CARBON AND NITROGEN REMOVAL FROM DAIRY WASTEWATER IN A LABORATORY SEQUENTIAL BATCH REACTOR SYSTEM

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Abstract. A 15 L laboratory scale Sequential Batch Reactor system was used to process a synthetic dairy wastewater. This effluent has similar composition than dairy factory crude wastewater (~ 3000 mgCOD/L, ~ 60 mgTKN/L, ~ 80 mgN-NO_3^-/L). The reactor operation is fully automatic. It consists on a controlled reactor load and drainage, a mixer and an aerator system automatically commanded, and a controlled purge pumping at a fixed flow rate. Additionally, a recycle with a controlled pump was installed to connect on-line sensors: temperature, dissolved oxygen and pH measurements. The reactor operation consists in 2 cycles by day. A typical configuration cycle is:

- Reactor feed (anoxic fill): 30 minutes.
- Anoxic phase at constant volume: 30 minutes.
- Aeration phase: 9.5 hours.
- Settling phase: 60 minutes.
- Drawing phase: 10 minutes.
- Idle time: 20 minutes.

In the experiments, a 4 days HRT and a 20 days θc were used during six months. The reactor temperature was maintained at 20 +/- 2 ºC. The obtained average removal efficiencies were highly satisfactory (94-99% for COD and 90-99% for total nitrogen) and the output average liquid quality were: COD < 90 mg/L, BOD_5 < 15 mg/L and total nitrogen < 5 mgN/L. Although the input nitrate in the system is removed by denitrification, the sludge growth was the main responsible for the nitrogen removal in the reactor. Autotrophic processes are negligible. Reactor sludge bulking was the sole operative problem that was detected in some occasions, resulting in a necessary programmed polymer addition (PRAESTOL 644) to overcome this problem. Further research is needed to reach the maximum loading capacity of this kind of reactor fed with dairy wastewater and controlling settling properties of the sludge. On-line and off-line data is being processed to establish an adequate model of reactor dynamics related with carbon and nitrogen removal, and will be presented in a future paper.

Keywords: Sequential Batch Reactor, Dairy wastewater, Heterotrophic growth.

1. Introduction

Many dairy factories employ continuous biological treatment systems in order to perform wastewater required depuration. In some cases, anaerobic/aerobic/anoxic series processes have been studied and applied (Castro et al., 2000) (Garrido et al., 2001). In other cases, single aerobic stages can be used to perform the complete wastewater depuration, spending sometimes high energy costs due to the required continuous aeration.

The Sequential Batch Reactor (SBR) process appears as a feasible alternative which could reach the complete depuration requirements, allowing continuous control and regulation of its critical operative parameters (stages duration, air supply, etc.), which is advantageous to overcome the problem of establishing optimal operation condition. This process operation is especially useful for small plants with a batch production and batch effluent generation, and only one reactor is required as treatment facility.

The aim of our study, in which this work is the first step, is to generate a phenomenological based model and a optimal control law for minimizing the operation costs of a dairy wastewater treatment by SBR. In the present paper a lab-scale reactor operation fed with dairy wastewater, data acquisition and performance analysis is presented. On-line and off-line data is being processed to establish an adequate model of reactor dynamics related with carbon and nitrogen removal, and will be presented in a future paper.

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2. Materials and Methods

2.1. Influent

The lab-scale 15 L reactor was fed with a semi-synthetic dairy wastewater, prepared diluting whole milk and adding sodium nitrate solution, to simulate the wastewater generated in an existing dairy plant.

Sodium nitrate is added to simulate industrial wastewater, since the dairy plant employs nitric acid and sodium hydroxide during washing routines. The influent is prepared daily and maintained at 4ºC in a 15 L tank. A previous experimental study was carried out to determine the average composition of an industrial dairy plant wastewater. Influent average characteristics were the following: 3000 mgCOD/L, 60 mgNTK/L, pH = 6.2 and 80 mgN-NO_3^-/L.

2.2. Operating Conditions

The reactor employed is cylindrical with a semi-spherical bottom. It has 20 L of maximum liquid capacity, and its geometrical features are the next:

- ☐ Height: 60 cm.
- ☐ Diameter: 23 cm.

The reactor operation is fully automatic. It consists on automatic load and drainage commanded with a control level system with electrodes, a mixer automatically activated, an aerator system composed by a automatically activated compressor and an air flow meter, and a controlled purge pumping at a fixed flow rate. Additionally, a recycle with a controlled pump was installed to connect on line sensors: temperature, dissolved oxygen and pH measurements. There is a cleaning system implemented to rinse the recycle system and probes. It consists on a clean water tank and commanded solenoid valves, and twice a cycle the cleaning system is activated by closing recycle valves and opening cleaning valves.

The data acquisition, historical management, alarms, reports and operator interface is carried out by an SCADA system. It was implemented as an Intellution FIX ® application. Figure 1 shows a diagram of the system.

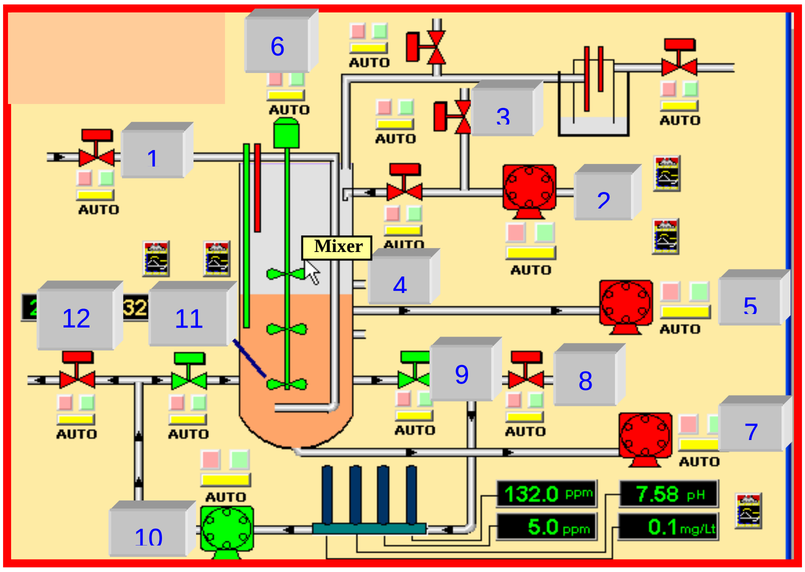

Fig. 1. Full automatic operation.

The SBR operates in 2 cycles by day (12 hours each). The operation times employed for cycles are shown in figure 2.

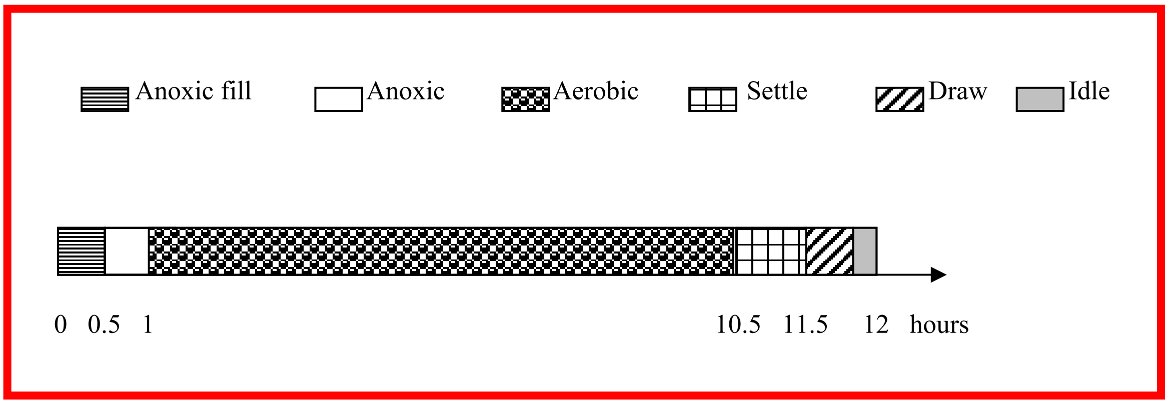

Fig. 2. Cycle setting times.

Operation scheme (see figure 1 for elements identification):

- ☐ Feeding stage: Before cycle begin, a preparation step is set: during 2 minutes feeding pump 2 is ON and remaining influent in pipes is throw out to a discharge tank (valve 4 closed and 3 open). Also, the recycle pump 10 and recycle valves 9 and 11 are ON, to allow probes stabilization. At 8:30 (or 20:30) feed starts by closing valve 3 and open valve 4. The feeding end is controlled by a maximum level electrode. Feeding time (~ 30 min. average) is registered for each cycle by the SCADA.
- ☐ Mixing system: During feeding, anoxic and aeration stages, mixer 6 and recycle pump 10 are ON.
- ☐ Aeration stage: The air supply is commanded by valve 1.
- ☐ Washing recycle system: Two washing steps at 14:30 (or 2:30 for night cycle) and 18:30 (6:30) are programmed. During 2 minutes, valves 9 and 11 are closed while 8 and 12 are open, and clear water is recycled from a 15 L tank.
- ☐ Purging: At the end of aeration phase, purging is activated (mixed liquor) by pump 7. Purging time is fixed at 6 minutes (modifiable by SCADA). The volume removed at this stage, corresponds each cycle to at 1/40 of total reactor liquor (~ 15 L). In these conditions, we obtain an operation cellular residence time of ~ 20 d.
- ☐ Settle: After aeration stage, valve 1, the mixer 6 and recycling pump (10) are OFF. The duration of this phase is ~ 1 hour.
- ☐ Draw: After previous stage, draw pump (5) is put ON. The draw end is commanded by a minimum level electrode. The duration of this phase is recorded each cycle by the SCADA.
- ☐ Temperature control: Temperature was kept stable by employing air conditioning in the laboratory.

2.3. Experimental Data

The reactor was operated and its removal efficiency evaluated for about ten months, and five cycles in selected standard conditions were fully analyzed. In this period, the following parameters were measured:

On Line Measurements. The reactor had available on-line measurements employing the following probes: DO (Dissolved Oxygen/non-stirring YSI 0551910), pH (Cole Palmer 27009-11), ORP (Oxidation Reduction Potential/Cole Palmer 27009-31) and Temperature (Cole Palmer 35613-05). The aeration flow rate was manually registered/adjusted every 30 minutes. Ammonia (Cole Palmer 27504-00) and Nitrate (Cole Palmer 35802-31) probes were initially employed, but due to calibration problems and unreliable data they were discarded. Additionally, employing the DO measurements, the k_{la} (oxygen mass transfer coefficient) value was determined for each cycle throughout the transient in-situ method.

Off Line Measurements. Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD_5), Total Kjeldhal Nitrogen (TKN), Ammonia Nitrogen ($N\text{-}NH_4^+$), Volatile and Total Suspended Solids (VSS and TSS), Sludge Volumetric Index (SVI), Nitrate and Nitrite were analyzed employing the techniques indicated in table 1:

Table 1. Techniques summary

Parameter	Principle	Reference
VSS, TSS	Gravimetric method	1
BOD_5	Respirometric method	1
COD	Dichromate oxidation/espectrophotometric method	1
TKN	Kjeldhal digester/titration method	1
$N\text{-}NH_4^+$	Titration method	1
$N\text{-}NO_3^-$, $N\text{-}NO_2^-$	HPLC	2
SVI	Imhoff cone	1

1 - APHA (1995).

2 - Column IC - pakA , detector UV (~ 210 nm), Mobile phase: K_2HPO_4/KH_2PO_4. Oven Temperature 35°C, flow rate = 1.2 $ml.min^{-1}$.

The total influent, total effluent and the centrifuged mixed liquor were analyzed. Settling velocity and SVI were also measured to evaluate sludge settling properties.

3. Results and Discussion

3.1. Reactor Removal Efficiency

Average soluble COD and TKN removal efficiency during ten months of operation are presented in table 2. In this table, the removal efficiency calculated at the end of aeration period (9.5 hs) and after 4.5 hs. of aeration are presented.

Table 2. Reactor efficiency after 4.5 and 9.5 hours of aeration

Parameter	At 4.5 hours of aeration	At 9.5 hours of aeration
COD	93-99 %	94-99 %
TKN	85-95 %	90-99 %

The data exhibited a high and constant COD removal efficiency, and a high TKN removal efficiency. It can be seen that for the actual organic load, no better efficiencies are obtained for longer periods than 4.5 hours of aeration excepting for some TKN values. In order to evaluate the maximum loading capacity of reactor, further research is needed.

Effluent COD and BOD_5 data from typical cycles in 2004 are shown in figure 3.

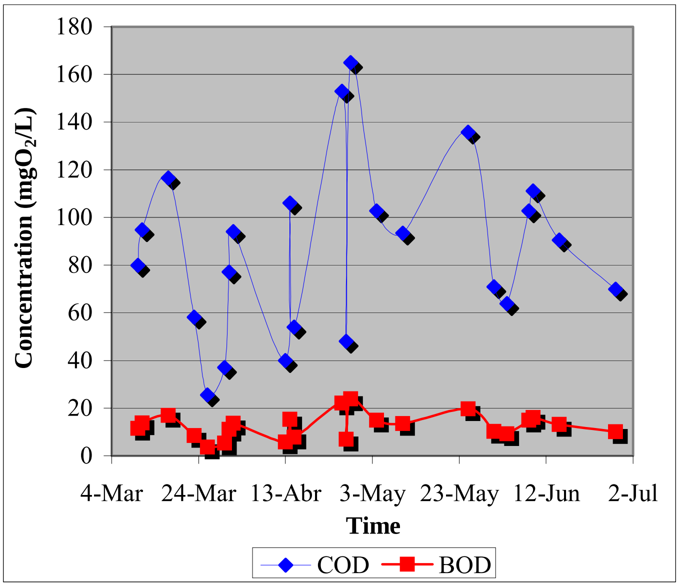

Fig. 3. Organic effluent quality.

The mean effluent soluble COD is 85 mg/L. This is a relatively high value, but with a very low BOD_5 (acceptable organic content). At this point, the corresponding BOD_5/COD ratio is near 0.15, indicating that there is an important inert fraction of COD that could came from biomass decay. Average TKN and ammonia effluent concentrations are very low, and allowable to be discharged (< 5 mg/L), according with Uruguayan Regulations (DINAMA, 1979): 2.4 and 0.8 mgN/L respectively (mean value of 20 samples).

In figure 4, typical COD, pH and DO profiles during a standard cycle are shown.

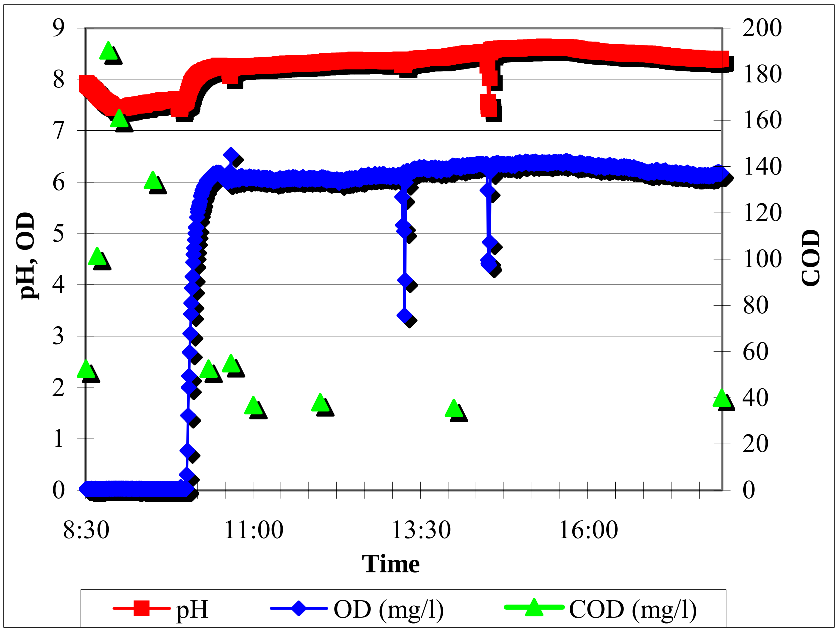

Fig. 4. COD, pH and DO evolution during a cycle.

As be seen, DO quickly reaches a stable value after aeration begins, corresponding with significant COD reduction. The cycle pH profile showed an initial reduction associated by the reactor load. Once the filling ends, a slightly increase is registered corresponding to anoxic reaction (denitrification). Then, pH increase significantly during aerobic phase. This behavior could be explained by CO_2 stripping effects (Chang, 1996), and not by the biological reaction. The COD evolution has an initial increase during feeding. However, the COD obtained after the end of feeding is around 50% of the expected COD, even considering the amount of organic matter disappeared by denitrification. This observation, which is systematically observed, can be explained because part of the organic matter is adsorbed (or stocked) in the sludge. After that, a reduction is registered, due to anoxic consumption.

From these profiles it is shown that 4.5 hours of aeration is enough to complete the reaction, in agreement with the removal efficiencies evaluated in table 2.

Typical nitrate and nitrite concentrations are very low in the liquor as it is shown in figure 5. This result shows a complete denitrification (at the end of anoxic phase very low nitrate and nitrite concentration are detected) and a negligible nitrification process (the nitrate and nitrite concentration do not increase at aeration stage), in accordance with the high influent C/N relation employed (~ 50).

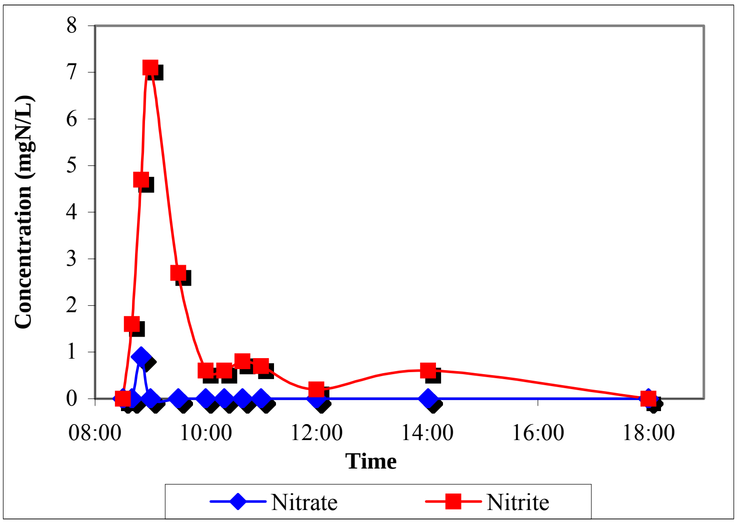

Fig. 5. Nitrate and nitrite cycle profiles.

An estimated nitrogen mass balance in the standard cycle is presented in figure 6:

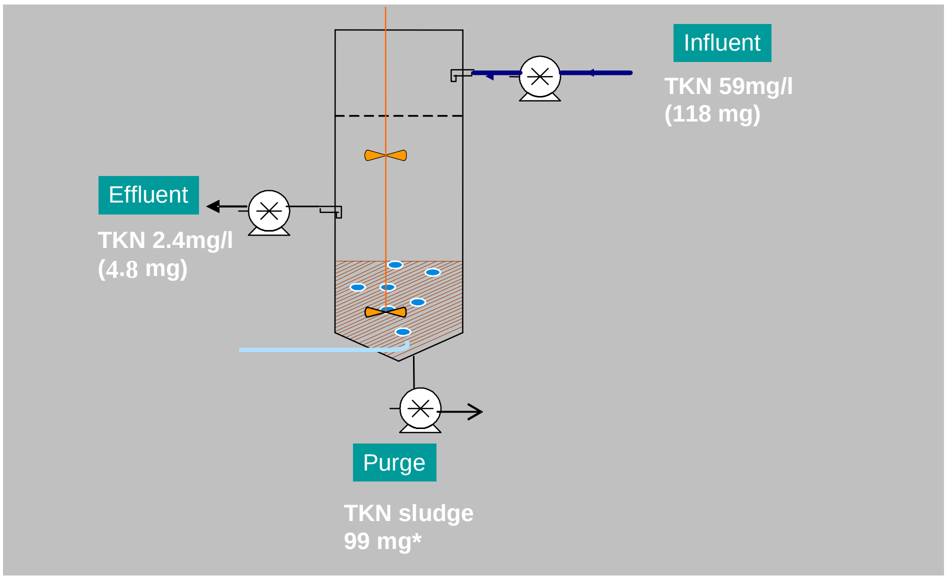

Fig. 6. Nitrogen balance.

The purged sludge (*) contents most of the nitrogen which enter each cycle with the influent. This balance is an estimation considering the %N of sludge as 12% of Volatile Suspended Solids (Orhón, 1994). Although the input nitrate in the system is removed by denitrification, the sludge growth was the main responsible for the nitrogen removal in the reactor.

3.2. Biomass Content

Volatile Suspended Solids evolution during ten months is shown in figure 7.

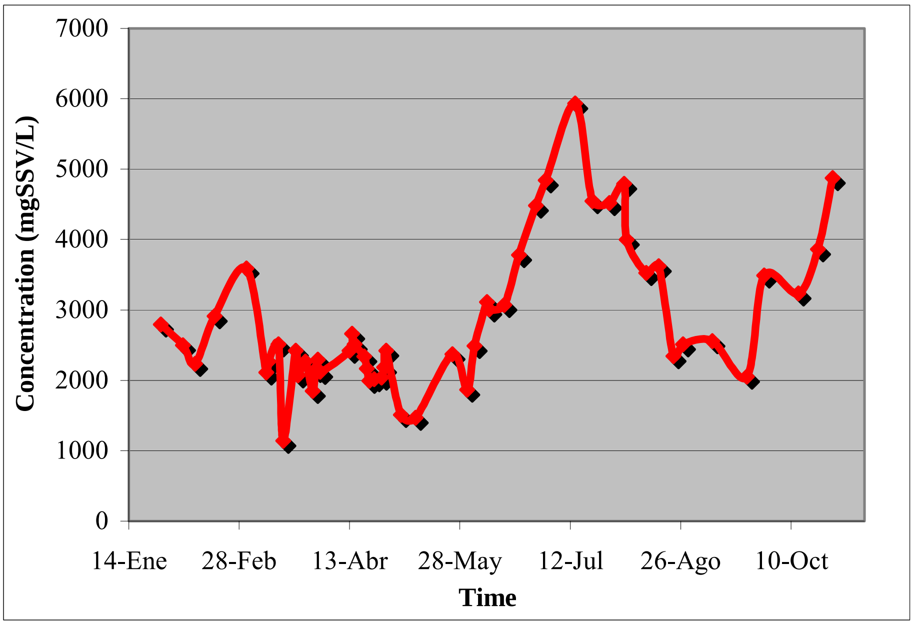

Fig. 7. Sludge amount in reactor.

VSS values were maintained between 2 and 4 g/L most of the time. In some occasions the sludge lost its settling capacity (washing out through the reactor), increasing the corresponding SVI and effluent VSS values. To overcome bulking, the programmed flocculant addition to the reactor (PRAESTOL 644 from Stockhausen GmbH & Co.) was conducted. To avoid sludge wash out, the reactor daily purge was closed and VSS increased during part of May-June and October.

3.3. k_{la} Measurements

In order to characterize oxygen mass transfer, kla measurements were conducted. This coefficient will be useful for modeling purposes. In table 3, the obtained values for this mass transfer coefficient are presented (average of 5 cycles). These values showed a low and reproducible mass transfer resistance.

Table 3. k_{la} determination

Average k_{la} (s^{-1})	Standard Deviation (s^{-1})
0,26	0,04

3.4. Experimental Denitrifying Rate

An estimation of denitrifying rate during the anoxic phase was performed based on cycle performance throughout a mass balance. The result obtained is shown in figure 8. This figure shows a fast denitrification reaction. The maximum denitrification specific rate obtained (ηd ~ 0.009 gN-NO_3^-/gVSS/h), corresponds to a substrate between readily biodegradable (0.03) and particulate substrate (0.004) (Orhón, 1994). By making the same analysis on other cycles a dispersion of results is observed.

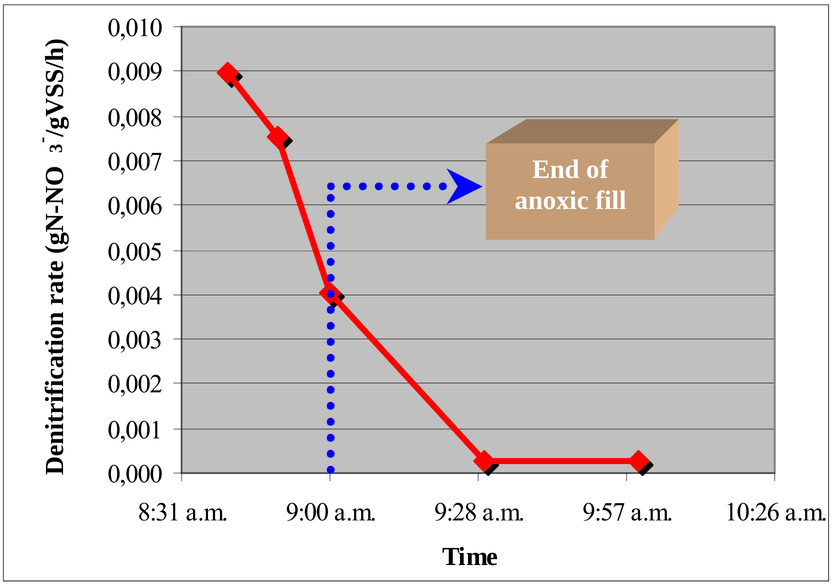

Fig. 8. Denitrification specific rate evolution. Anoxic phase exceptionally extended to 10:00 a.m..

3.5. Maximum Specific Growth Rate Determinations

To evaluate the maximum specific heterotrophic growth rate [μ(max)], respirometric batch tests were carried out for reactor sludge, three times during reactor operation. The procedure employed is described by Kappeler and Gujer (1992). It is based on OUR (Oxygen Uptake Rate) measurements throughout the experience. In figure 9 is shown a resume of results obtained [Ln(OUR/OUR$_0$) vs. Time].

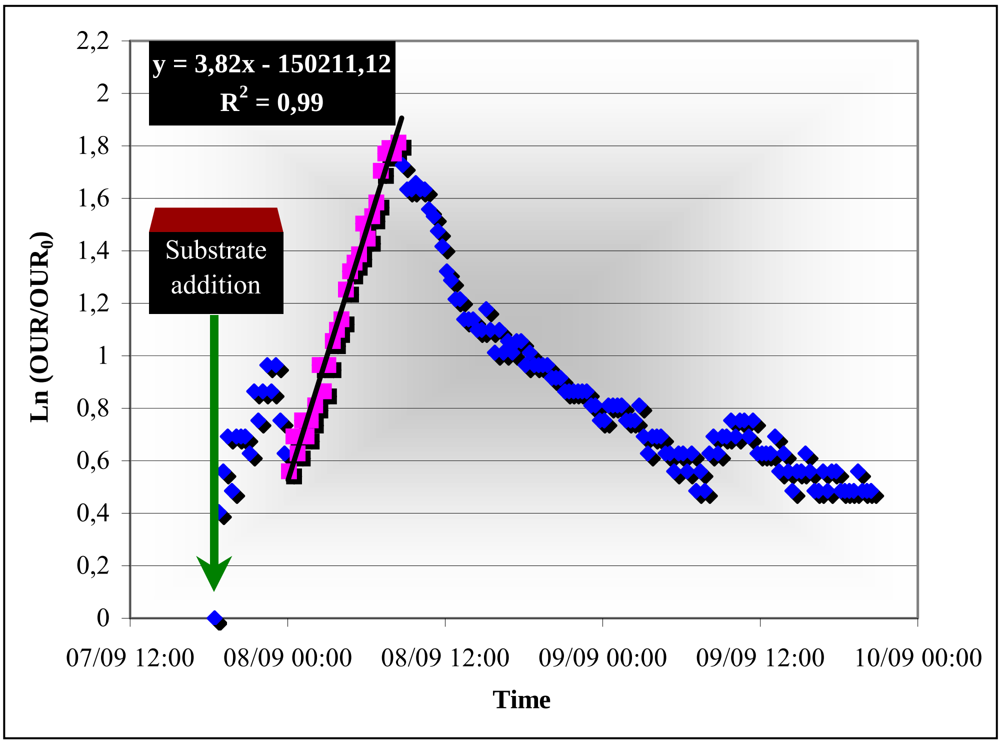

Fig. 9. Typical respirometric profile.

The previous data show a μ(max) value of 2 – 3.8 d^{-1}. This is consistent with the corresponding reported values in the literature (2.5 to 2.9 d^{-1} for dairy wastewaters) (Grady and Lim, 1980) (Castro et. al., 2000) (Orhón et al, 1993).

3.6. Aerobic Decay Rate Coefficient Determination

This parameter was experimentally evaluated aerating the sludge without exogenous substrate in the system, and evaluating volatile suspended solids at the beginning and ending of aeration period. The estimated value is obtained through the following expression:

$$b_H \approx \frac{\frac{\Delta X}{\Delta t}}{X_{av}} \qquad (1)$$

Where:

- ☐ ΔX: Decay biomass concentration (mgSSV/L).
- ☐ Δt: Process duration (d).
- ☐ X_{av}: Average biomass concentration in the system (mgSSV/L).
- ☐ b_H: Aerobic decay rate coefficient (d^{-1}).

The value obtained for this parameter (b_H) range from 0.06 to 0.13 d^{-1} (mean value ~ 0.09 d^{-1}). These results are comparable with reported in literature (Orhón, 1994).

4. Conclusions

A SBR reactor with 4 days HRT (Hydraulic Residence Time) and 20 days θc (Cellular Residence Time) were used. The obtained average removal efficiencies were highly satisfactory (~ 97% for COD, ~ 90% for total nitrogen) and the output average liquid quality were very good (COD < 90 mg/L, BOD_5 < 15 mg/L, total nitrogen < 5 mgN/L). Although the input nitrate in the system is removed by denitrification, the sludge growth was the main responsible for the nitrogen removal in the reactor. Autotrophic processes in the system are negligible. Additional treatment steps to reach adequate output effluent quality are not required, which result industrially advantageous from the point of view of total process simplicity.

For the employed loading rate, an adequate aeration time is less than 4.5 hours. In the studied conditions, the kinetic parameters [μ(max), b_H] obtained were similar to those found in the literature for similar substrates. On-line and off-line data is being processed to establish an adequate model of reactor dynamics related with carbon and nitrogen removal, and will be presented in a future paper.

Further research is needed to reach the maximum loading capacity of this reactor fed with dairy wastewater and to find optimal operating condition in order to improve sludge settling.

References

APHA. (1995). *Standard Methods for the Examination of Water and Wastewater*, United Baltimore Press, USA.

Castro, G., De Oliveira, R., Viñas, M. (2001). Eliminación de Carbono y Nitrógeno en Efluentes Lácteos con un Reactor Discontinuo Secuencial (SBR). *II Encuentro de Ingeniería Química.* Montevideo, Uruguay.

Chang, C. H., Hao, O. J. (1996). Sequential Batch Reactor System for Nutrient Removal: ORP and pH Profiles. *J. Chem. Tech. Biotechnol. 67*, 27-38. Horz.

DINAMA. (1979). *DECRETO 253/79 (Con las modificaciones de los Decretos 232/88, 698/89 y 195/91 incluidas),* Montevideo, Uruguay.

Ferrari, A., Gutiérrez, M. S., Benítez, A., Lema, P., Etchebehere, C., Travers, D. (2005). Effect of the Flocculant Periodical Addition on the Performance of a Sequential Batch Reactor Treating Dairy Wastewater. *Submitted paper in ENPROMER 2005.* Costa Verde, Rio de Janeiro, Brazil.

Garrido, J. M., Omil, F., Arrojo, B., Méndez, R., Lema, J.M. (2001). Carbon and Nitrogen Removal from a Wastewater of an Industrial Dairy Laboratory with a Coupled Anaerobic Filter - Sequencing Batch Reactor System. *Wat. Sci. and Tech., 43 (3),* 249 - 256.

Grady, C. P. L. Jr., Lim, H. C. (1998). *Biological Wastewater Treatment*. New York, NY: Marcel Dekker, Inc.

Kappeler, J., Gujer, W. (1992). Estimation of Kinetic Parameters of Heterotrophic Biomass under Aerobic Conditions and Characterization of Wastewater for Activated Sludge Modelling. *Wat. Sci. and Tech., 25 (6),* 125 - 140.

Orhon, D., Görgün, E., Germirli, F., Artan, N. (1993). Biological Treatability of Dairy Wastewaters. *Wat. Sci. and Tech., 27 (4),* 625-633.

Orhón,_D., Artan, N. (1994). *Modelling of Activated Sludge Systems*. Technomic Publishing Co., Switzerland. ISBN 1-56676-101-8.

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