PRACTICAL EXPERIENCES WITH START-UP AND OPERATION OF A CONTINUOUSLY AERATED LAB-SCALE SHARON REACTOR

S.W.H. Van Hulle*, S. Van Den Broeck*, J. Maertens*, K. Villez*, G. Schelstraete*, E.I.P. Volcke* and P.A. Vanrolleghem*
*Department for Applied Mathematics, Biometrics and Process Control (Biomath), Ghent University, Coupure Links 653, B-9000 Gent, Belgium

SUMMARY

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. Combination of such a SHARON process with the Anammox process, where ammonium is oxidised with nitrite to nitrogen gas under anoxic conditions, leads to cost-efficient and sustainable nitrogen removal from concentrated streams. In this study practical experiences during start-up and operation of a lab-scale SHARON reactor are discussed. Special attention is given to the start-up in view of possible toxic effects of high ammonium and nitrite concentrations (up to 4000 mgN/l) on the nitrifier population and because the reactor was inoculated with sludge from a SBR reactor operated under completely different conditions. Because of these considerations, the reactor was first operated as a SBR to prevent biomass wash out 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, such as mixing, evaporation and wall growth, concerning the operation of a continuously aerated SHARON reactor 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 g/l d, indicating the possibility of the SHARON process to treat highly loaded nitrogen streams.

INTRODUCTION

Biological nitrogen removal in wastewater with high nitrogen contents can become a major cost factor, in particular when the wastewater contains only small amounts of biologically degradable carbon compounds (Seyfried *et al.*, 2001). Conventionally nitrogen removal in these wastewaters is achieved using nitrification/denitrification. In such systems, nitrifying bacteria oxidize ammonium to nitrate under oxic conditions, and nitrate is subsequently or simultaneously reduced to dinitrogen 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 (about 35°C) and maintaining an appropriate sludge retention time (SRT) of 1 to 1.5 days, so that ammonium oxidizers are maintained in the reactor, while nitrite oxidizers are washed out and further nitrification of nitrite to nitrate is prevented. In this way, significant aeration cost savings are realized in comparison with conventional nitrification to nitrate. The SHARON process is very suitable to reduce the load of streams with high ammonium concentration (~1gNH_4^+-N/l), rather than to meet strict effluent standards. It is 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 operational since January 1999 at the Rotterdam Sluisjesdijk sludge treatment plant process (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 ammonium by the recently discovered Anammox (ANaerobic AMMonium OXidizers) organisms, that combine ammonium and nitrogen to form nitrogen gas (Jetten *et al.*, 1999). With the combined SHARON-Anammox process, low nitrogen effluent concentrations can be obtained, while aeration costs are significantly reduced, no additional carbon source is needed and sludge production is very low.

In case the SHARON influent contains ammonium and bicarbonate on an equimolar basis, which can be reasonably assumed for sludge digestion reject water, the protons produced during conversion of half of the ammonium are balanced 'exactly' via carbon dioxide stripping. For the high-concentrated streams to which the SHARON process is typically applied, the protons produced during ammonium conversion over 50% would cause a significant pH drop, preventing further nitrification. So theoretically, when assuming equimolar amounts of ammonium and bicarbonate in the influent of the SHARON reactor, its effluent will contain the required nitrite:ammonium ratio of 1:1. This is the ratio that is theoretically needed to feed the Anammox reactor. This simplified reasoning is represented schematically in Figure 1.

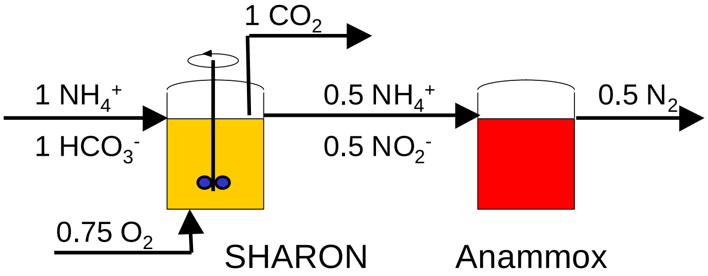

Figure 1. The combined SHARON-Anammox process

Very interesting and useful tools to further optimize the combined SHARON-Anammox system are modelling and simulation environments such as WEST® (Vanhooren *et al.*, 2003) or Matlab (The Mathworks Inc., www.mathworks.com). With such a simulation tool a large number of virtual experiments can be conducted in order to investigate the behaviour of the combined system under different operating conditions. However, in order to have a correct representation of reality by these simulations, parameters of the biological processes have to be assessed. Therefore a lab-scale SHARON reactor was constructed for estimation of the model parameters and for testing of promising operational conditions.

In this contribution practical experiences during start-up and operation of a lab-scale SHARON reactor are discussed in view of this objective. The experimental performance of the SHARON reactor under different conditions is discussed in view of its coupling with an Anammox unit.

MATERIALS AND METHODS

SHARON reactor

A lab-scale SHARON reactor was constructed in the BIOMATH lab. The reactor is a 2 litre continuously stirred tank reactor (CSTR) without biomass retention. Its schematic representation is presented in Figure 2. The synthetic influent is pumped with a peristaltic pump from the 5 litre influent vessel to the reactor. The pump flow rate of this influent pump determines both the hydraulic residence time and the sludge residence time (SRT), since both residence times are equal and defined as the ratio of the volume to the flow rate. 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 effluent flow rate since the withdrawal point is situated at the 2 litre mark.

The reactor is aerated through a pumice stone using air from a compressor (1 bar). The temperature of the reactor can be controlled between 15°C and 70°C, although the normal operational temperature is 35°C, as is usual in practice for the Sharon process. In the reactor the dissolved oxygen (DO) and pH are measured. The pH is controlled through the Labview® software (National Instruments, www.ni.com) installed on the computer with the addition of acid (HCl) and base ($NaHCO_3$). Data logging and control of the pH are also done with the Labview® software.

In a first series of experiments (days 0-170), the influent did not contain bicarbonate. In order to compensate for the proton production during the conversion of the high ammonium concentrations, base ($NaHCO_3$) was added in such a way that the reactor pH was kept constant at 7.1.

In a second series of experiments, bicarbonate was added to the influent in a 1:1 bicarbonate:ammonium molar ratio, while the pH was allowed to vary freely in a broad pH control range (pH 6-8).

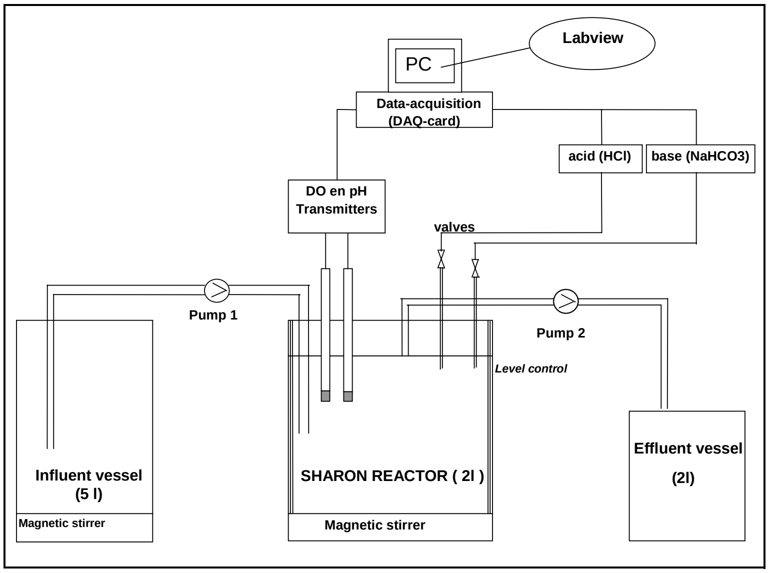

Figure 2. Schematic representation of the SHARON reactor (adopted from Van Den Broeck, 2002)

Influent and chemical analyses

The reactor is fed with synthetic influent which composition is described in Table 1. The ammonium and bicarbonate concentrations vary according to the type of experiment conducted, although normally the influent ammonium concentration is 2000 mg NH_4^+-N/l. The trace element composition is according to Visniac and Santer (1957) adjusted by Capalozza (2001).

Concentrations of NH_4^+-N, NO_2^--N and NO_3^--N were analysed on a daily basis using spectrophotometric methods (Dr Lange GmbH, Germany). TSS (Total Suspended Solids) concentrations were determined according to Standard Methods (APHA, 1992).

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 ammonium and nitrite concentrations typical for the SHARON process. Secondly, an inoculum from the sequencing batch reactor (SBR) in the BIOMATH lab (Lee and Vanrolleghem, 2003) was used. Since the organisms in the SBR are not adapted to high ammonium and nitrite concentrations, special attention was given to the start-up with this inoculum.

Table 1. Composition of the synthetic influent of the SHARON reactor

Main compounds	Concentrations (mg/l)
$(NH_4)_2SO_4$	Depends on experiment; 1000 mg N/l = 4714 mg $(NH_4)_2SO_4$/l
$NaHCO_3$	Depends on experiment; 1000 mg C/l = 6994 mg $NaHCO_3$
KH_2PO_4	500
$MgSO_4.7H_2O$	300
Trace compounds	
$FeSO_4.7H_2O$	7.5
$PbCl_2$	1.7
$ZnCl_2$	3.6
$Cr(NO_3)_3.9H_2O$	13.3
$CuCl_2.2H_2O$	9.2
$MnSO_4.H_2O$	9.25
$NiSO_4.6H_2O$	3.1
$CoCl_2.6H_2O$	1.7
$(NH_4)_6Mo_7O_4.4H_2O$	2.4
$CaCl_2$	12.2
EDTA	375

RESULTS AND DISCUSSION

Start-up of the SHARON reactor: fast method versus 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 a HRT of 2.5 days after 24 hours of adaptation of the biomass to the reactor. The pH and temperature were fixed at 6.9 and 35°C respectively. Different influent concentrations, ranging from 300 to 800 mgNH_4^+-N/l, 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 mgNH_4^+-N/l is shown in Figure 3. The first 3 days all incoming ammonium is oxidised to nitrate. After approximately 1 SRT nitrite starts to build up in the effluent, indicating the successful washout of the nitrite oxidisers. After 3 SRT however ammonium builds up in the effluent, indicating the washout of ammonium oxidisers too. From Figure 3 it can thus be concluded that directly imposing short residence times on the nitrifying organisms coming from a 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 ammonium and nitrite (Antonissen *et al.*, 1976) can be put forward as a possible explanation.

Slow start-up method. Since the fast start-up method had no success, a slow start-up method was tested. This time inoculum from a SBR reactor was used. Special attention was given to the start-up in view of possible toxic effects on the nitrifier population and because of the fact that the reactor was inoculated with sludge from a SBR reactor operated under completely different conditions (T=15°C, SRT = 10 d, ammonium load 9 mgNH_4^+-N/l d). The ammonium oxidisers were therefore allowed to adapt slowly to the changed conditions (Van Den Broeck, 2002).

The SHARON reactor was first operated as a SBR to prevent biomass washout, while the influent ammonium load was stepwisly increased from 600 to 1480 mgNH_4^+-N/ld. The temperature too was stepwisly increased from 23.4°C to 35°C. The pH was fixed at 7.1. Every 12 hours 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 ammonium was oxidised to nitrate. The reactor was then switched to normal chemostat operation with a SRT of 2.7 days. This time the nitrite oxidisers were washed out since the incoming ammonium was now oxidised to nitrite 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 startup with a general nitrifying sludge instead of 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.

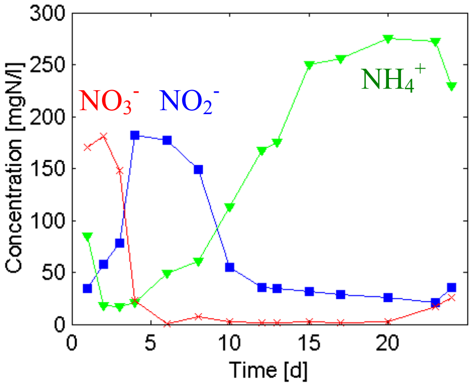

Figure 3. Fast start-up method for the SHARON reactor: evolution of NO_2^--N, NO_3^- -N and NH_4^+-N as function of the time, indicating the washout of nitrifying organisms

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 accurate as possible in order to compare experimental results with modelling results. For instance, evaporation and wall growth, among others, can hinder however the interpretation of experimental results.

Low biomass concentration. The reactor is designed to operate at an effluent ammonium:nitrite ratio of 1:1 and a HRT of 1.6 days, although the HRT at start-up was 2.7 days. For an influent ammonium concentration of 2000 mg NH_4^-N/l the amount of ammonium nitrified would then be 1000 mg NH_4^-N/l (N^{nitr}). According to Petersen *et al.* (2002) the concentration of ammonium oxidisers (X_{NH}) in the reactor can be calculated by the following equation.

$$X_{NH} = Y_{NH} \frac{SRT}{HRT} \frac{N^{nitr}}{1 + b_{NH} SRT} = 0.15 \frac{1.6}{1.6} \frac{1000}{1 + 0.2\ 1.6} \approx 110 mgCOD/l$$

with Y_{NH} the growth yield for ammonium oxidisers on ammonium (mgCOD/mg NH_4^-N) and 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 ammonium and no carbon source is used, leads to 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.

Evaporation. Water evaporation is not negligible and can amount to more than 10% of the influent flow when operating a 2 litre lab-scale reactor at 35°C. This evaporation was detected because the nitrogen mass balance, assuming the influent and effluent flow rate to be the same, over the reactor did not close. In other words, the nitrogen concentration (in the form of nitrite, nitrate and ammonium) coming out the reactor was higher than the nitrogen concentration in the reactor:

$$C_{NH_4^+}^{in} \leq \left(C_{NH_4^+}^{out} + C_{NO_2^-}^{out} + C_{NO_3^-}^{out} \right)$$

with C^{in} the concentration of ammonium in the influent and C^{out} the concentration of ammonium nitrite and nitrate in the effluent. This difference could only be explained by evaporation since numerous tests and dilution series were performed to exclude any measurement errors. Because of this evaporation the influent and effluent flow rates would differ.

Dilution by pH control. The base addition for pH control leads to a certain dilution. For example, the first 40 days of operation about 200 ml/d of an 84 g/l $NaHCO_3$ solution was added. Because of the dilution and evaporation effects the nitrogen concentrations presented hereafter are expressed in percentage of total nitrogen in the effluent.

Stripping of CO_2 from the influent. Due to CO_2 stripping from the influent vessel, the influent pH and bicarbonate concentration varies during the experiment. This has however no effect on the neutralising capacity of the influent since for every mole of CO_2 stripped a OH^- ion is produced. The loss in buffering capacity is therefore converted to an equivalent pH increase. Generally batches of 5-litre influent are made, hence after approximately 3 days the influent is finished, since the design HRT is 1.6 days. The pH evolution of 3 different influent batches when 12 g/l $NaHCO_3$ is added to the influent is depicted in Figure 4. It can be seen that the influent pH increases about 1 unit because of stripping. Of course this pH increase would stop if all bicarbonate is stripped.

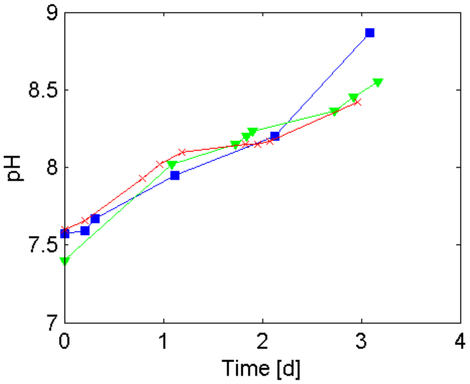

Figure 4. pH evolution in 3 different influent batches, showing a pH increase because of stripping

Mixing with air. Proper mixing of the Sharon reactor has to be ensured. However, during operation it was noticed that ammonium oxidisers are very sensitive to shear by mechanical stirring. Therefore, mixing of the SHARON reactor is performed by the air blown into the reactor.

Wall growth. Measures had to be taken to prevent wall growth, since in a chemostat the sludge residence time has to equal the hydraulic residence time. Also wall growth could induce anoxic conditions in the reactor and favour the growth of denitrifiers. Biomass of the reactor is therefore scraped off the walls every day.

Protozoa. Finally it can be mentioned that protozoa can disturb the SHARON reactor, mainly if batches of real wastewater are used. A possible solution is to lower the pH in the reactor to 6 for 2 hours (van Dongen *et al.*, 2001a&b).

Experimental results

The SHARON reactor is now running for more than 300 days after successful start-up with the slow start-up method. Several instabilities occurred, but on the other hand several successful operational periods can be distinguished.

pH control. During the first 45 days of operation the influent ammonium concentration was 4000 mg NH_4^-N /l and HRT was set at 2.7 days resulting in an ammonium load of 1480 mg NH_4^-N /l d. After 45 days the influent ammonium concentration and HRT were decreased to 2000 mg NH_4^-N /l and 1.6 days, resulting in an ammonium load of 1666 mg/l d. For both influent concentrations on average 80 % oxidation of ammonium to nitrite was proven to be feasible at a controlled pH of 7.1, indicating the possibility of the SHARON process to treat highly concentrated nitrogen streams. Disturbances in 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, indicating the successful wash out of nitrite oxidisers.

No pH control. The influence of the bicarbonate:ammonium ratio on the behaviour of the SHARON reactor was also investigated. Bicarbonate was added to the influent and the pH was only controlled to stay within the range 6-8. The influent ammonium concentration and HRT were kept at 2000 mg NH_4^+-N/l and 1.6 days. Addition of a 1:1 bicarbonate:ammonium ratio in the influent led to a 1:1 nitrite:ammonium effluent composition as predicted by previous simulation studies (Volcke *et al.*, 2002). This effluent is an Anammox suited effluent.

Experiments with a bicarbonate:ammonium ratio of 1:2 resulted in only 1/4 oxidised ammonium because of the depletion of bicarbonate. A bicarbonate:ammonium ratio of 3:2 resulted in approximately 3/4 oxidised ammonium because of the abundance of bicarbonate. Results of the tests with a varying bicarbonate:ammonium ratio are given in Figure 5.

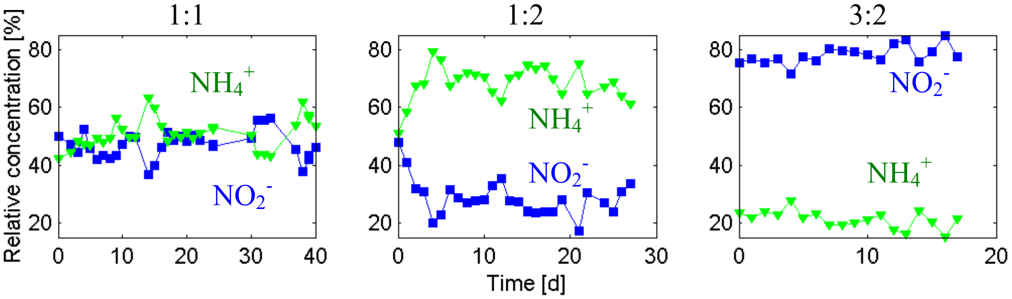

Figure 5. Daily NO_2^--N (■), and NH_4^+-N (∇) relative concentrations in experiments with varying bicarbonate:ammonium ratio's (1:1: left, 1:2: middle, 3:2: right)

CONCLUSIONS

In this study practical experiences during start-up and operation of a lab-scale SHARON reactor are discussed. Special attention was given to the start-up in view of possible toxic effects of ammonium and nitrite on the nitrifier population and because the reactor was inoculated with sludge from a SBR reactor operated under completely different conditions such as temperature and SRT. The sludge retention time is, for example, 10 days in the SBR reactor and only 1.5 days in the SHARON reactor. Because of these considerations, the reactor was first operated as a SBR to prevent biomass wash out 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 some practical considerations, such as wall growth, water evaporation and CO_2 stripping from the influent were discussed. These considerations are not trivial, since the reactor will be used for kinetic characterisation and modelling studies. Therefore, the exact conditions in the reactor have to be known.

Finally the performance of the SHARON reactor under different conditions is discussed in view of its coupling with an Anammox unit. On average 80 % nitrification was proven to be feasible for ammonium loads up to 1.5 g NH_4-N /l, indicating the possibility of the SHARON process to treat highly concentrated nitrogen streams. Results of experiments with different bicarbonate:ammonium ratios showed very good agreement to previous simulation studies. A 1:1 a bicarbonate:ammonium ratio yielded an Anammox suited effluent.

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