

# DEAMMONIFICATION PROCESS FOR TREATMENT OF AMMONIUM RICH WASTEWATER

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## ABSTRACT

The article presents the results obtained from laboratory-scale pilot plant experiments with deammonification process as a new method for biological treatment of ammonium rich streams with a low content of biodegradable organic matter. Deammonification with less than half of oxygen consumption and with no need for carbon source is the least resources consuming pathway for transfer of ammonium to nitrogen. The studies were conducted for one year with two main objectives: to cultivate such microorganisms that could accomplish deammonification for nitrogen removal from digested supernatant and to evaluate the influence of different environmental factors on the process performance. Important environmental factors, monitored during this process, included ammonium, nitrite, nitrate and oxygen concentrations as well as the influence of pH and temperature. The results obtained during the experiments indicated that it was possible to maintain stable conditions and achieve high deammonification process efficiency around 85 % of nitrogen removal with the maximum value of 92 %. Analysis of the experimental data showed that low oxygen concentrations below 0.5 mg O<sub>2</sub>/l in combination with pH-values around 8.2-8.3 and temperature of 30 °C were required to successfully operate the deammonification process. A dissolved oxygen concentration was the main parameter that influenced the process performance in the reactor.

## KEYWORDS

Anammox; deammonification; digester supernatant; leachate; nitrogen removal

## INTRODUCTION

Nitrogen removal from wastewater has been introduced in Sweden and in many other countries mainly with the implementation of technology based on biological nitrification and denitrification processes. Substantial part of influent nitrogen is in ammonium form. Two main processes for nitrogen removal (nitrogen is also removed through assimilation with excess sludge) can be described as:

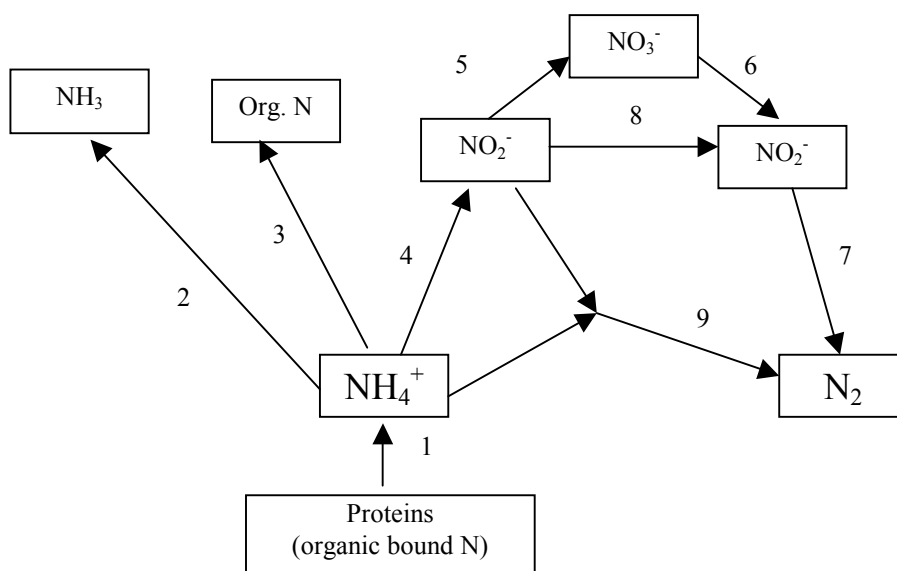
- Oxidation of ammonium to nitrite, followed by oxidation of nitrite to nitrate in oxygen rich environment (nitrification process)
- Reduction of nitrate to nitrite, followed by the reduction of nitrite to nitrogen gas (denitrification process)

In different municipal and industrial processes many nitrogen rich streams are produced with a low content of biodegradable organic matter. The examples of such streams are: leachate from solid waste landfills, supernatants from dewatering of digested sludge at wastewater treatment plants, water from explosive works in tunnels and wastewater from fertilizer's production.

During last years the deammonification process as a new technology for treatment of nitrogen rich wastewaters have been developed and studied (Hellings et al., 1998; Helmer et al., 2000; Van Dongen et al., 2001; Mulder et al., 2000; Siegrist et al., 1998). The introduction of deammonification process can reduce the costs of plant operation and make the management more sustainable. Due to low growth rates of microorganisms the process favors systems with long sludge retention times (biofilm systems), which continuously receive wastewater rich in ammonium and nitrites. In the experiments, the Kaldnes rings were used as biofilm carriers.

### THEORETICAL BACKGROUND

Reaction routes in removal of ammonium nitrogen are schematically presented in Figure 1. The experiments focused on the new pathways (pathways 4-9). The reactions involved are shown in Table 1. Based on these reactions, alkalinity changes as well as oxygen and organic material (as COD) demand can be calculated (see Table 2).



**Figure 1.** Different routes involving transfer of ammonium to nitrogen gas (1- ammonification, 2- ammonia stripping, 3- assimilation, 4 – nitrification, 5- nitrification, 6- denitrification, 7 – denitrification, 9- Anammox).

Implementation of the traditional biological technology with nitrification and denitrification, as performed at municipal wastewater treatment plants (pathway 4 + 5 + 6 + 7), need 4.57 g O<sub>2</sub>/g N and about 4 g COD/g N (Trela, 2000). This method applied for the ammonium rich streams with a low content of biodegradable organic matter has one big disadvantage, as external material (for example methanol) must be dosed. This results in high costs of chemicals as well as generation of an extra volume of sludge (which has to be taken care of), produced due to addition of organic matter.

The deammonification process with oxidation of half of the ammonia to nitrite (pathway 4) (oxygen demand based on an initial ammonium concentration is 38% of the oxygen needed for oxidation of all ammonium to nitrate) followed by Anammox in which equal amounts of ammonium and nitrite nitrogen reacts to form nitrogen gas (pathway 9) (without any need for organic material) is the least

resources consuming pathway for transfer of ammonium to nitrogen (Helmer et al., 2000; Johansson et al., 1998; Siegrist et al., 1998).

**Table 1.** Reaction routes in removal of nitrogen.

Reaction route	Main reaction	Process name	Organisms involved	Status (general knowledge)	Examples of studies at KTH
1	$C_3H_7O_2N + 5O_2 \rightarrow 4CO_2 + HCO_3^- + NH_4^+ + H_2O$	Ammonification	Bacteria	Well-known process	
2	$NH_4^+ + OH^- \rightarrow NH_3 + H_2O$	Ammonia stripping	- (physical process)	Well-known process	
3	$4CO_2 + HCO_3^- + NH_4^+ + H_2O \rightarrow C_3H_7O_2N + 5O_2$	Assimilation	Bacteria Algae (plants)	Well-known process	
4	$NH_4^+ + 1.5O_2 \rightarrow NO_2^- + 2H^+ + H_2O$	Nitritation	Nitrosomonas	Well-known process	Tendaj, 1985; Mossakowska, 1997, 1994; Plaza, 2001
5	$NO_2^- + 0.5O_2 \rightarrow NO_3^-$	Nitrataion	Nitrobacter	Well-known process	
6	$C + 2NO_3^- \rightarrow 2NO_2^- + CO_2$	Denitratation	Denitrification bacteria	Well-known process	Plaza, 1995, 1991, 1990; Trela, 2000, 1998
7	$3C + 2H_2O + 4NO_2^- \rightarrow 2N_2 + 4OH^- + 3CO_2$	Denitritation	Denitrification bacteria	Well-known process	
8	-	-	-	Difficulties ammonium nitrite, only	to ensure oxidation to
9	$NH_4^+ + NO_2^- \rightarrow N_2 + 2H_2O$	Anammox	Planctomycetales	New process observed during the last decade	Trela, 2001; La Rocca, 2001; Horeglad, 2001; Plaza et al., 2002

**Table 2.** Resources needed, alkalinity changes and energy obtained during reactions 4-7 and 9.

Reaction No	Process	Alkalinity change	Oxygen need	COD need without assimilation	COD need incl. assimilation
4	Nitritation	-2 ekv/mol N	3.43 g O <sub>2</sub> /gN	-	
5	Nitrataion	-	1.14 g O <sub>2</sub> /gN	-	
4+5	Complete nitrification	-2 ekv/mol N	4.57 g O <sub>2</sub> /gN	-	
6	Denitratation	-	-	1.72 g COD/gN	2.4 g COD/gN
7	Denitritation	-	-	1.14 g COD/gN	1.6 g COD/gN
6+7	Complete denitrification	+1 ekv/mol N +1 ekv/mol N	-	2.86 g COD/gN	4.0 g COD/gN
9	Anammox	-	-	-	
4+9	Deammonification ½ of ammonia to nitrite (reaction 4), followed by reaction 9	-1 ekv/mol N	1.72 g O <sub>2</sub> /gN	-	-
4+8+7	”Modified traditional nitrogen removal”	-1 ekv/mol N	3.43 g O <sub>2</sub> /gN	1.72 g COD/gN	2.4 g COD/gN
4+5+6+7	”Traditional nitrogen removal”	-1 ekv/mol N	4.57 g O <sub>2</sub> /gN	2.86 g COD/gN	4.0 g COD/gN

## MATERIALS AND METHODS

### Description of the laboratory-scale pilot plant

The laboratory-scale pilot plant (Figure 2, Table 3) consisted of two reactors, which were filled with Kaldnes rings as carrier material for fixed film growth. Each reactor was equipped with mixer, aeration system (at the beginning of the experiment) and thermostat. The pH level was corrected with a continuous dosage of  $\text{Na}_2\text{CO}_3$  solution to the first reactor.

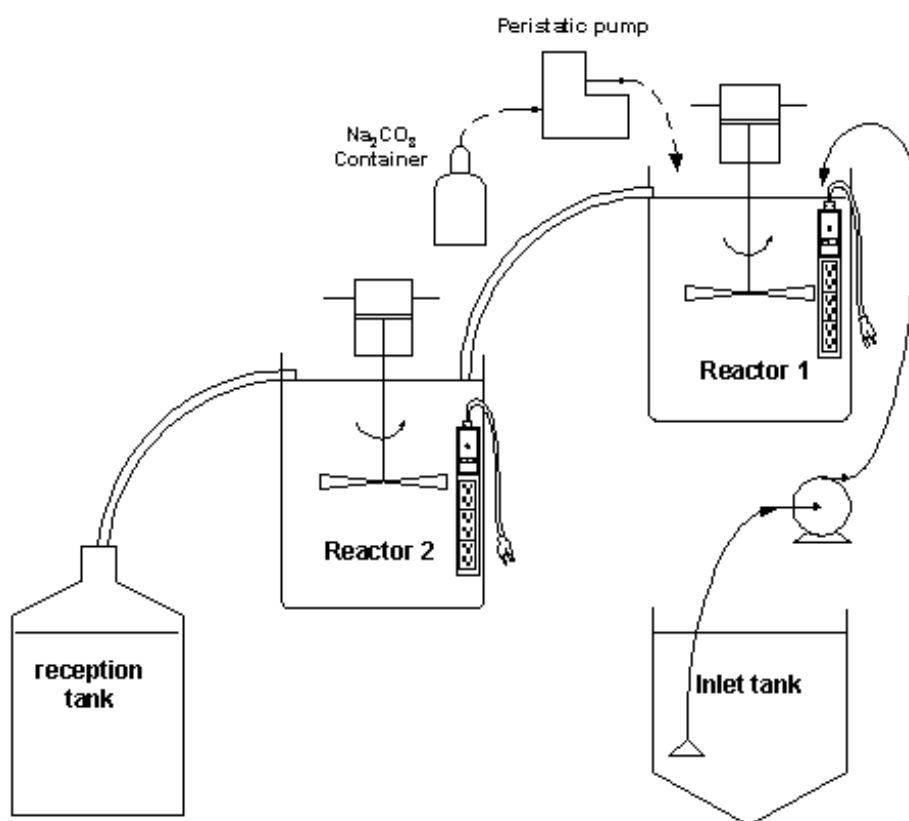


Figure 2. Laboratory-scale pilot plant scheme.

Table 3. Design parameters of laboratory-scale pilot plant.

PARAMETER	VALUE	
	Reactor 1	Reactor 2
Volume ( $\text{dm}^3$ )	8	7.5
Volume of Kaldnes filling (%)	20	20
Kaldnes area ( $\text{m}^2$ )	0.8	0.75

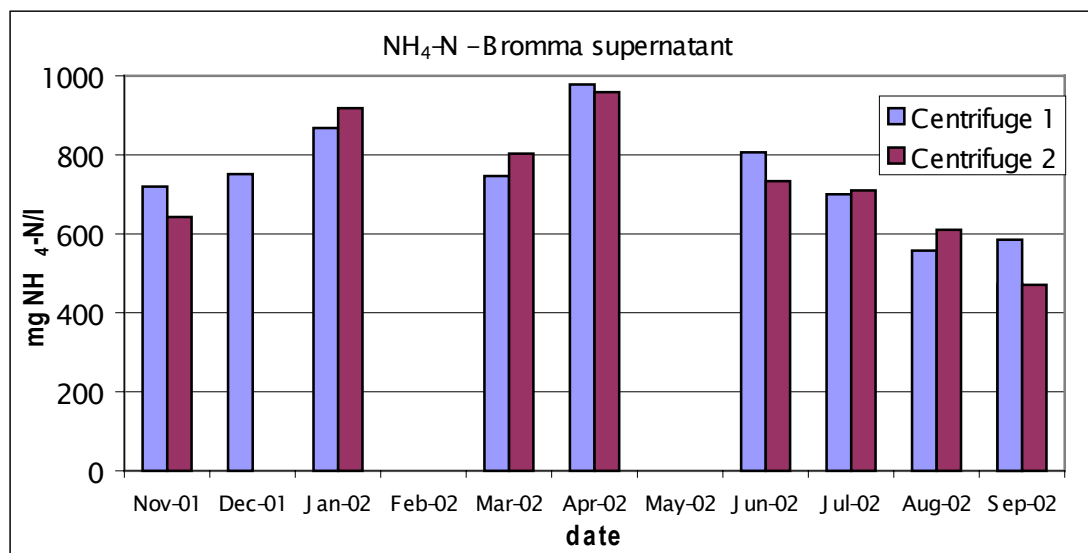
Table 4. Characteristics of supernatant from the Bromma WWTP.

	COD $\text{mg O}_2/\text{l}$	$\text{NH}_4\text{-N}$ $\text{mg N/l}$
Mean value	273.1	724.0
Minimum	150.0	470.6
Maximum	391.0	978.0
Standard deviation	71.7	150.9

The pilot plant was continuously fed with supernatant from dewatering of the digested sludge at the Bromma WWTP. Supernatant was analysed for  $\text{NH}_4\text{-N}$  and COD (filtrated) (Table 4). A big difference between the maximum and minimum concentration of ammonium nitrogen could be

observed (Figure 3). Due to the fact that supernatant was taken from two centrifuges there are two readouts for each collection date.

The concentration of ammonia nitrogen at the pilot plant inlet varied around 200 mg NH<sub>4</sub>-N/l during the period of experiment. To obtain the required concentration, supernatant was diluted with tap water.



**Figure 3.** NH<sub>4</sub>-N concentration in supernatant from Bromma WWTP.

### Measurements and sampling procedure

Samples were collected twice a week, both from the inlet and outlet of each reactor. They were analysed for NH<sub>4</sub>-N, NO<sub>3</sub>-N and NO<sub>2</sub>-N. The parameters such as dissolved oxygen concentration, temperature and pH value were measured every day. Nitrogen forms in the samples were analysed with TECATOR – AQUATEC 5400 ANALYZER. The COD samples were analysed with HACH DR/2010. A dissolved oxygen concentration was determined using RUSSELL model: RL425 while the pH level was determined with ORION model: 210A equipment.

### Operational parameters

The laboratory-scale pilot plant was operating in the continuous mode under specific conditions of temperature, pH and dissolved oxygen concentration. Temperature in reactor 1 was kept in a steady way. There were no difficulties in keeping this parameter around 31.9°C (average value). The minimum temperature in reactor 1 was 30.2°C and the maximum 32.4°C. An average temperature in reactor 2 was 29.8°C with the minimum value of 29.1°C and the maximum of 31.5°C.

Contrary to temperature, the dissolved oxygen concentration appeared to be the most sensitive parameter. Even though it was supposed to be maintained at levels lower than 0.5 mg O<sub>2</sub>/l, there were much higher values measured. At the beginning there was no significant problem with the DO concentration in reactor 1. In next period, there were a few cases in which the DO value exceeded 0.5 mg O<sub>2</sub>/l. Despite the lowest possible speed of stirring, DO levels were kept high. The maximum concentration reached over 3 mg O<sub>2</sub>/l.

The pH had to be corrected during the operational period due to consumption of alkalinity. Although the acid was balanced by the counter-ion of ammonium, sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) was used continuously for pH correction in reactor 1. Average values of pH in both reactors were 8.1.

Experiment was conducted with the influent ammonium nitrogen concentration around 150-200 mg  $\text{NH}_4\text{-N/l}$ . An average value of ammonium surface load (ASL) calculated for the pilot plant during the experiment was  $0.92 \text{ g NH}_4\text{-N/m}^2\cdot\text{d}$  with a maximum value of  $1.2 \text{ g NH}_4\text{-N/m}^2\cdot\text{d}$ . In the last two months of the experiment ASL was quite stable in reactor 1 and its average value was  $1.05 \text{ g NH}_4\text{-N/m}^2\cdot\text{d}$ .

## RESULTS AND DISCUSSION

### Analysis of nitrogen compounds

**REACTOR 1.** Figure 4 presents variation of nitrogen forms in reactor 1. Over near a ten-month period  $\text{NH}_4\text{-N}$  concentration in the influent to the first reactor varied from 108 mg  $\text{NH}_4\text{-N/l}$  to 240 mg  $\text{NH}_4\text{-N/l}$ ; an average value was 185 mg  $\text{NH}_4\text{-N/l}$ . During the initial weeks of analysed period (about 3 weeks) of the experiment the process of partial nitrification was observed. Afterwards,  $\text{NH}_4\text{-N}$  was utilized to a high degree, with very low (near 0 mg/l) concentrations of  $\text{NO}_2\text{-N}$  and  $\text{NO}_3\text{-N}$  in effluent, what indicated the presence of the deammonification process. Such conditions were maintained for almost one and a half months, until the 28<sup>th</sup> of October 2001. From October 2001 to the end of January 2002 very high dissolved oxygen concentrations (on average 1.2 mg  $\text{O}_2\text{/l}$ ) caused an undesirable change in the process performance and the nitrification process occurred. This experience proved that the process was very sensitive to a lift of DO concentrations. Every increase of this parameter resulted in oxidation of  $\text{NO}_2\text{-N}$  into  $\text{NO}_3\text{-N}$ . On the 25<sup>th</sup> of January 2002 the dissolved oxygen concentration came back to the optimal level for the development of the deammonification process, which started to get more stabilized. It was possible to significantly suppress oxidation of  $\text{NO}_2\text{-N}$  to  $\text{NO}_3\text{-N}$ . Low nitrite and nitrate nitrogen concentrations together with a decrease in the  $\text{NH}_4\text{-N}$  concentration are the proof of the deammonification process in the first reactor. However, fluctuations in effluent  $\text{NH}_4\text{-N}$  concentrations were observed.

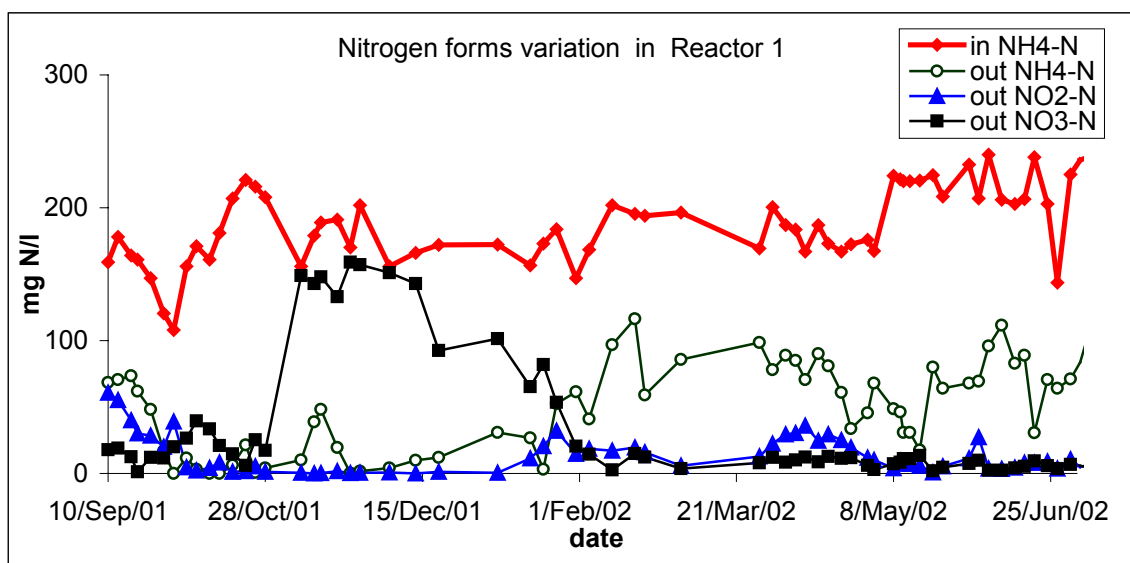


Figure 4. Nitrogen forms variation in reactor 1.

Analysing  $\text{NO}_2\text{-N}$  concentration in reactor 1, it can be noticed that only during the first month of the experimental phase  $\text{NO}_2\text{-N}$  concentration was relatively high. Afterwards, at higher DO concentration,  $\text{NO}_2\text{-N}$  was only an intermediate product of the nitrification process, so it was not accumulated. When the deammonification process stabilized,  $\text{NO}_2\text{-N}$  concentration was fluctuating to some degree, but within the acceptable range (from 0.8 mg  $\text{NO}_2\text{-N/l}$  to 36 mg  $\text{NO}_2\text{-N/l}$ ).

The  $\text{NO}_3\text{-N}$  concentration has been growing due to an increase of the dissolved oxygen concentration. From November 2001 to the end of January 2002, the nitrification process caused accumulation of  $\text{NO}_3\text{-N}$  in reactor 1. Except for that period  $\text{NO}_3\text{-N}$  concentrations were low. It is interesting to notice that after a period of high nitrate nitrogen concentrations the process gradually came back to a required course after changing process conditions, not reaching values higher than 31 mg  $\text{NO}_3\text{-N/l}$ .

REACTOR 2. Nitrogen forms variation in reactor 2 is shown in Figure 5. Behaviour of the bacterial culture in reactor 2 was affected not only by  $\text{NH}_4\text{-N}$  but also by  $\text{NO}_2\text{-N}$  and  $\text{NO}_3\text{-N}$  in the effluent from reactor 1. Considerable utilization of  $\text{NH}_4\text{-N}$  in the first reactor enabled an almost complete deammonification process in reactor 2. In September 2001 the influent  $\text{NH}_4\text{-N}$  concentration was gradually decreasing. At the same time there were changes in  $\text{NO}_2\text{-N}$  and  $\text{NO}_3\text{-N}$  concentrations. From October 2001 to the end of January 2002 higher concentrations of dissolved oxygen had a big impact on the process performance. Then, when the problem with too intensive oxidation inside of the reactors was solved, the process became stable and the bacterial culture adapted to new conditions. Since the middle of February 2002 the deammonification process appeared to dominate in the second reactor. Due to decreased removal of  $\text{NH}_4\text{-N}$  in reactor 1 much bigger quantity of  $\text{NH}_4\text{-N}$  passed to reactor 2, where the microorganisms were able to utilize it to a high degree.

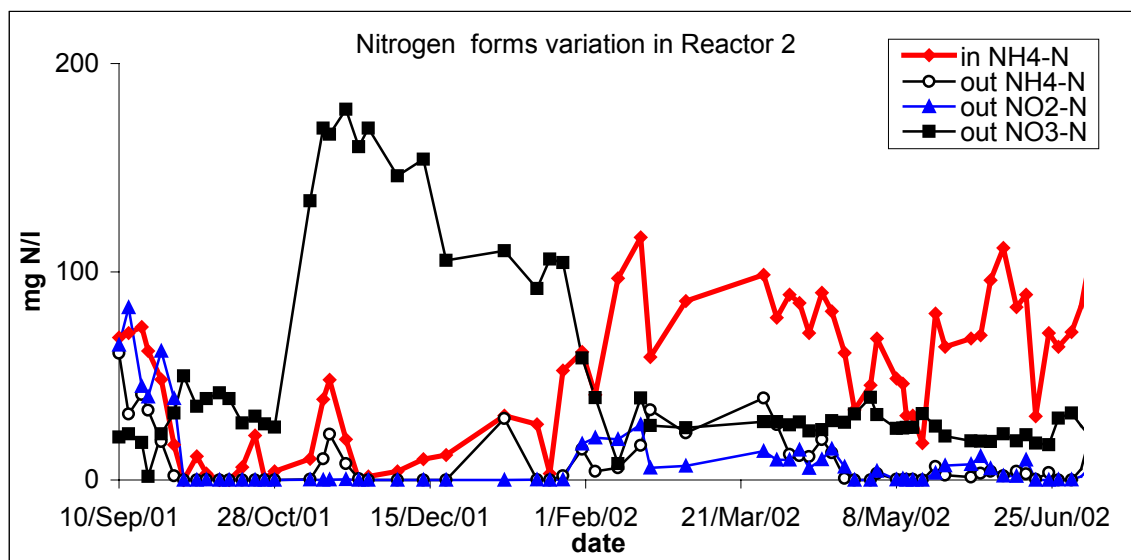


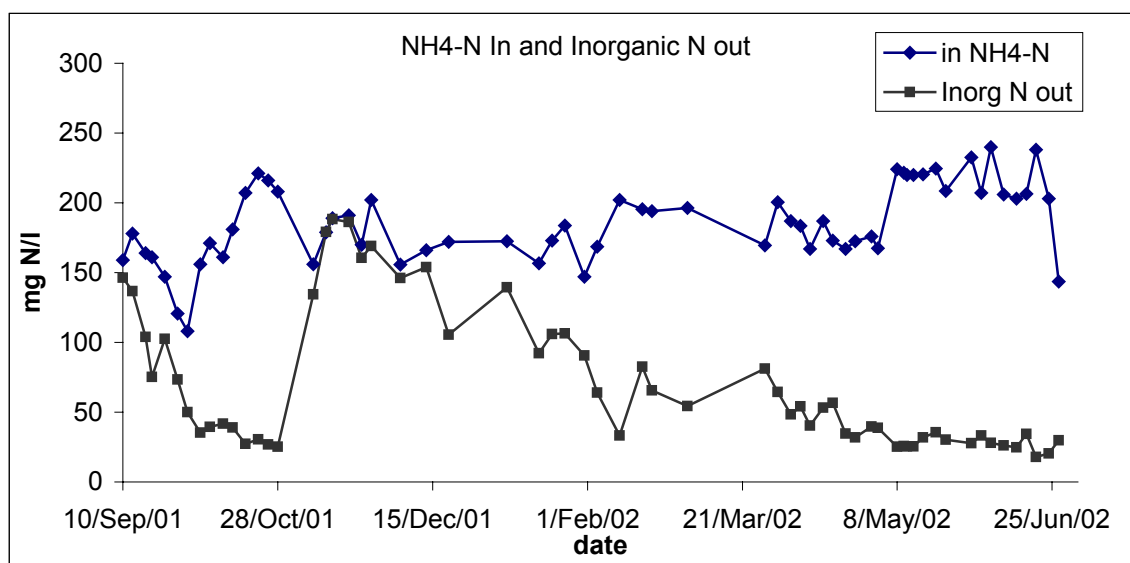
Figure 5. Nitrogen forms variation in reactor 2.

Looking into  $\text{NO}_2\text{-N}$  concentrations in reactor 2, it can be observed that only during September 2001 the  $\text{NO}_2\text{-N}$  concentration was quite high. On the first day of the experiment  $\text{NO}_2\text{-N}$  concentration of 60.8 mg  $\text{NO}_2\text{-N/l}$  was detected. In the following months,  $\text{NO}_2\text{-N}$  concentrations did not exceed 30 mg  $\text{NO}_2\text{-N/l}$ .  $\text{NO}_2\text{-N}$  was almost completely utilized in the deammonification process. However, during the period of high DO concentrations  $\text{NO}_2\text{-N}$  was oxidized to  $\text{NO}_3\text{-N}$ , as a part of the nitrification process.

In opposite to rather stable  $\text{NO}_2\text{-N}$  concentrations, the amount of  $\text{NO}_3\text{-N}$  was varying significantly (Figure 5). It was mainly caused by changes in DO concentration. Similar as for reactor 1 during the period November – January  $\text{NO}_3\text{-N}$  formation was enhanced. As soon as the problem with aeration was solved, nitrate concentrations decreased radically. Comparing the graphs of  $\text{NO}_3\text{-N}$  concentrations for both reactors, it can be noticed that they are very similar. This corresponds to the fact that higher dissolved oxygen concentrations happened at the same time in both reactors. Moreover, at that time reactor 2 was receiving the effluent from reactor 1 that was already rich in  $\text{NO}_3\text{-N}$ .

### Nitrogen removal efficiency

The deammonification process efficiency can be estimated by comparing the influent  $\text{NH}_4\text{-N}$  concentration with the concentration of inorganic nitrogen in the effluent from the pilot plant (Figure 6). There is a progressive alternation of the nitrogen removal process in the pilot plant. At first, until the end of October 2001 inorganic nitrogen at the outlet was only slightly lower than the ammonium concentration at the inlet. The effluent inorganic nitrogen concentration gradually decreased, even when the incoming ammonium concentration started to rise. During the next period due to increase in the DO concentration  $\text{NO}_3\text{-N}$  was produced in a high amount. It resulted in a sizable increase of the sum of inorganic nitrogen at the outlet. Afterwards, a gradually increasing gap between the influent  $\text{NH}_4\text{-N}$  concentration and the sum of inorganic nitrogen in the effluent has been noticed. Finally the process came to a stable mode. Though  $\text{NH}_4\text{-N}$  concentrations varied inorganic nitrogen at the outlet was steadily decreasing.



**Figure 6.**  $\text{NH}_4\text{-N}$  concentration at the inlet and inorganic nitrogen concentration at the outlet.

The quantity of inorganic nitrogen at the outlet proved that the deammonification process took place in the pilot plant. Influent  $\text{NH}_4\text{-N}$  concentrations increased to the average value of 214 mg  $\text{NH}_4\text{-N/l}$  during the last two months of the experiments while inorganic nitrogen in the effluent was very low and the mean value was 28 mg  $\text{N/l}$ . Concluding, it was proved that a stable deammonification process was achieved at the pilot plant. This can be clearly observed in Figure 7.



The results obtained for May and June 2002 assured the process stability and high activity of the bacterial culture. The overall nitrogen removal efficiency of the deammonification process in the pilot plant was about 85 % and reached the maximum value as 92 %.

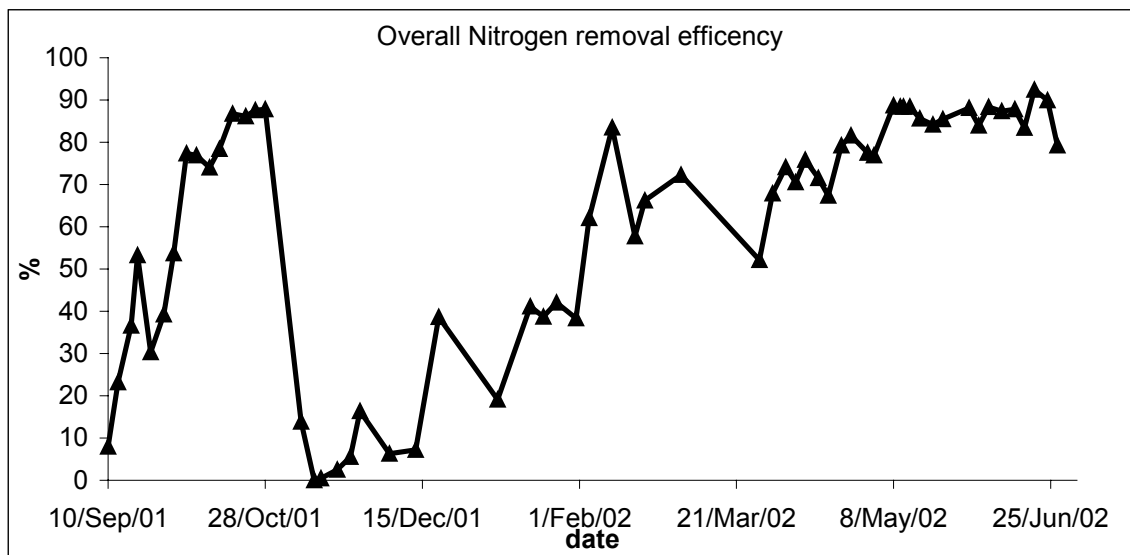


Figure 7. Overall nitrogen removal efficiency in the pilot plant.

## CONCLUSIONS

Performed laboratory-scale pilot plant studies enabled to evaluate new methods for biological removal of nitrogen in digester supernatant. This wastewater is characterised by a high ammonium nitrogen concentration and low BOD/nitrogen ratio.

The results from the experiments indicated that deammonification was the main mechanism for the nitrogen removal. Deammonification is defined as a two-step reaction: nitrification and anaerobic ammonium oxidation. The studies have shown that it is possible to significantly suppress the nitrate formation (to get only nitrification process) by use of low oxygen concentration (below 0.5 mg O<sub>2</sub>/l) in combination with pH-values around 8.1 at the temperature of about 30°C. Produced nitrite reacted with ammonium into N<sub>2</sub> in anaerobic ammonium oxidation (anammox process). Results from experiments proved that nitrogen removal could be obtained in one biofilm system at low DO concentration in the reactor. In both reactors of the pilot plant nitrification and Anammox processes took place. Evidently, Anammox was more efficient in the reactor 2 than in reactor 1, and that led to higher nitrogen removal efficiency in reactor 2.

It was difficult to get the stable process conditions during the experiment. At some periods the low difference between the influent ammonium nitrogen and the effluent inorganic nitrogen indicated that deammonification was insignificant. The main parameter influencing the process' performance was oxygen concentration in the reactor. It seems that deammonification is impaired by oxygen concentrations above about 0.5 mg O<sub>2</sub>/l. However, when the stable conditions have been secured, the nitrogen removal efficiency increased and during the last two months of this period was around 85 %, reaching even maximum value of 92 %.

Finally, it can be concluded that studies proved that it is possible to run deammonification as a continuous process for a long period (several months) with stable results. The process needs further

investigations both in reaction kinetics as in process applications. As dissolved oxygen concentration is crucial for the efficient performance of the deammonification process special focus should be put on the proper oxygen control system. In full-scale applications a proper DO control along with variable ammonium load is absolutely essential to obtain a good N-removal efficiency.

## ACKNOWLEDGEMENTS

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