



EOLI

Efficient Operation of Urban Wastewater Treatment Plants

Deliverable D1.4

Report on the evaluation of the FDI system

WP1. Process Experiments
Responsible: Germán Buitrón (UNAM)
Involved partners: POLIMI and UNAM

By
Roberto Canziani and Elena Ficara (POLIMI)
Cristina Verde, Dieter Wimberger and Jesús Mina (UNAM)

Contract Number ICA4-CT-2002-1001

TABLE OF CONTENTS

<u>I Introduction</u>	3
1.1 Nitrites Build Up Analysis. Reproducibility of the Fault	5
<u>II Analysis of the abnormal conditions</u>	5
2.1 Factors affecting Nitrite Oxidizing Bacteria and Ammonia Oxidizing Bacteria activity	5
2.2 Materials and Methods	5
<u>III Results</u>	7
3.1 Effect of dissolved oxygen on nitrification activity	7
3.2 Effect of pH on nitrification activity	7
3.3 Effect of the free ammonia concentration on the NOB activity	8
3.4 Effect of the aeration pattern on the endogenous decay rate	11
3.5 Micronutrients availability in the SBR feed	15
<u>IV Conclusions</u>	17
<u>V References</u>	18
<u>Annex 1 EOLI-Questionnaire Sensors and Actuators</u>	19
<u>Annex 2 Data of experiments for the assessment of the aeration pattern on the endogenous decay rate of nitrifying biomass</u>	28

I Introduction

To specify the functionality of the Fault Detection and Isolation task, FDI, considering practical situations, we captured, as first step, the information about the faults of the EOLI experiments made during the first year. Since, there are an infinite number of faults which could be considered in the difference cycles of the process, the most common and important faults and abnormal conditions are selected, using data of the four laboratories involved in the project (POLIMI, LBE-INRA, UU, UNAM).

The Annex A shows the questionnaire sent to the four laboratories, to capture the real faults of the installations. The sensors and actuators have a common template and the abnormal condition of the process has a free format. On the base of this pool, we analyze the more important and critical faults. The faults in sensors and actuators are generic and independent of the process; and the faults associated with a particular SBR of EOLI demand specific descriptions of the process behavior with strong interrelation between variables and usually historical data access.

On the base of a global device vulnerability index for each instrument and event in the process defined as:

$$DVI = \frac{1}{m} \sum_{j=1}^m FVI_j \quad \text{with} \quad FVI_j = \frac{1}{n} \sum_{i=1}^n F_{ij} W_{ij} \quad (1)$$

with:

n : the number of reactors (4),

m : the number of reported faults,

F_{ij} : the frequency of the fault j , in the reactor i , the specific fault of the FDI system were selected.

Figure 1 and 2 show the evaluation of the indexes and from them the following conclusions with the support of EOLI participants have been obtained.

1. The Dissolved Oxygen, DO, is the most important case to be considered between all the sensors for the aerobic phase in the SBR, since it is the only measurable state variable and the control strategies and the safety of the process are strong deteriorated when the sensor has a bias or gets damage,.
2. The highest priority for the fault diagnosis inside of the actuators set is given to the servo-valve, since faults in this electromechanical component are very frequently and their effect could interrupt the process sequence at the risk of damage the biomass.
3. Diverse faults and abnormal conditions of the process have been reported during the experiments; they are not generic, since each reactor has specific problems and some of them are not technical issues. However the nitrites build up is pointed out as the more interesting issue to be studied in detail from bioprocess point of view in collaboration with other EOLI partners.
4. Since, any FDI task requires a model with well defined normal conditions and the parameters identification given in D2.1 for the model EM1 of UNAM presented the highest confidence values, these parameters are considered to develop the diagnosis of abnormal conditions in the process using the respiration rate.

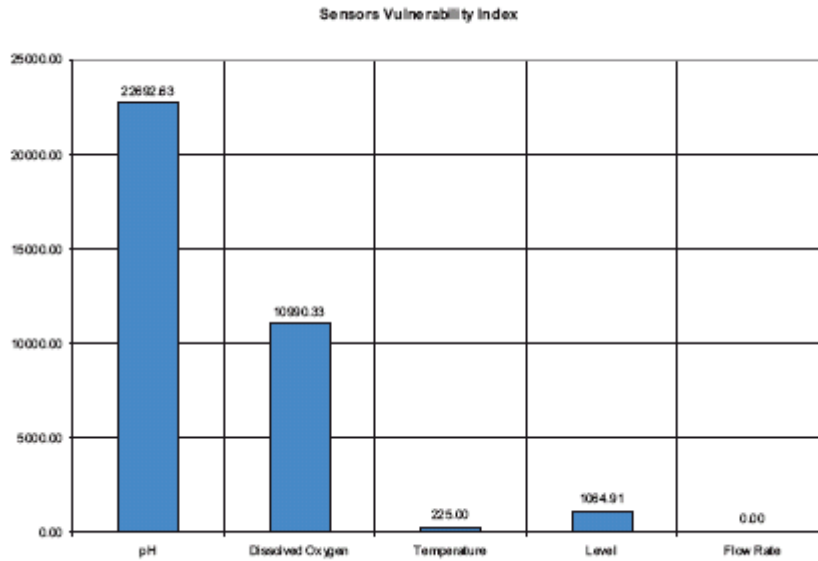


Figure 1: Indexes Evaluation of the SBR sensors

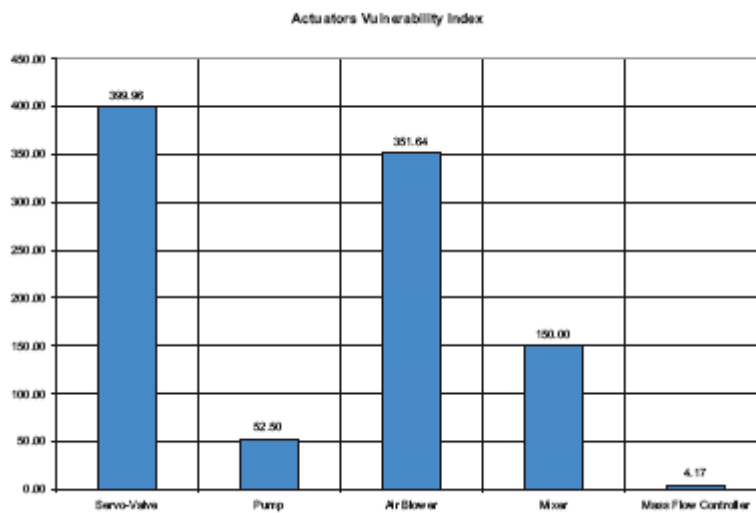


Figure 2: Indexes Evaluation for the SBR actuators

The main issue with the test and validation of any FDI algorithm in real conditions is the emulation and generation of fault in the process in real time without damage the installation and with the possibility to recover the normal condition in a short time. This is the case of the FDI algorithms for the SBR's. In particular, the constraints in the budget do not allow having specific devices in which physical damage can be emulated under control for sensor and actuators.

If historical data of the process with fault exists, the above issue can be solved using the data off-line to validate the FDI algorithm. However in the case of the SBR the data in abnormal conditions were not well characterized and enough rich to test the FDI algorithms. Moreover in the case of the damage membrane for the DO sensor, the developed membrane diagnosis procedure proposed to integrate electronic hardware with the standard sensor. This required a sensor during long time only for the WP6. The availability of sensor and actuator limited the conditions in which the FDI algorithm were tested. Other alternative to solve the problem of fault conditions generator is the design of a simulator in which the physical fault can be

introduced. This option was considered for the FDI algorithms developed in WP6 and the validation results are included as part of the D6.2.

1.1 Nitrites Build Up Analysis. Reproducibility of the Fault

Considering the analytical dynamic model EM2a given in D2.1 for POLIMI reactor which describes the behavior of the process during the reactor phase, a simulator was implemented to reproduce the reduction in the nitrification capacity and the growth of nitrifying organisms in the reactor. The idea was to design the FDI algorithm on the base of the model and the features reported by POLIMI. However the reproducibility of the phenomenon was not possible by simulations. It is to remark that the absence of a relationship between pH and biomass and substrate in the SBR reduced the possibility to detect and identify abnormal conditions. As consequence, a specific analysis was carried by POLIMI team trying to understand the causes of the nitrites build-up in its reactor using a set of experiments. The following paragraphs describe in detail results and conclusions of the analysis and they are the main contribution of this deliverable.

II Analysis of the abnormal conditions

2.1 Factors affecting Nitrite Oxidizing Bacteria and Ammonia Oxidizing Bacteria activity

Effluent nitrite concentrations well above toxicity threshold were experienced as the main abnormal condition during the two-years operation at the lab-scale POLIMI-SBR. The main origin of nitrite build-up is the unbalanced activity of ammonia and nitrite oxidisers. Usually, ammonia oxidation to nitrite is slower than the oxidation of nitrite to nitrates, therefore nitrite does not accumulate. However, in batch-fed reactors nitrite accumulation is observed more frequently than in continuously-fed systems.

An experimentation was conducted to try to find out whether specific environmental or operating conditions may favour ammonia oxidising bacteria (AOB) over nitrite oxidising bacteria (NOB), creating the conditions for unbalanced nitrification and thus possibly leading to nitrite build-up.

Specifically, this experimentation was focused on:

- the evaluation of the effect of pH, dissolved oxygen concentration (DO) and free ammonia concentration on the activity of ammonium and nitrite oxidizing bacteria;
- the identification of SBR operating conditions (aeration pattern, micronutrient support) which can prevent nitrite build-up.

In the literature, pH, free ammonia, DO and aeration patterns are reported to have a relevant effect on both groups of nitrifying bacteria. However, values are spread on very wide ranges and no conclusive statements can be made about the actual relevance of these effects at given experimental conditions. Therefore, batch tests were conducted to point out which of these aspects were indeed relevant in the case of the SBR sludge.

2.2 Materials and Methods

Nitrification activity was measured by pH-DO-stat titration performed with the laboratory titrator developed within this project (MARTINA instrument). The principle of operation and the experimental procedure applied to measure AOB and NOB activities has been already described in previous reports (see Deliverable 3.2).

Sludge samples were drawn either from the lab-scale POLIMI SBR or from a full scale WWTP, whose sludge was also used for the SBR inoculation, when poor nitrification occurred. However, even this latter sludge was occasionally showing low nitrification capacity, which could be due to the low temperature at which the WWTP was operated during the sampling period (January and February).

Tests to assess the effect of DO, pH and NH_3 concentration on the activity of AOB and NOB were performed as follows: a 0.5-L sludge sample was poured into the MARTINA reactor. An initial set of environmental parameters was fixed. A first activity test was performed by adding the appropriate substrate for the bacteria group whose activity had to be assessed (ammonium or nitrite, 5 mgN/L). When the added nitrogen had been fully oxidized, all parameters were kept constant but the one whose influence was under study, which was increased or decreased. The substrate was spiked again to assess the nitrification activity under the new condition. In some of the tests, substrate was supplemented a third -sometimes also a fourth- time, after having made a third change in the parameter under study. The maximum nitrification activity measured after each substrate spike was then plotted against the varied parameter.

Tests to assess the effect of the aeration pattern on the endogenous decay rate were performed as follows. A batch of 8 L of sludge was sampled and kept under aeration overnight to reach endogenous conditions. Initial nitrification (AOB and NOB) activity was measured. The sludge was then split into four batches, all continuously mixed, but characterized by different aeration patterns:

Batch A: not aerated;

Batch B: continuously aerated;

Batch C: aeration was cyclically kept on for 12 h and off for the following 12 h.

Batch D: aeration was cyclically kept on for 1 h and off for the following hour.

All batches were kept under endogenous conditions at room temperature (19-22 °C). Daily, pH was measured in all batches and corrected to remain in the range from 7.5 to 8.0. Nitrification activity, VSS and nitrates concentration were measured after 4 and 7 days.

The effect of the presence of micronutrients in the SBR feed was tested by monitoring NOB activity with and without micronutrient supplementation. The latter was automatically operated by pumping 1 ml/L of a micronutrient solution during the feeding phase. The composition of the micronutrient solution is the following (Larsen and Harremoës, 1994):

- $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$: 0.527 g/m³
- $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$: 0.228 g/m³
- $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$: 0.317 g/m³
- $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$: 0.231 g/m³
- $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$: 0.127 g/m³
- ZnCl_2 : 0.363 g/m³
- $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$: 3.7 g/m³

III Results

3.1 Effect of dissolved oxygen on nitrification activity

The effect of dissolved oxygen on nitrification activity was determined. Tests conditions were the following:

sludge concentration = 2-3 gVSS/L,

pH was adjusted to optimal, i.e.: 8.3 for the AOB test and 7.5 for the NOB test,

temperature = 21°C

DO = varied between 1.0 and 7.5 mg/L. Note that, during DO-stat titration, the DO concentration oscillates around the set point value of approximately ± 0.25 mg/L, therefore, set-point values lower than 1.0 could not be tested precisely.

AOB and NOB activity against DO is plotted in 3, together with the fitting Monod curve, whose best-fit parameters were estimated to be:

AOB: maximum activity = 3.0 mgN/gVSS/h; half saturation constant = 0.1 mgO₂/L

NOB: maximum activity = 2.6 mgN/gVSS/h; half saturation constant = 0.4 mgO₂/L

Error bars indicate that, as stated earlier, the DO concentration during the activity test oscillated around the set point value.

These results confirm the higher affinity of AOB for oxygen, as it has been reported in previous experimentations. Barnes e Bliss (1983) indicated a range for the DO half saturation constant of 0.25-0.5 mg/L for AOB and of 0.34-2.5 mg/L for NOB. Hanaki et al. (1990) found that AOB activity did not vary significantly at DO-concentrations below 0.5 mgO₂/L, while the NOB activity halved. However, the DO level during the aerobic react phase of the POLIMI SBR was normally above 1 mgO₂/L, so that DO limitation does not seem to be a major cause for nitrite build-up in this reactor.

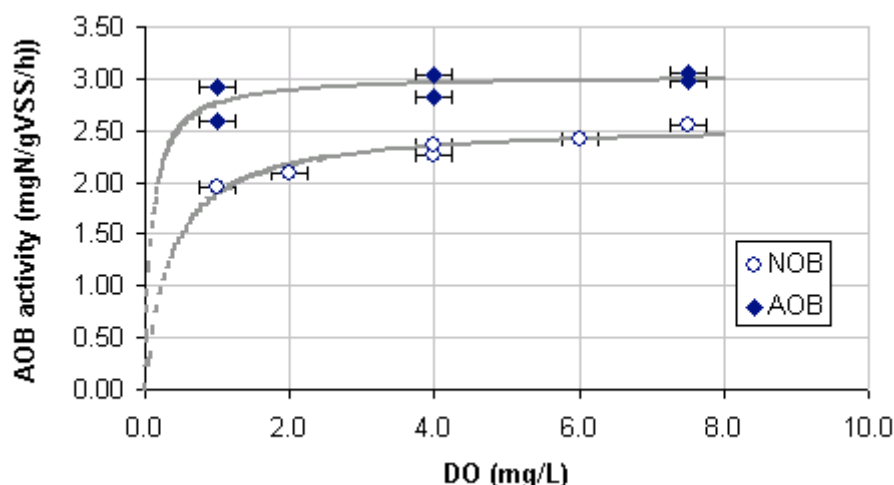


Figure 3: Effect of dissolved oxygen on the activity of NOB and AOB.

3.2 Effect of pH on nitrification activity

The effect of pH on nitrification activity was studied (4). Tests conditions were the following:

Range of sludge concentration during the experiments = 2.0-3.8 gVSS/L,

pH = varied between 7.5 and 8.3 which is the usual range within which the pH of the POLIMI SBR remains during the aerobic react phase.
 Temperature = 21°C,
 DO = 7.5 mg/L

As for the NOB activity, a decreasing trend with pH can be observed. However, this reduction (from 3.42 ± 0.39 mgN/gVSS/h at pH 7.5 to 2.88 ± 0.57 mgN/gVSS/h at pH 8.3, average reduction of 16%) seems to be moderate. On the contrary, AOB activity increased with pH with both tested sludge samples. For the SBR sludge, AOB activity increased from 2.5 at pH 7.5 to 3.4 mgN/gVSS/h at pH 8.3 (+37%). An even higher increase (+57%) was observed for the sludge drawn from the full scale WWTP.

It can be concluded that by working at pH higher than 8.2 during the aerobic phase of the SBR cycle, nitrite build up can occur due to a higher activity of AOB than NOB.

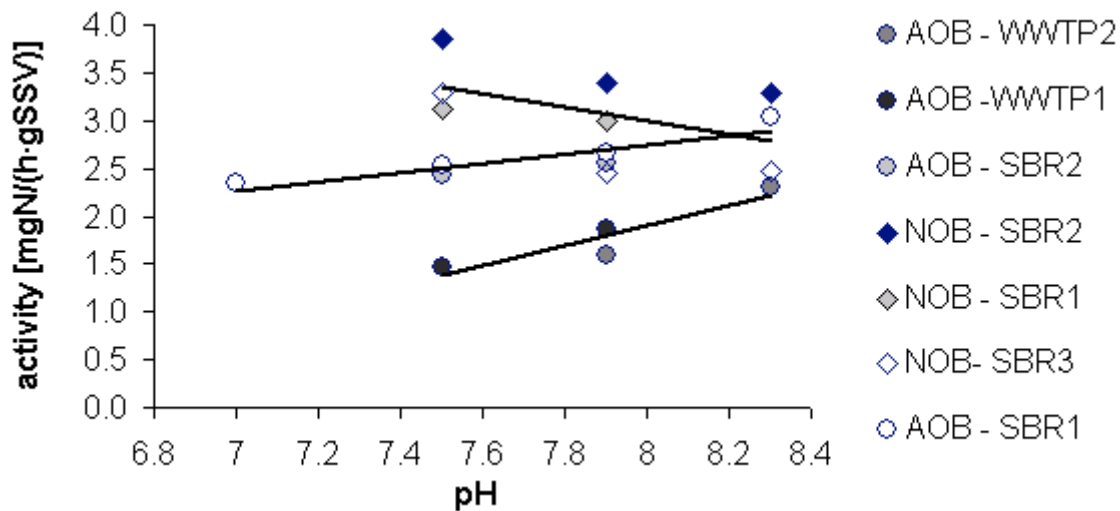


Figure 4: Effect of pH on the activity of AOB (circles) and NOB (diamonds).

3.3 Effect of the free ammonia concentration on the NOB activity

The third evaluation was aimed at assessing the effect of the free ammonia concentration on the NOB activity. Actually, this bacteria group is known to be inhibited by free ammonia levels as low as 1 mgN/L (Anthonisen et al., 1976), which are easily exceeded in batch-fed reactors such as SBRs.

To assess NOB activity of mixed cultures (such as activated sludge samples) in the presence of a constant ammonia concentration, an AOB selective inhibitor is needed to avoid concomitant oxidation of ammonium to nitrite. ATU is known to fully inhibit AOB and to be ineffective on NOB. However, the sensitivity of activated sludge samples to organic compounds may vary widely according to their specific growth conditions. Therefore, a preliminary test was aimed to verify the non-inhibitory effect of ATU to NOB. In Figure 5 the DO-stat titration curve obtained in this test is shown. A sludge sample under endogenous conditions was spiked with nitrites (5 mgN/L) before and after the addition of 15 mg/L of ATU. These two activity values were found to be very close, therefore no ATU inhibition was observed. It is worth noting that, after ATU inhibition, a reduction of the endogenous titration

rate was clearly observed, indicating that ammonification, and therefore a slow nitrification was taking place in the sludge sample, which was as high as 0.2 mgN/gVSS/h.

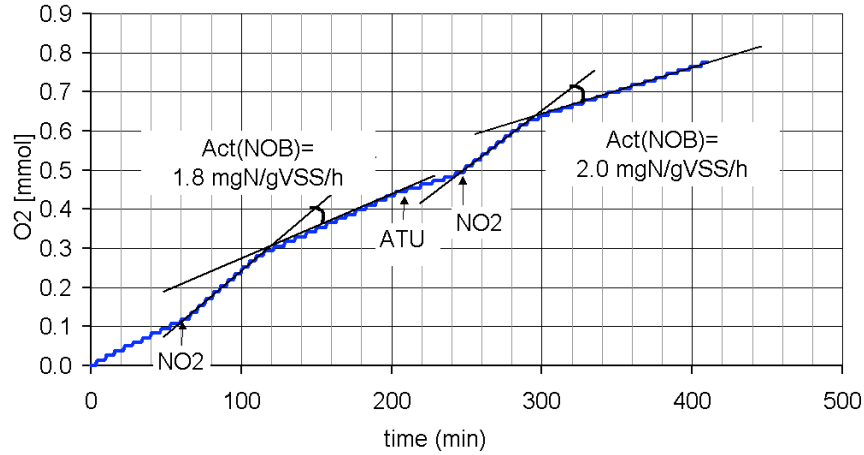
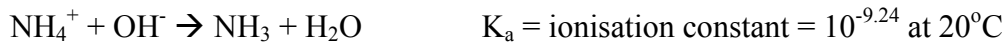


Figure 5: DO-stat titration curve for the assessment of the effect of ATU on NOB activity.

The effect of free ammonia was evaluated by measuring the decrease in NOB activity under increasing free-ammonia concentration (FA). The latter can be modified by varying the total ammoniacal nitrogen ($TAN = N-NH_3 + N-NH_4^+$), or by varying the suspension pH. A typical DO-stat titration curve obtained in these tests is shown in Figure 6. The ratio between dissociated (i.e. free) ammonia and TAN can be derived by their chemical equilibrium:



thus, the following equation is known to relate free-ammonia to TAN and pH:

$$[NH_3] = [TAN] \cdot \frac{10^{pH}}{K_a / K_w + 10^{pH}} \quad \text{with: } K_a/K_w = \exp[6334/(273+t)];$$

where: K_w = ionisation constant for water = $0.69 \cdot 10^{-14}$ at 20°C ;
t = temperature in $^\circ\text{C}$.

Tests were performed on sludge samples freshly drawn from the lab-scale SBR, whose AOB activity was inhibited by addition of 15 mg/L of ATU. Tests were conducted at pH of 7.5 and 8.3 which corresponds to the lowest and highest pH range observed in the SBR. Sludge concentration varied within 2.3 and 3.8 gVSS/L and temperature within 22.5 and 26.5 $^\circ\text{C}$. The following TAN were tested (three/four concentrations per each inhibition test):

Tests at pH = 7.5: 0, 20, 40, 50, 80, 100, 150, 200, 400, 600, 800 mgTAN/L;

Tests at pH = 8.3: 0, 20, 40, 50, 60, 80, 100, 150, 200, 300, 400, 600, 700, 800, 1000 mgTAN/L.

The percentage inhibition of NOB activity was plotted against both TAN (Figure 7) and FA (Figure 8). One can observe that similar inhibition responses were observed in the presence of different TAN concentrations but similar FA. Thus, it can be concluded that FA more than TAN is responsible for the observed inhibition. Inhibition (I) versus FA data were fitted with several inhibition functions (Webb, 1963; Kroiss et al., 1992), obtaining the following R^2 :

- linear: $I = a \cdot FA + b$ $R^2 = 0.954$;
- non-competitive: $I = FA / (a + FA)$ $R^2 = 0.925$;
- competitive: $I = FA / [a + b(1 + I/a)]$ $R^2 = 0.878$;
- exponential: $I = 1 - \exp(-a \cdot FA)$ $R^2 = 0.972$;
- S-curve: $I = 1 - 1 / [1 + (FA/a)^b]$ $R^2 = 0.958$.

The exponential curve gave the best fitting, as shown in Figure 9.

However, when considering TAN concentrations in the SBR reactor, they never exceeded 15 mgN/L corresponding, at 22°C and at pH 8.3 (maximum pH value which is normally reached at the end of the aerobic react phase, i.e. when TAN is at its minimum), to a FA of 1.25 mgN/L. At this concentration, less than 2% inhibition would be predicted by the best-fitting curve. It can thus be concluded that FA inhibition on NOB activity cannot be relevant in the SBR working conditions.

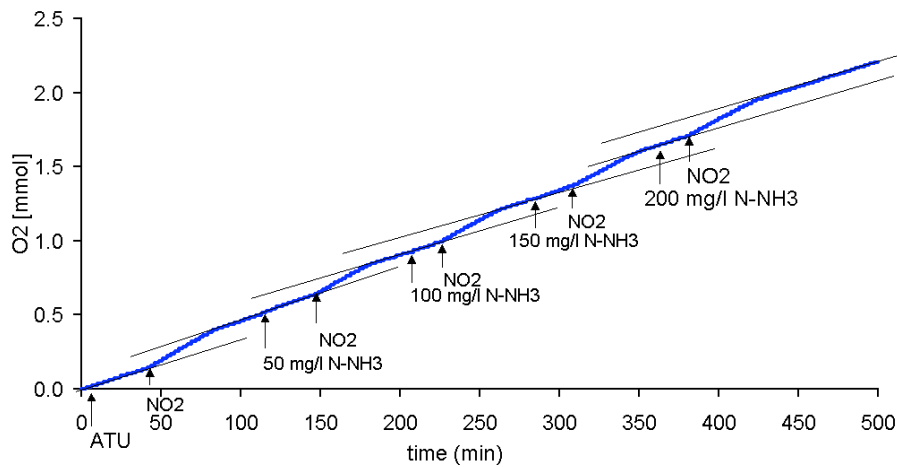


Figure 6: Typical titration curve of DO-stat tests for the assessment of the effect of increasing FA concentration on NOB activity.

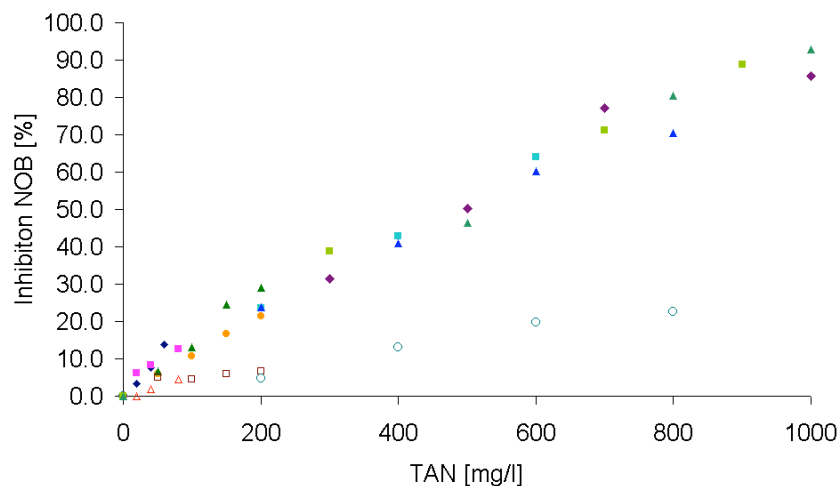


Figure 7: NOB activity inhibition by total ammonical nitrogen (TAN). Tests at pH 8.3 (full indicators) and at pH 7.5 (empty indicators).

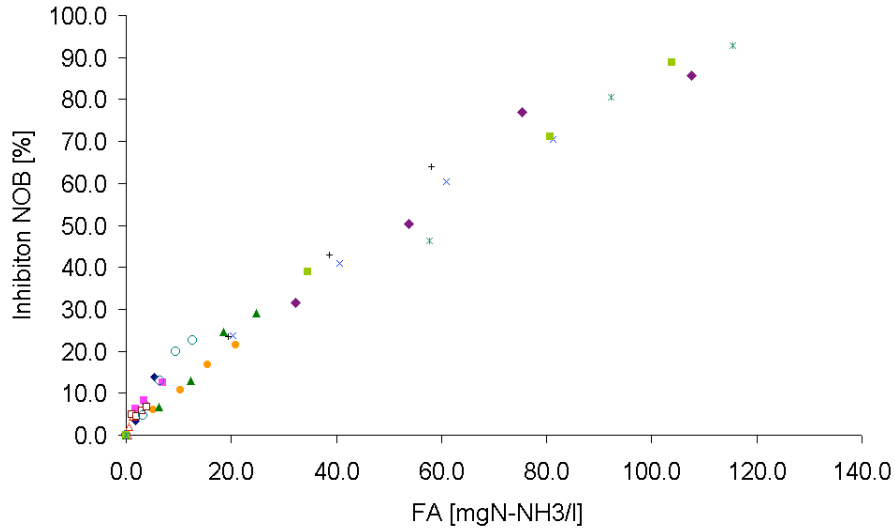


Figure 8: NOB activity inhibition by free ammonia (FA). Tests at pH 8.3 (solid symbols) and at pH 7.5 (open symbols).

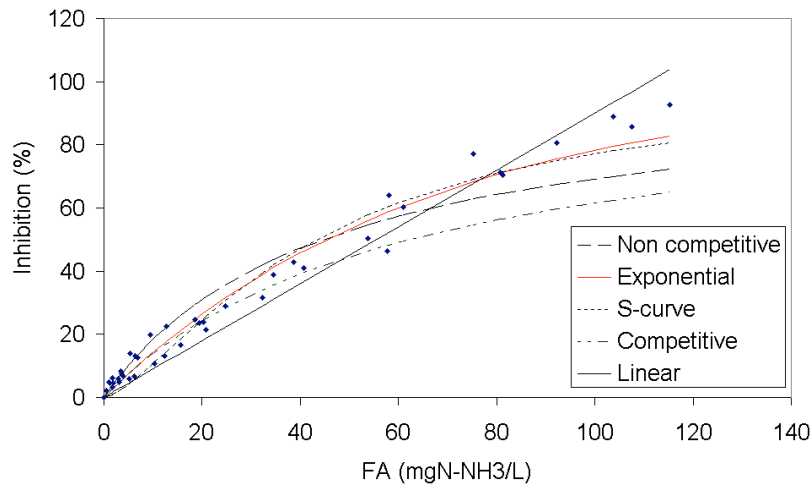


Figure 9: Fitting experimental data by several inhibition curves.

3.4 Effect of the aeration pattern on the endogenous decay rate

Three tests were performed to assess the effect of the aeration pattern on the endogenous decay rate of activated sludge bacteria. For this purpose, sludge was kept under endogenous conditions with, without and with alternate aeration. Batch A was not aerated and kept mixed by a magnetic stirrer set at the minimum velocity avoiding sludge settling. The redox potential (ORP_{AgCl}), initially above +180 mV, decreased to -200 mV after 10 hours without aeration. Therefore, this batch can be considered to be under anaerobic conditions. Batch B was continuously mixed and aerated, the DO level and redox were respectively 7-8 mg/L and +30-40 mV after 1 day. Batch B was fully aerobic for 12h/d and anoxic for the remaining 12h. Finally, in batch D, with cyclic aeration (1h), each time the aerator was switched off, DO decreased slowly and anoxic conditions were reached after approximately 40 min (redox potential was then around -50 mV). Therefore, it can be stated that this sludge was under aerobic conditions for 80% of the time and under anoxic conditions for the remaining 20%.

Endogenous oxygen uptake rate (OUR_{end} , $mgO_2/L/h$), ammonium and nitrites oxidation rates ($mgN/L/h$) were measured after 4 and 7 days.

As for the endogenous respiration, the average ratio between OUR_{end} at time 0 ($OUR_{end}(0)$) and at time t ($OUR_{end}(t)$) for the four batches are reported in Figure 10. An excellent reproducibility was obtained. By assuming a constant decay rate, b , for biomass, a negative exponential trend would be expected (i.e.: $OUR_{end}(t)=OUR_0 \cdot \exp(-b \cdot t)$). On the contrary, data indicate a much slower decay during the first four days than the one observed from day 4 to day 7. This may be explained by supposing that some substrate was “stored” or “biologically absorbed” and subsequently used for growth during the first endogenous period leading to a lower net decay rate. By comparing the four batches, after 7 days a relevant difference can be observed between the decrease in the aerobic respiration capacity of the anaerobic batch and the others, possibly due to the long exposure to anaerobic conditions. No significant difference can be observed among batches B, C, D.

The average trend in the loss of VSS concentration is plotted in Figure 11. A satisfying correspondence among the three repetitions was found for all data but those related to batch C at day 4. A limited loss in VSS was observed within day 0 and day 4, while a higher VSS loss was measured between day 4 and day 7 which appeared to be more relevant for the more aerobic batches (B and D) than for the anaerobic one. An intermediate VSS loss was observed for batch C (50% anoxic, 50% aerobic).

By comparing Figure and Figure , it appears that the loss in aerobic respiration capacity was more relevant than the loss in VSS content. As a matter of fact, biomass decay and lysis converts living biomass into particulate cell debris (measured as VSS), which is then hydrolyzed to soluble products (not accounted as VSS). Data related to the anaerobic batch indicate that under these low redox conditions the loss in respiration capacity is the highest, while, on the contrary, VSS did not decrease accordingly, possibly due to a less efficient production of hydrolytic enzymes and therefore to a slower conversion of particulate organic matted into soluble products.

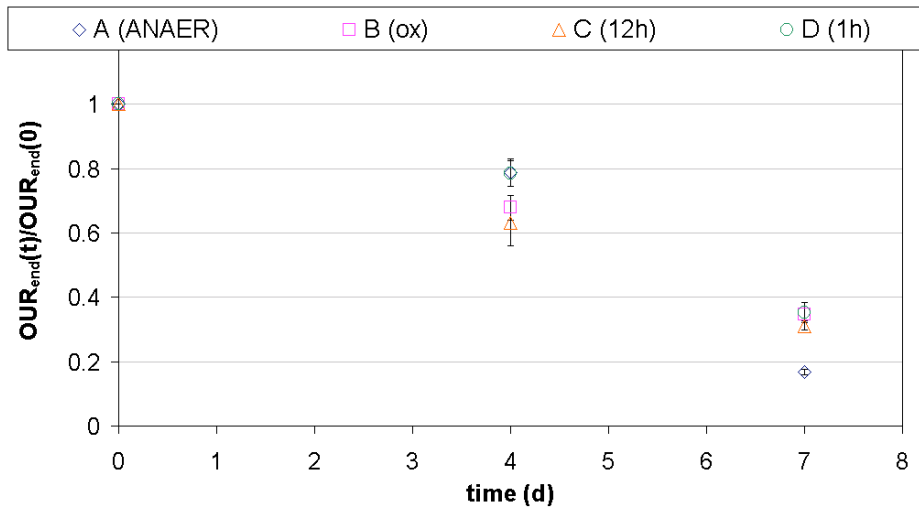


Figure 10: Trend of the loss in aerobic respiration capacity for the four sludge batches. Error bars refer to the standard deviation calculated on 3 repetitions.

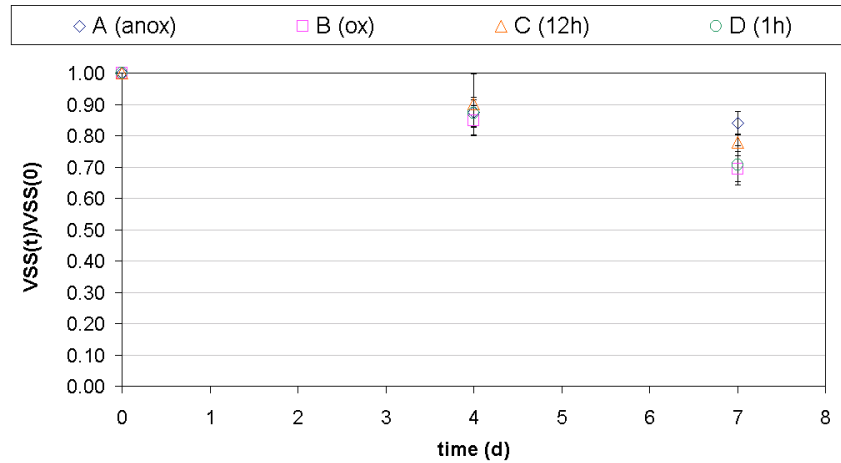


Figure 11: Trend of the loss in VSS concentration for the four sludge batches. Error bars refer to the standard deviation calculated on 3 repetitions.

As for the nitrification capacity, Figure 2 and Figure 3 depict the time trend and loss in nitrification activity for both ammonium and nitrite oxidisers. AOB activity was found to decrease much faster in batches B and D in which aerobic conditions prevailed. Less than 20% of the initial activity was measured after 7 days. Batch C was only slightly more performing. On the contrary, a significantly higher nitrification activity was observed for the sludge kept under anaerobic conditions, which lost about 40% of the initial activity in 7 days. The same can be stated for NOB, although the higher data dispersion makes differences less significant. Moreover, it appears that NOB activity was higher than AOB activity for all batches but the anaerobic one, in which a similar activity was measured for both nitrifying bacteria groups. The presence of a balanced nitrifying population is indicated by a NOB/AOB activity ratio higher than 1. As it is shown in Figure 4, this is the case for all aerobic/partially-aerobic batches, while, in the anaerobic batch this ratio slightly decreases with time. It can therefore be concluded that under aerobic/partially-aerobic conditions, on the one hand, the rate of loss in sludge nitrification capacity increases but, on the other hand, NOB are favoured over AOB, thus preventing the risk of an unbalanced nitrifiers population. On the contrary, anaerobic conditions better preserve nitrification capacity, but may reduce the nitrification over the nitrification capacity of the sludge.

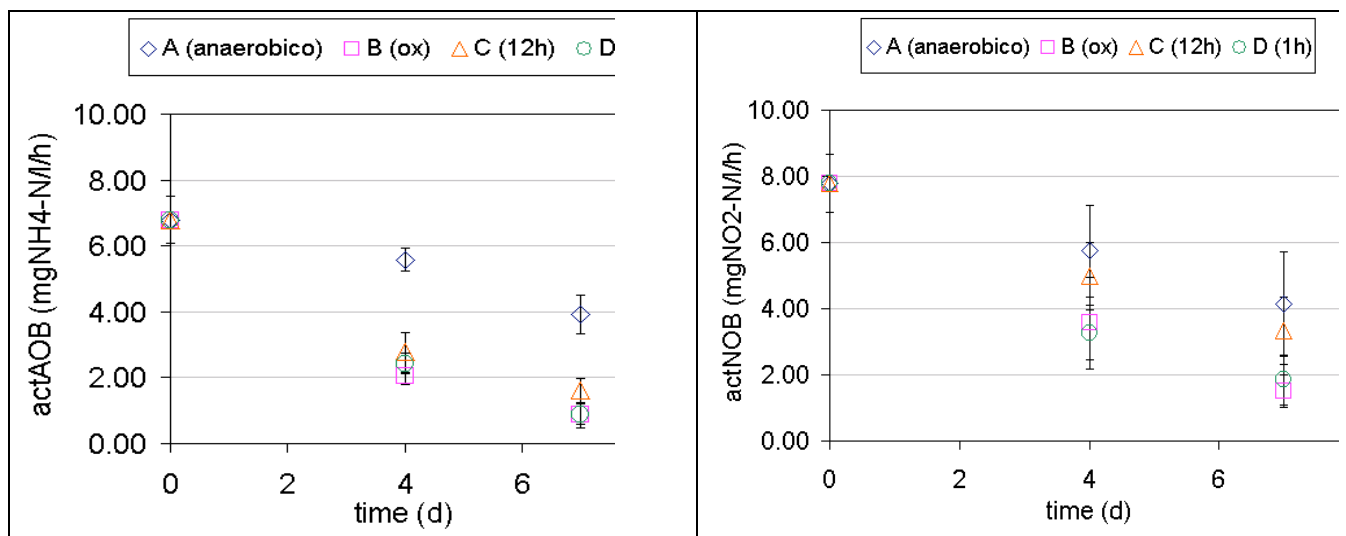


Figure 12: Trend of AOB and NOB activity for the four sludge batches. Error bars refer to the standard deviation calculated on 3 repetitions.

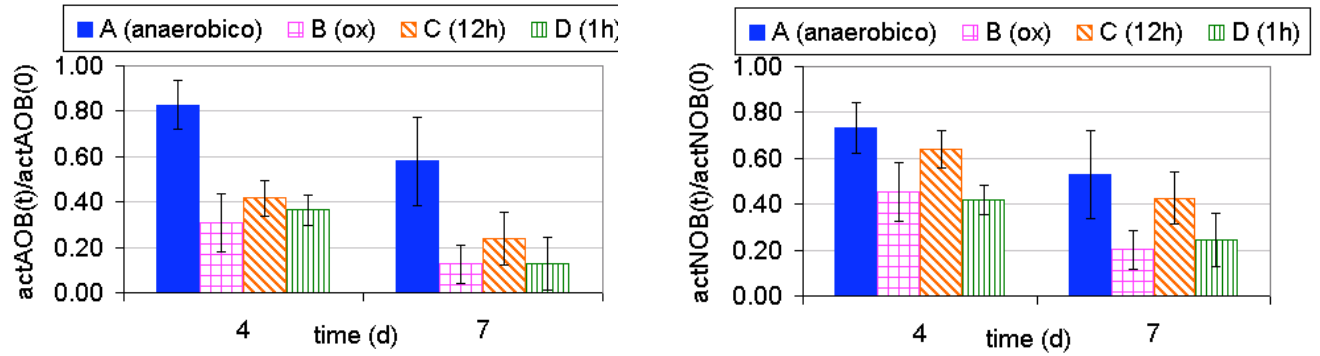


Figure 13: Loss in AOB and NOB activity for the four sludge batches. Error bars refer to the standard deviation calculated on 3 repetitions.

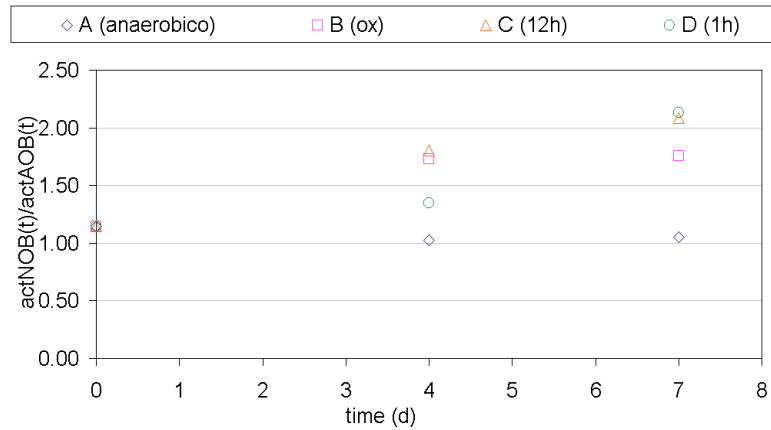


Figure 14: Trend of the ratio between NOB and AOB activity for the four sludge batches.

It can be observed that the decrease in nitrification activity is not well described by a negative exponential curve as it is the case when a first order decay is the sole active process on nitrifying biomass. On the contrary, nitrifiers may also grow on ammonia coming from ammonification of biomass debris and this may reduce the net decay rate.

Therefore, in order to estimate the decay rate of nitrifying biomass, the ASM1 model was modified by considering a two step nitrification (as suggested by Nowak et al., 1995) and it was implemented in AQUASIM. Standard values were adopted for all parameters except for nitrifiers half saturation constant for oxygen, which were set to the values estimated by pH-DO-stat tests (see previous paragraph). The initial concentration of Nitrifiers was set at a value which could fit the observed nitrification activity at day “0”: it resulted to be 1.6%, for AOB and 0.7%, for NOB, of the VSS concentration. Experimental data of AOB and NOB activity in the following days were fitted by choosing different decay rates for AOB and NOB which were assumed to be different under aerobic/anoxic and anaerobic conditions (Figure 15).

Results are summarized in Table 1. Aerobic/anoxic decay rate were found to be quite high, although few data are available on decay rate measured by taking into account biomass growth on lysis products. Note that experiments were performed at a quite high temperature (23°C).

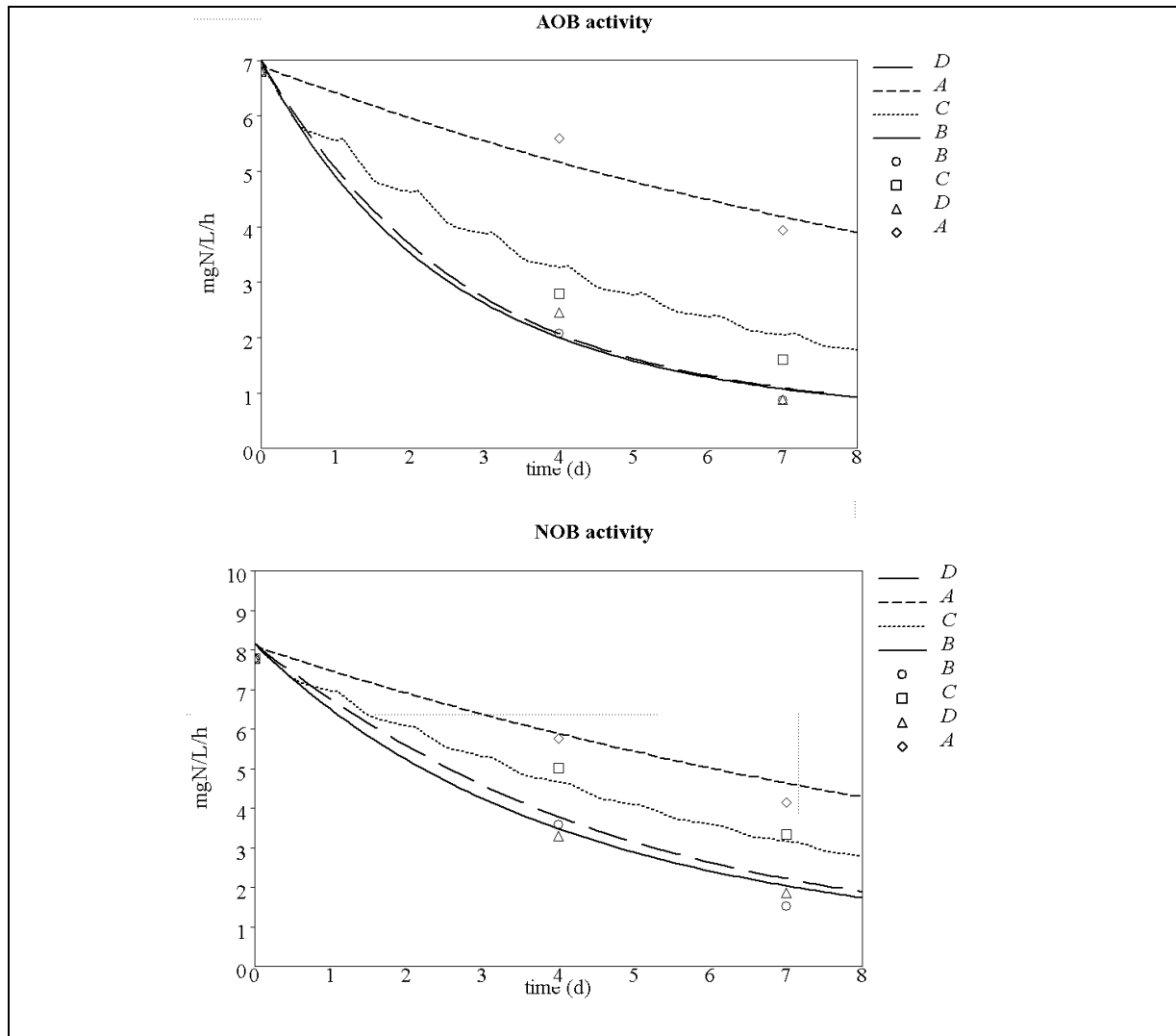


Figure 15: Simulation of the evolution of nitrification activity by ASM1.

Table 1: Estimates of the decay constant for nitrifiers

Redox condition	decay rate (d^{-1})	
	AOB	NOB
Aerobic	0.45	0.30
Anoxic	0.08	0.08
Anaerobic	0.08	0.08

3.5 Micronutrients availability in the SBR feed

Finally, the limitation of NOB growth by micronutrients availability in the SBR feed was also studied. A micronutrient solution was therefore supplemented for approximately 1 month and then suspended for three weeks, supplemented again for another month and suspended for one more month. During this period NOB activity was monitored (Figure 216). It can be clearly observed that NOB activity grew as long as micronutrients were supplemented, while it decreased during non-supplemented periods, resulting to be significantly lower than AOB activity. Nitrites build up was not observed since the SBR loading was kept low (Nitrogen loading = 60 mgN/L/d). However, nutrient limitation seems to be a key-factor for nitrites build-up in lab-scale SBR fed on synthetic sewage, while in would be presumably much less relevant for full-scale plants fed on real sewage.

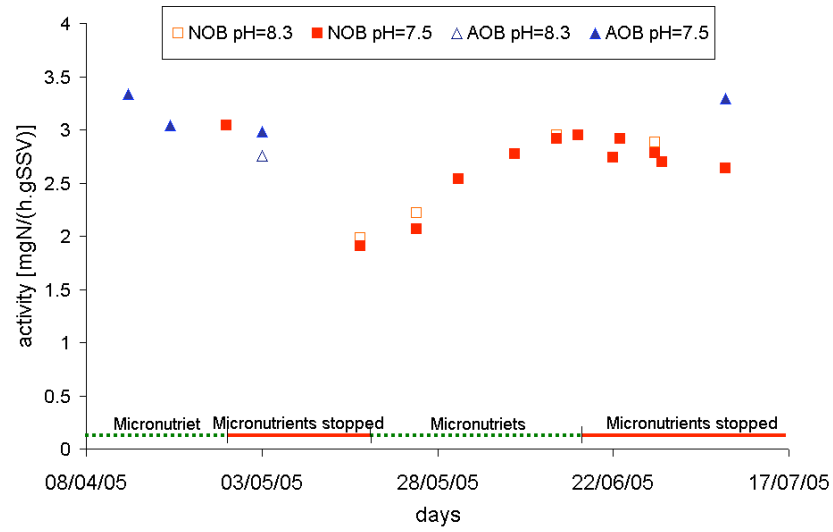


Figure 26: Effect of the supplementation of micronutrients on the nitrification activity of the SBR sludge.

IV Conclusions

The following conditions were found as being favourable to ammonia oxidisers over nitrite oxidisers, thus possibly leading to nitrite build-up:

- very low oxygen concentration (less than 1 mg/L) during the aerobic react phase;
- high pH values (above 8.3) during the aerobic react phase;
- free ammonia concentration above 10-20 mgN/L;
- prolonged endogenous periods under anaerobic conditions, which may take place during the anoxic mixed-react phase under low loading conditions;
- addition of micronutrients to the SBR feed appeared to be fundamental to support a balanced nitrifying population.

Considering the above described Abnormal Condition Nitrite Build-Up identified for the process of the POLIMI SBR reactor, and the safe process framework, WP6 concluded the following remarks:

The diagnostic procedure based on signal processing described in Deliverable 6.1 could not be applied and therefore verified through WP1 due to following reasons:

- Sufficient historical process data with reliable diagnostic information required for the procedure was not available.
- The validated model EM2a reported in Deliverable 2.3 by EOLI, could not be used to reproduce the abnormal conditions of the experiments analysis above. As a consequence this model could not be used for the generation of the data sets with diagnostic information as required by the procedure.

As a consequence, the only information that can be leveraged are recommended conditions for the reactor operation, as described formerly. These can be used for a simple monitoring system (see Figure 27, [Iserman, 1997]), that evaluates the corresponding process signals and informs the operator if conditions are unfavorable (i.e. known to cause an effect on the NOB activity) and could cause nitrite build-up to occur.

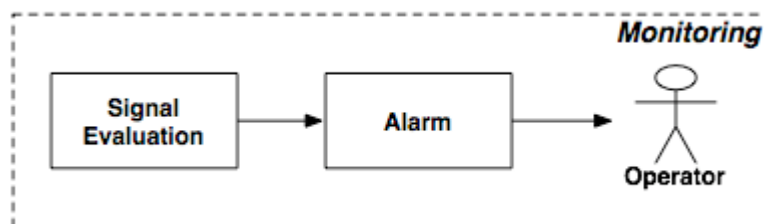


Figure 27 Monitoring with Threshold

For more elaborate process fault diagnosis, further investigation and experiments will be required, to either provide historical data sets with diagnostic information (e.g. nitrite concentration measurements) or to elaborate a more adequate process model that allows an analytical approach or at least the generation of data sets for a procedure based on feature extraction and classification.

V References

- Anthonisen A.C., Loehr R.C., Prakasam T.B.S. and Snirath E.G. (1976). Inhibition of nitrification by ammonia and nitrous acid. *J. W.P.C.F.* 48(5), 835-852.
- Bliss, P.J; Barnes, D (1986). Modelling nitrification in plant-scale activated sludge. *Water Sci. Technol.* 18(6), 139-148.
- Hanaki K. and Wantawin C. (1990). Nitrification at low levels of dissolved oxygen with and without organic loading in a suspended-growth reactor. *Water Res.* 24(3), 297-302.
- Isermann R. (1997): "Supervision, Fault-Detection and Fault-Diagnosis Methods - An Introduction"
- Kroiss H., Schweighofer W., Frei W. and Matsché N. (1992). Nitrification inhibition – a source identification method for combined municipal/industrial wastewater treatment plants. *Wat. Sci. Technol.*, 26(5-6), 1135-1146.
- Larsen T. and Harremoes P. (1994). Degradation mechanisms of colloidal organic matter in biofilm reactors. *Water Res.* 28(6), 1443-1452.
- Nowak O., Svandal K., Schweighofer P. (1995) The dynamic behaviour of nitrifying activated sludge systems influenced by inhibiting wastewater compounds. *Wat. Sci.Tech.* 31, (2), pp.115-124.
- Webb J.L. (1963). *Enzyme and Metabolic inhibitors*. Vol. 1, Academic Press, NY, London.

Annex 1 EOLI-Questionnaire Sensors and Actuators

Please provide a table for each equipment installed in your reactor. We did not include a table for the hardware sensors developed in WP3, but may be André and Nicola can help us to define some abnormal conditions with the new equipments which can be considered in the WP6 and WP7 (Development of the Fault Diagnosis and Isolation System and Supervision system development).

We classified 3 fault levels for each equipment and the rate indicates the number of occurrences of the fault during the reactor operation hours (#fault/hr).

The levels are:

- 1) “**Fuzzy**” means that the behavior is abnormal but still reasonable (noisy or spectrum out of shape)
- 2) “**Confuse**” (creasy) means that it is evident that the behavior is substantially abnormal, but the duration of this condition is too short to diagnostic the fault or the fault is intermittent.
- 3) “**Blind**” means the signal has no relation with any expected value and this effect persists during a long time.

The working months means the number of total months which the equipment has been used without maintenance or replacement.

The rows of the tables indicate the most common faults for each equipment, but if an important case is missing, please don't hesitate to add a row that describes the condition. If you think we missed important cases or you don't understand what we want, please give us a comment about it.

1. Faults in Sensors and Transmitters

1.1. PH, type of sensor _____ Manufacturer _____
installed in _____

Effect or Fault	Fuzzy Rate	Confused Rate	Blind Rate	Working months
Lost power				
Transmitter disconnected				
Lost temperature compensation				
Dirty membrane				
Cracked membrane				
Broken Electrode				
Drift				
Human				
Worn-out				
Unknown				
Others/describe _____				
General remarks _____				

1.2. Dissolved Oxygen, type of sensor _____ Manufacturer _____
installed in _____

Effect or Fault	Fuzzy Rate	Confused Rate	Blind Rate	Working months
Lost power				
Disconnected transmitter				
Lost temperature compensation				
Dirty membrane				
Cracked Membrane				
Broken Electrode				
Drift				
Human				
Worn-out				
Unknown				
Reading out of the normal conditions				
Others/describe _____				
General remarks _____				

1.3. Temperature, type of sensor _____ Manufacturer _____
installed in _____

Effect or Fault	Fuzzy Rate	Confused Rate	Blind Rate	Working months
Lost power				
Disconnected transmitter				
Open circuit in the transducer				
Leak of the thermal fluid (thermocouple)				
Thermal well dirty or debris filled (thermocouple)				
Drift				
Human				
Worn-out				
Unknown				
Others/describe _____				
General remarks _____				

1.4. Flow rate meter, type of sensor _____ installed in _____

Effect or Fault	Fuzzy Rate	Confused Rate	Blind Rate	Working months
Lost power				
Drift				
Human				
Worn-out				
Unknown				
Others/describe _____				
General remarks _____				

1.5. Level, type of sensor _____ Manufacturer _____
 installed in _____

Effect or Fault	Fuzzy Rate	Confused Rate	Blind Rate	Working months
Lost power				
Disconnected transmitter				
Lost temperature compensation (sonic or ultrasonic)				
Receptor dirty (sonic or ultrasonic)				
Plates dirty (capacitor sensor)				
Inappropriate tank ground (capacitor sensor)				
Sewage flow out of the reactor				
Sewage reach erroneous level				
Human				
Worn-out				
Unknown				
Drift				
Others/describe _____				
General remarks _____				

1.6. Light intensity, type of sensor Optic installed in _____ (LBE)

Effect or Fault	Fuzzy rate	Confuse Rate	Blind Rate
Dirty			
Worn-out			
Human			
Unknown			

2. Faults in Actuators

2.1. Servo-valve, type of actuator _____ Manufacturer _____
installed in _____

Effect or Fault	Fuzzy Rate	Confused Rate	Blind Rate	Working months
Signal conversion stage failed				
Modulator system disconnected or failed				
Valve Not moving				
Not closing fully				
Not opening fully				
Movement sluggish				
Human				
Worn-out				
Unknown				
Others/describe _____				
General remarks _____				

2.2 Pump, type of actuator _____ Manufacturer _____
installed in _____

Effect or Fault	Fuzzy Rate	Confused Rate	Blind Rate	Working months
Lost power				
Inlet or outlet valve blocked (positive displacement pump)				
Piston blocked (positive displacement pump)				
Rising vibration				
Human				
Worn-out				
Unknown				
Others/describe _____				
General remarks _____				

2.3 Air blower, type of actuator _____ Manufacturer _____
 installed in _____

Effect or Fault	Fuzzy Rate	Confused Rate	Blind Rate	Working months
Lost power				
System blocked				
Human				
Worn-out				
Unknown				
Not moving				
Others/describe _____				
General remarks _____				

2.4 Mixer, type of actuator _____ installed in _____

Effect or Fault	Fuzzy Rate	Confused Rate	Blind Rate	Working months
Lost power				
Decreasing power in the motor				
Eccentric rotation or rising vibration				
Human				
Worn-out				
Unknown				
Others/describe _____				
General remarks _____				

3. Abnormal Process Conditions

In the following, you can find the questionnaire that we have put together for abnormal process conditions that you think might occur and/or have occurred in the SBR. Abnormal conditions are to be understood as process conditions that are not considered by control and that you consider to be outside of the normal operating conditions of your process.

Please provide one filled in questionnaire for each case of abnormal condition.

Questionnaire (Template for each specific case)

Abnormal Condition: (Name, short name)

1. Description
2. Description of observations or reasoning indicating the abnormal condition
3. Description of actual or possible operator action
4. Occurred or expected to occur in your EOLI dedicated reactor? (Yes/no/don't know)
5. Frequency or expected frequency of occurrence (low, medium, high)
6. Importance/criticality you would assign to this abnormal condition (1=low-5=high)
7. References or literature that you consider to be related to this abnormal condition

Fault Occurrence Statistics for Device													
SENSORS		Uruguay	Italia	México	Francia	Fault Frequency Normalization				Fault Weighting			
Fault	pH	Type	Type	Type	Type	times / 6 months				F=33.33 , C=66.66 , B=100			
		Electrode	InLab 412 ²	720A ²		Uruguay	Italia	México	Francia	Uruguay	Italia	México	Francia
		P - 6m	P - 6m	P - 0.5m		0	6	0	0	0	100	0	0
1	Lost Power		B- 1 t/m			0	0	12	0	0	0	33.33	0
2	Transmitter Disconnected			F- 2 t/m		0	0	12	0	0	0	100	0
3	Broken Transmitter			B- 2 t/m		0	0	24	0	0	0	33.33	0
4	Dirty Membrane			F- 4 t/m		0	0	0	0	0	0	33.33	0
5	Drift	F- 2 t/w				48	0	0	0	33.33	0	0	0
6	Worn-Out		F- 1 t/6m	F- 1 t/5y		0	1	0.1	0	0	33.33	33.33	0
7	Human		C- 1 t/w			0	24	0	0	0	66.66	0	0
8	Unknown		F- 5 t/h			0	21600	0	0	0	33.33	0	0
										Device Vulnerability Index =			
										22692.63			
Fault	Dissolved Oxygen	Type	Type	Type	Type	Fault Frequency Normalization				Fault Weighting			
		Electrode	COS 3s ²	COS4 COM 223 ²⁴		times / 6 months				F=33.33 , C=66.66 , B=100			
		P - 6m	P - 6m	P - 10m		Uruguay	Italia	México	Francia	Uruguay	Italia	México	Francia
1	Lost Power		B- 1 t/m			0	6	0	0	0	100	0	0
2	Dirty Membrane	F- 2 t/d	C- 1 t/h	F- 1 t/w		360	4320	24	0	33.33	66.66	33.33	0
3	Cracked Membrane	F- 1 t/3m		B- 1 t/10m		2	0	0.6	0	33.33	0	100	0
4	Drift			F- 1 t/w		0	0	24	0	0	0	33.33	0
5	Worn-Out		F- 1 t/6m			0	1	0	0	0	33.33	0	0
6	Human		C- 3 t/w			0	72	0	0	0	66.66	0	0
7	Unknown		B- 1 t/m			0	6	0	0	0	100	0	0
										Device Vulnerability Index =			
										40990.33			
Fault	Temperature	Type	Type	Type	Type	Fault Frequency Normalization				Fault Weighting			
		Electrode	Pt100 ²	Inc. in DOx Sensor		times / 6 months				F=33.33 , C=66.66 , B=100			
		P - 6m	P - 6m			Uruguay	Italia	México	Francia	Uruguay	Italia	México	Francia
1	Lost Power		B- 1 t/m			0	6	0	0	0	100	0	0
2	Dirty Electrical Connection		B- 1 a 2 t/m			0	12	0	0	0	100	0	0
										Device Vulnerability Index =			
										225.00			
Fault	Level	Type	Type	Type	Type	Fault Frequency Normalization				Fault Weighting			
		3 Metal Elect.		Level Switch ²		times / 6 months				F=33.33 , C=66.66 , B=100			
		P - 6m				Uruguay	Italia	México	Francia	Uruguay	Italia	México	Francia
1	Transmitter Disconnected	B- 1 t/w				24	0	0	0	100	0	0	0
2	Inappropriate Tank Ground	B- 1 t/6m				1	0	0	0	100	0	0	0
3	Sewage Flow Out of Reactor	C- 1 t/2d				90	0	0	0	66.66	0	0	0
4	Sewage Reach Err. Level	C- 1 t/d ~10%				180	0	0	0	66.66	0	0	0
5	Dirty Electrodes	?- 1 t/w				24	0	0	0	33.33	0	0	0
										Device Vulnerability Index =			
										664.91			
Fault	Flow Rate	Type	Type	Type	Type	Fault Frequency Normalization				Fault Weighting			
		Rotameter				times / 6 months				F=33.33 , C=66.66 , B=100			
		P - 6m				Uruguay	Italia	México	Francia	Uruguay	Italia	México	Francia
0	No Fault					0	0	0	0	0	0	0	0
										Device Vulnerability Index =			
										0.00			
ACTUATORS						Fault Frequency Normalization				Fault Weighting			
Fault	Servo-Valve	Type	Type	Type	Type	times / 6 months				F=33.33 , C=66.66 , B=100			
		Solenoid ¹				Uruguay	Italia	México	Francia	Uruguay	Italia	México	Francia
		P - 6m				0	0	0	0	0	0	0	0
1	Not Closing Fully	C- 1 t/w				24	0	0	0	66.66	0	0	0
2	Not Opening Fully	C- 1 t/w				24	0	0	0	66.66	0	0	0
										Device Vulnerability Index =			
										399.96			
Fault	Pump	Type	Type	Type	Type	Fault Frequency Normalization				Fault Weighting			
		Peristaltic	Peristaltic ³	Peristaltic ⁵		times / 6 months				F=33.33 , C=66.66 , B=100			
		P - 6m	P - 6m	P - 10m		Uruguay	Italia	México	Francia	Uruguay	Italia	México	Francia
1	Lost Power		B- 1 t/m			0	6	0	0	0	100	0	0
2	Line Clogging	?- 1 t/6m				1	0	0	0	33.33	0	0	0
3	Worn-Out		B- 1 t/6m			0	1	0	0	0	100	0	0
4	Human	C- 1 t/6m		F- 1 t/4m		1	0	1.5	0	66.66	0	33.33	0
5	Unknown			F- 1 t/m		0	0	6	0	0	0	33.33	0
										Device Vulnerability Index =			
										52.50			
Fault	Air Blower	Type	Type	Type	Type	Fault Frequency Normalization				Fault Weighting			
		Rec. Air Comp.	Mem. Air Comp.	Air Compressor		times / 6 months				F=33.33 , C=66.66 , B=100			
		P - 6m	P - 6m	P - 10m		Uruguay	Italia	México	Francia	Uruguay	Italia	México	Francia
1	Lost Power	B- 1 t/6m	B- 1 t/m			1	6	0	0	100	100	0	0
2	Blocked Diffuser	F- 1 t/d				180	0	0	0	33.33	0	0	0
3	Broken Air Line	?- 1 t/6m				1	0	0	0	33.33	0	0	0
4	Worn-Out		B- 1 t/6m			0	1	0	0	0	100	0	0
5	Unknown			F- 1 t/m		0	0	6	0	0	0	33.33	0
										Device Vulnerability Index =			
										351.64			
Fault	Mixer	Type	Type	Type	Type	Fault Frequency Normalization				Fault Weighting			
		Helix Impeller	Mec. Var-Speed			times / 6 months				F=33.33 , C=66.66 , B=100			
		P - 6m	P - 6m			Uruguay	Italia	México	Francia	Uruguay	Italia	México	Francia
1	Lost Power		B- 1 t/m			0	6	0	0	0	100	0	0
										Device Vulnerability Index =			
										150.00			

Fault	Mass Flow Controller	Type	Type	Type	Type	Fault Frequency Normalization				Fault Weighting				Fault Vulnerability Index
				GFC3 ⁷		times / 6 months				F=33.33 , C=66.66 , B=100				
				P - 4y		Uruguay	Italia	México	Francia	Uruguay	Italia	México	Francia	
1	Worn-Out			B- 1t/3y		0	0	0.1666	0	0	0	100	0	4.17
										Device Vulnerability Index =				4.17
General Remarks:														
1. There are 6 solenoid valves operating in each reactor. One of them, in each reactor, fails 1 t/w.														
2. This sensor is cleaned and calibrated 1 t/w.														
3. Pipes cleaning 1 t/m.														
4. Drift appears when the membrane is dirty. Membrane change is done 1 t/10m of operation.														
5. The piping tube must be replaced in average 1 t/15d. When the tubing is changed the pump must be recalibrated.														
6. One week before the failure, the valve do not closed fully and a small leak of air was observed during decantation.														
Notation:														
P _i : Indicates the period during the equipment has been used.														
t: Times (occurrence).														
d, w, m, y: Respectively means, Days, Weeks, Moths and Years.														
F, C, B, ? : Respectively means, Fuzzy, Confuse, Blind and Not Indicated.														

Annex 2 Data of experiments for the assessment of the aeration pattern on the endogenous decay rate of nitrifying biomass

First repetition		specific AOB activity (mgN/(h*gSSV))	specific NOB activity (mgN/(h*gSSV))			
test day	Batch	pH-stat titration	DO-stat titration	TSS (g/L)	VSS (g/L)	VSS/TSS
0	All	2.75	3.13	3.24	2.45	0.76
4	A (anaer)	2.89	2.33	2.65	2.06	0.78
	B (ox)	0.82	1.21	2.98	2.18	0.73
	C (12h)	1.33	2.22	3.22	2.44	0.76
	D (1h)	1.14	1.22	3.01	2.22	0.74
7	A (anaer)	1.57	1.11	2.81	2.13	0.76
	B (ox)	0.74	0.83	2.192	1.616	0.74
	C (12h)	1.03	1.14	2.636	1.976	0.75
	D (1h)	0.63	0.59	2.376	1.748	0.74

Second repetition		specific AOB activity (mgN/(h*gSSV))	specific NOB activity (mgN/(h*gSSV))			
test day	Batch	pH-stat titration	DO-stat titration	TSS (g/L)	VSS (g/L)	VSS/TSS
0	All	2.82	2.61	3.33	2.664	0.80
4	A (anaer)	2.42	2.23	3.1	2.28	0.74
	B (ox)	1.00	1.37	2.9	2.12	0.73
	C (12h)	0.98	1.80	2.925	2.13	0.73
	D (1h)	0.95	1.32	2.99	2.195	0.73
7	A (anaer)	2.12	2.16	2.82	2.125	0.75
	B (ox)	0.52	1.14	2.395	1.815	0.76
	C (12h)	0.69	1.72	2.845	1.995	0.70
	D (1h)	0.45	1.25	2.885	2.035	0.71

Third repetition		specific AOB activity (mgN/(h*gSSV))	specific NOB activity (mgN/(h*gSSV))			
test day	Batch	pH-stat titration	DO-stat titration	TSS (g/L)	VSS (g/L)	VSS/TSS
0	All	2.03	2.9	3.82	3	0.79
4	A (anaer)	1.89	2.63	3.675	2.79	0.76
	B (ox)	0.88	2	3.535	2.575	0.73
	C (12h)	1.1	2.11	3.6	2.715	0.75
	D (1h)	1.01	1.59	3.51	2.66	0.76
7	A (anaer)	1.53	2.12	3.42	2.555	0.75
	B (ox)	0.2	0.51	3.265	2.22	0.68
	C (12h)	0.59	1.84	3.265	2.33	0.71
	D (1h)	0.3	1.02	2.725	1.92	0.70

