Construction, start-up and operation of a continuously aerated laboratory-scale SHARON reactor in view of coupling with an Anammox reactor

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Abstract

In this study practical experiences during start-up and operation of a laboratory-scale SHARON reactor are discussed, along with the construction of the reactor. Special attention is given to the start-up in view of possible toxic effects of high nitrogen concentrations (up to 4 000 mgN·ℓ^{-1}) on the nitrifier population and effects of inoculation with sludge from an SBR reactor operated under completely different conditions. Because of these considerations, the reactor was first operated as an SBR to prevent biomass washout and to allow the selection of a strong nitrifying population. A month after the inoculation the reactor was switched to normal chemostat operation. As a result the nitrite oxidisers were washed out and only the ammonium oxidisers persisted in the reactor.

In this contribution also some practical considerations concerning the operation of a continuously aerated SHARON reactor, such as mixing, evaporation and wall growth are discussed. These considerations are not trivial, since the reactor will be used for kinetic characterisation and modelling studies. Finally the performance of the SHARON reactor under different conditions is discussed in view of its coupling with an Anammox unit. Full nitrification was proven to be feasible for nitrogen loads up to 1.5 gTAN-N·ℓ^{-1}·d^{-1}, indicating the possibility of the SHARON process to treat highly loaded nitrogen streams. Applying different influent concentrations led to different effluent characteristics indicating the need for proper control of the SHARON reactor.

Keywords: SHARON, nitrification, start-up, control

Nomenclature

Anammox	=	anaerobic ammonium oxidation
b_{NH}	=	decay rate for ammonium oxidisers
C^{in}	=	influent concentration
C^{out}	=	effluent concentration
DO	=	dissolved oxygen
HRT	=	hydraulic residence time
K_e^{NH}	=	ammonia/ammonium equilibrium constant
K_e^{NO}	=	nitrite/nitrous acid equilibrium constant
i_{nbm}	=	nitrogen content of the biomass
N^{nitr}	=	amount of TAN nitrified
SBR	=	sequencing batch reactor
SHARON	=	single reactor high activity ammonia removal over nitrite
SRT	=	sludge residence time
TAN	=	total ammonia nitrogen (TAN = NH_4^+ + NH_3)
TIC	=	total inorganic carbon (TIC = CO_3^{2-} + HCO_3^- + H_2CO_3)
TNO_2	=	total nitrite nitrogen (TNO_2 = NO_2^- + HNO_2)
TSS	=	total suspended solids
X_{NH}	=	concentration of ammonium oxidisers
X^{out}	=	effluent biomass concentration
Y_{NH}	=	growth yield for ammonium oxidisers

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Introduction

Partial nitrification techniques, such as the continuously aerated SHARON process, have been denoted for quite a while as very promising for improved sustainability of wastewater treatment (Abeling and Seyfried, 1992). Conventionally nitrogen removal in these wastewaters is achieved using nitrification/denitrification. In such systems, nitrifying bacteria oxidise ammonium to nitrate under oxic conditions, and nitrate is subsequently or simultaneously reduced to di-nitrogen gas, under anoxic conditions. Recently however, novel processes for nitrogen removal were developed, for example the combined SHARON-Anammox process (Van Dongen et al., 2001a;b).

In the SHARON (Single reactor High activity Ammonia Removal Over Nitrite) process, partial nitrification of ammonium to nitrite is established by working at high temperature (above 25°C) and maintaining an appropriate sludge retention time (SRT) of 1 to 1.5 d, so that ammonium oxidisers are maintained in the reactor, while nitrite oxidisers are washed out and further nitrification of nitrite to nitrate is prevented as explained in detail below. In this way, significant aeration cost savings are realised in comparison with conventional nitrification to nitrate. The SHARON process is very suitable to reduce the load of streams with high ammonium concentration (~1 gTAN-N·ℓ^{-1}), rather than to meet strict effluent standards. It is typically applied for treating sludge digestion reject water in order to relieve the main wastewater treatment plant (WWTP) to which this stream is subsequently recycled. A full-scale SHARON process

is operating since January 1999 at the Rotterdam Sluisjesdijk sludge treatment plant (Van Kempen et al., 2001).

The nitrite produced in the SHARON process can be used as an electron acceptor for the oxidation of the remainder of the TAN by the recently discovered Anammox organisms (ANaerobic AMMonium OXidisers), that combine almost equimolar amounts of TAN and TNO_2 to form nitrogen gas (Jetten et al., 1999).

Benefits of the combined SHARON-Anammox process compared to the SHARON process with denitrification are the reduction by 50% of the aeration costs, since only half of the ammonium is converted, the omission of the need for additional carbon source, the virtual absence of sludge production and the possibility to obtain low nitrogen effluent concentrations through the subsequent autotrophic Anammox reaction. The latter has been an inspiring starting point for the development of more sustainable municipal wastewater treatment systems (Jetten et al., 1997).

An experimental study on the treatment of ammonium-rich wastewater by the combined SHARON-Anammox process performed by Van Dongen et al. (2001a) showed that the combined SHARON-Anammox system can perform stably over long periods and that the process is ready for full-scale implementation.

The SHARON process in detail

The continuously aerated SHARON process consists basically of a completely mixed reactor without sludge retention, operating at high temperature (above 25°C). The maximum specific growth rates at 20°C of both populations are approximately the same ($0.8 \cdot d^{-1}$ and $0.79 \cdot d^{-1}$ respectively) but the activation energies differ (68 and 44 $kJ \cdot mol^{-1}$) (Hao et al., 2002). Hence the maximum specific growth rate of the ammonium oxidisers will increase faster than the maximum specific growth rate of the nitrite oxidisers. Nitrite oxidisers can thus be washed out by sufficiently lowering the HRT, so that the dilution rate, which is the inverse of the HRT, becomes higher than the growth rate of nitrite oxidisers but lower than the growth rate of ammonium oxidisers. Consequently nitrite oxidisers will then not be able to persist in the reactor.

Not only washout of the nitrite oxidisers has to be accomplished, but also only half of the ammonium has to be oxidised to nitrite in order to produce an Anammox-suited effluent. This is accomplished in the following way. Oxidation of 1 mole of ammonium to nitrite produces 2 moles of protons according to Eq. (1).

$$NH_4^+ + \frac{3}{2}O_2 \rightarrow NO_2^- + 2H^+ + H_2O \qquad (1)$$
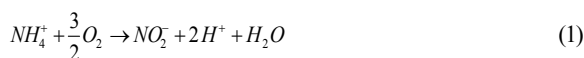
This production of protons leads to a significant pH decrease and consequently a stop in nitrification especially with highly loaded nitrogen streams that are generally treated by the SHARON reactor. However, normally SHARON influent contains bicarbonate next to ammonium. This bicarbonate is stripped by the air in the form of CO_2:

$$H^+ + HCO_3^- \rightarrow CO_2 + H_2O \qquad (2)$$
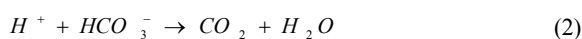
This means that for every mole of ammonium oxidised, 2 moles of bicarbonate are stripped. In case the SHARON influent has a molar TIC:TAN ratio of 1:1, the protons produced during conversion of half of the ammonium are equal to the protons taken up via carbon dioxide stripping. Hence, ammonium oxidation stops at 50% conversion due to acidification and an Anammox-

suited effluent is produced.

However, the influent TIC:TAN ratio is not always 1:1. For example, data from Izzet et al. (1991) show that this ratio varies between 1.02 and 1.44 with an average of 1.2 for sludge digestion reject water. This type of wastewater is typically used as an example for SHARON influent. This begs the question: What will be the result of this varying TIC:TAN ratio on the reactor performance. The influent ammonium concentration will also vary over time affecting reactor performance. Process control therefore seems necessary since the Anammox reactor benefits from a constant influent composition.

In this contribution the experimental performance of the SHARON reactor under different influent conditions is discussed in view of its coupling with an Anammox unit. Practical experiences during start-up and operation of a lab-scale SHARON reactor are also presented. The results of this study can be used in a simulation and control study similar to the study performed by Wyffels et al. (2004). Therefore the results will also be discussed in view of this study.

Materials and methods
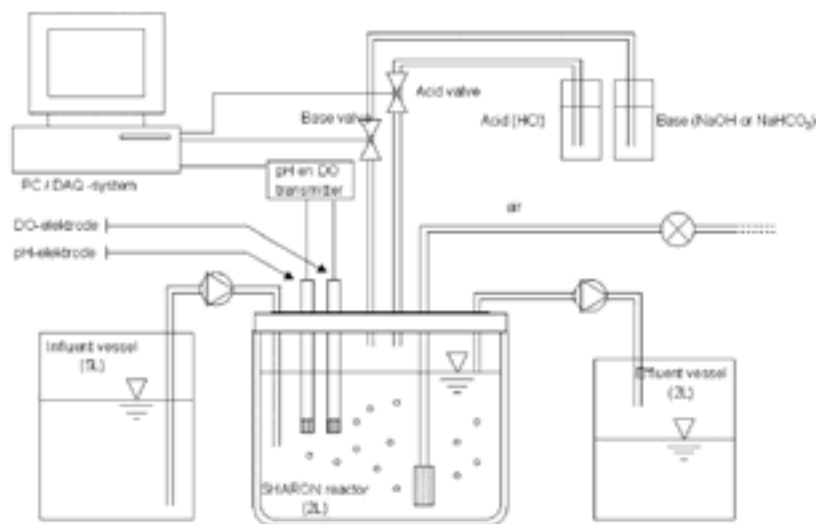
The SHARON reactor

Hardware and software

The SHARON reactor is a 2 ℓ continuously stirred tank reactor (CSTR) without biomass retention. Its schematic representation is shown in Fig. 1. The synthetic influent is pumped with a peristaltic pump from the 5 ℓ influent vessel to the reactor. The pump flow rate of this influent pump determines both the HRT and the SRT, since both residence times are equal and defined as the ratio of the volume to the flow rate. The flow rate can vary between 0.2 to 6 ℓ/d, which gives the possibility of operating at HRTs of between 10 and 0.33 d. The lower limit of the applied HRT is, however, determined by the growth rate of the ammonium oxidisers.

The effluent is pumped out of the reactor with a second peristaltic pump that operates at a higher flow rate than the first pump. The influent flow rate is, however, equal to the actual effluent flow rate since the withdrawal point is situated at the 2 ℓ mark. The reactor is aerated through a pumice stone using air from a compressor (1 bar overpressure). The temperature of the reactor can be controlled at between 20°C and 70°C, although the normal operating temperature is 35°C, as is usual for the SHARON process. In the reactor the dissolved oxygen (DO) and the pH are measured every 10 s using the Labview® software (National Instruments, www.ni.com). The pH is controlled by addition of acid (HCl) and base ($NaHCO_3$ or NaOH) based on the measured pH. A set-point is defined together with a pH boundary. If the pH exceeds this boundary a valve is briefly opened and acid or base is dosed. More details concerning the hardware and software of the SHARON reactor, in particular the structure of the Labview® software can be found in Van Hulle et al. (2003).

Inoculum

Two different inocula were tried as inoculum for the SHARON reactor. First, an inoculum from the SHARON reactor of the WWTP of Rotterdam (Mulder et al., 2001) was used. This SHARON reactor was operating under alternating oxic/anoxic conditions. These organisms are already adapted to the short residence times and high nitrogen concentrations typical for the SHARON process. Secondly, an inoculum from the sequencing batch reactor (SBR) in the BIOMATH lab (Lee and Vanrolleghem, 2003)

Figure 1
Schematic representation of the lab-scale SHARON reactor used in this study

<table>
<tr><td colspan="2" align="center">TABLE 1
Composition of the synthetic influent of the
SHARON reactor used in this study</td></tr>
<tr><th>Main compounds</th><th>Concentrations [mg·ℓ⁻¹]</th></tr>
<tr><td>$(NH_4)_2SO_4$</td><td>Depends on experiment; 1 000 mg TAN-N·ℓ⁻¹ = 4 714 mg $(NH_4)_2SO_4$·ℓ⁻¹</td></tr>
<tr><td>$NaHCO_3$</td><td>Depends on experiment; 1 000 mg C·ℓ⁻¹ = 6 994 mg $NaHCO_3$·ℓ⁻¹</td></tr>
<tr><td>KH_2PO_4</td><td>1 000</td></tr>
<tr><td>$MgSO_4.7H_2O$</td><td>600</td></tr>
<tr><td>Trace compounds</td><td></td></tr>
<tr><td>$FeSO_4.7H_2O$</td><td>15</td></tr>
<tr><td>$PbCl_2$</td><td>3.4</td></tr>
<tr><td>$ZnCl_2$</td><td>7.2</td></tr>
<tr><td>$Cr(NO_3)_3.9H_2O$</td><td>8</td></tr>
<tr><td>$CuCl_2.2H_2O$</td><td>6</td></tr>
<tr><td>$MnSO_4.H_2O$</td><td>19</td></tr>
<tr><td>$NiSO_4.6H_2O$</td><td>2</td></tr>
<tr><td>$CoCl_2.6H_2O$</td><td>3.4</td></tr>
<tr><td>$(NH_4)_6Mo_7O_{24}.4H_2O$</td><td>4.8</td></tr>
<tr><td>$CaCl_2$</td><td>5</td></tr>
<tr><td>EDTA</td><td>150</td></tr>
</table>

was used. Since the organisms in the SBR are not adapted to high nitrogen concentrations, special attention was given to the start-up with this inoculum.

Influent

The reactor is fed with synthetic influent with composition given in Table 1. The ammonium and bicarbonate concentrations vary according to the type of experiment conducted. The influent concentrations used were 500, 1 000, 2 000 and 4 000 mgTAN-N·ℓ⁻¹. The trace element composition is according to Visniac and Santer (1957) with the addition of Pb^{2+}, Cr^{3+} and Ni^{2+} as adjusted by Capalozza (2001).

Microscopy

The microscopic observations were performed by using an optical microscope, Olympus CX40 (Olympus, Japan) equipped with a video camera Ikegami ICD-46E (Ikegami Electronics Inc., USA). A drop of mixed liquor was carefully deposited on a glass slide and covered with a cover slip before being observed through the microscope.

Chemical analyses

Concentrations of TAN, TNO_2 and NO_3^- were analysed on a daily basis using spectrophotometric methods (Dr Lange GmbH, Germany). TSS (total suspended solids) concentrations were determined according to *Standard Methods* (1992). DO was measured by an Ingold (Mettler Toledo) Clarck type oxygen electrode and pH was measured with a glass electrode (Mettler Toledo HA 405-DXK-S8/120).

Results and discussion

### Start-up of the SHARON reactor: Fast method vs. slow method

Fast start-up method

Initially the reactor was inoculated with sludge from the SHARON reactor of Rotterdam. In order to start up in a fast way the reactor was set in CSTR mode with an HRT of 2.5 d after a 24 h lasting adaptation period of the biomass to the reactor. The pH and temperature were controlled at 6.9 and 35°C respectively. Different influent concentrations, ranging from 300 to 800 mg TAN-N·ℓ⁻¹, were used, but all start-ups had the same outcome. As an example the results of a start-up with an influent concentration of 300 mg TAN-N·ℓ⁻¹ are shown in Fig. 2. During the first 3 d all incoming TAN is oxidised to nitrate. After approximately 1 SRT TNO_2 starts to build up in the effluent, indicating the successful washout of the nitrite oxidisers. After 3 SRT, however, TAN builds up in the effluent, indicating the washout of ammonium oxidisers too. From Fig. 2 it can thus be concluded that directly imposing short residence times on the nitrifying organisms coming from this full-scale SHARON reactor did not result in a stable operation of the SHARON process in contrast to the findings of Van Dongen et al. (2001a; b). Toxic effects of ammonia and nitrous acid (Anthonisen et al., 1976) can be put forward as a possible explanation.

Slow start-up method

Since the fast start-up method proved to be unsuccessful, a slow start-up method was tested. This time inoculum from an SBR reactor was used. Special attention was given to the start-up in view of possible toxic effects on the nitrifier population originating from an SBR reactor operated under completely different conditions (T=15°C, SRT = 10 d, influent load = 9 mg TAN-N·ℓ⁻¹·d⁻¹). The ammonium oxidisers were therefore allowed to adapt slowly to the changed conditions (Van Den Broeck et al., 2004).

The SHARON reactor was first operated as an SBR to prevent biomass washout, while the influent TAN load was increased step-wise from 600 to 1 480 mgTAN-N·ℓ⁻¹·d⁻¹. The temperature
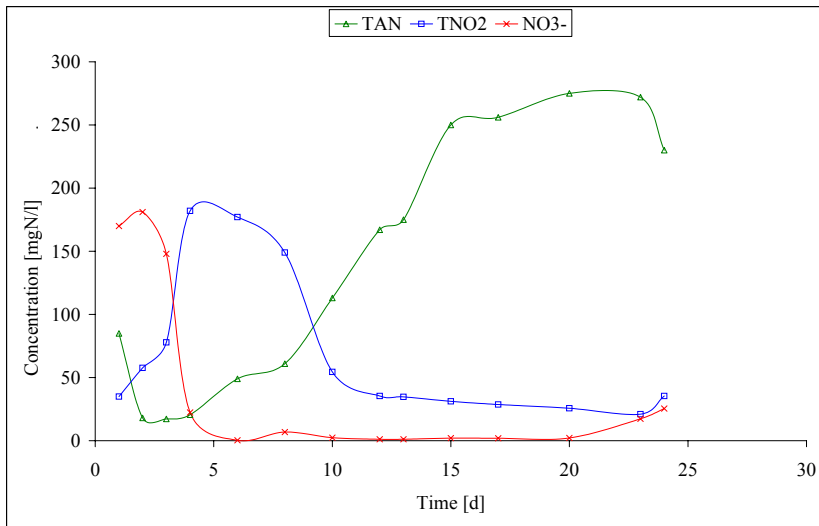
Figure 2
Fast start-up method for the SHARON reactor: Evolution of TNO_2 (□), NO_3^- (x) and TAN (▽), indicating the washout of nitrifying organisms
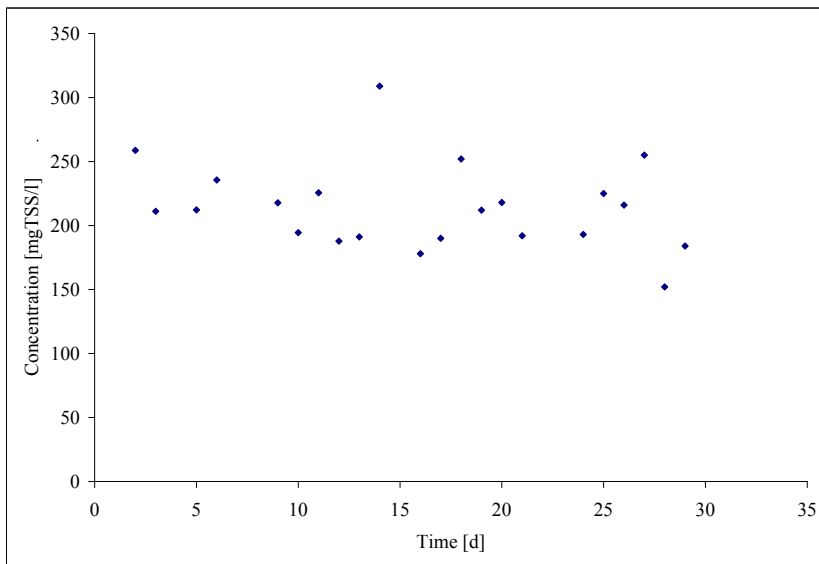
Figure 3
TSS concentrations for a 1-month period that the SHARON reactor was operated with an influent concentration of 2 000 mg TAN-N·ℓ^{-1}, indicating the low biomass concentration

too was increased step-wise from 23.4°C to 35°C. The pH was fixed at 7.1. Every 12 h the sludge was allowed to settle and the effluent was withdrawn. A month after the inoculation of the reactor, a stable nitrifying population was established since all incoming TAN was oxidised to nitrate. The reactor was then switched to normal chemostat operation with an SRT of 2.7 d. This time the nitrite oxidisers were washed out since the incoming TAN was now oxidised to TNO_2 only and no nitrate was formed. After start-up the reactor was operated as discussed in the next paragraph.

Concerning the slow start-up method, it appears more appropriate to start out with a general nitrifying sludge instead of a dedicated SHARON sludge, since the former one is more readily available. However, even though the fast start-up method was unsuccessful, probably the slow start-up method would have worked also with the SHARON sludge.

Practical considerations concerning the SHARON reactor

Apart from the careful start-up, some other practical considerations can be pointed out when operating a continuously aerated SHARON reactor at high temperatures.

Indeed, the conditions in the reactor have to be known as accurately as possible in order to compare experimental results with modelling results. For instance, evaporation and wall growth, among others, can hinder the interpretation of experimental results.

Low biomass concentration

The reactor is designed to operate at an effluent TAN:TNO_2 ratio of 1:1 and an HRT of 1.54 d, although the HRT at start-up was 2.7 d. For an influent TAN concentration of 2 000 mg TAN-N·ℓ^{-1} the amount of TAN nitrified would then be 1 000 mg TAN-N·ℓ^{-1} (N^{nitr}). According to Petersen et al. (2003) the concentration of ammonium oxidisers (X_{NH}) in the reactor can be calculated by Eq. (3):

$$X_{NH} = Y_{NH} \frac{SRT}{HRT} \frac{N^{nitr}}{1 + b_{NH} SRT}:$$

$$= 0.15 \frac{1.54}{1.54} \frac{1000}{1 + 0.1 \cdot 1.55} \approx 130 \, mgCODL^{-1} \quad (3)$$

where:
  Y_{NH} the growth yield for ammonium oxidisers on TAN (mg COD mgTAN-N·ℓ^{-1})
  b_{NH} the decay rate for ammonium oxidisers (d^{-1}) (Wiesmann, 1994).

The combination of this low ammonium oxidiser concentration and the absence of other biomass in the reactor (since synthetic influent with only TAN and no carbon source is used) results in a reactor operation that is very sensitive to disturbances. Any disturbance can only be dealt with by the ammonium oxidisers and can lead to the malfunctioning of the reactor.

The low biomass concentration is also reflected in the low TSS concentrations. As an example the TSS values for a 1-month period in which the SHARON reactor was operated with an influent concentration of 2 000 mg TAN-N ℓ^{-1} are depicted in Fig. 3.

Evaporation

Water evaporation is not negligible and can, depending on the air flow rate, amount to more than 20% of the influent flow when operating a 2 ℓ lab-scale reactor at 35°C. The effect of evaporation was detected because, assuming that the influent and effluent flow rate were the same, the nitrogen mass balance over the reactor was skewed. The nitrogen concentration (in the form of TNO_2, nitrate and TAN and incorporated in the biomass) coming out of the reactor was higher than the nitrogen concentration in the reactor, as expressed by Eq. (4):

$$C_{TAN}^{in} \leq \left(C_{TAN}^{out} + C_{TNO_2}^{out} + C_{NO_3^-}^{out} \right) + i_{nbm} X^{out} \quad (4)$$

where:

C^in the concentration of TAN in the influent

C^out the concentration of TAN, TNO_2 and nitrate in the effluent

i_{nbm} the nitrogen content of the biomass

X^out the biomass concentration in the effluent.

This difference could only be explained by evaporation since numerous tests and dilution series were performed to exclude measurement errors. Because of this evaporation the influent and effluent flow rates would differ. This evaporation was also noticed by Fux et al. (2002). It will also lead to an increase of the sludge age since the sludge age is determined by the outflow rate as the sludge age is the ratio of the sludge mass to the outflow waste rate. If the sludge age is increased above the minimal sludge age for nitrite oxidisers, then these organisms can grow in and nitrate will be produced.

The amount of water evaporated can be calculated as follows. According to Perry and Green (1998) the vapour pressure of water at 35°C and 1 atm is 0.056 atm. From the ideal gas law it can be calculated that 1 m^3 of air contains 39.6 moles. Hence 1 m^3 of saturated air contains 2.3 moles or 41.25 g or 41.25 mℓ of water, assuming a water density of 1 kg·m^-3. If dry air enters the SHARON reactor and saturated air leaves the SHARON reactor, then for every m^3 of air that enters the reactor 41.25 mℓ of water is removed. Normally the air-flow rate to the SHARON reactor is 3 to 8 ℓ·min^-1 or 4.32 to 11.52 m^3·d^-1. Therefore every day 178 to 475 mℓ of water is taken up by the air. Compared to an HRT of 1.55 d or an equivalent inflow rate of 1.3 ℓ·d^-1 about 14 to 37% of the flow evaporates. This was also checked experimentally. A batch reactor with a controlled temperature of 35°C was filled with 2 ℓ of water. After 24 h of aeration the water volume reduction was measured. This experiment was repeated at different airflow rates and the resulting evaporation, expressed as a percentage of the initial water volume, is depicted in Fig. 4.

In order to partially circumvent water evaporation, the air was subsequently saturated with water before entering the SHARON reactor.

Dilution by pH control

The base addition for pH-control leads to a certain dilution. For example, during the first 40d of operation of the SHARON reactor about 200 mℓ·d^-1 of a 1 M NaHCO_3 solution was added.

Stripping of CO_2 from the influent

Due to CO_2 stripping from the influent
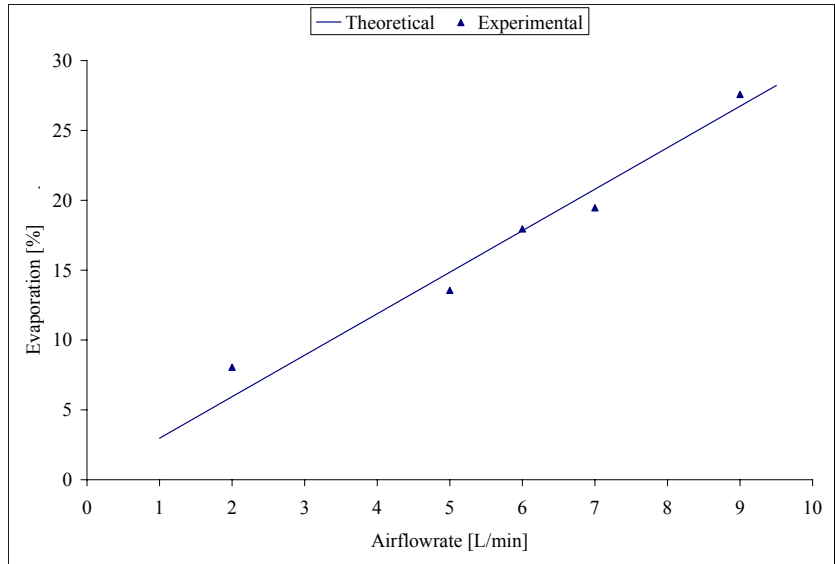
Figure 4
*Theoretical (-) and experimental (▼) percentage water evaporation in the SHARON reactor*
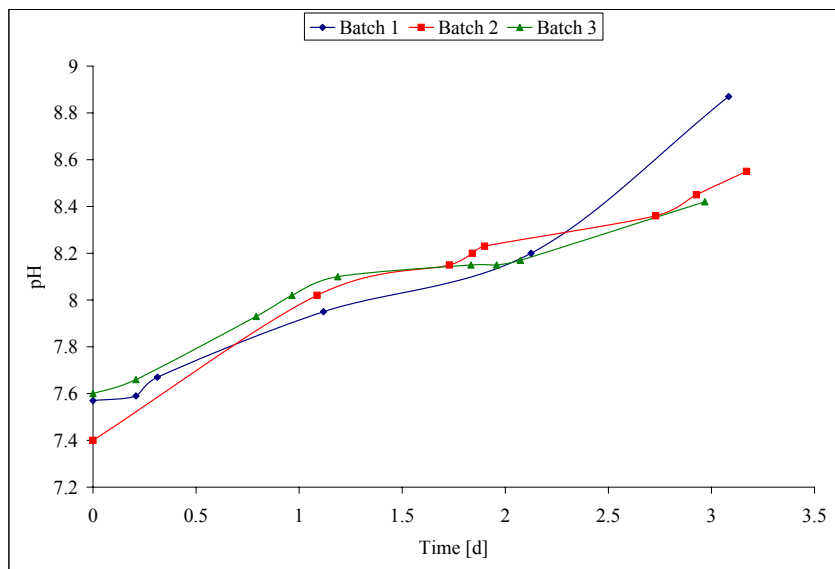
Figure 5
pH-evolution in 3 different influent batches, showing a pH increase because of CO_2 stripping

vessel, the influent pH and TIC concentrations vary over time. This has, however, no effect on the neutralising capacity of the influent since for every mole of CO_2 stripped one mole of OH^- ions is produced. The loss in buffering capacity is therefore converted to an equivalent pH increase. Generally batches of 5 ℓ influent are prepared, hence after approximately 3 d the influent is used up, since the design HRT is 1.54 d. The pH evolution of 3 different influent batches when 12 g·ℓ^-1 NaHCO_3 is added to the influent is depicted in Fig. 5. It can be seen that the influent pH increases by about 1 unit because of CO_2 stripping. This pH increase will stop when the entire TIC is stripped.

Mixing with air

Proper mixing of the SHARON reactor has to be ensured. However, during start-up and operation it was noticed that ammonium oxidisers are very sensitive to shear by mechanical stirring. Therefore, mixing of the SHARON reactor is performed by air blown into the reactor. The reduction of nitrifying activity by shear stress was also noticed by Ghyoot et al. (1999), among others, when operating a membrane bioreactor.
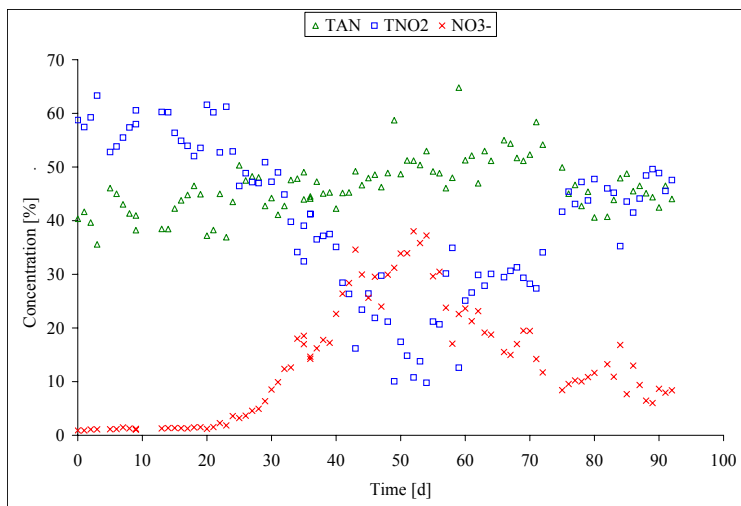
Figure 6
The evolution of TNO$_2$ ($\square$), NO$_3^-$ (x) and TAN ($\triangledown$) showing the ingrowth of nitrite oxidisers after changing the influent concentration from 1 000 mgTAN-N·ℓ$^{-1}$ to 500 mgTAN-N·ℓ$^{-1}$ on day 1

Wall growth

Measures had to be taken to prevent wall growth, since in a chemostat the SRT has to equal the HRT. Wall growth could increase the SRT and favour the growth of nitrite oxidisers. Wall growth could also induce anoxic conditions in the reactor and favour the growth of denitrifiers. Biomass of the reactor was therefore scraped off the walls every day.

Ingrowth of nitrite oxidisers

The SHARON reactor was operated for more than 500 d with influent concentrations of 4 000, 2 000 and 1 000mg TAN-N·ℓ$^{-1}$ at an HRT of 1.54 d. During this period malfunctioning of the reactor occurred due to biological instabilities, technical failures, software and computer crashes. At no point in time did nitrite oxidisers grow into the reactor resulting in nitrate build-up. However, decreasing the influent concentration from 1 000 mgTAN-N·ℓ$^{-1}$ to 500 mgTAN-N·ℓ$^{-1}$ led to the ingrowth of nitrite oxidisers even though the HRT was maintained at 1.54 d, indicating that in addition to temperature effects inhibition of nitrous acid, ammonia and/or salinity may play a role in the competition between ammonium and nitrite oxidisers. Decreasing the HRT from 1.5 d to 1.2 and 1 d led to a decreasing nitrate concentration as indicated in Fig. 6. Strangely, after the occurrence of this ingrowth, nitrate persisted in the reactor.

Protozoa

Protozoa can cause problems in the operation of the SHARON reactor, mainly if batches of real wastewater are used (Van Dongen et al., 2001a). A possible solution is to lower the reactor pH to 6 for 2 h or to incorporate non-aerated periods. Non-aerated periods, however, clearly have a negative effect on the nitrogen conversion by nitrifiers. A pH-lowering in the SHARON reactor can be attained by reducing the influent flow under constant aeration. After one or two hours the pH will decline to approximately 6. This does not require large aeration intensity because conversion rates are relatively small. When anoxic periods have to be regularly provided to prevent protozoa growth, the SHARON reactor has to be 30% larger to maintain good TNO$_2$ formation (Van Dongen et al., 2001a&b).

Protozoa can be observed via simple microscopic examination. This was done regularly during the operation of the SHARON reactor. No protozoa were ever observed, possibly because synthetic influent was used. Concentrations are expressed in percentage of the total effluent nitrogen concentrations for easy comparison.

Experimental results

The SHARON reactor was run for more than 2 years after successful start-up with the slow start-up method. Several instabilities occurred, but on the other hand several successful operating periods can be distinguished.

With pH control

During the first 45 d of operation the influent TAN concentration was 4 000 mgTAN-N·ℓ$^{-1}$ and HRT was set at 2.7 d resulting in an ammonium load of 1 480 mgTAN-N ·ℓ$^{-1}$d^{-1}. After 45 d the influent ammonium concentration and HRT were decreased to 2 000 mgTAN-N ·ℓ$^{-1}$ and 1.6 d, resulting in an ammonium load of 1 666 mgTAN-N·ℓ$^{-1}$·d^{-1}. For both influent concentrations on average 80% oxidation of TAN to TNO$_2$ was observed to be feasible at a pH controlled at 7.1 (results not shown), indicating the possibility of the SHARON process treating highly concentrated nitrogen streams.

However, periods of reduced performance occurred and indicated that pH-control is not enough to produce a stable effluent. The concentrations of nitrate were always below 20 mgNO$_3^-$-N·ℓ$^{-1}$, indicating the successful washout of nitrite oxidisers.

Without pH control

The influence of the TIC:TAN-ratio on the behaviour of the SHARON reactor was also investigated. NaHCO$_3$ was added to the influent and the pH was only controlled to stay within the range 6 to 8. The SHARON reactor was operated at different TAN influent concentrations (2 000, 1 000, 500 mgTAN-N·ℓ$^{-1}$) and TIC:TAN ratios (1:1, 0.5:1, 1.5:1). In all cases the HRT was 1.54 d. Influent concentrations and/or ratios were only changed if a sufficiently long steady state was achieved. Typically this steady state had a duration of 15 to 30 d or 10 to 20 times the HRT. Average steady state values are summarised in Fig. 7 together with the calculated HNO$_2$ and NH$_3$ concentrations. These concentrations are calculated based on Eqs. (5) and (6):

$$[NH_3] = \frac{[TAN]}{1 + \dfrac{10^{-pH}}{K_e^{NH}}} \tag{5}$$

$$[HNO_2] = \frac{[TNO_2]}{1 + \dfrac{K_e^{NO}}{10^{-pH}}} \tag{6}$$

where:
   K_e^{NH} and K_e^{NO} the ammonia/ammonium and nitrite/nitrous acid equilibrium constants.

Effluent TAN and TNO$_2$ concentrations are expressed as a percentage of total effluent nitrogen concentrations for easy comparison between the different operating modes. Transition from one operating mode to another one typically took about 8 d or 5 times the HRT as is usual in chemostat operation (Fig. 8).

For experiments with a TIC:TAN ratio of 0.5:1 and 1.5:1 and an influent TAN concentration of 1 000 mgTAN-N·ℓ$^{-1}$ the TNO$_x$ instead of the TNO$_2$ concentration is presented because of the
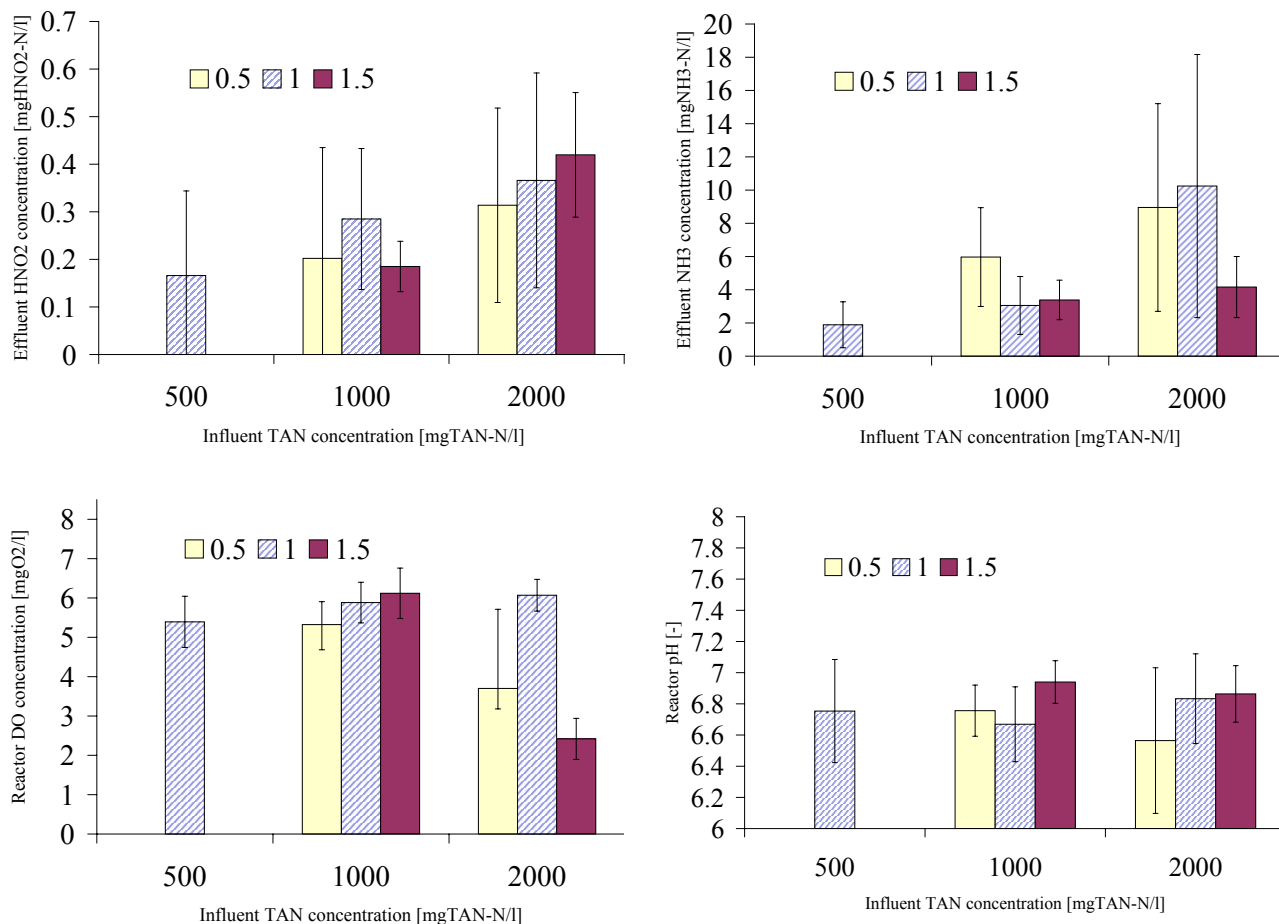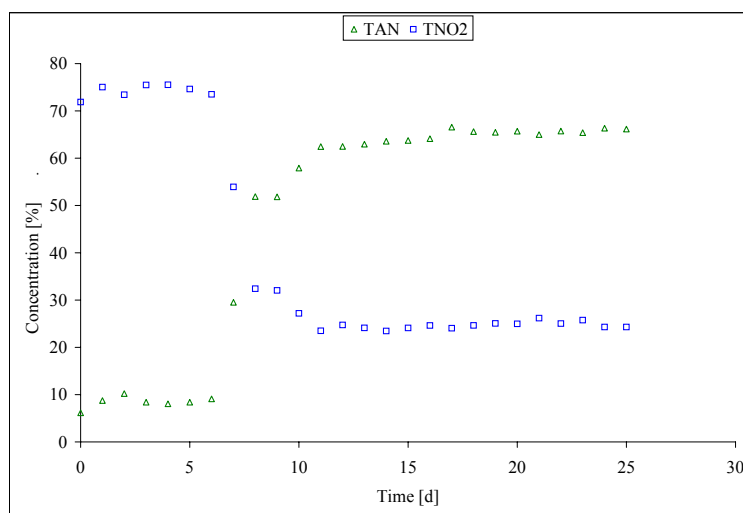
Figure 7
Average steady state results for different operating modes

ingrowth of nitrite oxidisers, which was discussed earlier. The experiments with a TIC:TAN ratio of 0.5:1 and 1.5:1 and an influent TAN concentration of 2 000 mgTAN-N·ℓ^{-1} have a low DO concentration, possibly because a defective DO electrode was used for the measurements.

When a TIC:TAN ratio of 1:1 is applied, then a TNO_2: TAN ratio of approximately 1:1 is obtained. This effluent is an Anammox-suited effluent. However, lower TAN influent concentrations but the same TIC:TAN ratio led to slightly higher TNO_2: TAN effluent compositions. This becomes clear from Fig. 9 where the relative concentrations of TNO_2 and TAN are depicted over three experimental periods, two with an influent TAN concentration of 1 000 mgTAN-N ℓ^{-1} and one with an influent concentration of 2 000 mgTAN-N·ℓ^{-1}.

This indicates that besides acidification, ammonia and nitrous acid inhibition also play a role. Indeed, at lower influent concentrations more TAN can be converted to TNO_2 before the same nitrous acid concentration is attained. Another reason for the observations relates to the fact that the aeration rate is not changed during the different experimental runs. This means that relatively less CO_2 can be stripped at higher influent concentrations, as the CO_2 uptake capability of the gas phase remains unaltered.

With a TIC:TAN ratio of 0.5:1 about 25% of the incoming TAN was oxidised, while a TIC:TAN ratio of 1.5:1 led to about

Figure 8
Transition of TNO_2 (□) and TAN (▽) concentration when switching to another operating mode on Day 7

75% of TAN oxidation. This confirms that a relationship exists between the influent TIC:TAN ratio and the effluent TNO_2:TAN ratio as was already predicted by a simulation study performed by Volcke et al. (2002).
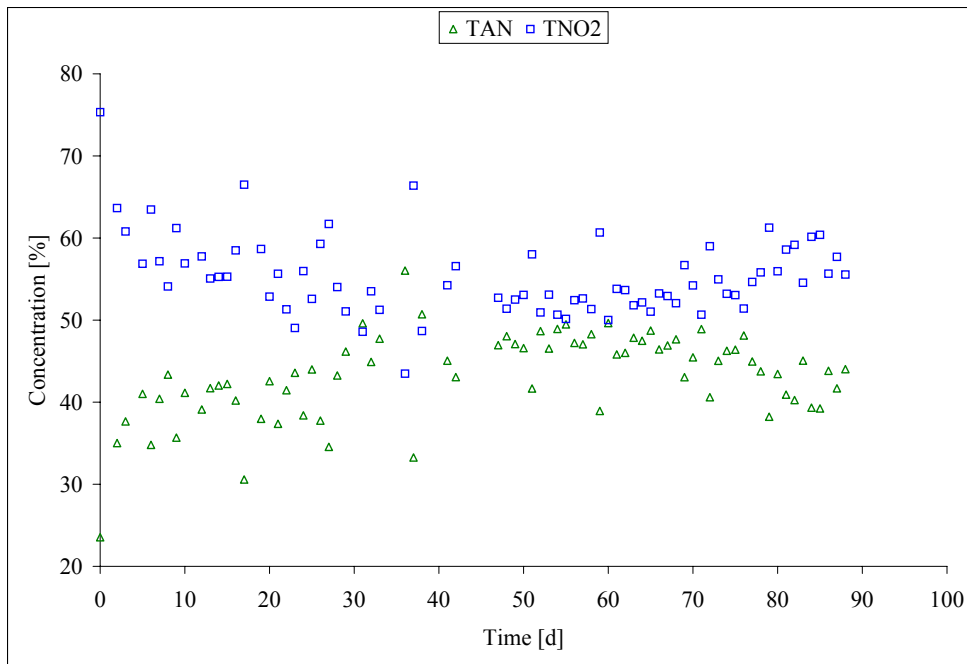
Figure 9
The evolution of TNO_2 (□) and TAN (▽) showing the effect of influent TAN concentration on effluent TAN and TNO_2 concentration. The numbers indicate the influent TAN concentration in $mgTAN\text{-}N \cdot \ell^{-1}$.

These results demonstrate the need for control of the SHARON reactor, since variations of the influent TAN concentration and the TIC:TAN ratio will occur in practice. These variations in the influent will lead to variations in the effluent, which are undesirable in view of the sensitivity of the Anammox process towards process disturbances, such as TNO_2 inhibition.

Conclusions

con-
structed?

In view of future modelling, simulation and control studies of a laboratory-scale SHARON reactor were undertaken. Start-up of the reactor was feasible by slowly adapting sludge originating from an SBR to the conditions typical for the SHARON process. A dedicated start-up was indeed necessary as the conditions in the SBR are completely different from the conditions in the SHARON reactor. The sludge retention time, for example, was 10 d in the SBR reactor and only 1.5 d in the SHARON reactor. The temperature in the SBR was 15°C, while the SHARON reactor was operated at 35°C. Also toxic effects of ammonia and nitrous acid on the nitrifier population were expected in view of the high nitrogen concentrations typical for the SHARON reactor. The start-up phase began with the operation of the reactor as an SBR to prevent biomass washout and to allow the selection of a strong nitrifying population. A month after the inoculation the reactor was switched back to normal chemostat operation. As a result the nitrite oxidisers were washed out and only the ammonium oxidisers persisted in the reactor.

After start-up the performance of the reactor could be assessed in view of its coupling with an Anammox unit. With pH controlled at 7.1, on average 80% nitrification was proven to be feasible for TAN loads up to 1.5g $TAN\text{-}N\cdot\ell^{-1}\cdot d^{-1}$, indicating the possibility of the SHARON process to treat highly concentrated nitrogen streams.

Results of experiments with different TIC:TAN ratios showed that both the influent TIC:TAN ratio and the influent TAN concentration influenced the resulting effluent concentrations, although generally it can be stated that the amount of TAN converted, or TNO_2 produced, is proportional to the influent TIC:TAN ratio. As such about 50% of the influent TAN is converted to TNO_2 when the influent TIC:TAN ratio is 1:1. Process control of the SHARON reactor will be necessary as a constant influent for the Anammox reactor is a prerequisite for the successful operation of the combined autotrophic nitrogen removal system. Indeed, varying influent TIC:TAN ratios will result in varying and thus non-optimal TNO_2:TAN ratios in the effluent of the SHARON reactor if no process control is applied.

When interpreting data of the lab-scale SHARON reactor for further modelling and control purposes some practical considerations, such as nitrate build-up, wall growth, water evaporation and CO_2 stripping from the influent should be considered. A list and quantification of these practical pitfalls were also presented in this contribution.

Acknowledgements

References

ABELING U and SEYFRIED CF (1992) Anaerobic-aerobic treatment of high strength ammonium wastewater-nitrogen removal via nitrite. *Water Sci. Technol.* **26** (5-6) 1007-1015.
ANTHONISEN AC, LOEHR RC, PRAKASAM TBS and SRINATH EG (1976) Inhibition of nitrification by ammonia and nitrous acid. *J. Water Pollut. Control Fed.* **48** 835-852.
CAPALOZZA C (2001) Design, Start-Up and Monitoring of a Pilot Sequencing Batch Reactor for Breeding Stable Nutrient Removal Sludge. M.Sc. Thesis, Ghent University, Faculty of Agricultural and Applied Biological Sciences. 147 pp.
FUX C, BOEHLER M, HUBER P, BRUNNER I and SIEGRIST H (2002) Biological treatment of ammonium-rich wastewater by partial nitrification and subsequent anaerobic ammonium oxidation (anammox) in a pilot plant. *J. Biotechnol.* **99** 295-306.
GHYOOT W, VANDAELE S and VERSTRAETE W (1999) Nitrogen removal from sludge reject water with a membrane-assisted bioreactor. *Water Res.* **33** 23-32.
HAO X, HEIJNEN JJ and VAN LOOSDRECHT MCM (2002) Model-based evaluation of temperature and inflow variations on a partial nitrification-ANAMMOX biofilm process. *Water Res.* **36** 4839-4849.

IZZET HB, WENTZEL MC and EKAMA GA (1991) The Effect of Thermophilic Heat Treatment on the Anaerobic Digestibility of Primary Sludge. Research Report No. W76, University of Cape Town, Cape Town, South Africa.

JETTEN MSM, HORN SJ and VAN LOOSDRECHT MCM. (1997) Towards a more sustainable wastewater treatment system. *Water Sci. Technol.* **35** (9) 171-180.

JETTEN MSM, STROUS M, VAN DE PAS-SCHOONEN KT, SCHALK J, VAN DONGEN UGJM, VAN DE GRAAF AA, LOGEMANN S, MUYZER G, VAN LOOSDRECHT MCM and KUENEN JG (1999) The anaerobic oxidation of ammonium. *FEMS Microbiol. Rev.* **22** 421-437.

LEE DS and VANROLLEGHEM PA (2003) Monitoring of a sequencing batch reactor using adaptive multiblock principal component analysis. *Biotechnol. Bioeng.* **82** 489-497.

MULDER JW, VAN LOOSDRECHT MCM, HELLINGA C and VAN KEMPEN R (2001) Full-scale application of the SHARON process for the treatment of rejection water of digested sludge dewatering. *Water Sci. Technol.* **43** (11) 27-134.

PERRY R and GREEN D (1998) *Perry's Chemical Engineer's Handbook* (7th edn.) McGraw-Hill, New York, USA.

PETERSEN B, GERNAEY K, HENZE M and VANROLLEGHEM PA (2003) Calibration of activated sludge models: A critical review of experimental designs. In: Agathos SN and Reineke W (ed.) *Biotechnology for the Environment: Wastewater Treatment and Modeling, Waste Gas Handling.* Kluwer Academic Publishers, Dordrecht, The Netherlands. 101-186.

STANDARD METHODS (1992) *Standard Methods for the Examination of Water and Wastewater* (18th edn.) American Public Health Association, Inc. (APHA) New York, USA.

VAN DEN BROECK S, VOLCKE EIP, VAN HULLE SWH and VANROLLEGHEM PA (2004) Kortsluiting leidt tot efficiënte stikstofverwijdering. *Het Ingenieursblad 2004* **(1-2)** 34-40 (In Dutch).

VAN DONGEN U, JETTEN MSM and VAN LOOSDRECHT MCM (2001a) *The Combined SHARON/Anammox Process*. IWA Publishing, London, UK.

VAN DONGEN U, JETTEN MSM and VAN LOOSDRECHT MCM (2001b) The SHARON®-Anammox® process for treatment of ammonium rich wastewater. *Water Sci. Technol.* **44** (1) 153-160.

VAN HULLE SWH, VAN DEN BROECK S and VANROLLEGHEM PA (2003) *The SHARON User Manual. Biomath Technical Report*, Ghent University, Belgium.

VAN KEMPEN R, MULDER JW, UIJTERLINDE CA and VAN LOOSDRECHT MCM (2001) Overview: Full scale experience of the SHARON process for treatment of rejection water of digested sludge dewatering. *Water Sci. Technol.* **44** (1) 145-152.

VISNIAC C and SANTER S (1957) The thiobacilli. *Bacteriol. Rev.* **21** 195-213.

VOLCKE EIP, VAN LOOSDRECHT MCM and VANROLLEGHEM PA (2002) Influence of operating parameters on the performance of a continuously aerated Sharon reactor. *Comm. Appl. Biol. Sci.* **67/4** 209-212.

WIESMANN U (1994) Biological nitrogen removal from wastewater. In: Fiechter A (ed.) *Advances in Biochemical Engineering/Biotechnology* **51**. Springer-Verlag, Berlin. 113-154.

WYFFELS S, VAN HULLE SWH, BOECKX P, VOLCKE EIP, VAN CLEEMPUT O, VANROLLEGHEM PA and VERSTRAETE W (2004) Modelling and simulation of oxygen-limited partial nitritation in a membrane-assisted bioreactor (MBR). *Biotechnol. Bioeng.* **86** 531-542.