SBRs on-line monitoring by set-point titration

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Abstract
The applicability of set-point titration for monitoring biological processes has been widely demonstrated in the literature. Based on published and on-going experiences, some operating procedures have been specifically developed to be applied to SBRs, so that real-time information about the process and/or the influent can be obtained. This, in turn, would allow plant operators to select the most appropriate actions properly and timely. Five operating modes are described for the monitoring of (1) influent toxicity, (2) influent N-content, (3) nitrification capacity, (4) end of the nitrification reaction, (5) nitrate effluent concentration, and are currently tested on the on-line titrator TITAAN (TITrimetric Automated ANalyser) which is in operation on a pilot scale SBR.

Keywords
Denitrification; nitrification; on-line monitoring; pH/DO-stat titration; SBR.

INTRODUCTION

The main challenge in the management of a sequencing batch reactor, for the biological treatment of urban and industrial wastewaters, is to ensure a stable treatment efficiency under highly variable influent quality and quantity. To help SBR operators to cope with this challenge, on-line instrumentation is fundamental, since it allows to characterize both influent variability and process efficiency. The more the on-line data are closely correlated with influent quality and SBR's treatment capacity, the more straightforward is to implement control strategies based on these data.

As it is widely demonstrated in the literature of the past decade, pH/DO-stat titration allows the assessment of two kind of information of great interest in the monitoring of an SBR for N-removal:

- tractability of the influent, in terms of sewage toxicity to the biomass and pollution load (e.g.: Massone et al., 1998; Ficara and Rozzi, 2001; Yuan et al., 2001; Rozzi et al., 2004; Ficara and Rozzi, 2004);
- the biomass treatment capacity, in terms of process rate (see e.g. Ramadori et al., 1980; Massone et al., 1996; Bogaert et al., 1997; Massone et al., 1998; Gernaey et al., 1998; Ficara et al., 2000; Petersen et al., 2002; Foxon et al., 2002).

Under aerobic conditions, pH/DO-stat titration operates as other respirometric techniques, but, differently from respirometry, pH-stat titration is applicable under anoxic conditions as well. Therefore, set-point titration was considered as a suitable technique to be applied to the monitoring of N-removing SBRs.

The following procedures, or modes, have been developed, based on the above listed literature, and experimented:

1) detection of influent acute toxicity to nitrifying biomass;
2) estimation of the influent nitrifiable nitrogen content;
3) measurement of the nitrification capacity of the SBR, assessed in terms of maximum rates of oxidation of ammonium to nitrite and of nitrite to nitrate, allowing to predict nitrite build up;
4) monitoring of ammonium oxidation process during the aerobic react phase of the SBR reactor;
5) estimation of nitrate concentration.

Here below, each mode is described. Measuring procedures and main outputs are presented, as they are implemented by an on-line automated titrator (TITAAN, TITrimetric Automated ANalyser). This titrator is the result of a cooperation between SPES s.c.r.l. (Fabriano, I) and the Technical University of Milan (Politecnico di Milano, I). Main components of this instrument are outlined in Fig. 1.

Experimental titration curves, reported in the following paragraphs, were obtained during an on-going experimentation on the monitoring of a 500 L pilot-scale SBR fed on urban wastewater.

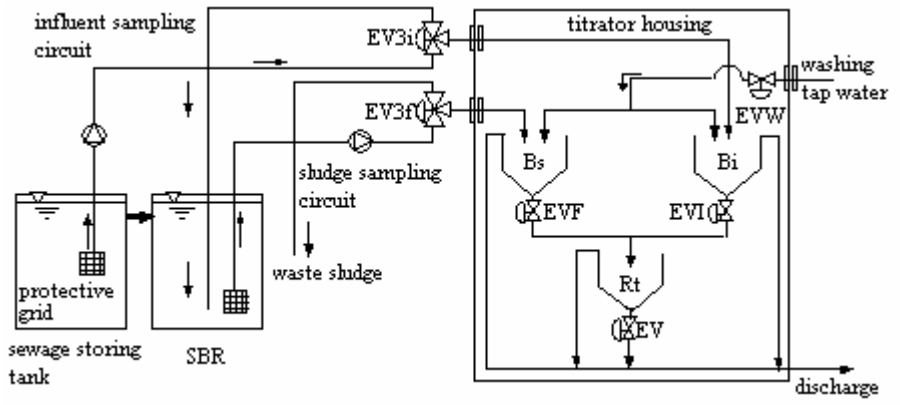

Figure 1 : Main components of the on-line titrator (TITAAN). Bs: sludge storing beaker; Bi: influent storing beaker; Rt: measuring and titration beaker; EV_3: actuated 3-way solenoid valves; EV: actuated solenoid valves.

MODES OF OPERATION

Mode 1: Detection of influent acute toxicity to nitrifying biomass

It consists in performing a pH/DO-stat titration on a SBR sludge sample, to which a selected amount of influent is dosed, to assess the activity of ammonium oxidizing bacteria (AOB) and nitrite oxidising bacteria (NOB). Influent acute toxicity is detected when biomass activities are found to be significantly lower than historical values.

Simplified titration curves obtained according to the following procedure are reported in Fig. 2a, typical experimental data are reported in Fig. 2b.

The titration reactor *Rt* is partially filled with the influent sample on which the toxicity test has to be performed. Then, activated sludge is withdrawn from the SBR tank (normally at the end of the aerobic phase) and loaded in *Rt* (test time t = 0). A known amount of NH_4Cl is dosed in order to ensure a non-limiting ammonium concentration during the whole nitrification test. The aerator is switched on for a short time (1–5 minutes) and titrants, NaOH and H_2O_2, are dosed until set-point values are reached, for both pH and DO. pH/DOstat titration starts when pH and DO set-points have been reached ($t = t_{sp}$). Starting from t_{sp}, titrant volumes are recorded and the corresponding cumulated curves can be drawn (Fig. 2a). Looking at these curves, three or four phases with different titration rates can be observed.

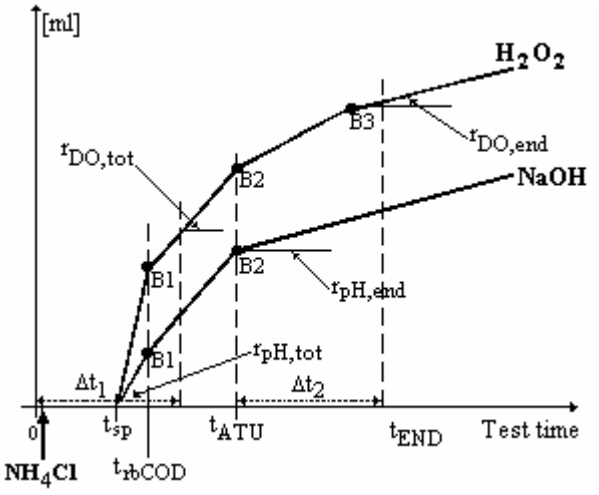

Figure 2a : Scheme of titration curves in MODE 1.

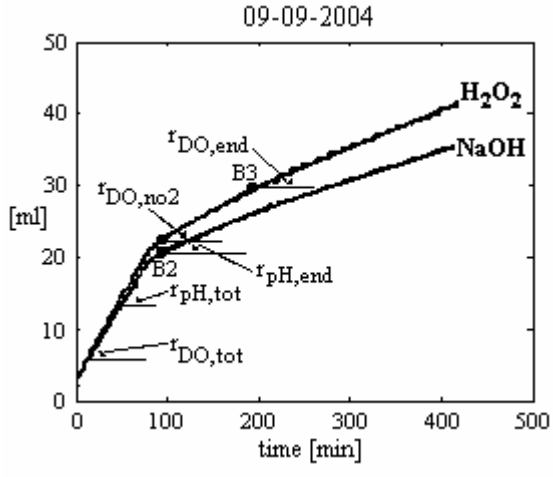

Figure 2b : Actual titration curves : B2 and B3 are automatically detected.

The titration rate is highest initially (at $t<t_{rbCOD}$), because all of the following processes occur simultaneously:

a) O_2 consumption/CO_2 production by heterotrophic oxidation of the rbCOD;
b) O_2 consumption/protons production by autotrophic ammonium oxidation;
c) O_2 consumption by autotrophic nitrite oxidation;
d) O_2 consumption/CO_2 production by heterotrophic oxidation of slowly biodegradable COD (sbCOD);
e) O_2 consumption/CO_2 production by heterotrophic endogenous respiration.

The value of t_{rbCOD} depends on the rbCOD content of the wastewater. If it is very low, it can happen that $t_{rbCOD}<t_{sp}$, and the first bending point (B_1) cannot be observed, as it is the case shown in Fig. 2b. For this reason, a fixed time interval Δt_1 is defined, according to the result of calibration tests, within which the rbCOD is, normally, fully oxidised.

In the following phase ($t>\Delta t_1$), titration rates are due to processes b) to e). The system can identify both slopes ($r_{DO,TOT}$ and $r_{pH,TOT}$) automatically and in real time at a given accuracy.

Once these slopes have been calculated, a peristaltic pump doses allylthiourea in the titration reactor ($t=t_{ATU}$), in order to inhibit ammonium oxidizing bacteria (AOB). A second bending point (B_2) is observed in both titration curves. Later on, only processes c) to e) are active and, therefore, can be titrated. The value of $r_{pH,end}$ is now assessed, which includes endogenous heterotrophic respiration and sbCOD oxidation.

A third bending point on the hydrogen peroxide titration curve (B_3) could be detected, due to the complete oxidation of nitrites. An algorithm tries to identify the bending point B_3. Actually, if ammonium oxidation is slower than nitrite oxidation, no nitrite build-up occurs in the titration reactor. In this case, B_2 and B_3 take place almost simultaneously, making the detection of B_3 impossible. Therefore, a fixed time interval Δt_2 (e.g. $\Delta t_2 = 0.5t_{ATU}$) is established, after which the system calculates the value of $r_{DO,end}$, even if B_3 has not been detected.

By taking into account the stoichiometry of the two nitrification reactions and from the two slopes "r_i" [$mL_{titrant}$/min] of each titration curve, activities (ACT, [$mgN_{oxidized}\ L_{sludge}^{-1}\ h^{-1}$]) of both ammonium and nitrite oxidizing bacteria are calculated as follows:

$$\mathrm{ACT(AOB)} = \frac{(r_{pH,tot} - r_{pH,end}) \cdot M_{NaOH} \cdot f_{stNaOH} \cdot 60\mathrm{min/h}}{\theta^{(T-20)} \cdot V_{sludge}} \qquad (1)$$

$$\mathrm{ACT(NOB)} = \left[\frac{(r_{DO,tot} - r_{DO,end}) \cdot 32\, mgO_2/mmol \cdot M_{O2} \cdot 60\,\mathrm{min}/h}{\theta^{(T-20)} \cdot V_{sludge}} - \mathrm{ACT(AOB)} \cdot f1_{stO2} \right] \cdot \frac{1}{f2_{stO2}} \qquad (2)$$

where:

f_{stNaOH} = 7 mgN/mmolNaOH : stoichiometric factor (oxidation of ammonium to nitrite);
$f1_{stO2}$ = 3,42 $mgO_2/mgN\text{-}NH_4^+$: stoichiometric factor (oxidation of ammonium to nitrite);
$f2_{stO2}$ = 1,14 $mgO_2/mgN\text{-}NH_4^+$: stoichiometric factor (oxidation of nitrite to nitrate);
T : test temperature in degrees Celsius;
θ=1,08 : temperature compensation coefficient for nitrifying bacteria (activity normalized at 20°C);
M_{NaOH} : molarity of the NaOH titrant;
M_{O2} : molarity of the H_2O_2 titrant (in $mmolO_2$/mL);
V_{sludge} : volume of sludge used for the test [L];
$r_{pH,tot}$, $r_{DO,tot}$: slope of the cumulated NaOH/H_2O_2 curve when rbCOD has been consumed;
$r_{pH,end}$: slope of the cumulated NaOH curve when AOB activity has been inhibited;
$r_{DO,end}$: slope of the cumulated H_2O_2 curve when heterotrophic respiration is the only active process.

Toxicity, which is the output of this procedure, is evaluated by comparing actual AOB and NOB activities with their mean values, calculated in a number "*n*" of previous tests and by taking into account the variability of these mean values, expressed as coefficient of variation (CV, % ratio between the standard deviation and the mean).

$$\mathrm{TOX\ (\%)} = \left[\frac{\mathrm{ACT_{mean}} - \mathrm{ACT_{measured}}}{\mathrm{ACT_{mean}}} \right] \cdot 100 \qquad (3)$$

If TOX (%) > 1.2 CV(%), influent acute toxicity is diagnosed and an alarm is generated on the control panel.

Mode 2: Estimation of the nitrifiable nitrogen content of the SBR influent

It consists of a pH/DO-stat test that allows estimating the nitrifiable nitrogen content of the sewage that feeds the SBR. The test is performed by adding a known volume of influent to an activated sludge sample under endogenous conditions. In order to reduce the test duration it is convenient to work with concentrated sludge samples – e.g. drawn from the bottom of the SBR tank at the end of the settling phase or during the idle phase – and with limited influent volumes. A simplified titration curve obtained according to the following procedure is reported in Fig. 3a, typical experimental data are reported in Fig. 3b.

The sludge is withdrawn from the SBR and stored in *Bs*. The influent sample is loaded in *Bi*. Then, the titration reactor is filled with influent and then with sludge up to the working volume.

After reaching set-point values ($t=t_{sp}$), NaOH titration curve is built and, as for Mode 1, it is characterized by two/three phases at different titration rates. The system waits for a fixed time interval (Δt_1) before $r_{pH,TOT}$ is assessed and then the fitting algorithm keeps on calculating titration rates until the bending point B_2 (corresponding to the end of ammonium oxidation) is detected. Then, $r_{pH,end}$ is estimated.

The volume of alkaline titrant added to compensate the acidity produced by nitrification (V_{NIT}) is assessed from the total volume of titrant added (V_{TOT}) as follows:

a) by subtracting the titrant volumes dosed to compensate:

a1) the acidifying effect of the endogenous respiration: $\Delta V_{end} = r_{pH,end} \cdot (t_{END} - t_{sp})$;

a2) the acidifying effect of the rbCOD respiration: $\Delta V_{rbCOD} = V_{\Delta t1} - r_{pH,TOT} \cdot (\Delta t_1 - t_{sp})$;

b) by adding the volume due to nitrification during the time interval $0–t_{sp}$, when no set-point titration was taking place: $\Delta V_{initial} = (r_{pH,TOT} - r_{pH,end}) \cdot t_{sp}$, i.e. it has been assumed that nitrification during $0–t_{sp}$ was taking place at the same rate as later on.

Therefore, the total volume of titrant corresponding to the nitrifiable nitrogen present at the beginning of the test (V_{NIT}) is:

$$V_{NIT} = V_{TOT} - r_{pH,end} \cdot (t_{END} - t_{sp}) - [V_{\Delta t1} - r_{pH,TOT} \cdot (\Delta t_1 - t_{sp})] + (r_{pH,TOT} - r_{pH,end}) \cdot t_{sp} \qquad (4)$$

The corresponding nitrifiable nitrogen (in mgN/L), which is the output of this procedure, is obtained by taking into account the stoichiometric relation between nitrogen oxidation and acidity production:

$$[N] = \frac{V_{NIT} \cdot f_{st\,NaOH} \cdot M_{NaOH}}{V_{influent}} \qquad (5)$$

$V_{influent}$ = volume of influent added [L].

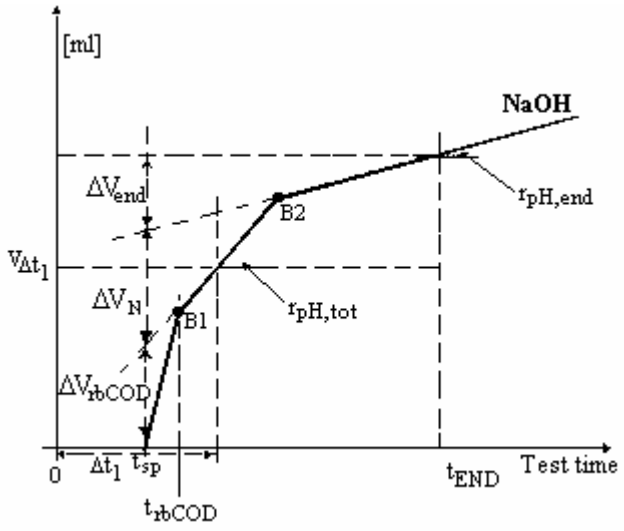

Figure 3a : Scheme of a pH-stat titration curve in Mode 2

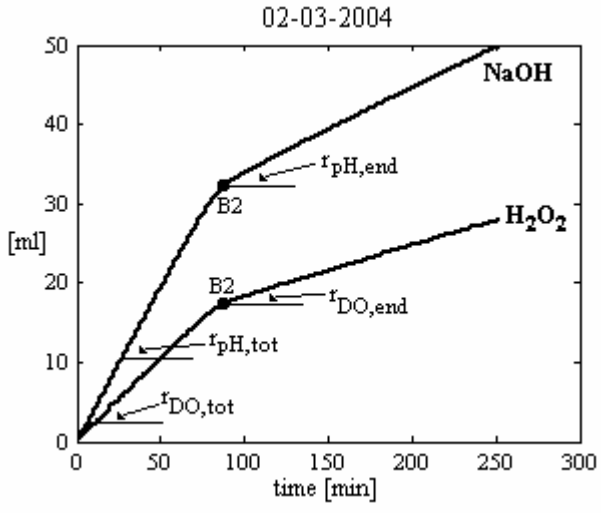

Figure 3b: Experimental titration curves in Mode 2

Mode 3: Measurement of the nitrification capacity of the SBR, assessed in terms of oxidation rates of both ammonium to nitrite and of nitrite to nitrate

Mode 3 is a pH/DO-stat test for the measurement of maximum activities of AOB and NOB which characterize the nitrification capacity of the SBR activated sludge. This type of test can be performed at the end of the idle phase of the SBR reactor, on sludge samples under endogenous conditions. A simplified titration curve according to Mode 3 is reported in Fig. 4a.

The sludge sample is loaded into *Rt*. At the beginning, the control routine brings pH and DO to their set-point values. Then, at time t_{sp}, a known amount of NH_4Cl is dosed by a peristaltic pump to trigger the nitrification process. Titration rates are then measured on-line ($r_{pH,tot}$, $r_{DO,tot}$, $r_{pH,end}$ and $r_{DO,end}$, in Fig. 4a).
Nitrification activities are calculated as in Mode 1 by taking into account that, in the idle phase, the SBR sludge is more concentrated than in the react phase, by a factor V_{Max}/V_{Min} ($V_{Min,}$ V_{max}: SBR volume after effluent decanting/during the react phase). The SBR nitrification capacity (C_{NIT}. $mgN_{oxidised}$/cycle), which is the output of this procedure, is calculated from the lowest between AOB and NOB activity (ACT_{min}):

$$C_{NIT} = ACT_{min} \cdot t_{AER} \cdot V_{Max} \quad (6)$$

where t_{AER} is the duration of the aerobic react phase.

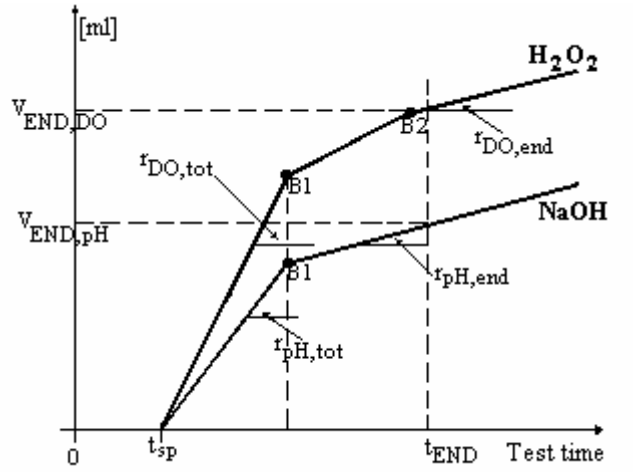

Figure 4a : Scheme of a pH/DO-stat titration test in Mode 3 and 4.

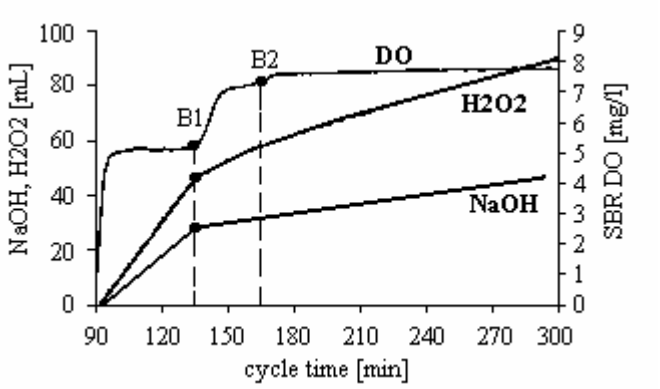

Figure 4b : Actual titration test in Mode 4. The DO trend measured in the SBR is reported for comparison.

Mode 4: Monitoring of nitrification during the aerobic phase in the SBR reactor

It's a working mode that allows to monitor the evolution of nitrification in the SBR during the aerobic phase. This makes it possible to identify the end of the nitrification process as soon as it takes place in the SBR. The sludge sample is withdrawn from the SBR at the end of the anoxic phase, so that nitrification takes place simultaneously in the titration beaker and in the SBR tanks.

The sludge is loaded into R_t and the set-point titration is performed as in Mode 3 (see Fig. 4a), but no extra nitrogen is added. As for Mode 3, Mode 4 can estimate AOB and NOB activities, but its main output is the ending time of the nitrification process in the SBR, i.e. bending points B_1 and B_2. If data obtained by the automated titrator are collected by a supervision system, this information would be used to stop the aerobic react, moving to the following phase before the scheduled time, thus saving energy under low-loading conditions. On the contrary, in the case of a nitrogen overload, or of a reduced nitrification capacity, the aerobic phase could be prolonged, allowing complete nitrification to take place. In Fig. 4b, an experimental titration test in Mode 4 is reported together with the dissolved oxygen concentration (DO) measured in the SBR reactor, during the same aerobic react phase (cycle time t=90–300 min). When bending points are identified on the titration curves, the DO value in the SBR also shows a bending point, indicating the end of a biological reaction (i.e. ammonium oxidation for B1 and nitrite oxidation for B2).

Mode 5: Estimation of nitrate concentration at the end of the anoxic phase

This pH-stat test is aimed at the determination of the amount of nitrates present at the end of the anoxic phase, which should be zero, (Mode 5a) or of the amount of nitrates produced by nitrification that are to be denitrified during the following SBR anoxic phase (Mode 5b). A simplified titration curve obtained according to Mode 5 is reported in Fig. 5a, typical experimental data are reported in Fig. 5b.

The sludge sample is withdrawn from the SBR at the beginning/end of the aerobic phase, it is loaded in *Bs* and then transferred into *Rt*, where pH-stat titration is performed. Once the pH has reached the set-point value, at $t=t_{sp}$, a non-limiting amount of acetate is added. The observed acid titration rate is almost constant because the denitrification kinetics is almost zero order and independent from both nitrate and acetate concentrations. Therefore, when nitrates are completely reduced, the titration curve shows a sub-horizontal trend, indicating the end of the denitrification reaction, so the first titration volume V_1 is assessed. Now the system doses a fixed amount of nitrates ($N\text{-}NO_3^-{}_{, dosed}$) and performs a second titration, until another sub-horizontal trend is detected, pointing out volume V_2. The nitrate content of the sludge loaded at the beginning of the test ($N\text{-}NO_3^-{}_{, sludge}$) is evaluated by a simple proportion, because the ratio between titrated acid and nitrates reduced, with the same carbon source and on the same sludge sample, is constant:

$$N\text{-}NO_3^-{}_{, sludge} = N\text{-}NO_3^-{}_{, dosed} \cdot V_1/(V_2 - V_1) \qquad (7)$$

For SBR monitoring and for control purposes, the value obtained can be used, for instance, to evaluate the performance of the denitrification phase (Mode 5a) or to predict the duration of the following anoxic phase (Mode 5b).

In Fig. 5b, from the dosage of the known concentration of nitrates (15 mgN L^{-1}), an initial concentration of 20 mg $N\text{-}NO_3^-$ L^{-1} was estimated. Compared to the analytical determination, a discrepancy of less that 10% was found.

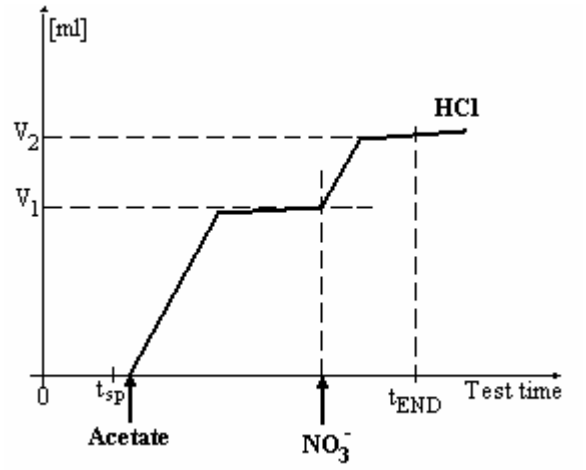

Figure 5a : Scheme of titration pH-stat test in Mode 5.

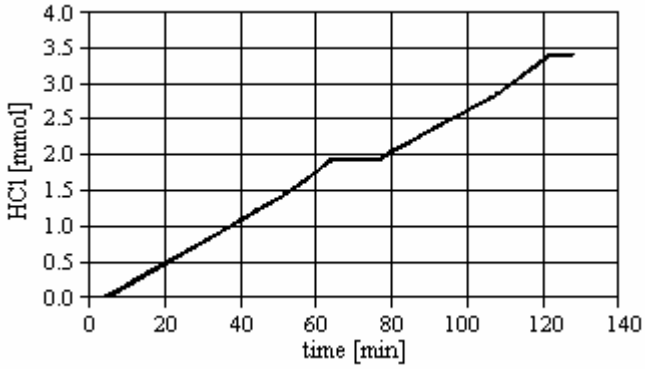

Figure 5b : Anoxic pH-stat titration test obtained on a nitrate containing sludge sample, to which a non limiting acetate concentration was added.

SYNCHRONIZATION BETWEEN THE TITRATOR OPERATION AND THE SBR CYCLE

The outputs of the on-line titrator (toxicity, activities, nitrogen content, nitrates concentration) are meant to be used for process control. For example, this means that the influent toxicity should be detected before the fill phase, or that the influent nitrogen content should be assessed before the beginning of the aerobic phase, while nitrate concentration should be determined before the beginning of the anoxic phase.

Since the on-line titrator can perform only one Mode at a time, sludge/influent sampling times, as well as titration tests, should be carefully scheduled by synchronising them with the SBR cycle time (t_{SBR}). As an example, Fig. 6 shows the scheduling for an SBR working according to a cycle including: anoxic fill, anoxic

react, aerobic react, settling, drawing and idle. Sludge/influent withdrawing are indicated by 'L' (as it means: "Loading TITAAN"). This "Loading" should take place at specific timings, scheduled at the end/beginning of each SBR-phase.
A possible operational sequence could be the following: (Mode5a)/(Mode1)/(Mode5b)/(Mode2)/(Mode3).

Of course, the feasibility of performing all the above steps depends on the length of each SBR phase. For example, in Mode 2 sludge is taken at the beginning of the idle phase, while in Mode 3 sludge is taken just before the end of the idle phase. If no idle phase is scheduled during that cycle (due to occasional operational needs), only one of these two Modes can be performed.
This sequence does not include Mode 4. In fact, if an instrumented SBR includes a DO probe, bending points may be evidenced without performing Mode 4 (see Fig. 6). A different sequence can be designed in case the assessment of the end of the nitrification phase is considered to be a priority.

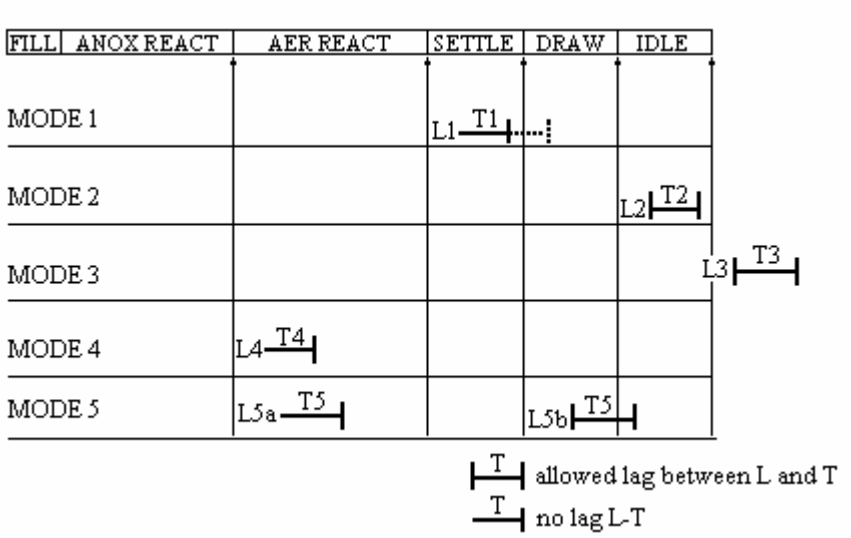

Figure 6 : Synchronization between the operation of the titration Modes and the SBR cycle-timing. L stands for "loading of the titration vessel"; T stands for "Titration".

CONCLUSIONS

Process monitoring of an SBR is necessary to ensure proper operation, especially if biological nitrogen removal is to be achieved. An automated titrator (TITAAN: TITrimetric Automated ANalyzer) has been developed, capable to perform several tests aimed to assess influent loadings and biological activity both for organic carbon and nitrogen removal.

Then, five set-point titration procedures (called Modes) were defined for the monitoring of an SBR (but applicable to continuous processes with minor modifications) to: (1) detect influent acute toxicity to nitrifying biomass; (2) estimate the influent N-content; (3) measure the nitrification capacity of the SBR; (4) monitor ammonia oxidation during the aerobic react phase of the SBR; and (5) estimate residual nitrate concentration at the end of the anoxic phase or at the end of SBR cycle.

Data collected during the titration tests are automatically elaborated by the master computer linked with TITAAN and results are stored in a permanent memory. It has to be stressed that TITAAN is a powerful tool in order to prevent faults in biological processes and to implement correct operational strategies as follows:

- Mode 1 gives toxicity early warning that allows the operator to divert part of the influent flow to a storage tank and/or to operate other countermeasures (e.g.: addition of powdered activated carbon).
- Mode 2 can activate the increase of the aeration capacity, well before the detection of low-DO conditions during the aeration phase (which can be too late); also, the duration of the aerobic phase can be increased if necessary.

- Modes 3 and Mode 4 allow checking whether both autotrophic populations (AOB and NOB) are working at their optimal rates. By looking at historical data, the operator can check whether nitrite oxidation rate trend is decreasing, leading to possible nitrite build-up, and consequently can take adequate early countermeasures to restore optimal conditions (i.e.: increasing the duration of the aeration phase and/or inoculate fresh fully nitrifying sludge).
- Historical data collected from Mode 5 can point out increasing trends of nitrate concentration so that adequate countermeasures can be taken by the operator (i.e.: dosing easily biodegradable organic carbon during the fill or the anoxic phase and/or increasing the duration of the anoxic phase).

A prototype of TITAAN is currently running at a pilot-scale SBR located at the WWTP of Trebbo di Reno (Bologna, Italy). A further paper summarising results of the on-going experimentation on the pilot-scale SBR will be submitted for publication soon.

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