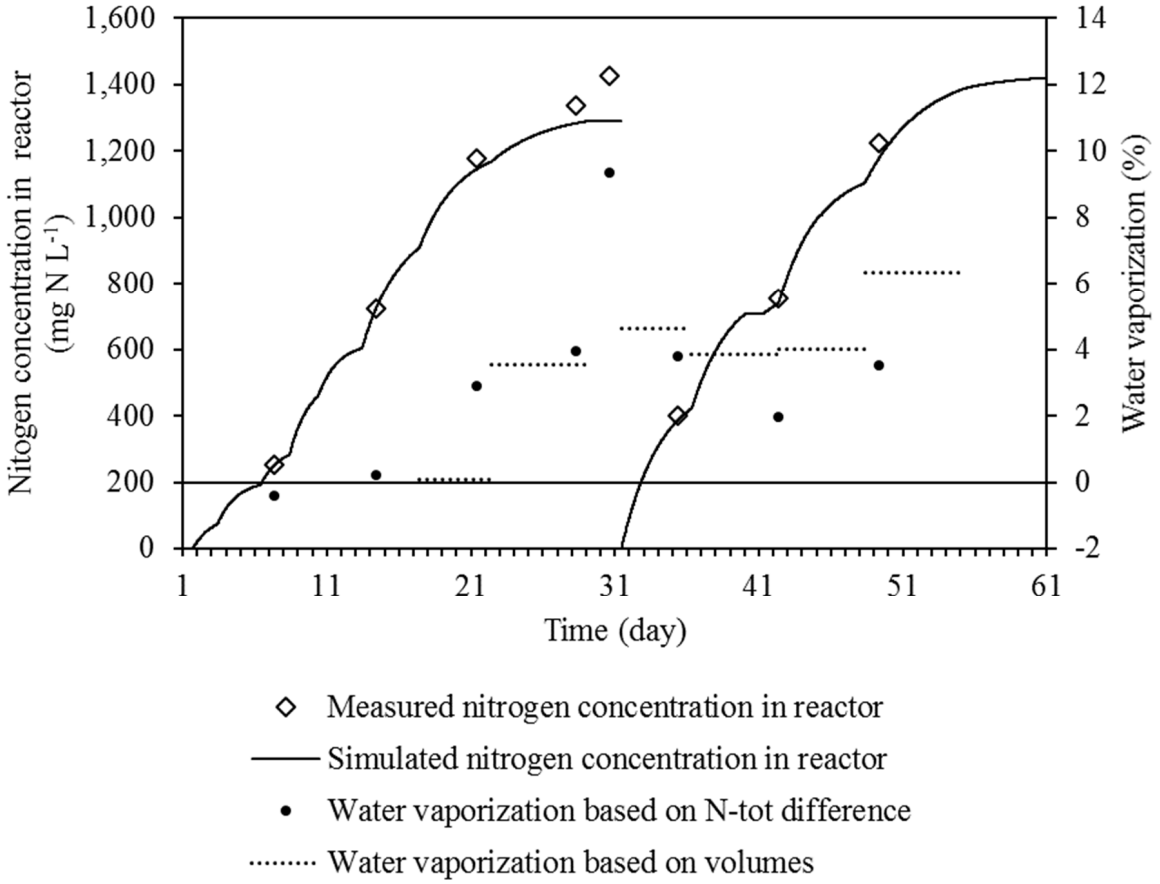

Figure 5.10. Water vaporization and simulated and measured total nitrogen concentration in the reactor.

If any nitrogen loss took place, it was too small to be measurable. The measured total nitrogen concentration in the reactor corresponded well with the simulated total nitrogen concentration in the reactor when no vaporization occurred and the estimated water vaporization based on total nitrogen concentration corresponded well with calculated water vaporization based on volume measurements (Figure 5.10).

5.4 Design of a pilot plant

There is a lot of things to consider when scaling up a process from bench scale to pilot scale. Some problems only occur when the process has a certain size and some actions are only possible when the process is small enough. A pilot plant for treatment of undiluted urine from 100 persons living in an apartment house was designed in order to give an idea of what

dimensions a pilot plant reactor for that purpose may need. Table 5.2 shows the data used when making the calculations for the design. The design parameters calculated and the equations used for the calculations can be seen in Table 5.3.

Table 5.2. Data used when designing the pilot plant.

	Designation	Value	Note
Number of people	N_p	100	Number chosen.
Volume urine secreted at home by one person	V_p	1 L day^{-1}	Value assumed based on the fact that one human secretes 1.4 L urine per day (Rose *et al.*, 2015).
Concentration of urea in urine	C_{urea}	7.6 g N L^{-1}	Average value. Urine contains between 9.3 and 23.3 g urea L^{-1}, which corresponds to 4.3–10.9 g N L^{-1} (Rose *et al.*, 2015). The molar mass of urea is 60.07 g mol^{-1}.
Nitrification rate	NR	2 g N m^2 d^{-1}	Value assumed based on data from earlier studies (Fumasoli *et al.*, 2016, Udert and Wächter, 2012, Udert et al., 2003b).
Nitrogen load per area	L_A	4 g N m^2 d^{-1}	Two times the nitrification rate. Approximately 50% of the ammonia will be oxidized (Udert *et al.*, 2003b).
Carrier area	$A_{carrier}$	1,277 mm^2	Z-400 from Veolia Water Technologies AB
Reactor volume per carrier	$RV_{carrier}$	0.0067 L	Reactor volume per carrier in this study (2 L/300 carriers). The carriers will maybe need less volume in a pilot-scale reactor.
Liquid volume per carrier	$LV_{carrier}$	0.006 L	Estimated liquid volume per carrier in this study (1.8 L / 300 carriers). The carriers will maybe need less volume in a pilot-scale reactor.

Table 5.3. Parameters calculated and the equations used for the calculations.

Parameter	Designation	Equation
Daily nitrogen load	L	$L = N_p \cdot V_p \cdot C_{urea}$
Total carrier area	A_{tot}	$A_{tot} = \frac{L}{L_A}$
Number of carriers	$N_{carrier}$	$N_{carrier} = \frac{A_{tot}}{A_{carrier}}$
Reactor volume	RV	$RV = N_{carrier} \cdot RV_{carrier}$
Liquid volume	LV	$LV = N_{carrier} \cdot LV_{carrier}$
Feed flow	F	$F = N_p \cdot C_p$
Hydraulic retention time	HRT	$HRT = \frac{LV}{F}$

6 Results

All data recorded continuously during the experiment are presented in Appendix IV. Concentrations presented in this section are measured concentration. Water vaporization are not accounted for, since the percentage of water that vaporized was low and varied throughout the experiment.

6.1 Urine solution and inlet concentrations

The urine was retrieved from a private container (60 L) for urine storage. The container had been filled with undiluted urine from one household (the most from just one man) during 2014–2015. The urine was collected in two containers of 2.5 L each and transported to the Department of Chemical Engineering, Lund University, on 2 March 2016. The two containers (labelled A and B) where then stored in a refrigerator at a temperature of 4 ºC. Inlet batches of diluted urine were made by mixing urine from the storage containers with tap water. Figure 6.1 shows how the ratio between ammonium and total nitrogen changed over time in the storage containers in the refrigerator (Figure 6.1a) and in the inlet container (Figure 6.1b).

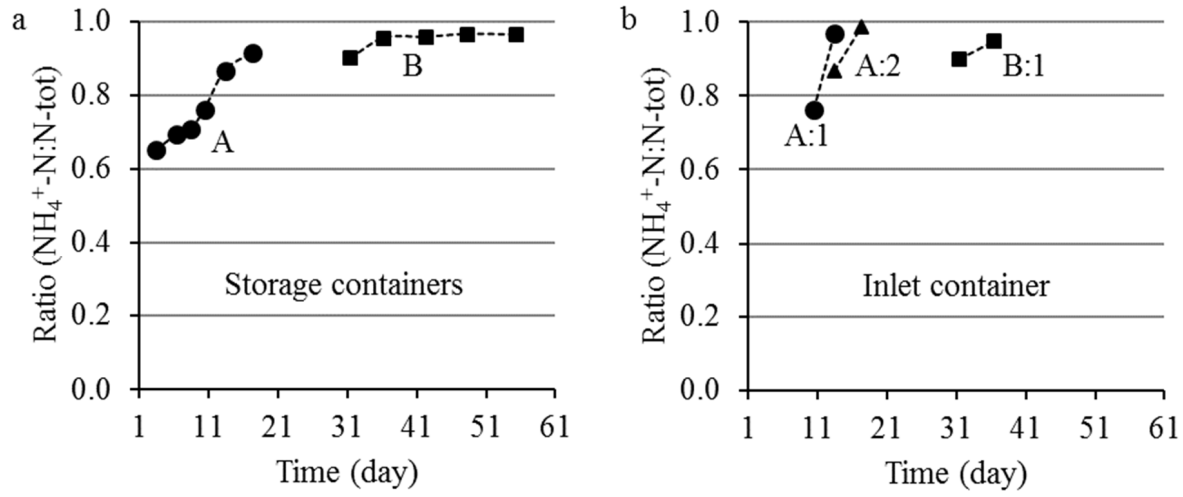

Figure 6.1: a) Change of ratio between ammonia nitrogen and total nitrogen in storage container A and B over time. b) Change of ratio between ammonia nitrogen and total nitrogen in the inlet container over time. Three measurements were conducted. Legend explanation: (From storage container (A/B):Test number)

Total nitrogen, COD, total phosphorus and phosphate were measured in most of the inlet batches. The concentration of these compounds in the storage containers were calculated from the measurements of the inlet batches and are presented in Table 6.1. Urine from the storage containers was diluted and fed to the reactor in 13 different batches, see Appendix I Table AI.1 and Table AI.2 for more information about the inlet batches.

Table 6.1. Concentrations in storage containers. [a]Batch 6 was not used in calculations of COD.

	Container A (avg. ± std. dev.)	**Container B (avg. ± std. dev.)**
N-tot (mg TN L^{-1})	10,210 ± 120	10,240 ± 160
COD (mg O_2 L^{-1})	11,980 ± 290	11,410 ± 220
P-tot (mg PO_4^{3-}-P L^{-1})	749 ± 12	785 ± 11
PO_4^{3-}-P (mg PO_4^{3-}-P L^{-1})	745 ± 18	782 ± 8
Data from batch:	2, 3, 4, 5, 6[a], 7	9, 10, 11, 12, 13

6.2 Start-up of the nitrification reactor

The reactor was operated during 57 days. During the first 12 days, it was clearly observed that the DO concentration decreased as the load was increased (Figure 6.2). Even though the total nitrogen concentration increased during the whole period the DO concentration stabilized for each loading step, indicating that the oxygen consumption was proportional to the bacterial activity. When the feed pump was turned off for some minutes during load changes the pH immediately dropped. When the feed pump then was turned on the pH first increased and then slowly decreased. This can be seen as peaks in Figure 6.2. One reason for this behaviour could be that the bacteria needed time to adapt to the new inlet concentration and load. At day 13 the feed flow was decreased in order to lower the load. The feed pump was turned off for 91 minutes during the change of flowrate. During that time, the new flow was measured. This is the reason for the different behaviour on day 13, compared to previous load changes. The variations in DO concentration during the first two days occurred because no stirring was performed.

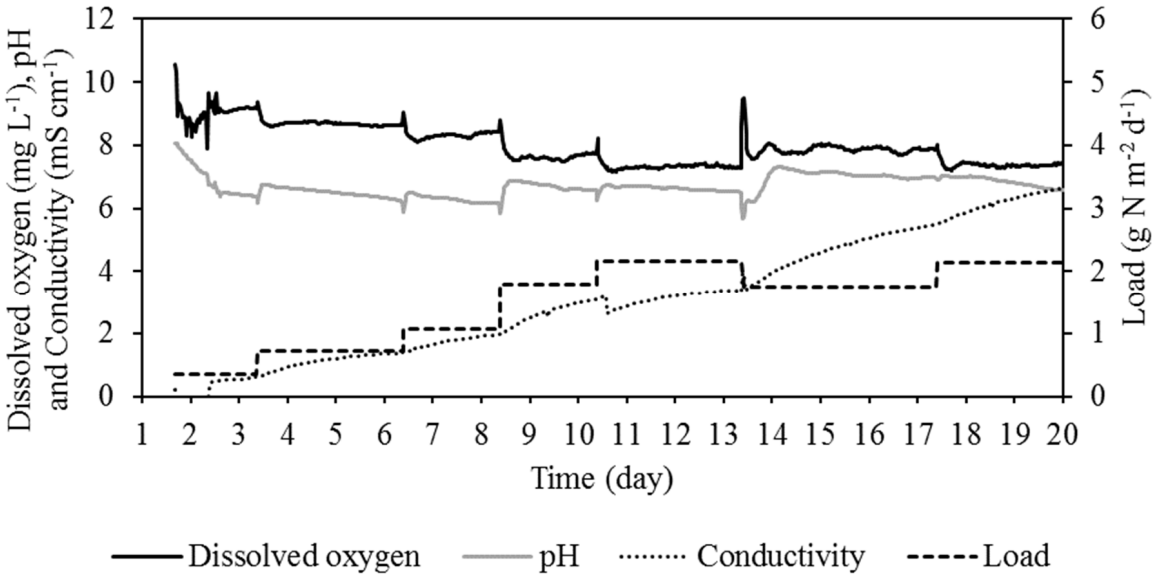

Figure 6.2. Data from continous measurements and load during the first 19 days of the experiment.

Figure 6.3 shows the concentration of nitrogen components in the outlet during the first 19 days of the experiment. The ammonium and the nitrate concentrations followed each other. At the 13th day the nitrite concentration was below 1 mg NO_2^--N L^{-1} in the outlet for the first time. No special action was performed to reduce the nitrite concentration. The highest measured nitrite concentration in the outlet was 17.7 mg NO_2^--N L^{-1} during these first 19 days.

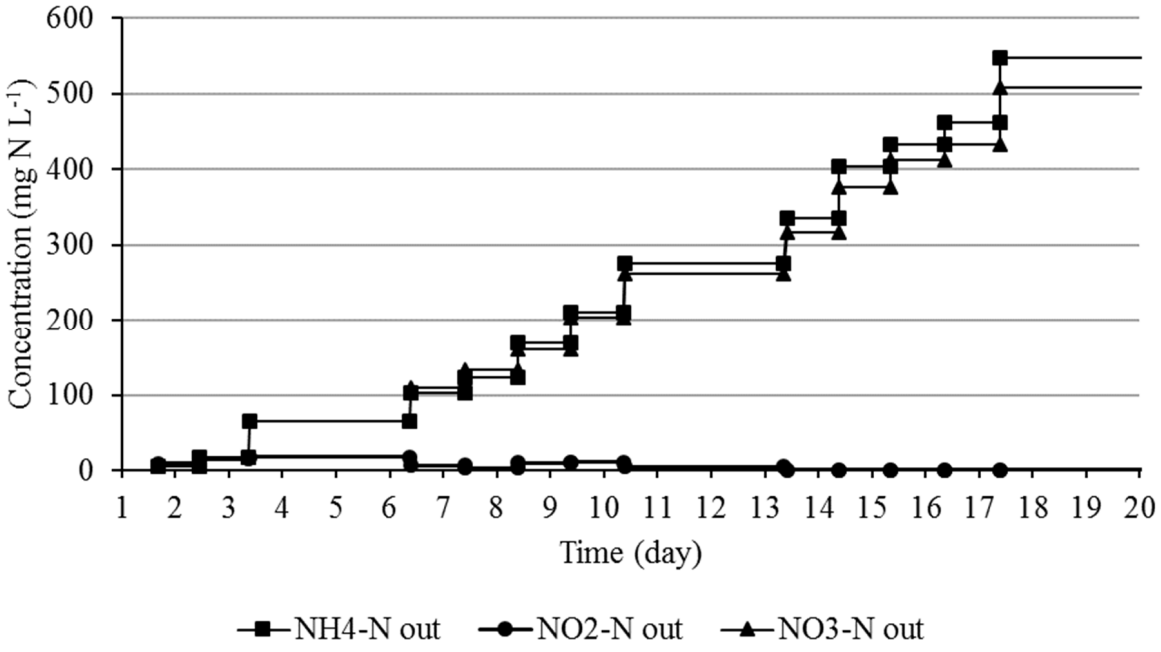

Figure 6.3. Concentrations of inorganic nitrogen compounds in the outlet during the start-up period. The nitrate concentration is plotted first at the 6th day because the nitrite concentration was above the interfering level (> 2 mg NO_2^--N L^{-1}) in the diluted samples the nitrate concentration was measured in.

6.3 Nitrite accumulation

Except for the very first period, nitrite was accumulated in the reactor two times during the experimental period (Figure 6.4). The first nitrite accumulation occurred at an inlet nitrogen concentration of 1,220 mg N L^{-1} and the second nitrite accumulation occurred at an inlet nitrogen concentration of 1,320 mg N L^{-1}. The load was 2.10 g N m^{-2} d^{-1} before the first accumulation and 1.54 g N m^{-2} d^{-1} before the second nitrite accumulation. The first nitrite accumulation was handled by increased air flow and reduced feed flow. The nitrite concentration was measured three times in the reactor during the period of the first nitrite accumulation. The nitrite concentration was 46.2 mg NO_2^--N L^{-1} at noon at the 22nd day. The 23rd day the nitrite concentration was 16.5 mg NO_2^--N L^{-1} in the morning and 13.2 mg NO_2^--N L^{-1} at noon. During the second nitrite accumulation the nitrite concentration in the rector was between 91.4 and 125 mg NO_2^--N L^{-1}, see day 28 to 31 in Figure 6.4. Both nitrite accumulations occurred after sudden increases of both pH and DO, see day 20 and 26 in Figure 6.4. Data from all analysis of samples from the reactor are presented in Appendix I Table AI.4.

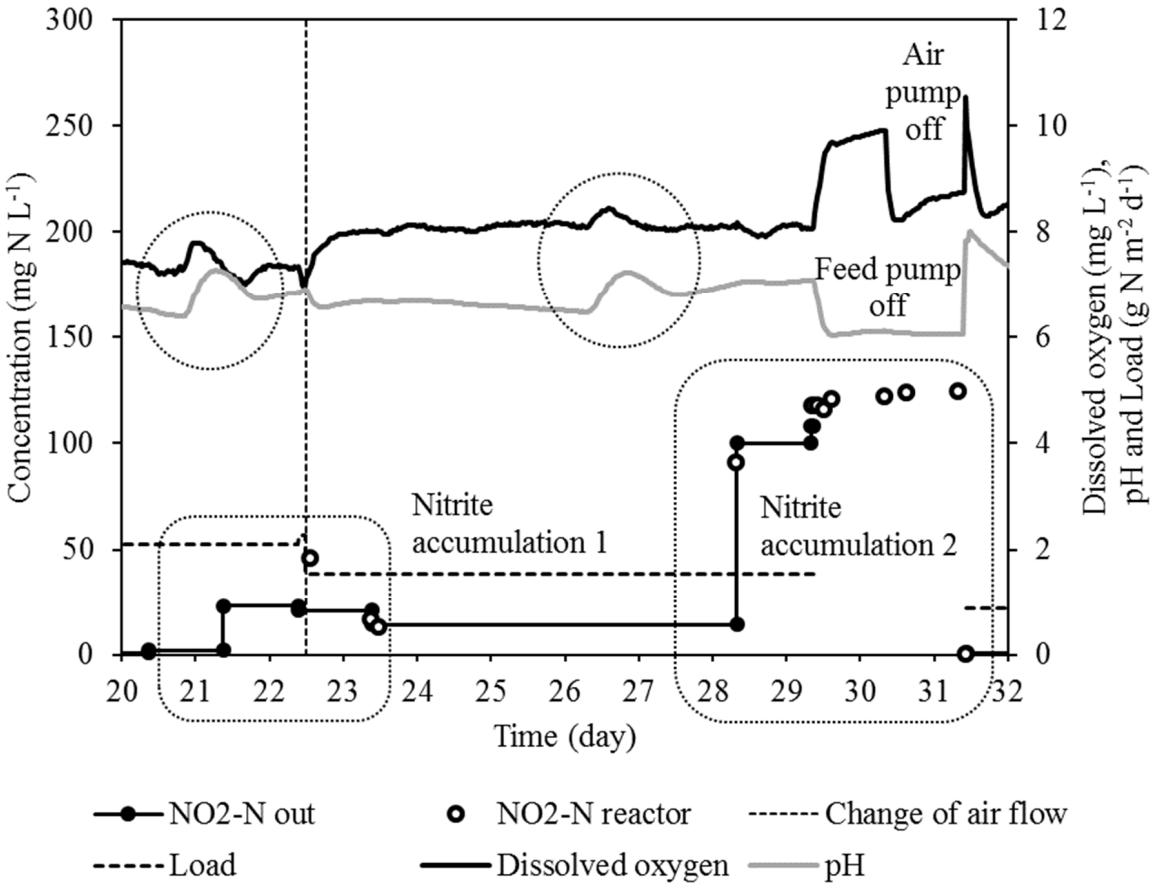

Figure 6.4. Nitrite accumulations during the start-up. The first accumulation was handled by increased air flow and decreased feed flow. To get rid of the nitrite accumulated during the second nitrite accumulation a dilution of the reactor content was performed at day 31.

Different actions were tried out to remove the nitrite during the second nitrite accumulation. First the feed flow was stopped at day 29 and then the air pump was turned off at day 30. These changes can be seen in Figure 6.4. When the feed flow was stopped pH decreased and the DO concentration increased. pH probably decreased because no more alkaline solution was added to the reactor and the AOB consumed the free ammonia available. The DO concentration increased because the bacterial activity decreased when no substrate was added. When the air pump was turned off the DO concentration decreased, because no oxygen was added. These actions did not have any impact on the nitrite concentration in this case. During the time the reactor was stopped the pH stabilized around 6.07. At day 31, around 20 L of tap water was poured into the reactor. The solution in the reactor was diluted until the nitrite concentration was below 0.6 mg NO_2^--N L^{-1}. Then a new start-up of the reactor was performed.

6.4 Start-up after nitrite accumulation

In the beginning of the second start-up the dissolve oxygen concentration and pH first increased and then decreased to stable levels, see Figure 6.5. The sudden increase in DO concentration appeared because the air pump was turned on. The increase in pH probably occurred because the bacteria had been less active during the period when the feed pump had been turned off. A similar decrease in pH can be seen in Figure 6.2 at the absolute start-up of the experiment. The process ran very stably after the second start-up.

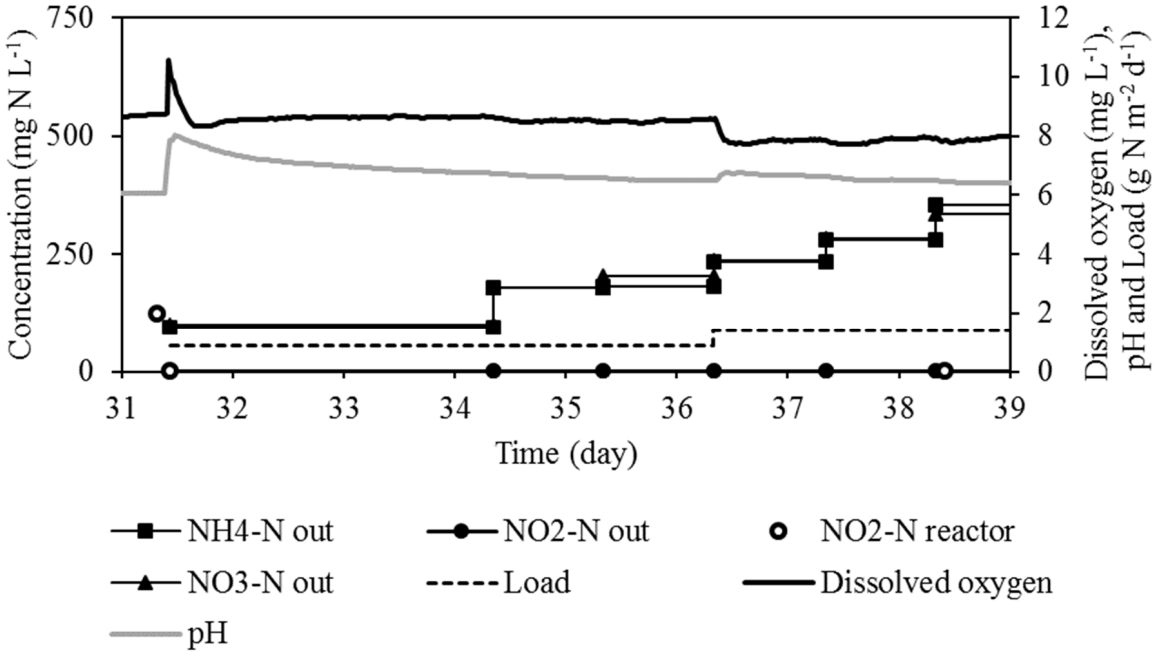

Figure 6.5. Start-up after the second nitrite accumulation.

6.5 Process stability after power outages

During the experimental period after the second start-up, two power outage occurred. The first occurred on the 40th day and the second on the 45th day, see Figure 6.6. The reactor started well after both the power outages, indicating that the reactor was robust against interruptions where both the air pump and the feed pump was stopped. The stirring equipment was also stopped during the power outages.

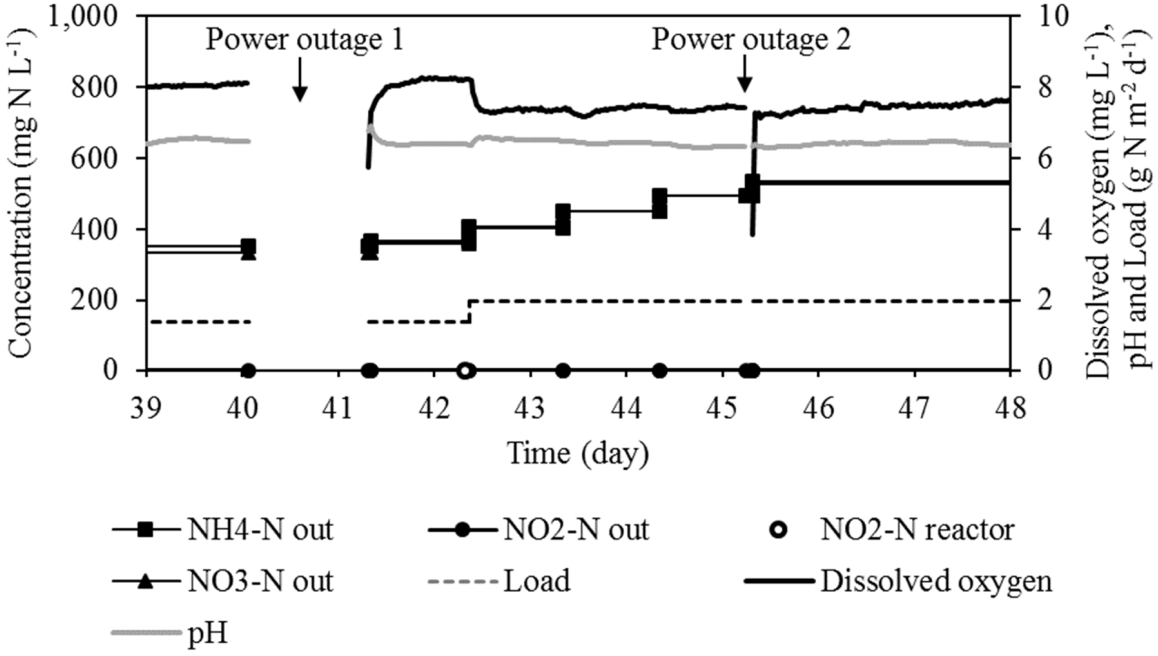

Figure 6.6. First and second power outage and the start-ups thereafter.

6.6 Process stability during the end of the experiment

Figure 6.7 shows the last 10 days of the experiment. The total nitrogen concentration in the inlet was increased to just above 1,400 mg N L^{-1} at day 48. Both DO concentration and pH were very stable during this period.

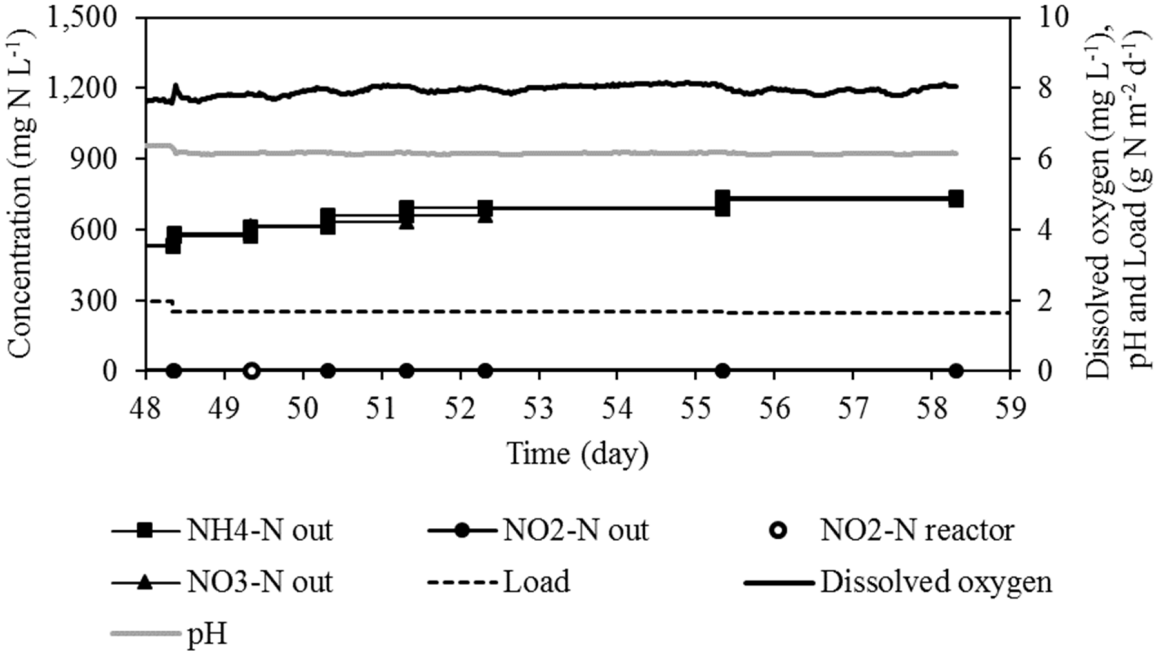

Figure 6.7. Concentrations of nitrogen compounds, DO concentration, pH, and load during the last 10 days of the experiment. The curves shows that the activity in the reactor was stable during this period.

The small variation in DO concentration (Figure 6.7) followed temperature variations (Figure 5.8). The relationship was probably due to variations in bacterial activity, which depends on the temperature.

6.7 Nitrate production and nitrification rate

Figure 6.8 shows the concentrations of nitrogen compounds in the inlet, the reactor and the outlet during the experimental period. The ammonium and nitrate concentration followed each other as the inlet nitrogen concentration increased. All data from analysis of outlet batches are presented in Appendix I Table AI.5.

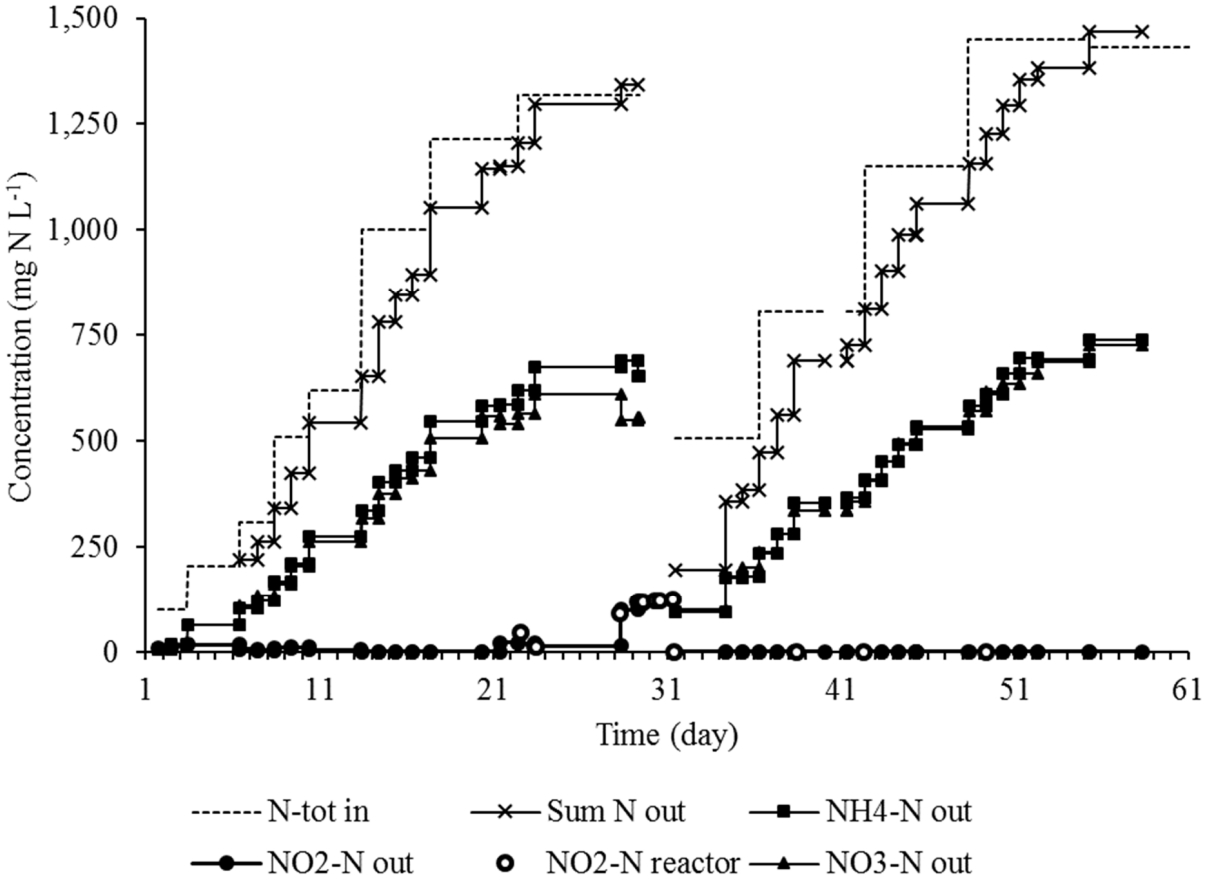

Figure 6.8. Inlet and outlet nitrogen concentrations and nitrite concentration in the reactor.

Figure 6.9 shows the ratio between nitrate and ammonium in the outlet batches. The ratios are plotted against the pH values that were measured in the outlet batches, and not against the average pH values in the reactor during the times the different batches were filled. The pH values in the outlet container was lower than the average values in the reactor. The reason for this may be suspended biomass that continued the oxidation reactions in the tube from the reactor and in the outlet container. The close to 1:1 ratio between nitrate and ammonia corresponded well with results from similar studies (Udert and Wächter, 2012; Udert et al., 2003b).

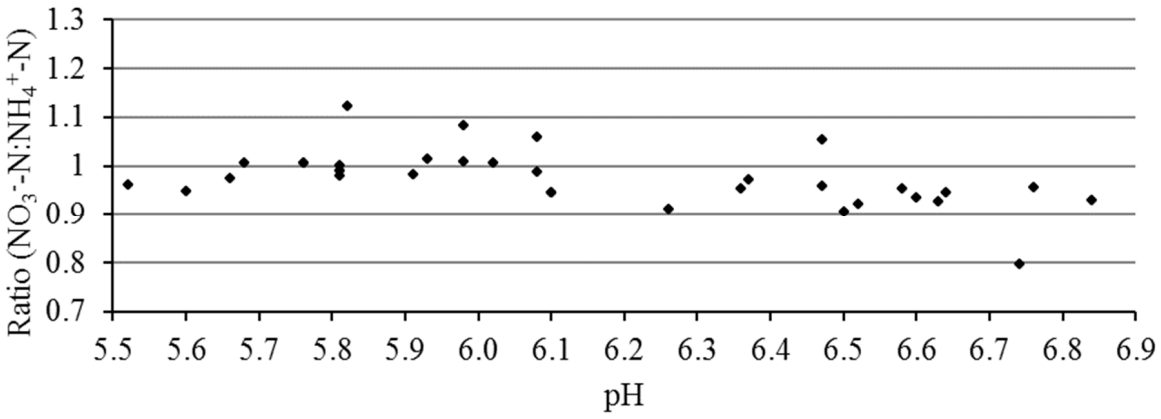

Figure 6.9. Ratio between nitrate and ammonium in the outlet plotted at measured pH values in the outlet.

The nitrification rate calculated from the outlet concentration of nitrate is dependent of the load and the dilution rate of compounds in the reactor. The nitrification rate calculated from the outlet concentration can be seen in Figure 6.10 together with the load. The maximum nitrification rate calculated from the outlet concentration was 0.97 g N m^{-2} d^{-1}.

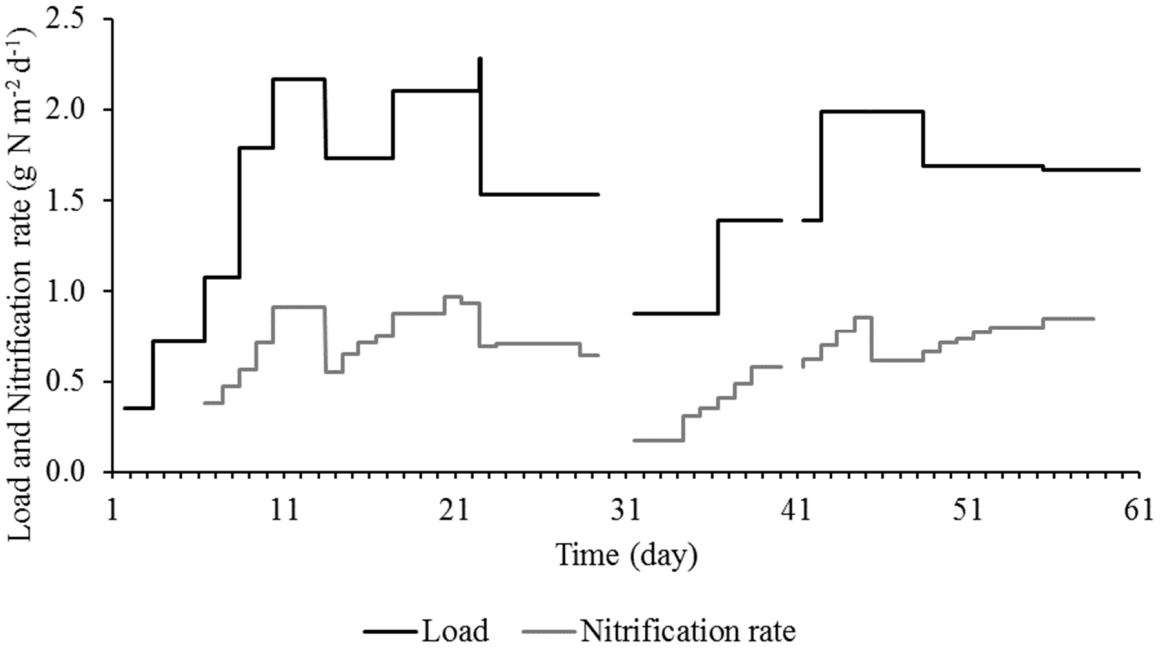

Figure 6.10. Load and nitrification rate calculated from the outlet average concentration of nitrate.

6.8 Pilot plant design

The results from the design calculations performed (Section 5.4) are presented in Table 6.2. Note that the reactor volume, the liquid volume and the hydraulic retention time are based on estimated data from the experiment presented in this work.

Table 6.2. Pilot plant design.

Parameter	Value
Daily nitrogen load	760 g N day^{-1}
Total carrier area	190 m^2
Number of carriers	150,000 carriers
Reactor volume	990 L
Liquid volume	890 L
Feed flow	69 ml min^{-1}
Hydraulic retention time	8.9 days

7 Discussion

During the first days, the activity of the AOB was probably slightly higher than the activity of the NOB, because a low but not negligible amount of nitrite was measured in the outlet. The small amounts of nitrite that were present in the reactor were removed during the first 12 days, probably due to an increase in the activity of the NOB.

As the total nitrogen concentration in the inlet was increased during the experiment, the concentrations of ammonium and nitrate in the outlet increased at approximately the same rate, i.e. the nitrified urine contained ammonia and nitrate in approximately the same amounts throughout the experiment, see Figure 6.8 and Figure 6.9.

Nitrite accumulation occurred two times. Before the accumulations were observed by analytical measurement a sudden increase in both DO concentration and pH could be seen (Figure 6.4). The increases were followed by sudden decreases. The changes were probably due to changes in bacterial activity, since there were nothing affecting the reactor from the outside at these time points. The increase of DO concentration indicates that the bacterial activity was decreased. It is likely that the activity of the AOB was decreased, since it is these bacteria that directly affects the pH. If a decrease in the activity of the NOB occurred at the same time is hard to say. One explanation for the scenario could be the following: The AOB was slightly inhibited, which resulted in a decrease in activity. When the activity of the AOB decreased the pH increased. Since ammonia is the substrate for AOB and that concentration increases with pH the AOB could have become more active again at the elevated pH, which may have resulted in the decrease of both DO concentration and pH. If this was the case, the sudden changes in activity of the AOB may be the reason for the nitrite accumulations that followed. After the sudden increases and following decreases the pH increased instead of decreased, as it had been before the sudden increases. These subsequently increases indicated that something was wrong with the system, since the system did not behave in that way at any other moments.

After the second start-up, the reactor ran stably and was robust against power-outages. The reason for the more stable performance may have been that the bacteria were more accustomed to the reactor conditions.

The pilot plant designed in this study only gives an idea of what dimension a pilot plant for treatment of urine from an apartment house where 100 people are living may require. A reactor volume as large as the one calculated here will probably not be needed, since less volume per carrier will probably be needed in a pilot-scale reactor than in a bench-scale reactor. In order to make a complete pilot plant design more investigations regarding optimal temperature, pH, DO and load have to be done.

8 Conclusions

Nitrite accumulations and inhibition of bacteria are two significant problems that may arise during start-up of a nitrification reactor. They arose during this experiment and they have arisen in similar experiments performed in other studies (Udert and Wächter, 2012; Fumasoli *et al.*, 2016).

Nitrite accumulation may be avoided by not exposing the bacteria to sudden changes or unfavourable conditions. The DO concentration and pH in the nitrification reactor should be monitored in order to follow the conditions in the reactor and thereby be able to make changes when the conditions are changed. To measure the nitrite concentration in the reactor regularly is important to be able to detect increases in nitrite concentration early. When the total nitrogen concentration in the inlet is not as controlled as in this experiment it is probably important to measure the conductivity in the reactor to follow changes in inlet concentrations. If the DO concentration and pH are regulated by adjusting the feed flow and air flow, it will probably work to study the changes in flows instead of the conditions in the reactor.

If nitrite accumulation occurs, it may be possible to get rid of the nitrite by lowering the load and increasing the DO concentration. The first accumulation in this experiment was handled in that way. If a lower load and an increased DO concentration does not have any affect, turning off both the feed pump and the air pump and adding acetate and hydrochloric acid may reduce the nitrite concentration by denitrification (Udert and Wächter, 2012). In extreme cases the nitrite concentration in the reactor can be decreased by diluting the solution in the reactor with water. However, dilution of the reactor content may require a new start-up of the reactor and may not be applicable for large-scale reactors.

9 Future work

The aim of this work was to study the start-up process of a nitrification reactor and to use the obtained information about the process to design a pilot-plant reactor. The results from this study was only sufficient for giving an idea of what dimension a pilot plant may require. More information about how reactors for nitrification of urine works is needed before a pilot plant can be constructed and run optimally. Investigations of how different temperatures, loads, pH and DO concentrations affect the nitrification process can probably give important information that can be useful when designing the pilot plant. To measure the amount of suspended solids produced in the reactor may also give useful information about the system.

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11 Appendices

Appendix I: Experimental data and primary data from manual analyses.

Appendix II: Comparison between results from analyses performed on unfiltered and filtered samples.

Appendix III: Validation of simulation by comparing simulated data with measured conductivity.

Appendix IV: Data from continuous measurements performed during day 1–58.

Appendix IV: Popular scientific summary (Swedish).

Appendix I

Table AI.1. Times for when the different inlet and outlet batches were in use. Times for stops are not included in the table.

Batch	Time, used from (day)	Time, used to (day)	Batch out (nbr)	Time, used from (day)	Time, used to (day)
IN1	1.6806		**OUT1**	1.6806	2.4583
		3.3667	**OUT2**	2.4590	3.3667
IN2	3.3833	6.3778	**OUT3**	3.3833	6.3778
IN3	6.3951		**OUT4**	6.3951	7.4042
		8.3840	**OUT5**	7.4042	8.3840
IN4	8.3965		**OUT6**	8.3965	9.3819
		10.3743	**OUT7**	9.3819	10.3743
IN5	10.3889	13.3597	**OUT8**	10.3889	13.3597
IN6	13.4229		**OUT9**	13.4229	14.3917
			OUT10	14.3917	15.3556
			OUT11	15.3556	16.3625
		17.3944	**OUT12**	16.3625	17.3944
IN7	17.4007		**OUT13**	17.4007	20.3562
			OUT14	20.3562	21.3722
		22.3896	**OUT15**	21.3722	22.3917
IN8	22.3910		**OUT16**	22.3917	23.3792
			OUT17	23.3792	28.3299
			OUT18	28.3299	29.3333
		29.3694	**OUT19**	29.3333	29.3694
IN9	31.4347		**OUT20**	31.4347	34.3472
			OUT21	34.3472	35.3361
		36.3299	**OUT22**	35.3361	36.3299
IN10	36.3299		**OUT23**	36.3299	37.3389
			OUT24	37.3389	38.3319
			OUT25	38.3319	41.3451
		42.3604	**OUT26**	41.3451	42.3604
IN11	42.3604		**OUT27**	42.3604	43.3361
			OUT28	43.3361	44.3451
			OUT29	44.3451	45.3181
		48.3458	**OUT30**	45.3181	48.3458
IN12	48.3458		**OUT31**	48.3583	49.3340
			OUT32	49.3340	50.3146
			OUT33	50.3146	51.3139
			OUT34	51.3139	52.3104
		55.3299	**OUT35**	52.3104	55.3299
IN13	55.3299	61.9583	**OUT36**	55.3299	58.3160

Table AI.2. Primary data from manual analysis of concentrations of substances in the inlet.
[a]*The ammonium ion concentration increased during storage (Figure 6.1).*

Batch (from storage container A/B)	**Dilution (vol% urine)**	**NH_4^+-N (mg NH_4^+-N L^{-1}) [a]**	**N-tot (mg TN L^{-1})**	**COD-tot (mg O_2 L^{-1})**	**P-tot (mg PO_4^{3-}-P L^{-1})**	**PO_4^{3-}-P (mg PO_4^{3-}-P L^{-1})**	**pH**
1 (A)	1	68.8	101.4	100.4	7.16	7.42	8.87
2 (A)	2	134.2	205.6	240	14.56	14.4	8.96
3 (A)	3	214	308.4	372	22.4	22	9.97
4 (A)	5	362	511.2	588	37.48	38.56	8.96
5 (A)	6	471.2	619.2	724	45.6	45.2	8.99
6 (A)	10	867	1,000	-	74.5	73.8	9.03
7 (A)	12	1,110	1,216	1,400	91.5	90.3	9.03
8 (A and B)	13	1,236	1,318	1,520	97.9	97.2	9.06
9 (B)	5	456	506.4	584	38.48	38.6	9.04
10 (B)	8	768	805	925	62.5	62.5	9.04
11 (B)	11	1,100	1,150	1,250	87.6	86.2	9.05
12 (B)	14	1,398	1,450	1,555	111	111	9.05
13 (B)	14	1,376	1,430	1,585	110	109	9.05

The concentrations of nitrite and nitrate were measured in batch number 2 and 10. The nitrite concentration was below 0.6 mg N L^{-1} in both batches and the nitrate concentration was below 5 mg N L^{-1} in both batches. These two levels are the lower limits of the measurement ranges for the used tests.

Table AI.3. Primary data from volumetric measurements. Sample volumes are accounted for.
[a]Because of feed flow changes, calculations were needed to get these values.

Batch	From (day)	To (day)	Time (min)	Volume (mL)
OUT13	17.4007	20.3562	4,256	1,963
OUT14	20.3562	21.3722	1,463	662
OUT15	21.3722	22.3896	1,465	722
SUM OUT	-	-	7,184	3,347
IN7	17.4007	22.3896	7,184	3,350
OUT16	22.4951	23.3792	1,273	343[a]
OUT17	23.3792	28.3299	7,129	2,078
OUT18	28.3299	29.3333	1,445	520
OUT19	29.3333	29.3694	52	45
SUM OUT	-	-	9,899	2,986
IN8	22.4535	29.3694	9,899	3,095[a]
OUT20	31.4347	34.3472	4,194	1,790
OUT21	34.3472	35.3361	1,424	663
OUT22	35.3361	36.3299	1,431	613
SUM OUT	-	-	7,049	3,066
IN9	31.4347	36.3299	7,049	3,215
OUT23	36.3299	37.3389	1,453	618
OUT24	37.3389	38.3319	1,430	623
OUT25	38.3319	40.0694	-	-
	41.3125	41.3451	2,549	1,165
OUT26	41.3451	42.3604	1,462	645
SUM OUT	-	-	6,894	3,051
IN10	36.3299	40.0694	-	-
	41.3125	42.3604	6,894	3,173
OUT27	42.3604	43.3361	1,405	617
OUT28	43.3361	44.3451	1,453	648
OUT29	44.3451	45.2458	-	-
	45.3125	45.3181	1,305	580
OUT30	45.3181	48.3458	4,360	1,898[a]
SUM OUT	-	-	8,523	3,743
IN11	42.3604	45.2458	-	-
	45.3139	48.3458	8,521	3,900
OUT31	48.3458	49.3340	1,423	388[a]
OUT32	49.3340	50.3146	1,412	415
OUT33	50.3146	51.3139	1,439	395
OUT34	51.3139	52.3104	1,435	410
OUT35	52.3104	55.3299	4,348	1,225
SUM OUT	-	-	10,057	2,833
IN12	48.3458	55.3299	10,057	3,025
OUT36	55.3299	58.3160	4,300	1,170

Table AI.4. Primary data from manual analysis of concentrations of substances in the reactor.

Time (day)	NH_4^+-N (mg NH_4^+-N L^{-1})	NO_2^--N (mg NO_2^--N L^{-1})	NO_3^--N (mg NO_3^--N L^{-1})	N-tot (mg TN L^{-1})	COD-tot (mg O_2 L^{-1})	P-tot (mg PO_4^{3-}-P L^{-1})	PO_4^{3-}-P (mg PO_4^{3-}-P L^{-1})
7.381	-	-	-	251.6	178	17.46	17
14.388	-	-	-	721.6	335	49.6	46.4
21.366	-	-	-	1178	492	79.9	75.8
22.556	-	46.2	-	-	-	-	-
23.372	-	16.5	-	-	-	-	-
23.467	-	13.15	-	-	-	-	-
28.315	-	91.4	-	1,336	636	91.7	89.4
29.349	-	118	-	-	-	-	-
29.418	-	118.2	-	-	-	-	-
29.506	-	116.2	-	-	-	-	-
29.604	-	120.8	-	-	-	-	-
30.316	729	122.4	581.6	-	-	-	-
30.615	670	124	560	-	-	-	-
31.318	-	124.8	-	-	-	-	-
31.428	-	< 0.6	-	-	-	-	-
35.342	-		-	400.8	314	25.2	25.2
38.413	-	1.39	-				
42.317	-	1.14	-	752	338	50.5	49.8
49.342	-	< 0.6	-	1,222	468	83.5	80.5

Table AI.5. Primary data from manual analysis of concentrations of substances in the outlet.

Batch (nbr)	NH_4^+-N (mg NH_4^+-N L^{-1})	NO_2^--N (mg NO_2^--N L^{-1})	NO_3^--N (mg NO_3^--N L^{-1})	N-tot (mg TN L^{-1})	COD-tot (mg O_2 L^{-1})	P-tot (mg PO_4^{3-}-P L^{-1})	PO_4^{3-}-P (mg PO_4^{3-}-P L^{-1})	pH
1	5.51	9.82	8.54[1]	-	-	-	-	7.27
2	19	15.68	23.9[1]	-	-	-	-	6.14
3	65.9	17.68	64.2[1]	-	-	-	-	6.09
4	103	7.24	109.2	-	-	-	-	6.08
5	124	3.84	134.4	-	-	-	-	5.98
6	169.6	10.72	161.6	347.2	230	23.60	22.28	6.58
7	210	11.24	204	431.2	225	-	-	6.37
8	275.2	5.08	262.4	539.2	-	-	-	6.36
9	334.8	0.71	316.8	652.8	-	-	-	6.64
10	404	0.925	376	796.8	-	-	-	6.84
11	432	0.67	412.8	846.4	310	57.04	54.96	6.76
12	461.6	< 0.6	432	867.2	-	-	-	6.60
13	546.4	0.767	507.2	1,062.4	-	-	-	6.63
14	584	1.97	560	1,146.0	-	-	-	6.47
15	587	23.1	542	1,156.0	534	79.00	76.30	6.52
16	620	21	566	1,182.0	-	-	-	6.26
17	674	14.3	610	1,284.0	-	-	-	6.50
18	691	100	551.2	1,348.0	-	-	-	6.74
19	-	-	-	-	-	-	-	-
20	95.2	0.893	100.4	196.0	-	-	-	6.47
21	177.9	< 0.6	179.4	378.0	-	-	-	5.98
22	180.5	< 0.6	203	399.0	-	-	-	5.82
23	234	< 0.6	237.6	486.0	-	32.16	31.24	5.93
24	280.4	< 0.6	282	590.4	-	-	-	6.02
25	353.6	< 0.6	334.8	-	-	-	-	6.10
26	368	< 0.6	358.4	-	-	-	-	5.66
27	408	< 0.6	404.8	-	-	-	-	5.81
28	451.1	< 0.6	452	929.0	378	61.50	60.00	5.81
29	493	< 0.6	496	-	-	-	-	5.76
30	534	< 0.6	528	-	-	-	-	6.08
31	584	< 0.6	572	-	-	-	-	5.81
32	611	< 0.6	616	-	-	-	-	5.68
33	659	< 0.6	634	1,276	340	86.70	85.80	5.52
34	695	< 0.6	660	-	-	-	-	5.60
35	694	< 0.6	688,5	-	-	-	-	5.81
36	739	< 0.6	727,5	-	-	-	-	5.91

Appendix II

Since all samples were analysed unfiltered, a few tests were performed to estimate what the results may have looked like if the samples had been filtered. The filter paper used for these tests were MUNKTELL General Purpose Filter Paper Article number: 120014, Grade: 1002.

The results from the tests performed for comparison between unfiltered and filtered samples can be seen in Table AII.1. The average values for the analysis of the different components can be seen in Table AII.2.

Table AII.1. Data for comparison between results from analysis of filtered and unfiltered samples. [a]*Value from unfiltered sample minus value from filtered sample.*

Sample	Dilution (times)	Unfiltered (mg L^{-1})	Filtered (mg L^{-1})	Absolute difference[a] (mg L^{-1})	Difference in percent of unfiltered value
NH_4^+-N OUT35	10	694	642	52	7.5
NO_2^--N OUT35	1	0.061[u]	-0.001[u]	-	-
NO_3^--N OUT35 (1)	30	690	705	-15	-2.2
NO_3^--N OUT35 (2)	30	687	696	-9	-1.3
NH_4^+-N IN13	20	1,376	1,296	80	5.8
P-tot IN13	10	110	106	4	3.6
PO_4^{3-}-P IN13 (1)	10	109	105	4	3.7
PO_4^{3-}-P IN13 (2)	10	109	106	3	2.8
NH_4^+-N OUT36 (A)	10	756	692	64	8.5
NH_4^+-N OUT36 (B)	10	722	649	73	10.1
NO_2^--N OUT35	1	0.08[u]	0.04[u]	-	-
NO_3^--N OUT35 (A)	30	720	735	-15	-2.1
NO_3^--N OUT35 (B)	30	714	732	-18	-2.5

(1) and (2) are analyses performed on the same dilutions. (A) and (B) are analyses performed on different dilutions. [u] The value was under the range of measurements.

Table AII.2. Resulting difference between results from analysis of filtered and unfiltered samples.

Substance	Difference in percent of unfiltered value (avg. ± std. dev.)	Number of tests
NH_4^+-N	8.0 ± 1.8%	4
NO_3^--N	-2.0 ± 0.5%	4
PO_4^{3-}-P	3.2 ± 0.6%	2
P-tot	3.6 ± 0.0%	1

Appendix III

It was possible to find a linear relationship between the simulated total nitrogen concentration in the reactor and the measured conductivity values. The linear equation was found by visually comparing the two data sets. The measured conductivity took some unexplainable steps during the experimental period. When those happened the linear equation had to be changed. See Table AIII.1 for the linear equations used.

Table AIII.1. Equations describing the linear relationships between measured conductivity and simulated total nitrogen concentration in the reactor. (y is conductivity and x is the simulated total nitrogen concentration in the reactor)

Start time (day)	End time (time)	Equation
1.68	10.58	$y = 6.5 x + 130$
10.60	31.43	$y = 6.5 x - 470$
31.49	37.93	$y = 6.5 x + 180$
37.95	58.30	$y = 6.5 x - 470$

The linear relationship between the simulated curve and the conductivity curve (Figure AIII.1) indicates that the reactor could be modelled as an ideal CSTR.

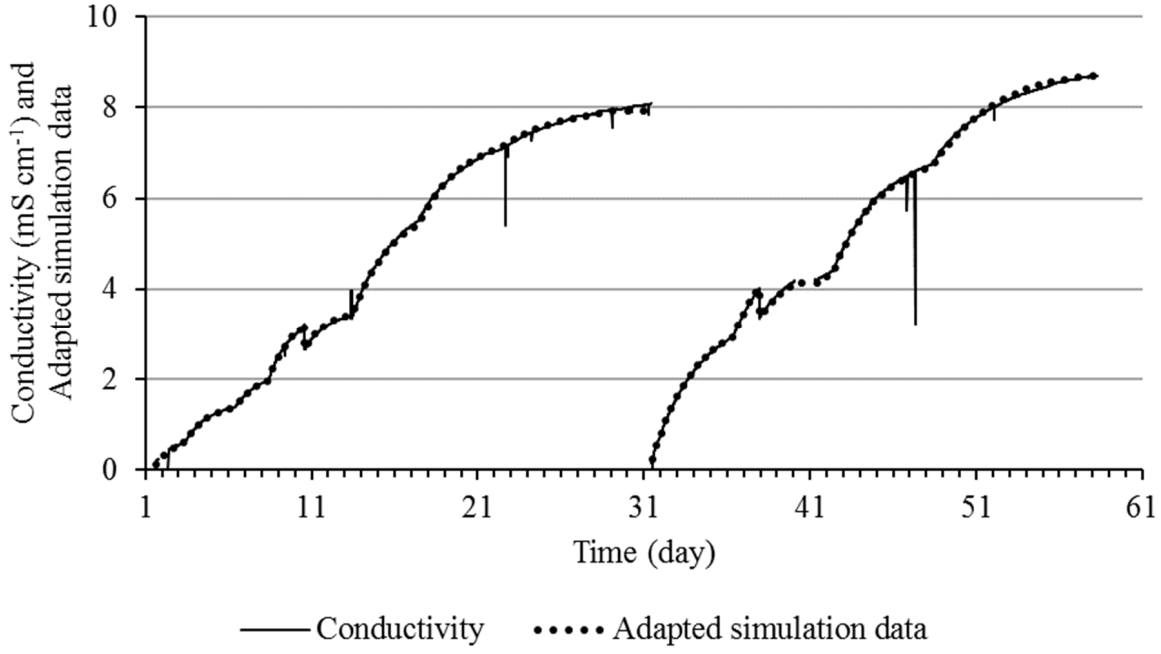

Figure AIII.1. Conductivity and adapted simulation data.

Appendix IV

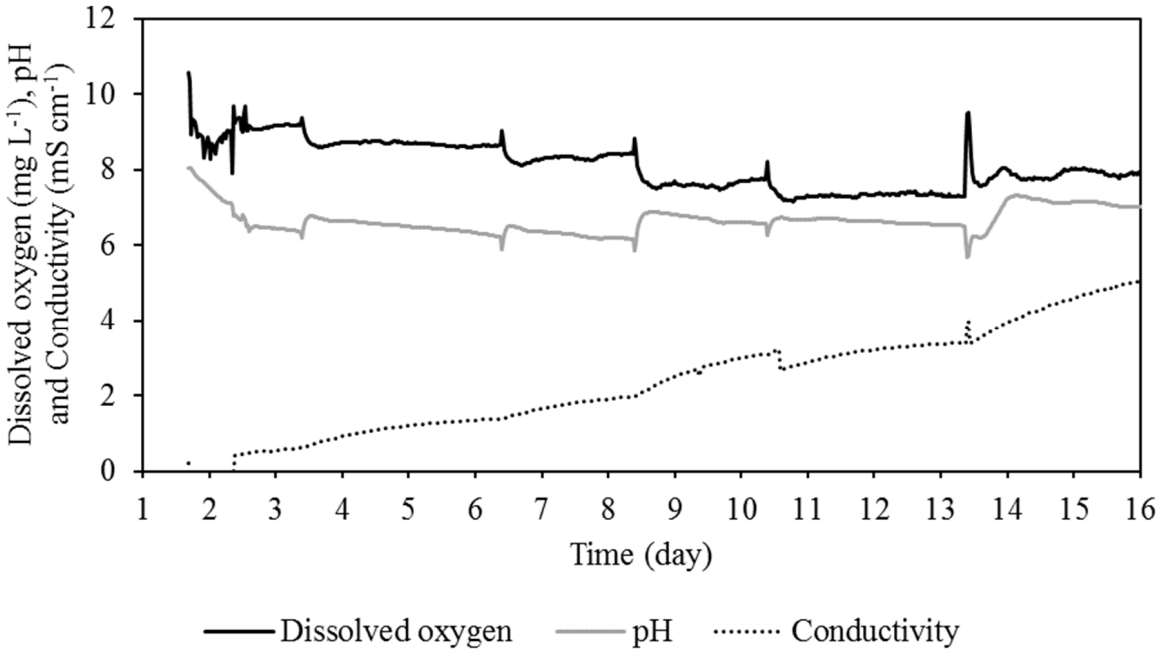

Figure AIV.1. Data from continuous measurements performed during day 1–16.

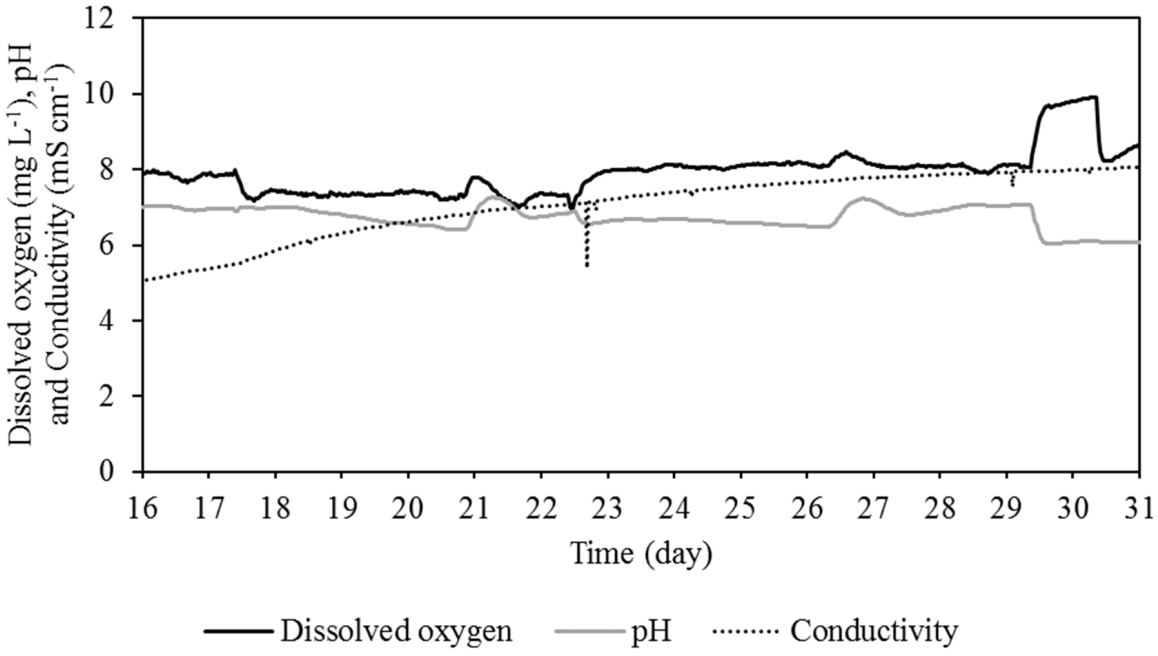

Figure AIV.2. Data from continuous measurements performed during day 16–31.

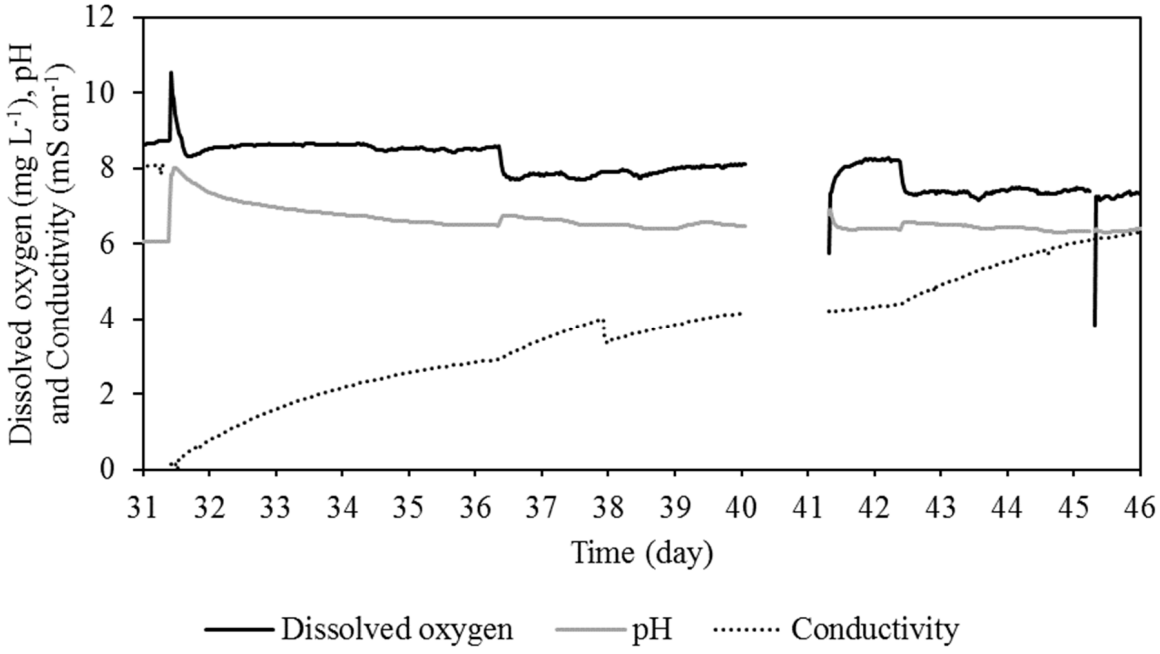

Figure AIV.3. Data from continuous measurements performed during day 31–46.

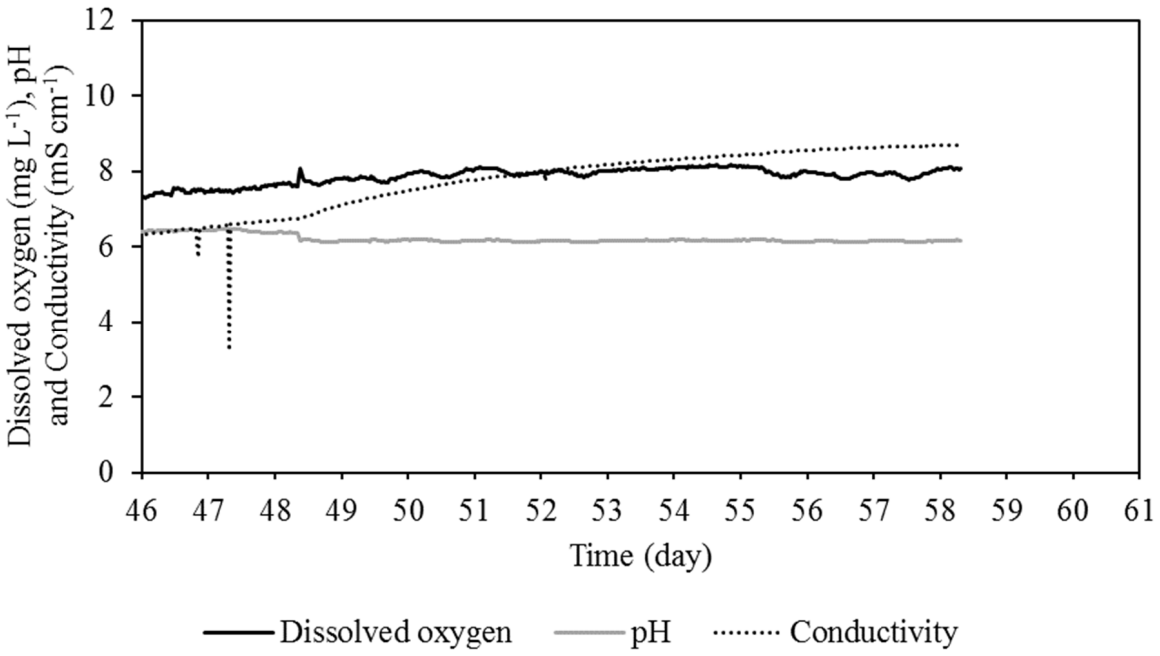

Figure AIV.4. Data from continuous measurements performed during day 46–58.

Appendix V

Stabilisering av urin för återanvändning av näringsämnen

I naturen finns kretslopp för näringsämnen. När människan via avloppsvattenreningsverk släpper ut näringsämnen i vattendrag eller i luften bryts kretsloppen. För att bli en del av de naturliga kretsloppen behöver näringsämnena som människan avger återföras till jorden.

Marianne Olofsson
Populärvetenskaplig sammanfattning av masteruppsatsen:
Stabilization of Urine by Nitrification in a Moving Bed Biofilm Reactor

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Dagens avloppsvattenreningsverk kan inte avskilja alla näringsämnen i avloppsvatten fullständigt, och en del av dem släpps ut i vattendrag eller i luften. För att minska belastningen på avloppsvattenreningsverk och återföra en stor del av näringsämnena till jordbruket kan urin separeras och behandlas separat. Separeringen kan ske med en toalett med två avlopp, i vilken urinen aldrig blandas med resten av avföringen.

Urin innehåller en kväveförening som kallas urea. När urea kommer i kontakt med ett naturligt förekommande enzym som kan bryta ner urea omvandlas urean till ammoniak. Ammoniak är en flyktig förening som luktar fränt. För att undvika att ammoniak avges från urinen behöver urinen stabiliseras. I jord finns bakterier som kan omvandla ammoniak till nitrat, genom att byta ut väteatomerna i ammoniak mot syreatomer. Samma typ av bakterier har man kunnat få att växa på specialutformade plastbitar, kallade rörligt bärarmaterial. Dessa bärare kan användas vid avloppsvattenrening.

Koncentrationen av kväveföreningar i urin är mycket högre än den i vanligt avloppsvatten. Bakterier på plastbärare som tidigare behandlat vanligt avloppsvatten kan därför behöva tid på sig att vänja sig vid de höga kvävekoncentrationerna i urin. Syftet med det här arbetet var att studera uppstarten av en reaktor för stabilisering av urin.

Uppstarten skedde genom att utspädd urin pumpades in till en reaktor med plastbärare. Allt eftersom tiden gick ökades andelen urin i inflödet och kvävekoncentrationen blev högre. Två gånger ackumulerades nitrit i reaktorn. Nitrit är mellanprodukten då ammoniak omvandlas till nitrat och ska helst inte ackumuleras i för stora mängder. Vid höga nitritkoncentrationer kan nämligen bakteriernas aktivitet minska, vilket gör att stabiliseringsprocessen försämras.

För att kunna upptäcka en nitritackumulering på ett tidigt stadium bör nitritkoncentrationen i reaktorn mätas kontinuerligt. Om en ökning av nitritkoncentrationen upptäcks tidigt kan eventuellt en nitritackumulering förebyggas. Genom att kontinuerligt studera pH och syrekoncentrationen i reaktorn kan processavvikelser upptäckas och åtgärdas.

Att kunna köra en sådan här processen stabilt är en förutsättning för att anläggningar ska byggas. Just nu planerar Malmö stad att bygga en sådan här anläggning, men mer information om hur en sådan här process fungerar behöver införskaffas innan bygget kan påbörjas och en reaktor kan köras på ett tillfredsställande sätt.

Nästa gång du möts av en toalett med två hål, bli glad, din urin kan komma till nytta!